

*Supporting Information*

**Asymmetric Suzuki-Miyaura Cross-Coupling of 1-Bromo-2-naphthoates  
using the Helically Chiral Polymer Ligand PQXphos**

Yuto Akai, Laure Konnert, Takeshi Yamamoto, and Michinori Suginome<sup>\*</sup>

Department of Synthetic Chemistry and Biological Chemistry, Graduate School of Engineering,  
Kyoto University Katsura, Nishikyo-ku, Kyoto 615-8510, Japan,  
Tel: 075-383-2720, Fax: 075-383-2722, E-mail: suginome@sbchem.kyoto-u.ac.jp

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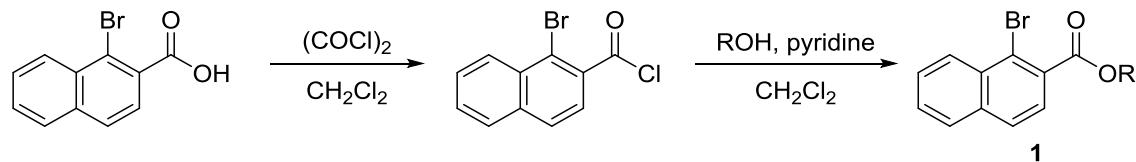
## 1. General

All reactions were carried out under an atmosphere of nitrogen with magnetic stirring.  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were recorded on a Varian 400-MR spectrometer at ambient temperature.  $^1\text{H}$  NMR data are reported as follows: chemical shift in ppm downfield from tetramethylsilane ( $\delta$  scale), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, quint = quintet, m = multiplet, and br = broad), coupling constant (Hz), and integration.  $^{13}\text{C}$  NMR chemical shifts are reported in ppm downfield from tetramethylsilane ( $\delta$  scale).  $^{31}\text{P}$  NMR chemical shifts are reported in ppm downfield from  $\text{H}_3\text{PO}_4$  (85%). All  $^{13}\text{C}$  NMR and  $^{31}\text{P}$  NMR spectra were obtained with complete proton decoupling.

Toluene and THF were dried and deoxygenized using an alumina/catalyst column system (GlassContour Co.),  $[\text{PdCl}(\eta^3\text{-C}_3\text{H}_5)]_2$  (TCI), distilled water (Nacalai tesque), 1-bromo-2-naphthoic acid (TCI), methanol (Nacalai tesque), ethanol (Nacalai tesque), 2-propanol (Nacalai tesque), 3-pentanol (TCI), 2,4-dimethyl-3-pentanol (TCI), dicyclohexylmethanol (Aldrich), cyclohexanol (Wako), cyclooctanol (TCI), t-butyl alcohol (Wako), phenol (TCI), 2,6-dimethylphenol (TCI), oxalyl chloride (Wako), 1-naphthaleneboronic acid (Wako), 2-methylphenylboronic acid (Wako), 4-methyl-1-naphthaleneboronic acid (Alfa Aesar), 4-methoxy-1-naphthaleneboronic acid (Aldrich), 4-fluoro-1-naphthaleneboronic acid (Aldrich), 1-pyreneboronic acid (Wako), 2,3-dimethylphenylboronic acid (Wako), 2,5-dimethylphenylboronic acid (Wako), 5-fluoro-2-methylphenylboronic acid (Wako), 4-fluoro-2-methylphenylboronic acid (Alfa Aesar), lithium aluminium hydride (Wako), and potassium hydroxide (Nacalai tesque) were used as received from the commercial sources. 1,1,2-trichloroethane (Wako), dimethylformamide (Nacalai tesque), and pyridine (Wako) were purchased from the commercial sources and distilled before use. Potassium phosphate (Nacalai tesque) was purchased and dried prior to use. PQXphos **L1-L6** were synthesized by the method reported previously.<sup>S1</sup>

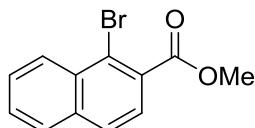
## 2. Experimental Procedures and Spectral Data for New Compounds

### 2.1 Preparation of Aryl Bromide

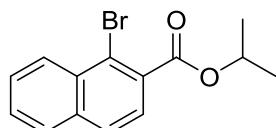


**Scheme S1.** Synthesis of aryl bromide 1

**General procedure:** To a solution of 1-bromo-2-naphthoic acid (1.11 g, 4.40 mmol) in  $CH_2Cl_2$  (20 mL) was added oxalyl chloride (416  $\mu L$ , 4.84 mmol) at 0 °C. The mixture was stirred at room temperature for 1 h. After evaporation of the solvent, to the mixture was added pyridine (1.0 mL, 13.3 mmol), alcohol (13.3 mmol), and  $CH_2Cl_2$  (20 mL). The mixture was stirred at room temperature for 18 h. The resulting mixture was quenched by water and extracted with  $CH_2Cl_2$ . After drying with anhydrous  $MgSO_4$ , the concentrated mixture was purified by column chromatography (hexane: $CH_2Cl_2$  = 2:1) to give a desired product.



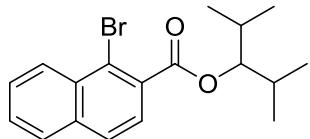
**Methyl 1-bromo-2-naphthoate (1A):** 95% yield;  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  8.46 (d,  $J$  = 8.4 Hz, 1H), 7.85 (d,  $J$  = 8.0 Hz, 1H), 7.84 (d,  $J$  = 8.4 Hz, 1H), 7.58-7.70 (m, 3H), 4.01 (s, 3H);  $^{13}C$  NMR (100 MHz,  $CDCl_3$ )  $\delta$  167.9, 135.2, 132.3, 131.2, 128.6, 128.2, 128.1, 128.1, 127.8, 125.7, 122.6, 52.7; IR (ATR)  $\nu$  2925, 1717, 1456, 1429, 1265, 1234, 1219, 1126, 1003, 866, 827, 760  $cm^{-1}$ ; HRMS (ESI)  $m/z$  calcd for  $C_{12}H_9BrO_2+H^+$  ( $M+H^+$ ): 264.9859, found: 264.9851.



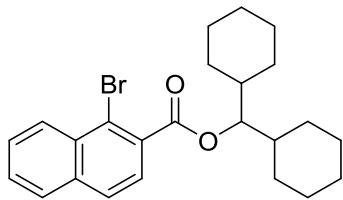
**Isopropyl 1-bromo-2-naphthoate (1B):** 82% yield;  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  8.44 (d,  $J$  = 8.8 Hz, 1H), 7.84 (d,  $J$  = 8.0 Hz, 1H), 7.83 (d,  $J$  = 8.8 Hz, 1H), 7.56-7.67 (m, 3H), 5.36 (sep,  $J$  = 6.0 Hz, 1H), 1.44 (d,  $J$  = 6.0 Hz, 6H);  $^{13}C$  NMR (100 MHz,  $CDCl_3$ )  $\delta$  167.1, 135.0, 132.2, 132.2, 128.4, 128.2, 128.0, 127.9, 127.8, 125.6, 122.0, 69.7, 21.8 (2C); IR (ATR)  $\nu$  2986, 1722, 1460, 1373, 1271, 1242, 1172, 1099, 978, 926, 823, 788, 762  $cm^{-1}$ ; HRMS (ESI)  $m/z$  calcd for  $C_{14}H_{13}BrO_2+H^+$  ( $M+H^+$ ): 293.0172, found: 293.0162.



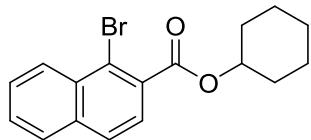
**Pentan-3-yl 1-bromo-2-naphthoate (1C):** 87% yield;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.44 (d,  $J$  = 8.0 Hz, 1H), 7.85 (dd,  $J$  = 8.0 Hz, 1.2 Hz, 1H), 7.84 (d,  $J$  = 8.4 Hz, 1H), 7.56-7.67 (m, 3H), 5.12 (quin,  $J$  = 6.0 Hz, 1H), 1.77 (dq,  $J$  = 7.6 Hz, 6.0 Hz, 4H), 1.03 (t,  $J$  = 7.6 Hz, 6H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  167.5, 134.9, 132.4, 132.3, 128.4, 128.2, 128.0, 127.9, 127.8, 125.5, 121.9, 78.8, 26.5 (2C), 9.8 (2C); IR (ATR)  $\nu$  2966, 1728, 1558, 1456, 1265, 1234, 1150, 1136, 1109, 1045, 974, 930, 822, 758  $\text{cm}^{-1}$ ; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{16}\text{H}_{17}\text{BrO}_2+\text{H}^+$  ( $\text{M}+\text{H}^+$ ): 321.0485, found: 321.0475.



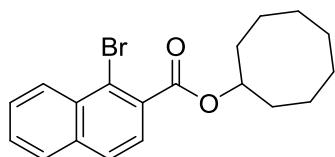
**2,4-Dimethylpentan-3-yl 1-bromo-2-naphthoate (1D):** 80% yield;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.46 (d,  $J$  = 8.4 Hz, 1H), 7.84 (dd,  $J$  = 7.2 Hz, 0.8 Hz, 1H), 7.84 (d,  $J$  = 8.4 Hz, 1H), 7.56-7.70 (m, 3H), 4.97 (t,  $J$  = 6.0 Hz, 1H), 2.07 (dsep,  $J$  = 6.4 Hz, 6.0 Hz, 2H), 1.03 (t,  $J$  = 6.4 Hz, 12H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  167.5, 135.0, 132.4, 132.3, 128.5, 128.1, 128.0, 127.9, 127.8, 125.7, 122.1, 84.7, 26.6 (2C), 19.7 (2C), 17.5 (2C); IR (ATR)  $\nu$  2963, 1717, 1558, 1325, 1276, 1240, 1171, 1117, 972, 935, 893, 810, 754  $\text{cm}^{-1}$ ; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{18}\text{H}_{21}\text{BrO}_2+\text{H}^+$  ( $\text{M}+\text{H}^+$ ): 349.0798, found: 349.0787.



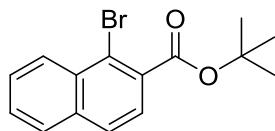
**Dicyclohexylmethyl 1-bromo-2-naphthoate (1E):** 80% yield;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.47 (d,  $J$  = 8.4 Hz, 1H), 7.84 (d,  $J$  = 8.0 Hz, 2H), 7.56-7.72 (m, 3H), 5.00-5.04 (m, 1H), 1.56-1.78 (m, 12H), 1.15-1.34 (m, 10H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  167.3, 135.0, 132.4, 132.1, 128.6, 128.1, 128.0, 127.9, 127.8, 125.8, 122.3, 83.3, 38.6 (2C), 30.0 (2C), 27.7 (2C), 26.4 (2C), 26.3 (2C), 26.1 (2C); IR (ATR)  $\nu$  2929, 2851, 1728, 1683, 1653, 1558, 1265, 1238, 1167, 1124, 975, 754  $\text{cm}^{-1}$ ; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{24}\text{H}_{29}\text{BrO}_2+\text{H}^+$  ( $\text{M}+\text{H}^+$ ): 429.1424, found: 429.1411.



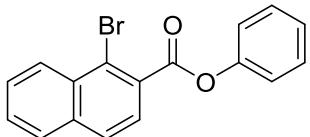
**Cyclohexyl 1-bromo-2-naphthoate (1F):** 92% yield;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.44 (d,  $J$  = 8.4 Hz, 1H), 7.84 (dd,  $J$  = 8.0 Hz, 1.2 Hz, 1H), 7.83 (d,  $J$  = 8.4 Hz, 1H), 7.56-7.67 (m, 3H), 5.13 (sep,  $J$  = 4.0 Hz, 1H), 2.02-2.07 (m, 2H), 1.80-1.85 (m, 2H), 1.51-1.70 (m, 3H), 1.41-1.51 (m, 2H), 1.29-1.39 (m, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  167.1, 135.0, 132.3 (2C), 128.4, 128.2, 128.0, 127.9, 127.8, 125.7, 122.0, 74.5, 31.6 (2C), 25.4, 23.7 (2C); IR (ATR)  $\nu$  2933, 1717, 1558, 1506, 1456, 1285, 1248, 1170, 1128, 1011, 980, 824, 756  $\text{cm}^{-1}$ ; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{17}\text{H}_{17}\text{BrO}_2+\text{H}^+$  ( $\text{M}+\text{H}^+$ ): 333.0485, found: 333.0473.



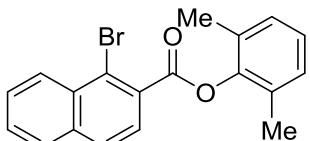
**Cyclooctyl 1-bromo-2-naphthoate (1G):** 93% yield;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.44 (d,  $J$  = 8.4 Hz, 1H), 7.84 (d,  $J$  = 8.4 Hz, 1H), 7.83 (d,  $J$  = 8.4 Hz, 1H), 7.56-7.66 (m, 3H), 5.30 (sep,  $J$  = 4.0 Hz, 1H), 1.90-2.07 (m, 4H), 1.74-1.83 (m, 2H), 1.55-1.66 (m, 8H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  167.0, 135.0, 132.4, 132.2, 128.4, 128.1, 128.0, 127.9, 127.8, 125.6, 122.0, 77.2, 31.4 (2C), 27.1 (2C), 25.4, 23.0 (2C); IR (ATR)  $\nu$  2920, 1717, 1558, 1273, 1244, 1169, 1138, 1031, 976, 924, 826, 758, 746  $\text{cm}^{-1}$ ; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{19}\text{H}_{21}\text{BrO}_2+\text{H}^+$  ( $\text{M}+\text{H}^+$ ): 361.0798, found: 361.0784.



**tert-Butyl 1-bromo-2-naphthoate (1H):** 63% yield;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.42 (d,  $J$  = 8.0 Hz, 1H), 7.83 (d,  $J$  = 8.0 Hz, 1H), 7.82 (d,  $J$  = 8.4 Hz, 1H), 7.55-7.65 (m, 3H), 1.67 (s, 9H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.9, 134.8, 133.4, 132.2, 128.3, 128.1, 127.9, 127.8, 127.7, 125.5, 121.3, 82.9, 28.2 (3C); IR (ATR)  $\nu$  1724, 1684, 1558, 1506, 1456, 1367, 1294, 1248, 1126, 822, 754, 654  $\text{cm}^{-1}$ ; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{15}\text{H}_{15}\text{BrO}_2+\text{H}^+$  ( $\text{M}+\text{H}^+$ ): 307.0328, found: 307.0317.

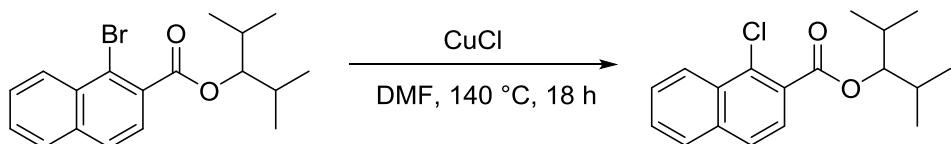


**Phenyl 1-bromo-2-naphthoate (1I):** 79% yield;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.51 (dd,  $J = 8.4$  Hz, 0.8 Hz, 1H), 7.87-7.93 (m, 3H), 7.69 (ddd,  $J = 8.4$  Hz, 7.2 Hz, 1.2 Hz, 1H), 7.64 (ddd,  $J = 8.0$  Hz, 6.8 Hz, 1.2 Hz, 1H), 7.45-7.50 (m, 2H), 7.29-7.35 (m, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  165.8, 150.8, 135.4, 132.4, 130.5, 129.6 (2C), 128.7, 128.4, 128.3, 128.3, 128.0, 126.2, 125.9, 123.4, 121.6 (2C); IR (ATR)  $\nu$  1734, 1718, 1684, 1558, 1489, 1456, 1229, 1190, 1109, 968, 822, 758, 745, 691  $\text{cm}^{-1}$ ; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{17}\text{H}_{11}\text{BrO}_2+\text{H}^+$  ( $\text{M}+\text{H}^+$ ): 307.0015, found: 307.0003.



**2,6-Dimethylphenyl 1-bromo-2-naphthoate (1J):** 78% yield;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.54 (d,  $J = 8.4$  Hz, 1H), 7.99 (d,  $J = 8.4$  Hz, 1H), 7.93 (d,  $J = 8.8$  Hz, 1H), 7.90 (d,  $J = 8.8$  Hz, 1H), 7.70 (ddd,  $J = 8.4$  Hz, 6.8 Hz, 1.2 Hz, 1H), 7.65 (ddd,  $J = 8.0$  Hz, 6.8 Hz, 1.2 Hz, 1H), 7.12-7.19 (m, 3H), 2.36 (s, 6H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  164.9, 148.2, 135.4, 132.4, 130.3, 130.2 (2C), 128.8 (2C), 128.4, 128.3, 128.2, 128.0, 126.1, 125.8 (2C), 123.5, 16.7 (2C); IR (ATR)  $\nu$  2358, 1684, 1653, 1647, 1558, 1506, 754  $\text{cm}^{-1}$ ; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{19}\text{H}_{15}\text{BrO}_2+\text{H}^+$  ( $\text{M}+\text{H}^+$ ): 355.0328, found: 355.0316.

## 2.2 Preparation of Aryl Chloride 1D'



**2,4-Dimethylpentan-3-yl 1-chloro-2-naphthoate:** To a solution of **1D** (349 mg, 1.0 mmol) in DMF (1.0 mL) was added CuCl (109 mg, 1.1 mmol). The mixture was stirred at 140 °C for 18 h. After filtration of the reaction mixture with  $\text{Et}_2\text{O}$ , the filtrate was extracted by  $\text{Et}_2\text{O}$  and washed by water, dried over  $\text{MgSO}_4$ . After evaporation of the solvent, the residue was purified by column chromatography (hexane: $\text{CH}_2\text{Cl}_2$  = 2:1) to give a desired product (254 mg, 84%);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.47 (d,  $J = 8.0$  Hz, 1H), 7.87 (d,  $J = 8.0$  Hz, 1H), 7.80 (d,  $J = 8.4$  Hz, 1H), 7.77 (d,  $J = 8.4$  Hz, 1H), 7.66 (dd,  $J = 6.8$  Hz, 6.4 Hz, 1H), 7.61 (dd,  $J = 6.8$  Hz, 6.4

Hz, 1H), 4.97 (t,  $J$  = 6.0 Hz, 1H), 2.06 (dsep,  $J$  = 6.4 Hz, 6.0 Hz, 2H), 1.01 (t,  $J$  = 6.4 Hz, 12H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.7, 135.1, 131.5, 131.2, 128.9, 128.1 (2C), 127.7, 126.9, 125.8, 125.7, 84.5, 29.6 (2C), 19.7 (2C), 17.4 (2C); IR (ATR)  $\nu$  2963, 1717, 1464, 1331, 1272, 1242, 1120, 989, 893, 812, 754  $\text{cm}^{-1}$ ; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{18}\text{H}_{21}\text{ClO}_2+\text{Na}^+$  ( $\text{M}+\text{Na}^+$ ): 327.1122, found: 327.1118.

### 2.3 General Procedure for Asymmetric Suzuki-Miyaura Cross-coupling (Tables 1–3 and Eq 1)

To a solution of (*P*)-(*R*)-PQXphos (27 mg, 4.0  $\mu\text{mol}$  phosphorous atom) in THF (300  $\mu\text{L}$ ) was added  $[\text{PdCl}(\eta^3\text{-C}_3\text{H}_5)]_2$  (0.01 M in THF, 100  $\mu\text{L}$ , 1  $\mu\text{mol}$ ),  $\text{K}_3\text{PO}_4$  (43 mg, 0.2 mmol), **1** (0.10 mmol), **2** (0.15 mmol),  $\text{H}_2\text{O}$  (40  $\mu\text{L}$ ) in this order. The reaction was stirred at 40 °C for 24–48 h. After the reaction, subsequent addition of MeCN (10 mL) resulted in precipitation of the polymer complex. The suspension was passed through a pad of Celite® using MeCN as an eluent. The crude product was subjected to PTLC (hexane/Et<sub>2</sub>O = 4/1) to give a desired product. Further purification was performed by GPC if necessary. The enantiomeric excesses of the products were determined by HPLC or SFC with a chiral stationary phase.

### 2.4 Gram-Scale Reaction (Table 3, entry 6)

To a solution of (*P*)-(*R*)-PQXphos (595 mg, 96  $\mu\text{mol}$  phosphorous atom) in THF (16 mL) was added  $[\text{PdCl}(\eta^3\text{-C}_3\text{H}_5)]_2$  (14.6 mg, 40  $\mu\text{mol}$ ),  $\text{K}_3\text{PO}_4$  (1.70 g, 8.0 mmol), **1** (1.40 g, 4.0 mmol), **2** (900 mg, 6.0 mmol),  $\text{H}_2\text{O}$  (1.6 mL) in this order. The reaction was stirred at 40 °C for 72 h. After the reaction, subsequent addition of MeCN (30 mL) resulted in precipitation of the PQXphos. The suspension was passed through a pad of Celite® using MeCN as an eluent. The crude product was isolated by column chromatography (hexane/Et<sub>2</sub>O = 4/1) to give a desired product **3Dg** (1.31 g, 87% yield). The enantiomeric excess of this compound was determined by SFC analysis.

### 2.5 Reuse of Catalyst (Scheme 1)

**[Initial Run]** To a solution of (*P*)-(*R*)-PQXphos (27 mg, 4.0  $\mu\text{mol}$  phosphorous atom) in THF (300  $\mu\text{L}$ ) was added  $[\text{PdCl}(\eta^3\text{-C}_3\text{H}_5)]_2$  (0.01 M in THF, 100  $\mu\text{L}$ , 1  $\mu\text{mol}$ ),  $\text{K}_3\text{PO}_4$  (43 mg, 0.2 mmol), **1D** (0.12 mmol, 42 mg), **2g** (0.10 mmol, 15 mg),  $\text{H}_2\text{O}$  (40  $\mu\text{L}$ ) in this order. The reaction was stirred at 40 °C for 40 hours. After the reaction, acetonitrile was added to the mixture to precipitate polymer complex. The insoluble materials were washed by acetonitrile to extract the product. After evaporation of the extract, the residue was purified by PTLC (hexane/Et<sub>2</sub>O = 4/1) to give a desired product (20 mg, 53%). The enantiomeric excesses of the

products were determined by SFC with a chiral stationary phase. The polymer catalyst remaining in the reaction vessel was dried under vacuum and used for the next run.

**[2nd and 3rd Runs]** To a mixture of polymer catalyst and THF (400  $\mu$ L) was added K<sub>3</sub>PO<sub>4</sub> (43 mg, 0.2 mmol), **1D** (0.12 mmol, 42 mg), **2g** (0.10 mmol, 15 mg), H<sub>2</sub>O (40  $\mu$ L) in this order. The reaction was stirred at 40 °C. After the reaction, acetonitrile was added to the mixture to precipitate polymer complex. The insoluble materials were washed by acetonitrile to extract the product. After evaporation of the extract, the residue was purified by PTLC (hexane/Et<sub>2</sub>O = 4/1) to give a desired product (2nd run, 25 mg, 66%; 3rd run, 26 mg 69%). The enantiomeric excesses of the products were determined by SFC with a chiral stationary phase.

## 2.6 Asymmetric Suzuki-Miyaura Coupling Using Helically Inversed PQXphos (Scheme 2)

(*R*)-**L6** (27 mg, 4.0  $\mu$ mol phosphorus atom) in 1,1,2-trichloroethane (0.6 mL) and THF (0.2 mL) was stirred at 60 °C for 24 h. To the mixture was added [PdCl( $\eta^3$ -C<sub>3</sub>H<sub>5</sub>)]<sub>2</sub> [0.01 M in THF/1,1,2-TCE (5/2), 100  $\mu$ L, 1  $\mu$ mol], and the solution was stirred at 60 °C for 10 min. To the mixture was added K<sub>3</sub>PO<sub>4</sub> (43 mg, 0.2 mmol), **1D** (35 mg, 0.1 mmol), **2f** (37 mg, 0.15 mmol), and H<sub>2</sub>O (40  $\mu$ L). The mixture was stirred at 40 °C for 48 h. After the reaction, subsequent addition of MeCN (10 mL) resulted in precipitation of the PQXphos. The suspension was passed through a pad of Celite® using MeCN as an eluent. The crude product was subjected to PTLC (hexane/Et<sub>2</sub>O = 4/1). Further purification was performed by GPC to give a desired product (44 mg, 93% yield). The enantiomeric excesses of the products were determined by SFC with a chiral stationary phase.

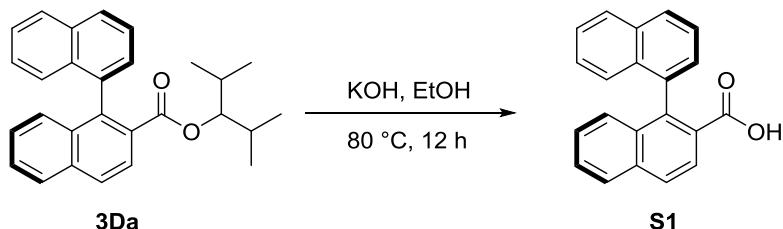
## 2.7 Reduction and Hydrolysis of (*S*)-**3Dg** (Scheme 3)

**[Reduction]** To a solution of (*S*)-**3Dg** (35 mg, 0.094 mmol) in Et<sub>2</sub>O (7.0 mL) was added lithium aluminum hydride (7 mg, 0.20 mmol) at 0 °C. The mixture was stirred at 80 °C for 1 h. After the reaction, the mixture was quenched by water and extracted with Et<sub>2</sub>O. After drying with anhydrous MgSO<sub>4</sub>, the concentrated mixture was purified by column chromatography (hexane/Et<sub>2</sub>O = 3/1) to give a desired product (*S*)-**4** (21 mg, 84% yield). The enantiomeric excess of this compound was determined by SFC analysis.

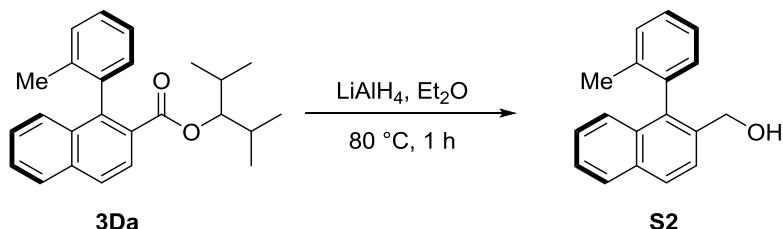
**[Hydrolysis]** To a solution of (*S*)-**3Dg** (131 mg, 0.35 mmol) in EtOH (30.0 mL) was added KOH (11 g, 200 mmol). The mixture was stirred at 80 °C for 12 hours. After the reaction, the mixture was extracted with Et<sub>2</sub>O and the extracts were washed with water. After drying with anhydrous MgSO<sub>4</sub>, the concentrated mixture was purified by column chromatography (hexane:Et<sub>2</sub>O = 1:1) to give a desired product (*S*)-**5** (94 mg, 98% yield). The enantiomeric

excess of this compound was determined by SFC analysis.

## 2.8 Determination of Absolute Configuration

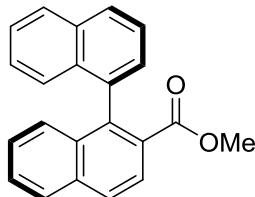


To a solution of **3Da** (26 mg, 0.066 mmol, 84% ee) in EtOH (7.0 mL) was added KOH (3.0 g, 53 mmol). The mixture was stirred at 80 °C for 12 hours. After the reaction, the mixture was extracted with Et<sub>2</sub>O and the extracts were washed with water. After drying with anhydrous MgSO<sub>4</sub>, the concentrated mixture was purified by column chromatography (hexane:Et<sub>2</sub>O = 1:1) to give a desired product **6** (16 mg, 83% yield). The enantiomeric excess of this compound was determined by SFC analysis. The absolute configuration was determined by comparing its optical rotation with reported data.<sup>S2a</sup> (**S1**): <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.10 (d, *J* = 8.4 Hz, 1H), 7.98 (d, *J* = 8.8 Hz, 1H), 7.92-7.95 (m, 3H), 7.52-7.57 (m, 2H), 7.45 (ddd, *J* = 8.4 Hz, 6.8 Hz, 1.2 Hz, 1H), 7.33 (dd, *J* = 6.8 Hz, 0.8 Hz, 1H), 7.21-7.30 (m, 3H), 7.15 (d, *J* = 8.0 Hz, 1H); [α]<sup>23</sup><sub>D</sub> -20.1 [c 0.810, CH<sub>2</sub>Cl<sub>2</sub>, 84% ee (S)].

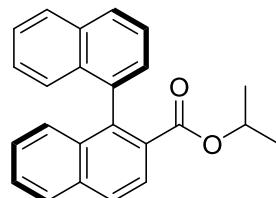


To a solution of **3Db** (42 mg, 0.12 mmol, 82% ee) in Et<sub>2</sub>O (7.0 mL) was added lithium aluminum hydride (9 mg, 0.24 mmol) at 0 °C. The mixture was stirred at 80 °C for 1 h. After the reaction, the mixture was quenched by water and extracted with Et<sub>2</sub>O. After drying with anhydrous MgSO<sub>4</sub>, the concentrated mixture was purified by column chromatography (hexane/Et<sub>2</sub>O = 3/1) to give a desired product **7** (23 mg, 80% yield). The enantiomeric excess of this compound was determined by SFC analysis. The absolute configuration was determined by comparing its optical rotation with reported data.<sup>S2b</sup> (**S2**): <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.81 (d, *J* = 8.8 Hz, 1H), 7.78 (d, *J* = 9.6 Hz, 1H), 7.61 (d, *J* = 8.4 Hz, 1H), 7.36 (ddd, *J* = 7.6 Hz, 6.8 Hz, 0.8 Hz, 1H), 7.15-7.28 (m, 5H), 7.05 (d, *J* = 7.2 Hz, 1H), 4.39 (d, *J* = 5.6 Hz, 2H), 1.82 (s, 3H); [α]<sup>22</sup><sub>D</sub> -33.7 [c 1.070, CH<sub>2</sub>Cl<sub>2</sub>, 81% ee (S)].

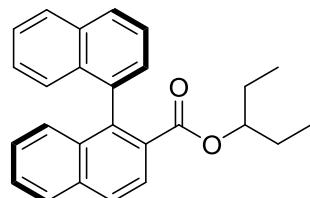
## 2.9 Spectral Data for New Compounds



**Methyl 1-(1-naphthyl)-2-naphthoate (3Aa):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.09 (d,  $J = 8.4$  Hz, 1H), 8.01 (d,  $J = 8.4$  Hz, 1H), 7.94–7.98 (m, 3H), 7.59 (dd,  $J = 8.4$  Hz, 6.8 Hz, 1H), 7.55 (ddd,  $J = 8.0$  Hz, 5.2 Hz, 2.4 Hz, 1H), 7.46 (ddd,  $J = 8.4$  Hz, 6.8 Hz, 1.6 Hz, 1H), 7.37 (dd,  $J = 6.8$  Hz, 1.2 Hz, 1H), 7.29–7.32 (m, 2H), 7.25–7.28 (m, 1H), 7.22 (d,  $J = 8.4$  Hz, 1H), 3.44 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  168.0, 140.2, 136.9, 134.8, 133.2, 133.1, 132.9, 128.7, 128.2, 128.1, 128.0, 127.8, 127.8, 127.6, 126.9, 126.7, 126.0, 126.0, 125.7, 125.7, 125.2, 51.8; IR (ATR)  $\nu$  3057, 2947, 2841, 1724, 1712, 1504, 1431, 1280, 1240, 1122, 831, 798, 763, 731  $\text{cm}^{-1}$ ; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{22}\text{H}_{16}\text{O}_2+\text{H}^+$  ( $\text{M}+\text{H}^+$ ): 313.1223, found: 313.1215;  $[\alpha]^{27}_D -20.1$  [c 1.240,  $\text{CH}_2\text{Cl}_2$ , 70% ee (S)].

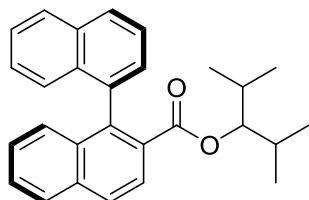


**Isopropyl 1-(1-naphthyl)-2-naphthoate (3Ba):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.07 (d,  $J = 8.8$  Hz, 1H), 8.00 (d,  $J = 8.8$  Hz, 1H), 7.92–7.99 (m, 3H), 7.56 (dd,  $J = 8.0$  Hz, 6.8 Hz, 1H), 7.53 (ddd,  $J = 8.4$  Hz, 6.4 Hz, 2.0 Hz, 1H), 7.45 (ddd,  $J = 8.4$  Hz, 5.2 Hz, 3.2 Hz, 1H), 7.25–7.35 (m, 5H), 4.74 (sep,  $J = 6.4$  Hz, 1H), 0.57 (d,  $J = 6.4$  Hz, 3H), 0.54 (d,  $J = 6.4$  Hz, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  167.7, 139.3, 137.3, 134.7, 133.3, 133.3, 133.1, 129.8, 128.0, 128.0, 127.9, 127.9, 127.6, 127.4, 127.1, 126.6, 126.4, 125.9, 125.8, 125.7, 125.1, 67.9, 20.8, 20.8; IR (ATR)  $\nu$  3057, 2977, 2931, 1699, 1371, 1278, 1103, 822, 766  $\text{cm}^{-1}$ ; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{24}\text{H}_{20}\text{O}_2+\text{H}^+$  ( $\text{M}+\text{H}^+$ ): 341.1536, found: 341.1530;  $[\alpha]^{28}_D -7.3$  [c 1.050,  $\text{CH}_2\text{Cl}_2$ , 79% ee (S)].

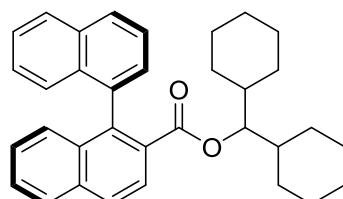


**3-pentyl 1-(1-naphthyl)-2-naphthoate (3Ca):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.14 (d,  $J = 8.4$

Hz, 1H), 8.02 (d,  $J$  = 8.8 Hz, 1H), 7.94 (m, 3H), 7.56 (dd,  $J$  = 8.4 Hz, 7.2 Hz, 1H), 7.54 (ddd,  $J$  = 8.4 Hz, 5.2 Hz, 4.8 Hz, 1H), 7.45 (ddd,  $J$  = 8.0 Hz, 4.4 Hz, 3.2 Hz, 1H), 7.36 (dd,  $J$  = 6.8 Hz, 1.2 Hz, 1H), 7.28-7.30 (m, 2H), 7.24-7.27 (m, 2H), 4.58 (sep,  $J$  = 2.4 Hz, 1H), 0.74-1.09 (m, 4H), 0.54 (t,  $J$  = 7.2 Hz, 3H), 0.43 (t,  $J$  = 7.6 Hz, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  167.9, 139.5, 137.4, 134.8, 133.3, 133.3, 133.2, 129.5, 128.1, 128.0 (2C), 127.8, 127.6, 127.5, 127.0, 126.6, 126.4, 126.1, 125.9, 125.7, 125.1, 77.4, 25.9, 25.7, 9.5, 9.2; IR (ATR)  $\nu$  3057, 2966, 2933, 1699, 1327, 1269, 1136, 833, 766  $\text{cm}^{-1}$ ; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{26}\text{H}_{24}\text{O}_2+\text{H}^+$  ( $\text{M}+\text{H}^+$ ): 369.1849, found: 369.1841;  $[\alpha]^{29}\text{D}$  -6.4 [c 1.105,  $\text{CH}_2\text{Cl}_2$ , 82% ee (S)].

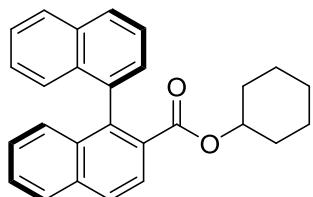


**2,4-Dimethylpentan-3-yl 1-(1-naphthyl)-2-naphthoate (3Da):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.13 (d,  $J$  = 8.8 Hz, 1H), 8.01 (d,  $J$  = 8.4 Hz, 1H), 7.95 (d,  $J$  = 2.4 Hz, 1H), 7.93 (d,  $J$  = 2.0 Hz, 1H), 7.92 (d,  $J$  = 8.4 Hz, 1H), 7.56 (dd,  $J$  = 8.0 Hz, 6.8 Hz, 1H), 7.52 (ddd,  $J$  = 8.0 Hz, 6.4 Hz, 1.6 Hz, 1H), 7.43 (ddd,  $J$  = 8.0 Hz, 5.2 Hz, 2.8 Hz, 1H), 7.38 (dd,  $J$  = 6.8 Hz, 1.2 Hz, 1H), 7.20-7.29 (m, 4H), 4.55 (t,  $J$  = 6.0 Hz, 1H), 1.46 (sep,  $J$  = 6.4 Hz, 1H), 1.39 (sep,  $J$  = 6.4 Hz, 1H), 0.66 (d,  $J$  = 6.8 Hz, 3H), 0.63 (d,  $J$  = 6.8 Hz, 3H) 0.50 (d,  $J$  = 6.4 Hz, 3H), 0.30 (d,  $J$  = 6.4 Hz, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  168.1, 139.4, 137.1, 134.7, 133.4, 133.3, 133.1, 129.6, 128.1, 128.0, 127.8, 127.8, 127.4, 127.3, 126.6, 126.4, 126.2, 126.0, 125.7, 125.2, 83.7, 29.1, 19.2, 19.2, 17.4 (2C), 16.5 (2C); IR (ATR)  $\nu$  2963, 2359, 1726, 1699, 1558, 1506, 1456, 1232, 1117, 800, 770  $\text{cm}^{-1}$ ; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{28}\text{H}_{28}\text{O}_2+\text{H}^+$  ( $\text{M}+\text{H}^+$ ): 397.2162, found: 397.2155;  $[\alpha]^{29}\text{D}$  -6.7 [c 1.030,  $\text{CH}_2\text{Cl}_2$ , 87% ee (S)].

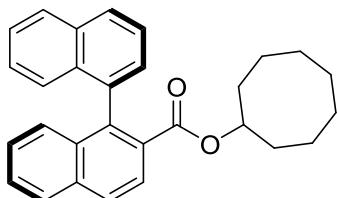


**Dicyclohexylmethyl 1-(1-naphthyl)-2-naphthoate (3Ea):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.16 (d,  $J$  = 8.8 Hz, 1H), 8.01 (d,  $J$  = 8.8 Hz, 1H), 7.95 (d,  $J$  = 1.6 Hz, 1H), 7.94 (s, 1H), 7.92 (d,  $J$  = 8.4 Hz, 1H), 7.56 (dd,  $J$  = 8.0 Hz, 6.8 Hz, 1H), 7.52 (ddd,  $J$  = 8.0 Hz, 6.8 Hz, 1.2 Hz, 1H), 7.44 (ddd,  $J$  = 8.0 Hz, 6.0 Hz, 2.4 Hz, 1H), 7.38 (dd,  $J$  = 6.8 Hz, 1.2 Hz, 1H), 7.19-7.28 (m, 4H), 4.59 (t,  $J$  = 6.0 Hz, 1H), 0.03-1.57 (m, 22H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  168.2, 139.3, 137.2,

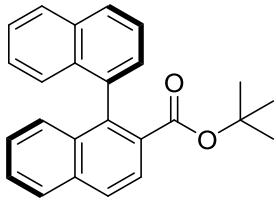
134.7, 133.4, 133.3, 133.2, 129.4, 128.1 (2C), 128.0, 127.8, 127.6, 127.4, 127.2, 126.5 (2C), 126.5, 126.0, 125.6, 125.1, 82.3, 37.9 (2C), 29.5, 29.2, 27.7, 26.2, 26.2, 26.1, 26.1, 26.0, 25.9, 25.8; IR (ATR)  $\nu$  2924, 2849, 1726, 1699, 1446, 1271, 1121, 907, 766, 729 cm<sup>-1</sup>; HRMS (ESI)  $m/z$  calcd for C<sub>34</sub>H<sub>36</sub>O<sub>2</sub>+H<sup>+</sup> (M+H<sup>+</sup>): 477.2788, found: 477.2777; [ $\alpha$ ]<sub>D</sub><sup>29</sup> -1.2 [c 1.735, CH<sub>2</sub>Cl<sub>2</sub>, 82% ee (S)].



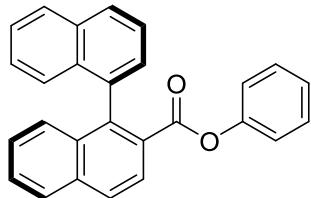
**Cyclohexyl 1-(1-naphthyl)-2-naphthoate (3Fa):** <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.07 (d,  $J$  = 8.4 Hz, 1H), 8.00 (d,  $J$  = 8.4 Hz, 1H), 7.96 (d,  $J$  = 4.0 Hz, 1H), 7.94 (d,  $J$  = 4.0 Hz, 1H), 7.93 (d,  $J$  = 8.0 Hz, 1H), 7.56 (dd,  $J$  = 8.0 Hz, 6.8 Hz, 1H), 7.53 (ddd,  $J$  = 8.0 Hz, 4.8 Hz, 2.8 Hz, 1H), 7.45 (ddd,  $J$  = 8.0 Hz, 3.6 Hz, 3.6 Hz, 1H), 7.35 (dd,  $J$  = 6.8 Hz, 1.2 Hz, 1H), 7.25-7.29 (m, 4H), 4.53 (tt,  $J$  = 9.6 Hz, 4.0 Hz, 1H), 1.25-1.34 (m, 5H), 0.99-1.12 (m, 2H), 0.87-0.97 (m, 1H) 0.52-0.65 (m, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  167.7, 139.2, 137.3, 134.7, 133.3, 133.2, 133.1, 129.9, 128.0 (2C), 127.9, 127.7, 127.4, 127.1, 126.6, 126.4, 125.9 (2C), 125.7, 125.2, 73.1, 30.7 (2C), 25.1 (2C), 23.4, 23.4; IR (ATR)  $\nu$  2930, 1699, 1327, 1279, 1111, 912, 764 cm<sup>-1</sup>; HRMS (ESI)  $m/z$  calcd for C<sub>27</sub>H<sub>24</sub>O<sub>2</sub>+H<sup>+</sup> (M+H<sup>+</sup>): 381.1849, found: 381.1840; [ $\alpha$ ]<sub>D</sub><sup>30</sup> -5.2 [c 1.030, CH<sub>2</sub>Cl<sub>2</sub>, 80% ee (S)].



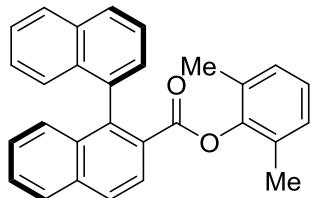
**Cyclooctyl 1-(1-naphthyl)-2-naphthoate (3Ga):** <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.06 (d,  $J$  = 8.8 Hz, 1H), 8.00 (d,  $J$  = 8.8 Hz, 1H), 7.95 (dd,  $J$  = 6.0 Hz, 0.8 Hz, 1H), 7.92-7.94 (m, 2H), 7.56 (dd,  $J$  = 8.0 Hz, 6.8 Hz, 1H), 7.52 (ddd,  $J$  = 8.0 Hz, 4.0 Hz, 4.0 Hz, 1H), 7.46 (ddd,  $J$  = 8.0 Hz, 4.8 Hz, 2.8 Hz, 1H), 7.34 (dd,  $J$  = 7.2 Hz, 1.2 Hz, 1H), 7.25-7.29 (m, 4H), 4.72 (sep,  $J$  = 4.0 Hz, 1H), 1.10-1.31 (m, 12H), 0.80-1.02 (m, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  167.7, 139.0, 137.3, 134.7, 133.4, 133.3, 133.1, 130.0, 128.0, 128.0, 127.9, 127.9, 127.7, 127.3, 127.1, 126.5, 126.4, 126.0, 125.9, 125.7, 125.2, 75.7, 30.0, 30.0, 27.1, 27.1, 25.0, 22.4, 22.3; IR (ATR)  $\nu$  2922, 1705, 1335, 1269, 1134, 945, 762 cm<sup>-1</sup>; HRMS (ESI)  $m/z$  calcd for C<sub>29</sub>H<sub>28</sub>O<sub>2</sub>+H<sup>+</sup> (M+H<sup>+</sup>): 409.2162, found: 409.2152; [ $\alpha$ ]<sub>D</sub><sup>30</sup> -10.5 [c 1.240, CH<sub>2</sub>Cl<sub>2</sub>, 80% ee (S)].



**tert-Butyl 1-(1-naphthyl)-2-naphthoate (3Ha):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.92-7.99 (m, 5H), 7.56 (dd,  $J = 8.4$  Hz, 6.8 Hz, 1H), 7.51 (ddd,  $J = 8.4$  Hz, 6.0 Hz, 2.4 Hz, 1H), 7.46 (ddd,  $J = 8.0$  Hz, 5.6 Hz, 2.4 Hz, 1H), 7.35 (dd,  $J = 6.8$  Hz, 1.2 Hz, 1H), 7.24-7.31 (m, 4H), 0.81 (s, 9H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  168.0, 138.2, 137.4, 134.5, 133.4, 133.3, 133.0, 131.4, 128.0, 127.9, 127.8, 127.6, 127.3, 127.2, 126.6, 126.5, 126.0, 125.8, 125.7, 125.2, 80.9, 27.1 (3C); IR (ATR)  $\nu$  2976, 1699, 1367, 1334, 1286, 1124, 853, 766  $\text{cm}^{-1}$ ; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{25}\text{H}_{22}\text{O}_2+\text{Na}^+$  ( $\text{M}+\text{Na}^+$ ): 377.1512, found: 377.1502;  $[\alpha]^{27}_D -21.5$  [ $c$  0.810,  $\text{CH}_2\text{Cl}_2$ , 68% ee (*S*)].

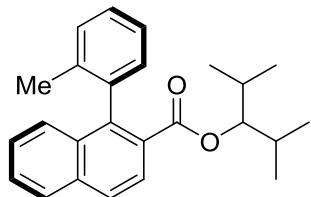


**Phenyl 1-(1-naphthyl)-2-naphthoate (3Ia):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.20 (d,  $J = 8.4$  Hz, 1H), 8.08 (d,  $J = 8.4$  Hz, 1H), 8.00 (d,  $J = 8.0$  Hz, 1H), 7.96 (d,  $J = 8.4$  Hz, 1H), 7.94 (d,  $J = 8.8$  Hz, 1H), 7.56-7.61 (m, 2H), 7.44-7.48 (m, 2H), 7.39 (d,  $J = 8.0$  Hz, 1H), 7.27-7.36 (m, 3H), 7.13-7.19 (m, 2H), 7.06 (dddd,  $J = 8.8$  Hz, 6.8Hz, 1.2 Hz, 1.2 Hz, 1H), 6.46-6.49 (m, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.4, 150.4, 140.3, 136.6, 135.0, 133.3, 130.0, 132.9, 129.0 (2C), 128.4, 128.2, 128.1, 128.0, 128.0, 127.9, 127.8, 127.3, 126.8, 126.2, 126.0, 125.8, 125.4, 125.1, 121.0 (2C); IR (ATR)  $\nu$  2962, 1728, 1591, 1265, 1188, 1099, 1068, 798, 762  $\text{cm}^{-1}$ ; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{27}\text{H}_{18}\text{O}_2+\text{H}^+$  ( $\text{M}+\text{H}^+$ ): 375.1380, found: 375.1370;  $[\alpha]^{30}_D +29.7$  [ $c$  0.640,  $\text{CH}_2\text{Cl}_2$ , 78% ee (*S*)].

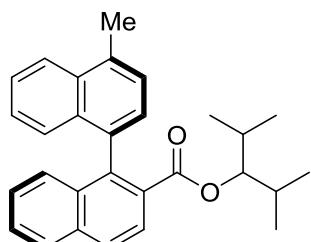


**2,6-Dimethylphenyl 1-(1-naphthyl)-2-naphthoate (3Ja):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.32

(d,  $J = 8.8$  Hz, 1H), 8.09 (d,  $J = 8.8$  Hz, 1H), 8.01 (d,  $J = 8.0$  Hz, 1H), 7.92 (d,  $J = 8.4$  Hz, 1H), 7.90 (d,  $J = 8.4$  Hz, 1H), 7.55-7.61 (m, 2H), 7.48 (dd,  $J = 6.8$  Hz, 1.2 Hz, 1H), 7.43 (ddd,  $J = 8.4$  Hz, 6.0 Hz, 2.0 Hz, 1H), 7.24-7.34 (m, 4H), 6.88-6.92 (m, 3H), 1.83 (s, 6H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  165.2, 148.2, 140.9, 136.5, 135.0, 133.4, 133.1, 130.0, 128.3 (2C), 128.2, 128.2, 128.1, 127.9, 127.9, 127.3, 126.8, 126.1 (2C), 126.1, 125.9, 125.7, 125.5 (2C), 125.1, 16.1 (2C); IR (ATR)  $\nu$  2926, 1746, 1230, 1159, 1109, 800, 772, 764  $\text{cm}^{-1}$ ; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{29}\text{H}_{22}\text{O}_2+\text{H}^+$  ( $\text{M}+\text{H}^+$ ): 403.1693, found: 403.1682;  $[\alpha]^{30}_{\text{D}} +29.7$  [c 0.945,  $\text{CH}_2\text{Cl}_2$ , 85% ee (*S*)].

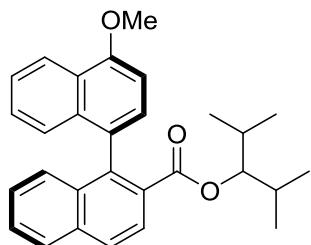


**2,4-Dimethylpentan-3-yl 1-(2-methylphenyl)-2-naphthoate (3Db):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.02 (d,  $J = 8.8$  Hz, 1H), 7.91 (d,  $J = 8.4$  Hz, 1H), 7.91 (d,  $J = 8.0$  Hz, 1H), 7.54 (ddd,  $J = 8.0$  Hz, 6.4 Hz, 0.8 Hz, 1H), 7.40 (ddd,  $J = 8.4$  Hz, 6.4 Hz, 1.2 Hz, 1H), 7.29-7.36 (m, 3H), 7.25 (ddd,  $J = 7.2$  Hz, 1.6 Hz, 0.8 Hz, 1H), 7.12 (dd,  $J = 6.8$  Hz, 1.2 Hz, 1H), 4.71 (t,  $J = 6.4$  Hz, 1H), 1.97 (s, 3H), 1.79 (sep,  $J = 6.8$  Hz, 1H), 1.71 (sep,  $J = 6.8$  Hz, 1H), 0.81 (d,  $J = 6.8$  Hz, 3H), 0.80 (d,  $J = 6.8$  Hz, 3H), 0.74 (d,  $J = 6.8$  Hz, 3H), 0.74 (d,  $J = 6.8$  Hz, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  168.1, 140.7, 138.7, 136.7, 134.6, 132.4, 129.6, 129.5, 128.4, 127.9, 127.6, 127.5, 127.4, 127.3, 126.6, 125.9, 125.4, 83.6, 29.4, 29.4, 20.0, 19.4, 19.4, 17.5, 17.2; IR (ATR)  $\nu$  2922, 1695, 1558, 1261, 1118, 937, 770, 760, 731  $\text{cm}^{-1}$ ; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{25}\text{H}_{28}\text{O}_2+\text{H}^+$  ( $\text{M}+\text{H}^+$ ): 361.2162, found: 361.2151;  $[\alpha]^{25}_{\text{D}} -40.5$  [c 1.020,  $\text{CH}_2\text{Cl}_2$ , 88% ee (*S*)]

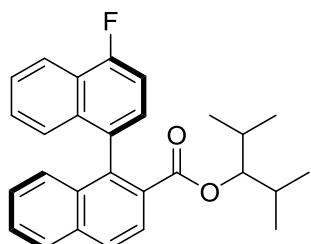


**2,4-Dimethylpentan-3-yl 1-(4-methyl-1-naphthyl)-2-naphthoate (3Dc):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.08 (d,  $J = 8.4$  Hz, 1H), 8.05 (d,  $J = 8.4$  Hz, 1H), 7.97 (d,  $J = 8.8$  Hz, 1H), 7.91 (d,  $J = 8.4$  Hz, 1H), 7.50 (dd,  $J = 8.0$  Hz, 4.0 Hz, 1H), 7.45 (ddd,  $J = 8.4$  Hz, 4.8 Hz, 2.8 Hz, 1H), 7.38 (dd,  $J = 7.2$  Hz, 0.8 Hz, 1H), 7.21-7.26 (m, 5H), 4.53 (t,  $J = 6.4$  Hz, 1H), 2.78 (d,  $J = 0.4$  Hz, 3H), 1.49 (sep,  $J = 6.8$  Hz, 1H), 1.42 (sep,  $J = 6.8$  Hz, 1H), 0.65 (d,  $J = 6.8$  Hz, 3H), 0.61 (d,

$J = 6.8$  Hz, 3H), 0.47 (d,  $J = 6.8$  Hz, 3H), 0.36 (d,  $J = 6.8$  Hz, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  168.1, 139.7, 135.3, 134.6, 134.0, 133.5, 133.2, 132.5, 129.7, 128.1, 127.8, 127.7, 127.3, 127.0, 126.9, 126.5, 126.1, 125.9, 125.6, 125.5, 124.2, 83.5, 29.1, 29.1, 19.5, 19.2, 19.2, 17.2, 16.6; IR (ATR)  $\nu$  2962, 1699, 1456, 1271, 1236, 1124, 947, 827, 750  $\text{cm}^{-1}$ ; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{29}\text{H}_{30}\text{O}_2+\text{H}^+$  ( $\text{M}+\text{H}^+$ ): 411.2319, found: 411.2308;  $[\alpha]^{29}_{\text{D}} -5.6$  [c 0.815,  $\text{CH}_2\text{Cl}_2$ , 92% ee (S)].

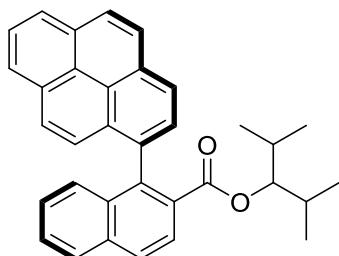


**2,4-Dimethylpentan-3-yl 1-(4-methoxy-1-naphthyl)-2-naphthoate (3Dd):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.34 (d,  $J = 8.4$  Hz, 1H), 8.07 (d,  $J = 8.8$  Hz, 1H), 7.98 (d,  $J = 8.4$  Hz, 1H), 7.93 (d,  $J = 8.0$  Hz, 1H), 7.51 (ddd,  $J = 8.0$  Hz, 6.0 Hz, 1.6 Hz, 1H), 7.42 (ddd,  $J = 8.0$  Hz, 6.8 Hz, 1.2 Hz, 1H), 7.23-7.32 (m, 4H), 7.17 (d,  $J = 8.4$  Hz, 1H), 6.91 (d,  $J = 8.0$  Hz, 1H), 4.55 (t,  $J = 6.0$  Hz, 1H), 4.08 (s, 3H), 1.53 (sep,  $J = 6.8$  Hz, 1H), 1.44 (sep,  $J = 6.8$  Hz, 1H), 0.69 (d,  $J = 6.8$  Hz, 3H), 0.62 (d,  $J = 6.8$  Hz, 3H), 0.48 (d,  $J = 6.8$  Hz, 3H), 0.41 (d,  $J = 6.8$  Hz, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  168.4, 155.2, 139.3, 134.6, 134.0, 133.7, 130.2, 129.1, 128.1, 127.8, 127.8, 127.3, 127.2, 126.5, 126.5, 126.3, 126.0, 125.4, 125.0, 122.0, 103.3, 83.5, 55.6, 29.2, 29.2, 19.3, 19.2, 17.2, 16.6; IR (ATR)  $\nu$  2924, 1718, 1683, 1558, 1506, 1456, 1236, 1088, 762  $\text{cm}^{-1}$ ; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{29}\text{H}_{30}\text{O}_3+\text{H}^+$  ( $\text{M}+\text{H}^+$ ): 427.2268, found: 427.2260;  $[\alpha]^{30}_{\text{D}} +1.7$  [c 1.870,  $\text{CH}_2\text{Cl}_2$ , 88% ee (S)].

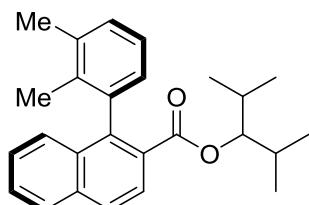


**2,4-Dimethylpentan-3-yl 1-(4-fluoro-1-naphthyl)-2-naphthoate (3De):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.20 (d,  $J = 8.4$  Hz, 1H), 8.11 (d,  $J = 8.8$  Hz, 1H), 8.01 (d,  $J = 8.4$  Hz, 1H), 7.95 (d,  $J = 8.0$  Hz, 1H), 7.53 (ddd,  $J = 8.4$  Hz, 6.8 Hz, 1.2 Hz, 1H), 7.50 (ddd,  $J = 8.4$  Hz, 6.8 Hz, 1.2 Hz, 1H), 7.20-7.32 (m, 6H), 4.56 (t,  $J = 6.4$  Hz, 1H), 1.53 (sep,  $J = 6.8$  Hz, 1H), 1.48 (sep,  $J = 6.8$  Hz, 1H), 0.68 (d,  $J = 6.8$  Hz, 3H), 0.65 (d,  $J = 7.2$  Hz, 3H), 0.54 (d,  $J = 6.8$  Hz, 3H), 0.37 (d,  $J =$

6.8 Hz, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  168.0, 158.5 ( $J_{\text{CF}} = 250.1$  Hz), 138.5, 134.7, 134.4 ( $J_{\text{CF}} = 4.7$  Hz), 133.4, 133.0 ( $J_{\text{CF}} = 4.6$  Hz), 129.9, 128.2, 127.9, 127.8, 127.5, 127.0, 126.8 ( $J_{\text{CF}} = 8.5$  Hz), 126.7, 126.4 ( $J_{\text{CF}} = 2.3$  Hz), 126.1, 126.0 ( $J_{\text{CF}} = 1.5$  Hz), 123.6 ( $J_{\text{CF}} = 16.3$  Hz), 120.6 ( $J_{\text{CF}} = 5.4$  Hz), 108.8 ( $J_{\text{CF}} = 20.1$  Hz), 83.7, 29.2 (2C), 19.3, 19.2, 17.3, 16.5; IR (ATR)  $\nu$  2966, 1718, 1684, 1558, 1506, 1456, 1246, 1126, 953, 841, 759  $\text{cm}^{-1}$ ; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{28}\text{H}_{27}\text{O}_2\text{F}+\text{H}^+$  ( $\text{M}+\text{H}^+$ ): 415.2068, found: 415.2057;  $[\alpha]^{30}_{\text{D}} -7.1$  [c 1.955,  $\text{CH}_2\text{Cl}_2$ , 90% ee (S)].

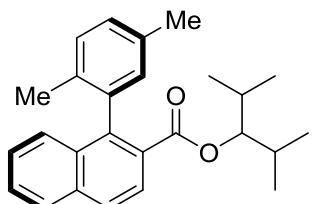


**2,4-Dimethylpentan-3-yl 1-(1-pyrenyl)-2-naphthoate (3Df):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.27 (d,  $J = 7.6$  Hz, 1H), 8.06-8.23 (m, 6H), 8.01 (d,  $J = 7.6$  Hz, 1H), 7.99 (d,  $J = 7.6$  Hz, 1H), 7.94 (d,  $J = 8.0$  Hz, 1H), 7.85 (d,  $J = 9.2$  Hz, 1H), 7.53 (ddd,  $J = 8.4$  Hz, 6.8 Hz, 1.2 Hz, 1H), 7.48 (d,  $J = 9.2$  Hz, 1H), 7.23 (ddd,  $J = 8.4$  Hz, 6.4 Hz, 1.2 Hz, 1H), 7.12 (dd,  $J = 8.4$  Hz, 0.8 Hz, 1H), 4.48 (t,  $J = 6.8$  Hz, 1H), 1.41 (sep,  $J = 6.8$  Hz, 1H), 1.38 (sep,  $J = 6.4$  Hz, 1H), 0.59 (d,  $J = 6.8$  Hz, 3H), 0.56 (d,  $J = 6.8$  Hz, 3H), 0.45 (d,  $J = 6.8$  Hz, 3H), 0.27 (d,  $J = 6.4$  Hz, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  167.9, 139.8, 134.6, 134.4, 133.5, 131.3, 130.9, 130.8, 129.9, 129.7, 128.1, 128.0, 127.8, 127.8, 127.4, 127.4 (2C), 127.2, 126.6, 126.0, 125.8, 125.5, 125.0, 124.9, 124.7, 124.6, 124.2, 83.5, 29.0, 29.0, 19.1, 19.1, 17.0, 16.5; IR (ATR)  $\nu$  2964, 1716, 1699, 1456, 1276, 1186, 847, 750, 721, 667  $\text{cm}^{-1}$ ; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{34}\text{H}_{30}\text{O}_2+\text{H}^+$  ( $\text{M}+\text{H}^+$ ): 471.2319, found: 471.2307;  $[\alpha]^{30}_{\text{D}} -67.1$  [c 2.165,  $\text{CH}_2\text{Cl}_2$ , 95% ee (S)].

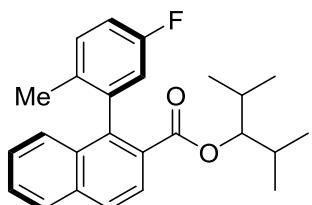


**2,4-Dimethylpentan-3-yl 1-(2,3-dimethylphenyl)-2-naphthoate (3Dg):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.01 (d,  $J = 8.8$  Hz, 1H), 7.91 (d,  $J = 8.0$  Hz, 1H), 7.90 (d,  $J = 8.8$  Hz, 1H), 7.53 (ddd,  $J = 8.4$  Hz, 6.8 Hz, 1.6 Hz, 1H), 7.32-7.40 (m, 2H), 7.22 (d,  $J = 7.6$  Hz, 1H), 7.14 (dd,  $J = 7.6$  Hz, 7.6 Hz, 1H), 6.98 (d,  $J = 7.6$  Hz, 1H), 4.71 (t,  $J = 6.0$  Hz, 1H), 2.36 (s, 3H), 1.87 (s, 3H), 1.76 (sep,  $J = 6.8$  Hz, 1H), 1.69 (sep,  $J = 6.8$  Hz, 1H), 0.81 (d,  $J = 7.2$  Hz, 3H), 0.79 (d,  $J = 6.8$  Hz, 3H), 0.72 (d,  $J = 6.8$  Hz, 3H), 0.72 (d,  $J = 6.8$  Hz, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$

168.3, 141.3, 138.7, 136.3, 135.1, 134.6, 132.6, 129.1, 128.5, 127.8, 127.6, 127.5, 127.3, 127.3, 126.5, 126.0, 125.0, 83.5, 29.4, 29.3, 20.4, 19.4, 19.4, 17.4, 17.1, 16.7; IR (ATR)  $\nu$  2958, 1695, 1558, 1456, 1327, 1273, 1265, 1119, 891, 772 cm<sup>-1</sup>; HRMS (ESI) *m/z* calcd for C<sub>26</sub>H<sub>30</sub>O<sub>2</sub>+H<sup>+</sup> (M+H<sup>+</sup>): 375.2319, found: 375.2309; [ $\alpha$ ]<sub>D</sub><sup>26</sup> -44.6 [c 1.460, CH<sub>2</sub>Cl<sub>2</sub>, 96% ee (S)].

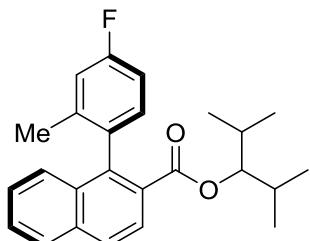


**2,4-Dimethylpentan-3-yl 1-(2,5-dimethylphenyl)-2-naphthoate (3Dh):** <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.00 (d, *J* = 8.8 Hz, 1H), 7.90 (d, *J* = 8.4 Hz, 2H), 7.53 (ddd, *J* = 8.4 Hz, 6.0 Hz, 2.4 Hz, 1H), 7.34-7.41 (m, 2H), 7.19 (d, *J* = 7.6 Hz, 1H), 7.13 (dd, *J* = 8.0 Hz, 1.6 Hz, 1H), 6.94 (d, *J* = 1.2 Hz, 1H), 4.71 (t, *J* = 6.0 Hz, 1H), 2.31 (s, 3H), 1.93 (s, 3H), 1.78 (sep, *J* = 6.8 Hz, 1H), 1.69 (sep, *J* = 6.8 Hz, 1H), 0.81 (d, *J* = 6.8 Hz, 6H), 0.74 (d, *J* = 6.8 Hz, 3H), 0.73 (d, *J* = 6.8 Hz, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  168.3, 140.8, 138.5, 134.6, 134.6, 133.6, 132.4, 130.3, 129.4, 128.5, 128.3, 127.9, 127.5, 127.4, 127.3, 126.6, 126.0, 83.6, 29.4 (2C), 20.9, 19.5, 19.4, 19.4, 17.4, 17.1; IR (ATR)  $\nu$  2962, 1699, 1558, 1464, 1329, 1261, 1234, 1121, 1096, 943, 899, 810, 768 cm<sup>-1</sup>; HRMS (ESI) *m/z* calcd for C<sub>26</sub>H<sub>30</sub>O<sub>2</sub>+H<sup>+</sup> (M+H<sup>+</sup>): 375.2319, found: 375.2309; [ $\alpha$ ]<sub>D</sub><sup>27</sup> -44.2 [c 0.965, CH<sub>2</sub>Cl<sub>2</sub>, 91% ee (S)].

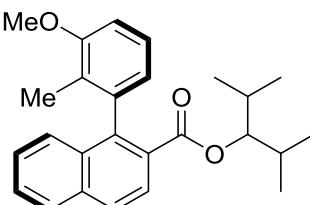


**2,4-Dimethylpentan-3-yl 1-(5-fluoro-2-methylphenyl)-2-naphthoate (3Di):** <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.04 (d, *J* = 8.4 Hz, 1H), 7.93 (d, *J* = 8.4 Hz, 1H), 7.92 (d, *J* = 6.4 Hz, 1H), 7.56 (ddd, *J* = 8.4 Hz, 6.8 Hz, 1.2 Hz, 1H), 7.41 (ddd, *J* = 8.4 Hz, 6.8 Hz, 1.2 Hz, 1H), 7.33 (d, *J* = 8.0 Hz, 1H), 7.26 (dd, *J* = 8.4 Hz, 6.4 Hz, 1H), 7.04 (ddd, *J* = 8.4 Hz, 8.4 Hz, 2.8 Hz, 1H), 6.87 (dd, *J* = 9.2 Hz, 2.8 Hz, 1H), 4.73 (t, *J* = 6.4 Hz, 1H), 1.91 (s, 3H), 1.80 (sep, *J* = 6.8 Hz, 1H), 1.75 (sep, *J* = 6.8 Hz, 1H), 0.84 (d, *J* = 6.8 Hz, 3H), 0.83 (d, *J* = 6.8 Hz, 3H), 0.81 (d, *J* = 7.2 Hz, 3H), 0.75 (d, *J* = 6.8 Hz, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  167.7, 160.9 (*J*<sub>CF</sub> = 243.1 Hz), 140.5 (*J*<sub>CF</sub> = 7.8 Hz), 139.5, 134.7, 132.4 (*J*<sub>CF</sub> = 3.1 Hz), 132.0, 130.8 (*J*<sub>CF</sub> = 8.5 Hz), 128.2, 128.0, 127.9, 127.6, 127.1, 126.9, 126.0, 116.5 (*J*<sub>CF</sub> = 20.9 Hz), 114.2 (*J*<sub>CF</sub> = 20.9 Hz), 83.8, 29.4, 29.4 (2C), 19.1, 17.4, 17.2; IR (ATR)  $\nu$  2924, 1717, 1558, 1488, 1254, 1130, 951,

889, 804, 760  $\text{cm}^{-1}$ ; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{25}\text{H}_{27}\text{FO}_2+\text{H}^+$  ( $\text{M}+\text{H}^+$ ): 379.2068, found: 379.2059;  $[\alpha]^{28}_{\text{D}} -37.5$  [c 1.705,  $\text{CH}_2\text{Cl}_2$ , 93% ee (S)].

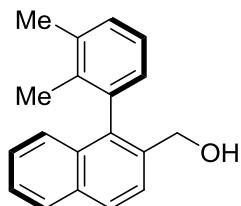


**(2,4-Dimethylpentan-3-yl 1-(4-fluoro-2-methylphenyl)-2-naphthoate (3Dj):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.01 (d,  $J = 8.8$  Hz, 1H), 7.92 (d,  $J = 8.4$  Hz, 1H), 7.91 (d,  $J = 8.4$  Hz, 1H), 7.55 (ddd,  $J = 8.0$  Hz, 6.8 Hz, 1.2 Hz, 1H), 7.42 (ddd,  $J = 8.4$  Hz, 6.8 Hz, 1.2 Hz, 1H), 7.31 (dd,  $J = 8.0$  Hz, 0.4 Hz, 1H), 7.08 (dd,  $J = 8.4$  Hz, 6.0 Hz, 1H), 7.04 (dd,  $J = 9.6$  Hz, 2.4 Hz, 1H), 6.96 (ddd,  $J = 8.8$  Hz, 8.8 Hz, 2.4 Hz, 1H), 4.72 (t,  $J = 6.4$  Hz, 1H), 1.96 (s, 3H), 1.83 (sep,  $J = 6.8$  Hz, 1H), 1.77 (sep,  $J = 6.8$  Hz, 1H), 0.83 (d,  $J = 6.8$  Hz, 6H), 0.77 (d,  $J = 6.8$  Hz, 3H), 0.77 (d,  $J = 6.8$  Hz, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  167.9, 162.3 ( $J_{\text{CF}} = 243.9$  Hz), 139.6, 139.2 ( $J_{\text{CF}} = 7.7$  Hz), 134.7, 134.5 ( $J_{\text{CF}} = 3.1$  Hz), 132.5, 130.9 ( $J_{\text{CF}} = 7.7$  Hz), 128.8, 128.0, 127.8, 127.4, 127.1, 126.8, 125.9, 116.3 ( $J_{\text{CF}} = 20.9$  Hz), 112.2 ( $J_{\text{CF}} = 20.9$  Hz), 83.7, 29.4 (2C), 20.1, 19.4, 19.4, 17.4, 17.2; IR (ATR)  $\nu$  2923, 1684, 1558, 1506, 1456, 1329, 1238, 1110, 895, 770  $\text{cm}^{-1}$ ; HRMS (ESI)  $m/z$  calcd for  $\text{C}_{25}\text{H}_{27}\text{FO}_2+\text{H}^+$  ( $\text{M}+\text{H}^+$ ): 379.2068, found: 379.2060;  $[\alpha]^{28}_{\text{D}} -34.1$  [c 1.730,  $\text{CH}_2\text{Cl}_2$ , 87% ee (S)].

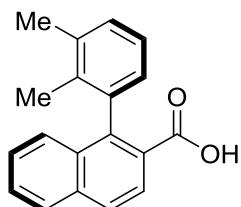


**2,4-Dimethylpentan-3-yl 1-(3-methoxy-2-methylphenyl)-2-naphthoate (3Dk):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.01 (d,  $J = 8.8$  Hz, 1H), 7.90 (d,  $J = 10.8$  Hz, 1H), 7.90 (d,  $J = 6.0$  Hz, 1H), 7.53 (ddd,  $J = 8.0$  Hz, 5.2 Hz, 3.2 Hz, 1H), 7.36-7.38 (m, 2H), 7.21 (dd,  $J = 8.4$  Hz, 8.0 Hz, 1H), 6.92 (d,  $J = 8.0$  Hz, 1H), 6.76 (dd,  $J = 7.6$  Hz, 0.8 Hz, 1H), 4.71 (t,  $J = 6.0$  Hz, 1H), 3.90 (s, 3H), 1.83 (s, 3H), 1.79 (sep,  $J = 6.8$  Hz, 1H), 1.71 (sep,  $J = 6.8$  Hz, 1H), 0.82 (d,  $J = 6.8$  Hz, 3H), 0.79 (d,  $J = 6.8$  Hz, 3H), 0.74 (d,  $J = 6.8$  Hz, 3H), 0.73 (d,  $J = 6.8$  Hz, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  168.1, 157.5, 140.6, 139.9, 134.6, 132.5, 128.3, 127.8, 127.6, 127.4, 127.3, 126.6, 125.9, 125.8, 125.5, 122.2, 109.2, 83.5, 55.4, 29.4, 29.3, 19.4, 19.4, 17.4, 17.1, 13.0; IR (ATR)  $\nu$  2960, 1730, 1684, 1653, 1558, 1456, 1242, 1126, 1088, 945, 835, 775  $\text{cm}^{-1}$ ; HRMS

(ESI)  $m/z$  calcd for  $C_{26}H_{30}O_3+H^+$  ( $M+H^+$ ): 391.2268, found: 391.2257;  $[\alpha]^{29}_D -33.7$  [c 1.725,  $CH_2Cl_2$ , 91% ee (S)].



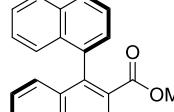
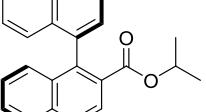
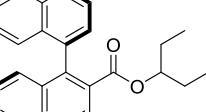
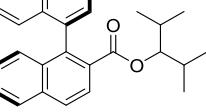
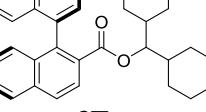
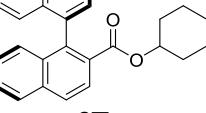
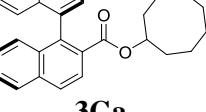
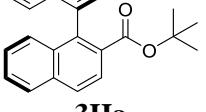
**[1-(2,3-dimethylphenyl)naphthalen-2-yl]methanol (4):**  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  7.91 (d,  $J = 8.4$  Hz, 1H), 7.89 (d,  $J = 8.8$  Hz, 1H), 7.71 (d,  $J = 8.4$  Hz, 1H), 7.46 (ddd,  $J = 8.0$  Hz, 6.8 Hz, 1.6 Hz, 1H), 7.35 (ddd,  $J = 8.4$  Hz, 6.8 Hz, 1.2 Hz, 1H), 7.26-7.30 (m, 2H), 7.21 (dd,  $J = 7.6$  Hz, 7.2 Hz, 1H), 7.00 (d,  $J = 7.2$  Hz, 1H), 4.50 (s, 2H), 2.39 (s, 3H), 1.84 (s, 3H);  $^{13}C$  NMR (100 MHz,  $CDCl_3$ )  $\delta$  137.8, 137.6, 137.2, 135.5, 135.4, 132.9, 132.5, 129.3, 127.9, 127.8, 127.7, 126.3, 126.1, 125.7, 125.7, 125.6, 63.5, 20.5, 16.4; IR (ATR)  $\nu$  3192, 2923, 2362, 1446, 1068, 1014, 827, 815, 744, 723  $cm^{-1}$ ; HRMS (ESI)  $m/z$  calcd for  $C_{19}H_{18}O+Na^+$  ( $M+Na^+$ ): 285.1250, found: 285.1243;  $[\alpha]^{24}_D -49.6$  [c 1.040,  $CH_2Cl_2$ , 96% ee (S)].

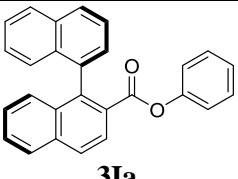
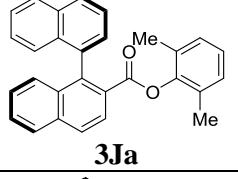
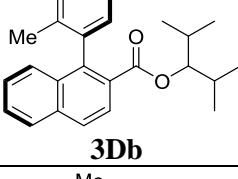
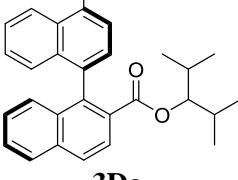
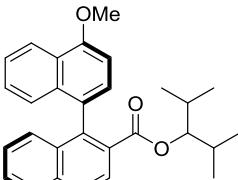
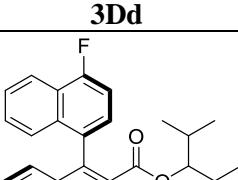
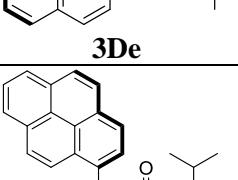
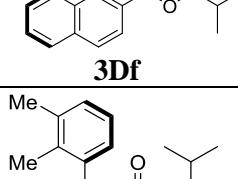


**1-(2,3-dimethylphenyl)-2-naphthoic acid (5):**  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  8.06 (d,  $J = 8.8$  Hz, 1H), 7.91 (d,  $J = 8.8$  Hz, 2H), 7.56 (ddd,  $J = 10.0$  Hz, 8.0 Hz, 1.6 Hz, 1H), 7.34-7.41 (m, 2H), 7.25 (d,  $J = 8.8$  Hz, 1H), 7.17 (dd,  $J = 8.0$  Hz, 7.2 Hz, 1H), 6.94 (d,  $J = 7.2$  Hz, 1H), 2.38 (s, 3H), 1.85 (s, 3H);  $^{13}C$  NMR (100 MHz,  $CDCl_3$ )  $\delta$  171.5, 143.1, 138.1, 136.5, 135.3, 135.2, 132.6, 129.3, 127.9, 127.9, 127.5, 127.1, 126.7, 126.2, 126.1, 125.1, 20.4, 16.5; IR (ATR)  $\nu$  2924, 2359, 1695, 1666, 1558, 1288, 939, 773  $cm^{-1}$ ; HRMS (ESI)  $m/z$  calcd for  $C_{19}H_{16}O_2-H^-$  ( $M-H^-$ ): 275.1078, found: 275.1082;  $[\alpha]^{25}_D -48.0$  [c 0.695,  $CH_2Cl_2$ , 97% ee (S)].

**3. Determination of the Enantiomeric Excesses of the Products by HPLC or SFC with a Chiral Stationary Phase**

**Table S1.** SFC separation conditions and retention times of products

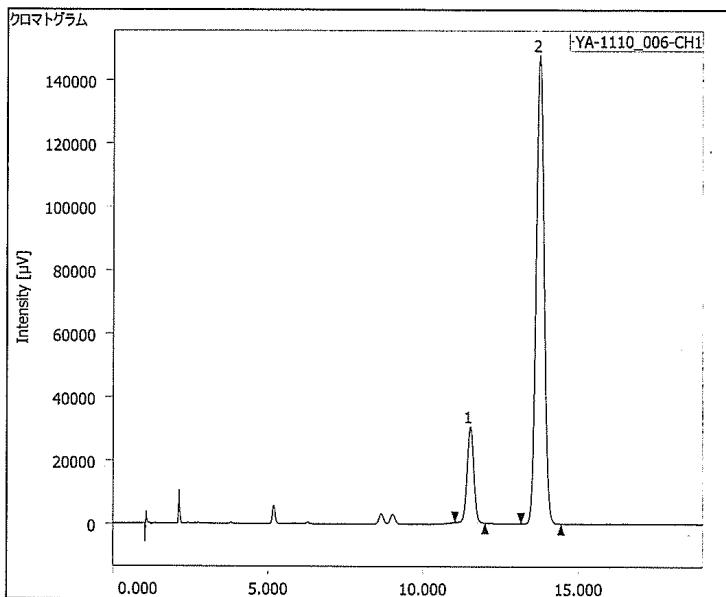
entry	Compound	column	Eluent	Flow rate (mL/min)	$t_R$ of ( <i>R</i> )- isomer (min)	$t_R$ of ( <i>S</i> )- isomer (min)
1	 <b>3Aa</b>	AD-H	<i>i</i> -PrOH/CO <sub>2</sub> 1/20	3.15	11.5	13.7
2	 <b>3Ba</b>	AD-H	<i>i</i> -PrOH/CO <sub>2</sub> 1/10	3.3	5.3	6.7
3	 <b>3Ca</b>	AD-H	<i>i</i> -PrOH/CO <sub>2</sub> 1/10	3.3	6.3	7.1
4	 <b>3Da</b>	AD-H	<i>i</i> -PrOH/CO <sub>2</sub> 1/10	3.3	6.2	7.0
5	 <b>3Ea</b>	AD-H	<i>i</i> -PrOH/CO <sub>2</sub> 1/5	3.6	8.8	7.3
6	 <b>3Fa</b>	AD-H	<i>i</i> -PrOH/CO <sub>2</sub> 1/5	3.6	5.0	6.8
7	 <b>3Ga</b>	AD-H	<i>i</i> -PrOH/CO <sub>2</sub> 1/5	3.6	6.2	7.9
8	 <b>3Ha</b>	AD-H	<i>i</i> -PrOH/CO <sub>2</sub> 1/10	3.3	5.6	9.1

9		AD-H	<i>i</i> -PrOH/CO <sub>2</sub> 1/5	3.6	7.0	10.0
10		AD-H	<i>i</i> -PrOH/CO <sub>2</sub> 1/5	3.6	8.2	13.2
11		AD-H	<i>i</i> -PrOH/CO <sub>2</sub> 1/5	3.6	2.1	1.9
12 <sup>a</sup>		AD-H	<i>i</i> -PrOH/hexane 1/19	0.6	8.2	12.0
13		AD-H	<i>i</i> -PrOH/CO <sub>2</sub> 1/10	3.3	8.6	7.4
14		AZ-H	<i>i</i> -PrOH/CO <sub>2</sub> 1/20	3.15	6.7	6.1
15		AD-H	<i>i</i> -PrOH/CO <sub>2</sub> 1/5	3.6	7.1	10.8
16		OZ-H	<i>i</i> -PrOH/CO <sub>2</sub> 1/20	3.15	3.9	3.4

17 <sup>a</sup>		AD-H	<i>i</i> -PrOH/hexane 1/49	0.6	10.4	7.8
18		AD-H	<i>i</i> -PrOH/CO <sub>2</sub> 1/20	3.15	3.4	3.1
19		AD-H	<i>i</i> -PrOH/CO <sub>2</sub> 1/20	3.15	3.1	2.6
20		OZ-H	<i>i</i> -PrOH/CO <sub>2</sub> 1/20	3.15	5.7	4.6
21		AZ-H	<i>i</i> -PrOH/CO <sub>2</sub> 1/10	3.3	8.3	9.2
22		AD-H	<i>i</i> -PrOH/CO <sub>2</sub> 1/10	3.3	9.2	10.3
23		AD-H	<i>i</i> -PrOH/CO <sub>2</sub> 1/5	3.6	9.3	10.3
24		AD-H	<i>i</i> -PrOH/CO <sub>2</sub> 1/5	3.6	6.9	5.6

<sup>a</sup> Determined by chiral HPLC.

1012 YA-1110\_006 2014/11/08 16:27:14

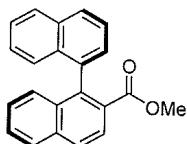


クロマトグラム情報

ユーザー名 JASCO  
更新日時 2012/10/12 19:51:37  
コメント  
HPLC システム名 JASCO SFC  
測定日 2012/10/12 19:32:36  
注入量 1.00 [ $\mu$ L]  
サンプル# 47  
プロジェクト名 Akai  
取込時間 19.0 [min]  
測定シーケンス P2\_IPA5%\_20min\_220nm  
コントロールメソッド

ピーク情報

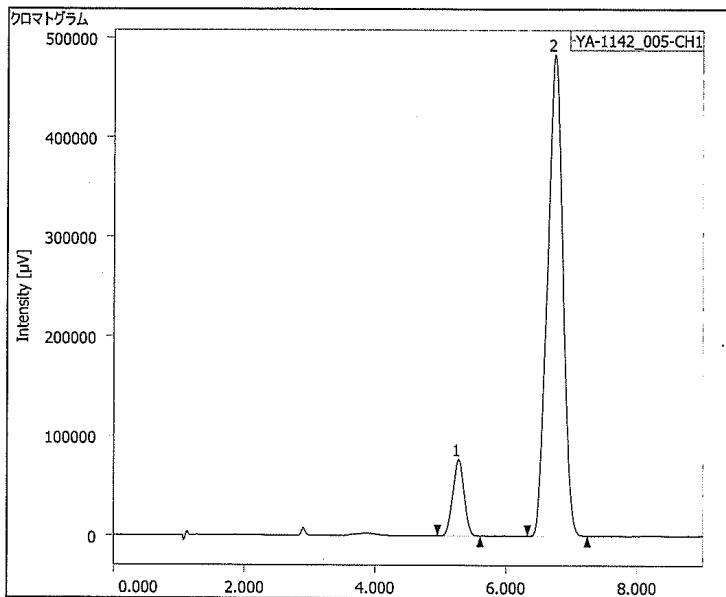
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2	Unknown	13.743	2622203	147749	85.287	82.958	N/A	13777	N/A



1 / 1

SFC trace for 3Aa

1101 YA-1142\_005 2014/11/08 16:24:24

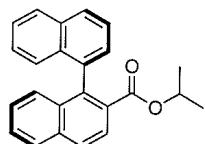


クロマトグラム情報

ユーザー名 JASCO  
更新日時 2012/11/01 18:41:48  
コメント  
HPLC システム名 JASCO SFC  
測定日 2012/11/01 18:32:46  
注入量 1.00 [ $\mu\text{L}$ ]  
サンプル# 12  
プロジェクト名 Akai  
取込時間 9.0 [min]  
測定シーケンス P2\_IPA10%,10min\_230nm  
コントロールメソッド

ピーク情報

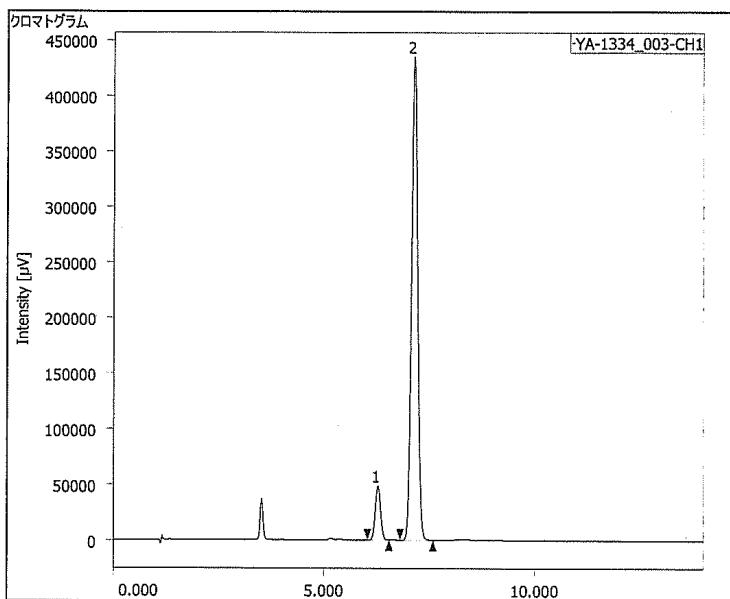
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1	Unknown	5.273	915145	76705	10.423	13.696	N/A	4391	3.909
2	Unknown	6.740	7865264	483333	89.577	86.304	N/A	3859	N/A



1 / 1

SFC trace for **3Ba**

0308 YA-1334\_003 2014/11/08 16:19:52

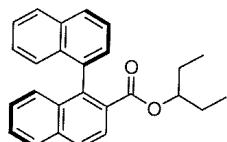


クロマトグラム情報

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更新日時 2013/03/08 16:46:39  
コメント  
HPLC システム名 JASCO SFC  
測定日 2013/03/08 16:32:37  
注入量 1.00 [μL]  
サンプル# 2  
プロジェクト名 Akai  
取込時間 14.0 [min]  
測定シーケンス P2\_JPA10%\_15min\_220nm  
コントロールメソッド

ピーク情報

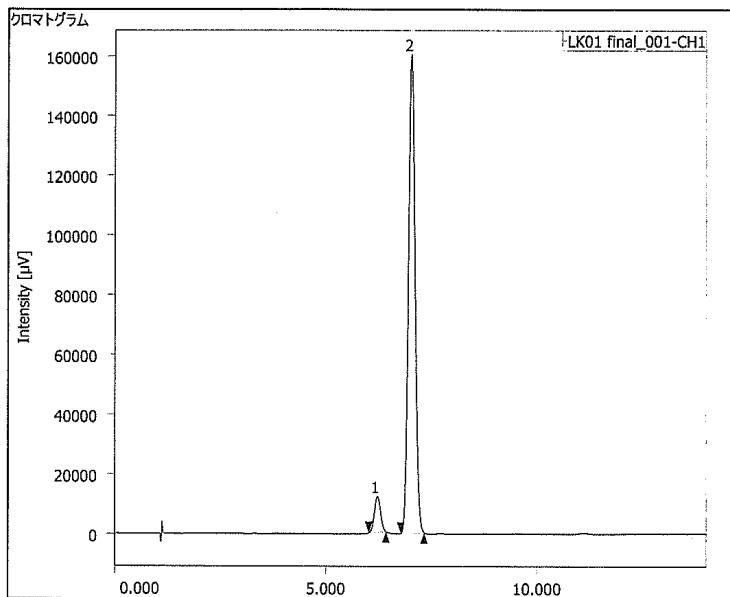
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2	Unknown	7.140	4416613	434882	91.197	89.984	N/A	11377	N/A



1 / 1

SFC trace for **3Ca**

0624\_LK01\_final\_001 2014/11/08 16:10:53

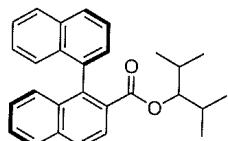


クロマトグラム情報

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更新日時 2014/06/24 15:20:14  
コメント  
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測定日 2014/06/24 14:52:50  
注入量 1.00 [ $\mu$ L]  
サンプル# 1  
プロジェクト名 Akai  
取込時間 14.0 [min]  
測定シーケンス P2\_IPA10%\_15min\_220nm  
コントロールメソッド

ピーク情報

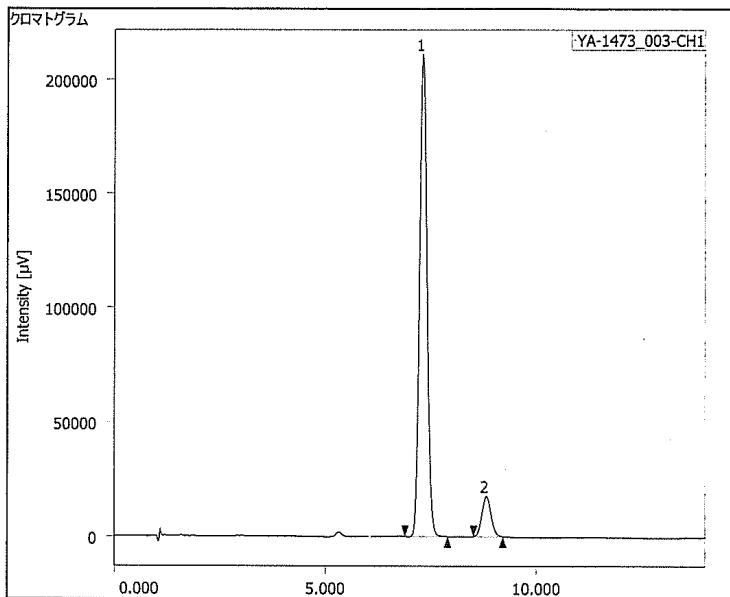
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1	Unknown	6.227	121323	12000	6.483	6.975	N/A	8558	2.903
2	Unknown	7.033	1750166	160039	93.517	93.025	N/A	9542	N/A



1 / 1

SFC trace for 3Da

0726 YA-1473\_003 2014/11/08 16:33:22

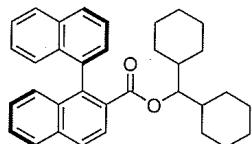


クロマトグラム情報

ユーザー名 JASCO  
更新日時 2013/07/26 18:36:16  
コメント  
HPLC システム名 JASCO SFC  
測定日 2013/07/26 18:22:14  
注入量 1.00 [ $\mu$ L]  
サンプル# 22  
プロジェクト名 Akai  
取込時間 14.0 [min]  
測定シーケンス P2\_JPA20%\_15min\_220nm  
コントロールメソッド

ピーク情報

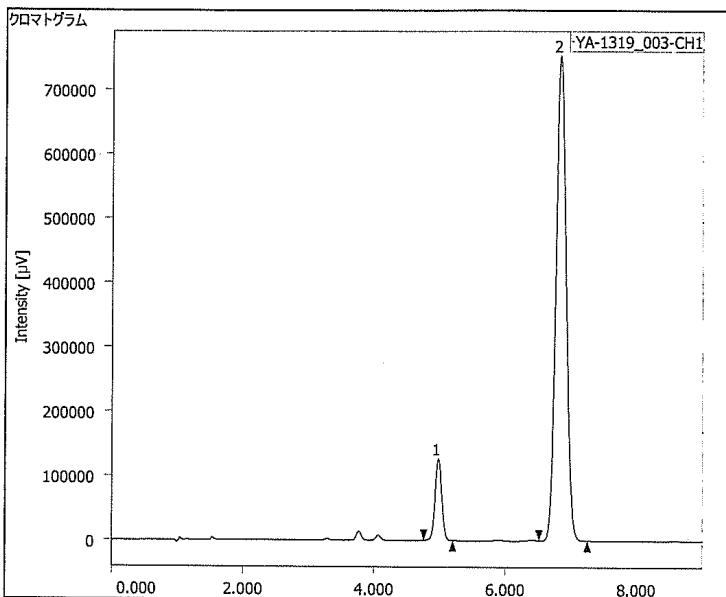
#	ピーク名	tR [min]	面積 [ $\mu$ V/sec]	高さ [ $\mu$ V]	面積%	高さ%	定量値	NTP	分離度
1	Unknown	7.313	2600063	211016	90.815	92.307	N/A	8202	4.209
2	Unknown	8.820	262973	17586	9.185	7.693	N/A	7986	N/A



1 / 1

SFC trace for 3Ea

0228 YA-1319\_003 2014/11/08 16:21:27

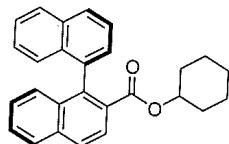


クロマトグラム情報

ユーザー名 JASCO  
更新日時 2013/02/28 20:35:01  
コメント  
HPLC システム名 JASCO SFC  
測定日 2013/02/28 20:25:59  
注入量 1.00 [ $\mu$ L]  
サンプル# 2  
プロジェクト名 Akai  
取込時間 9.0 [min]  
測定シーケンス P2\_IPA20%\_10min\_220nm  
コントロールメソッド

ピーク情報

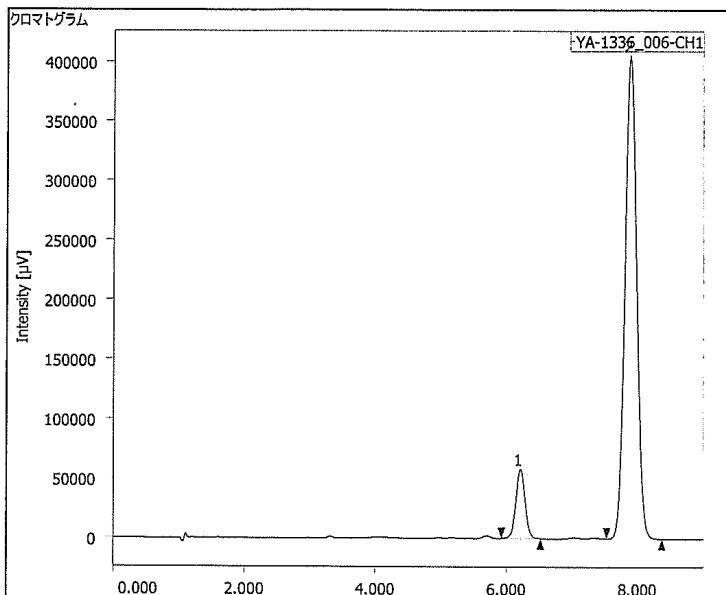
#	ピーク名	tR [min]	面積 [ $\mu$ V·sec]	高さ [ $\mu$ V]	面積%	高さ%	定量値	NTP	分離度
1	Unknown	4.973	803083	126722	10.031	14.413	N/A	11325	7.890
2	Unknown	6.830	8099978	752520	89.969	85.687	N/A	9192	N/A



1 / 1

SFC trace for 3Fa

0308 YA-1336\_006 2014/11/08 16:19:05

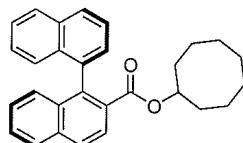


クロマトグラム情報

ユーザー名	JASCO
更新日時	2013/03/08 18:57:57
コメント	
HPLC システム名	JASCO SFC
測定日	2013/03/08 18:48:54
注入量	1.00 [ $\mu$ L]
サンプル#	4
プロジェクト名	Akai
取込時間	9.0 [min]
測定シーケンス	
コントロールメソッド	P2_IPA20%_10min_220nm

ピ�ク情報

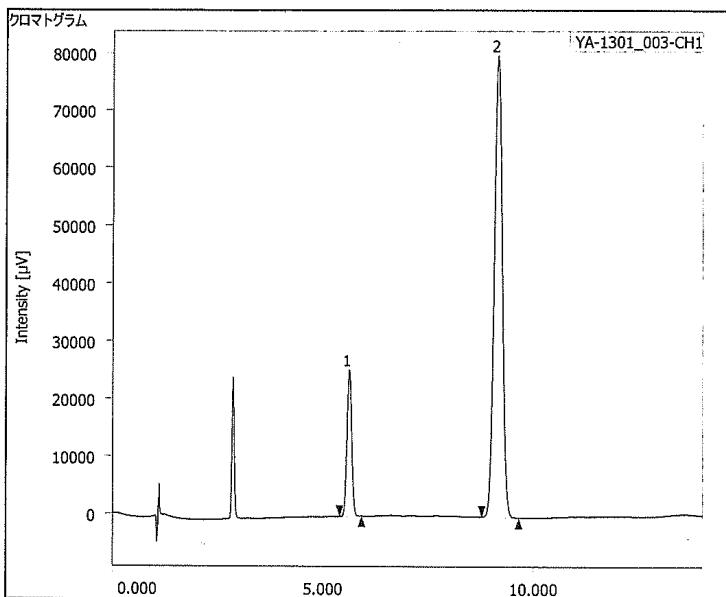
#	ピ�ク名	tR [min]	面積 [ $\mu$ V·sec]	高さ [ $\mu$ V]	面積%	高さ%	定量値	NTP	分類度
1	Unknown	6.210	564870	57952	10.063	12.506	N/A	9509	5.736
2	Unknown	7.880	5048657	405427	89.937	87.494	N/A	9185	N/A



1 / 1

SFC trace for 3Ga

0221 YA-1301\_003 2014/11/08 16:23:06

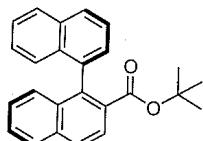


クロマトグラム情報

ユーザー名 JASCO  
更新日時 2013/02/21 18:50:21  
コメント  
HPLC システム名 JASCO SFC  
測定日 2013/02/21 18:36:19  
注入量 1.00 [ $\mu\text{L}$ ]  
サンプル# 2  
プロジェクト名 Akai  
取込時間 14.0 [min]  
測定シーケンス P2\_IPA10%\_15min\_220nm  
コントロールメソッド

ピーク情報

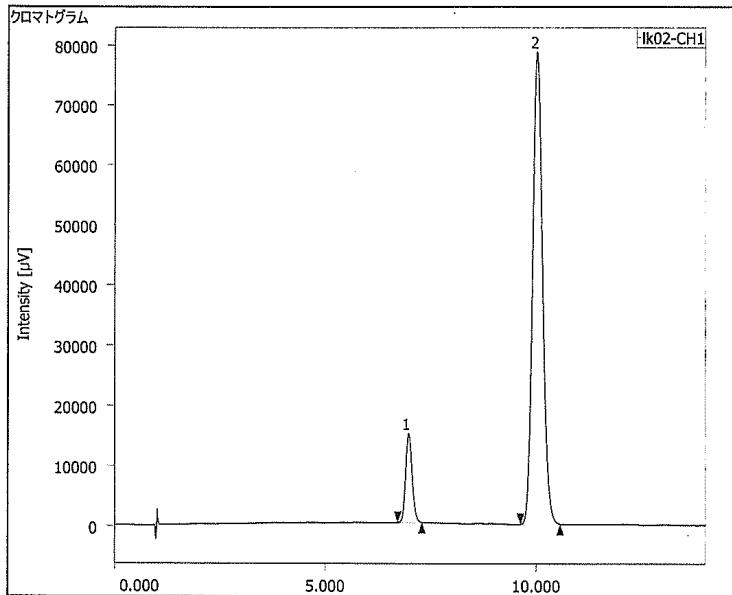
#	ピーク名	tR [min]	面積 [ $\mu\text{V}\cdot\text{sec}$ ]	高さ [ $\mu\text{V}$ ]	面積%	高さ%	定量値	NTP	分離度
1	Unknown	5.620	199526	25444	15.979	24.109	N/A	11739	12.759
2	Unknown	9.147	1049122	80095	84.021	75.891	N/A	11128	N/A



1 / 1

SFC trace for 3Ha

Akai-1 lk02 2014/11/08 16:34:18

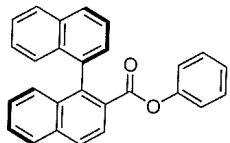


クロマトグラム情報

ユーザー名 JASCO  
更新日時 2014/06/26 15:41:21  
コメント  
HPLC システム名 JASCO SFC  
測定日 2014/06/26 15:03:09  
注入量 1.00 [ $\mu$ L]  
サンプル# 2  
プロジェクト名 Akai  
取込時間 14.0 [min]  
測定シーケンス Akai-1  
コントロールメソッド P2\_IPA20%\_15min\_220nm

ピーク情報

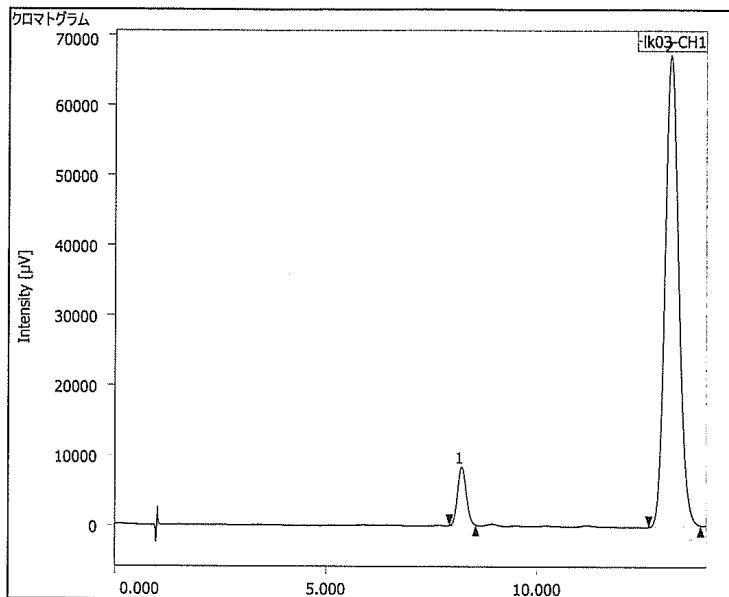
#	ピーク名	tR [min]	面積 [ $\mu$ V·sec]	高さ [ $\mu$ V]	面積%	高さ%	定量値	NTP	分離度
1	Unknown	6.980	157362	14817	10.988	15.876	N/A	10148	8.687
2	Unknown	10.017	1274750	78518	89.012	84.125	N/A	8936	N/A



1 / 1

SFC trace for 3Ia

Akai-1 lk03 2014/11/08 16:34:49

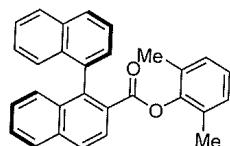


クロマトグラム情報

ユーザー名 JASCO  
更新日時 2014/06/26 15:41:20  
コメント  
HPLC システム名 JASCO SFC  
測定日 2014/06/26 15:18:13  
注入量 1.00 [ $\mu\text{L}$ ]  
サンプル# 3  
プロジェクト名 Akai  
取込時間 14.0 [min]  
測定シーケンス Akai-1  
コントロールメソッド P2\_IPA20%\_15min\_220nm

ピーク情報

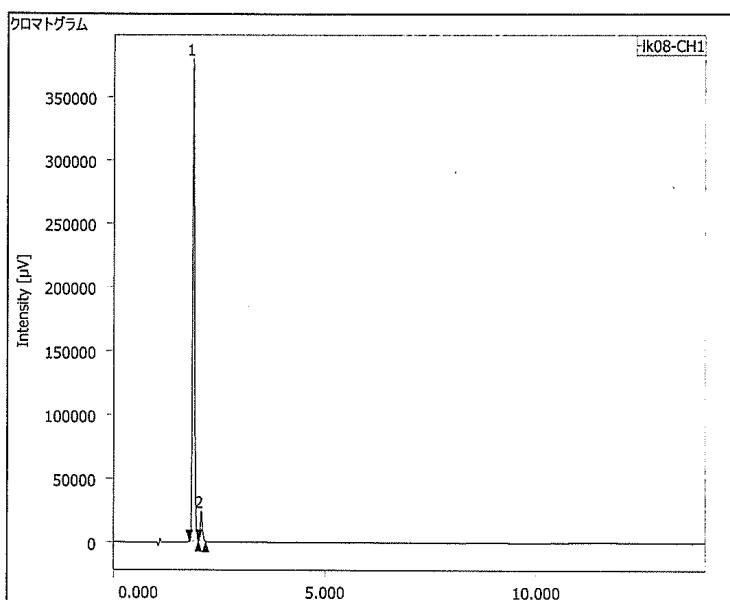
#	ピーク名	tR [min]	面積 [ $\mu\text{V}\cdot\text{sec}$ ]	高さ [ $\mu\text{V}$ ]	面積%	高さ%	定量値	NTP	分離度
1	Unknown	8.220	118561	8306	7.305	10.995	N/A	7513	10.267
2	Unknown	13.163	1504346	67238	92.695	88.005	N/A	8067	N/A



1 / 1

SFC trace for 3Ja

Akai-i lk08 2014/11/08 16:12:10

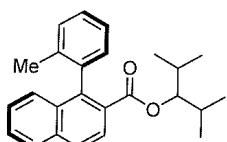


クロマトグラム情報

ユーザー名 JASCO  
更新日時 2014/07/03 18:36:12  
コメント  
HPLC システム名 JASCO SFC  
測定日 2014/07/03 17:37:54  
注入量 1.00 [ $\mu$ L]  
サンプル# 4  
プロジェクト名 Akai  
取込時間 14.0 [min]  
測定シーケンス Akai-1  
コントロールメソッド P2\_JPA20%\_15min\_220nm

ピーク情報

#	ピーク名	tR [min]	面積 [UV-sec]	高さ [mV]	面積%	高さ%	定量値	NTP	分離度
1	Unknown	1.877	1241115	378550	94.159	94.306	N/A	7994	2.333
2	Unknown	2.077	76990	22856	5.841	5.694	N/A	8917	N/A

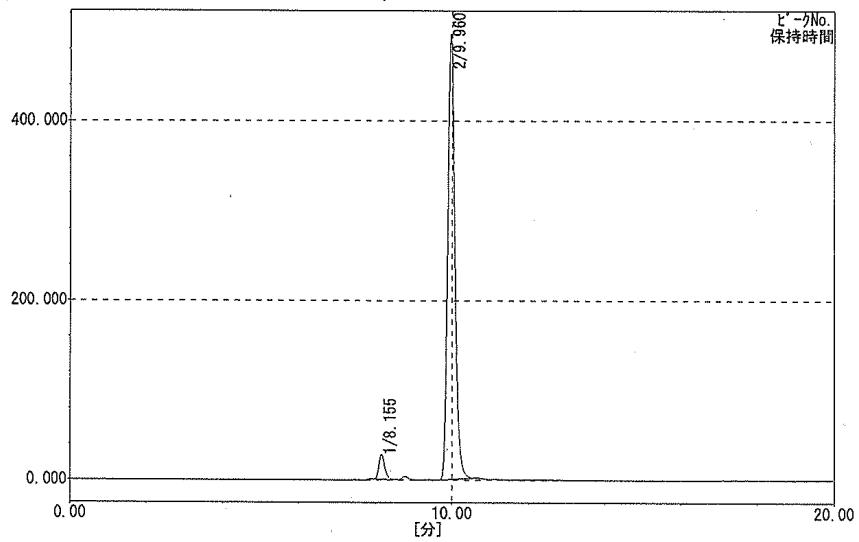


1 / 1

SFC trace for 3Db

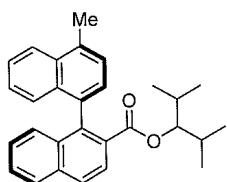
タイトル :  
 データ : C:\HPLCData\data\_nishikawa.mdb TN0540 lk29  
 日時 : 収集 2014/07/26 10:46:52 計算 2014/07/26 13:05:57  
 カップ番号 : 12  
 収集属性 : 独立 0.00 - 20.00 min  
 計算方法 : 百分率法

[mV]



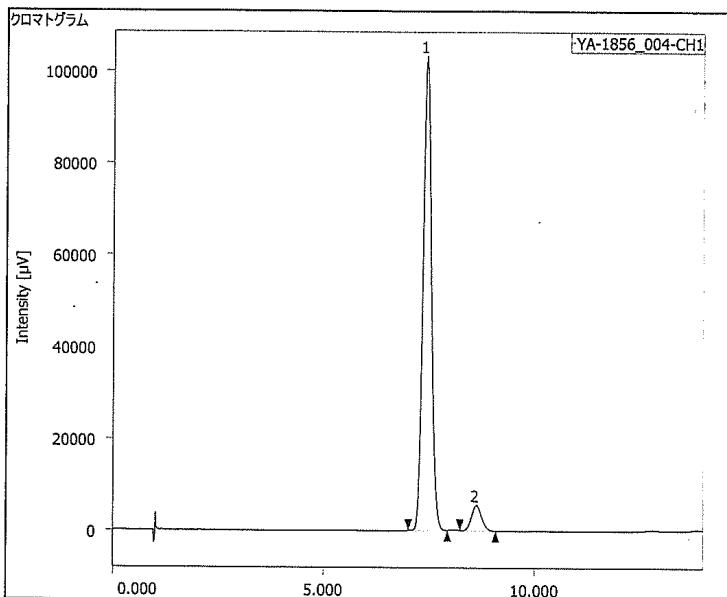
No.	Ch	時間[分]	高さ[mV]	半値幅[秒]	面積[mVx秒]	面積%	理論段数	分離能	非対称係数
1	1	8.155	27.17	9.51	284.70	4.17	14679	0.00	1.44
2	1	9.960	496.85	11.97	6545.35	95.83	13812	5.94	1.48

CH.1 Peak Not Found.



SFC trace for 3Dc

0902 YA-1856\_004 2014/11/08 15:58:10

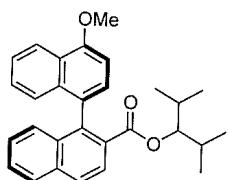


クロマトグラム情報

ユーザー名 JASCO  
更新日時 2014/09/02 12:37:23  
コメント  
HPLC システム名 JASCO SFC  
測定日 2014/09/02 12:23:22  
注入量 1.00 [ $\mu$ L]  
サンプル# 3  
プロジェクト名 Akai  
取込時間 14.0 [min]  
測定シーケンス P2\_IPA10%\_15min\_220nm  
コントロールメソッド

ピーク情報

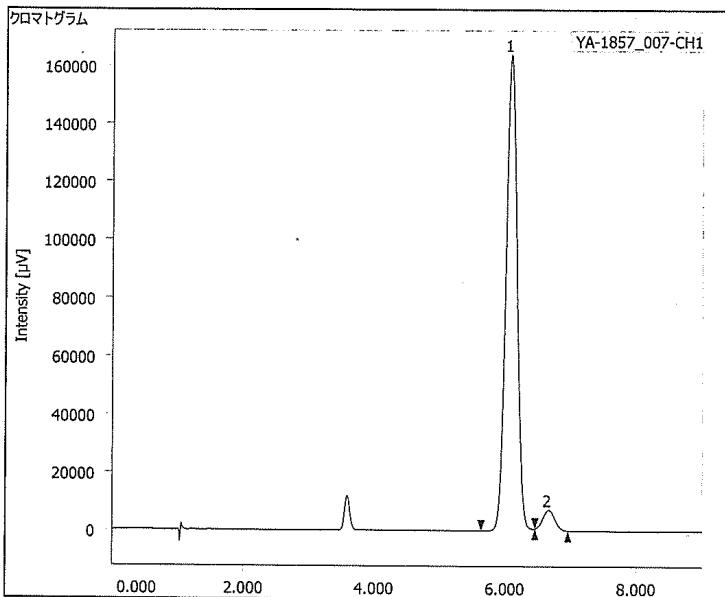
#	ピーカ名	tR [min]	面積 [ $\mu$ V·sec]	高さ [ $\mu$ V]	面積%	高さ%	定量値	NTP	分離度
1	Unknown	7.427	1408857	103072	93.941	94.960	N/A	6928	3.034
2	Unknown	8.630	90868	5471	6.059	5.040	N/A	6202	N/A



1 / 1

SFC trace for 3Dd

0903 YA-1857\_007 2014/11/08 15:56:35

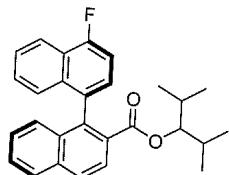


クロマトグラム情報

ユーザー名 JASCO  
更新日時 2014/09/03 12:48:20  
コメント  
HPLC システム名 JASCO SFC  
測定日 2014/09/03 12:34:58  
注入量 1.00 [ $\mu$ L]  
サンプル# 5  
プロジェクト名 Akai  
取込時間 9.0 [min]  
測定シーケンス P4\_IPA5%\_10min\_220nm  
コントロールメソッド

ピーク情報

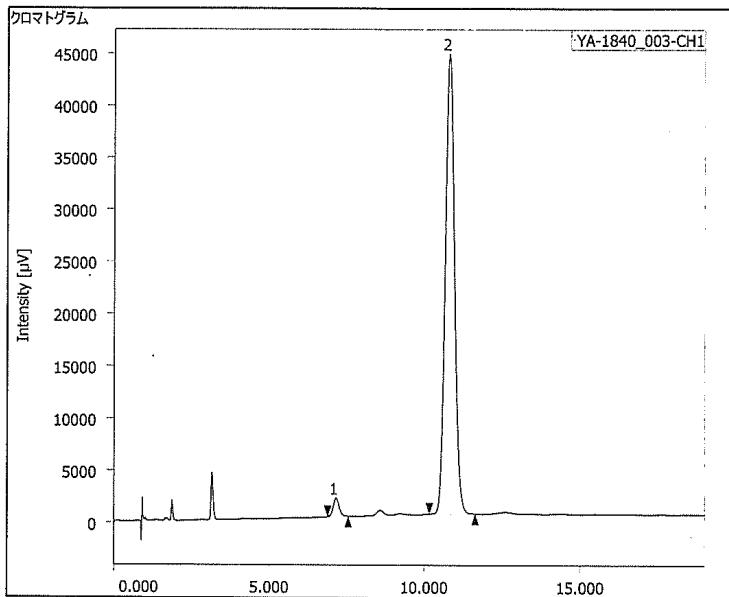
#	ピーク名	tR [min]	面積 [ $\mu$ V·sec]	高さ [ $\mu$ V]	面積%	高さ%	定量値	NTP	分離度
1	Unknown	6.080	1927675	163845	95.357	95.787	N/A	6102	1.780
2	Unknown	6.663	93850	7206	4.643	4.213	N/A	5943	N/A



1 / 1

SFC trace for 3De

0823 YA-1840\_003 2014/11/08 16:04:01

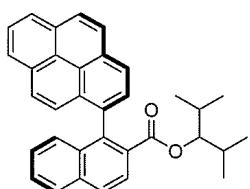


クロマトグラム情報

ユーザー名 JASCO  
更新日時 2014/08/23 16:31:32  
コメント  
HPLC システム名 JASCO SFC  
測定日 2014/08/23 16:12:31  
注入量 1.00 [ $\mu$ L]  
サンプル# 5  
プロジェクト名 Akai  
取込時間 19.0 [min]  
測定シーケンス P2\_IPA20%\_20min\_220nm  
コントロールメソッド

ピーク情報

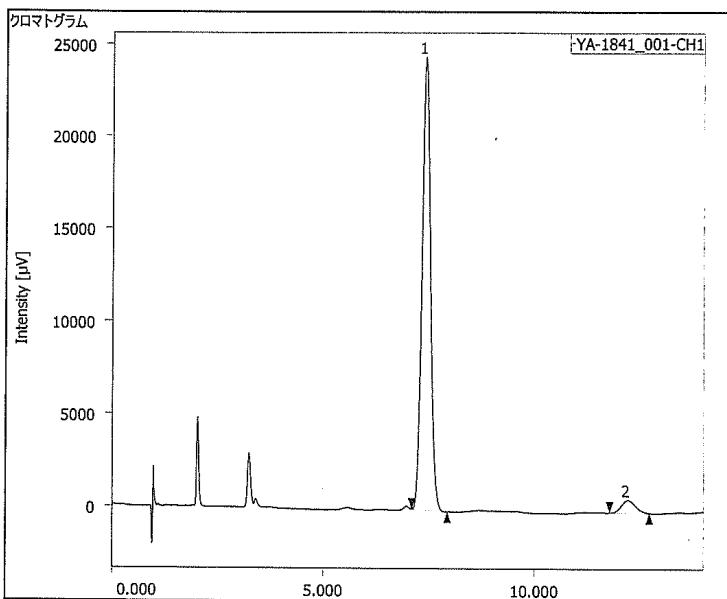
#	ピーク名	tR [min]	面積 [ $\mu$ V·sec]	高さ [ $\mu$ V]	面積%	高さ%	定量値	NTP	分離度
1	Unknown	7.143	23328	1750	2.505	3.819	N/A	6678	8.234
2	Unknown	10.797	907947	44072	97.495	96.181	N/A	6394	N/A



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SFC trace for (S)-3Df

0828 YA-1841\_001 2014/11/08 16:04:34

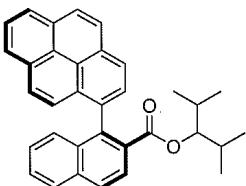


クロマトグラム情報

ユーザー名 JASCO  
更新日時 2014/08/28 11:01:47  
コメント  
HPLC システム名 JASCO SFC  
測定日 2014/08/28 10:05:23  
注入量 1.00 [ $\mu\text{L}$ ]  
サンプル# 23  
プロジェクト名 Akai  
取込時間 14.0 [min]  
測定シーケンス  
コントロールメソッド P2\_JPA20%\_15min\_220nm

ピーク情報

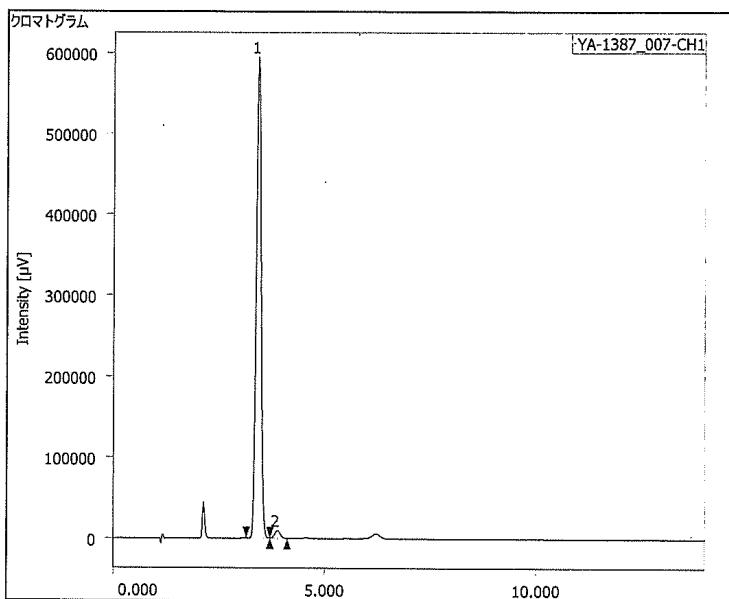
#	ピーク名	tR [min]	面積 [ $\mu\text{V}\cdot\text{sec}$ ]	高さ [ $\mu\text{V}$ ]	面積%	高さ%	定量値	NTP	分離度
1	Unknown	7.413	337963	24454	95.618	97.284	N/A	6722	9.909
2	Unknown	12.207	15489	683	4.382	2.716	N/A	6441	N/A



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SFC trace for (R)-3Df

0511 YA-1387\_007 2014/11/08 16:15:38

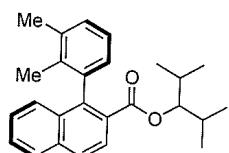


クロマトグラム情報

ユーザー名 JASCO  
更新日時 2013/05/11 12:07:52  
コメント  
HPLC システム名 JASCO SFC  
測定日 2013/05/11 11:53:51  
注入量 1.00 [μL]  
サンプル# 22  
プロジェクト名 Akai  
取込時間 14.0 [min]  
測定シーケンス P6\_IPA5%\_15min\_220nm  
コントロールメソッド

ピーク情報

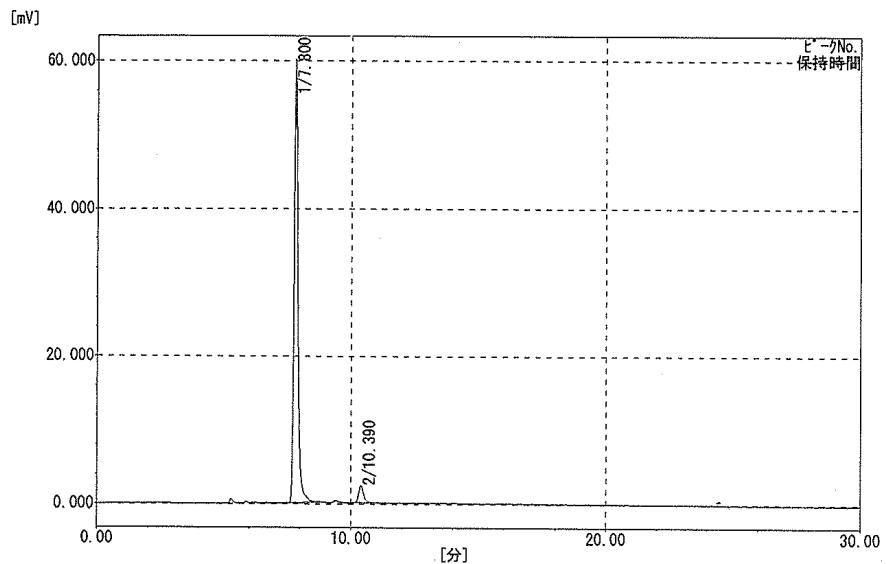
#	ピーク名	tR [min]	面積 [UV:sec]	高さ [AU]	面積%	高さ%	定量値	NTP	分離度
1	Unknown	3.427	4676967	594042	98.250	98.453	N/A	4325	2.059
2	Unknown	3.887	83296	9337	1.750	1.547	N/A	4210	N/A



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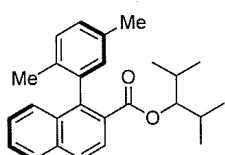
SFC trace for **3Dg**

タイトル : C:\HPLCData\Y\data\_Ke.mdb yz01660022 lk43  
 データ :  
 日時 : 収集 2014/08/02 11:11:25 計算 2014/08/02 11:44:49  
 カップ番号 : 11  
 収集属性 : 独立 0.00 ~ 30.00 min  
 計算方法 : 百分率法

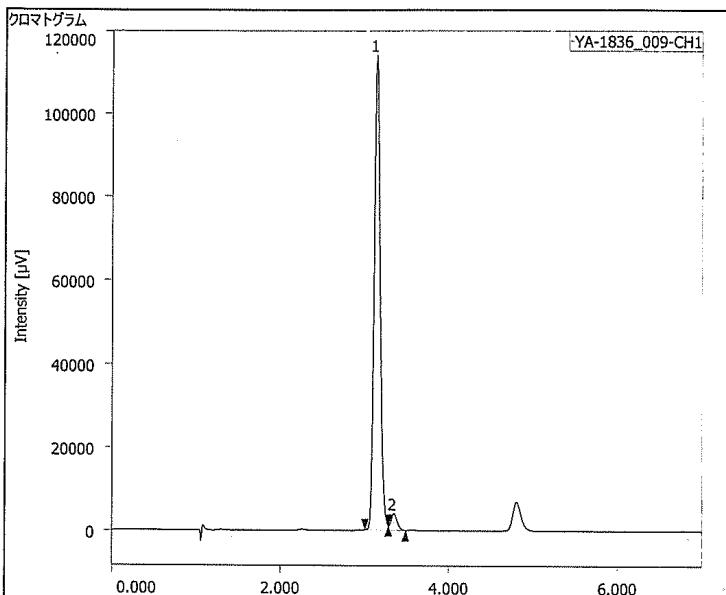


No.	Ch	時間 [分]	高さ [mV]	半値幅 [秒]	面積 [mVx秒]	面積%	理論段数	分離能	非対称係数
1	1	7.800	60.16	9.18	638.77	95.48	14393	0.00	1.66
2	1	10.390	2.34	11.78	30.26	4.52	15524	8.73	1.46

CH.1 Peak Not Found.



0824 YA-1836\_009 2014/11/08 16:02:34

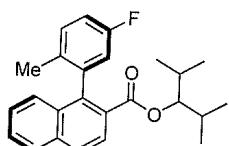


クロマトグラム情報

ユーザー名 JASCO  
更新日時 2014/08/24 20:20:52  
コメント  
HPLC システム名 JASCO SFC  
測定日 2014/08/24 20:03:26  
注入量 1.00 [ $\mu\text{L}$ ]  
サンプル# 14  
プロジェクト名 Akai  
取込時間 7.0 [min]  
測定シーケンス P2\_IPA5%\_8min\_220nm  
コントロールメソッド

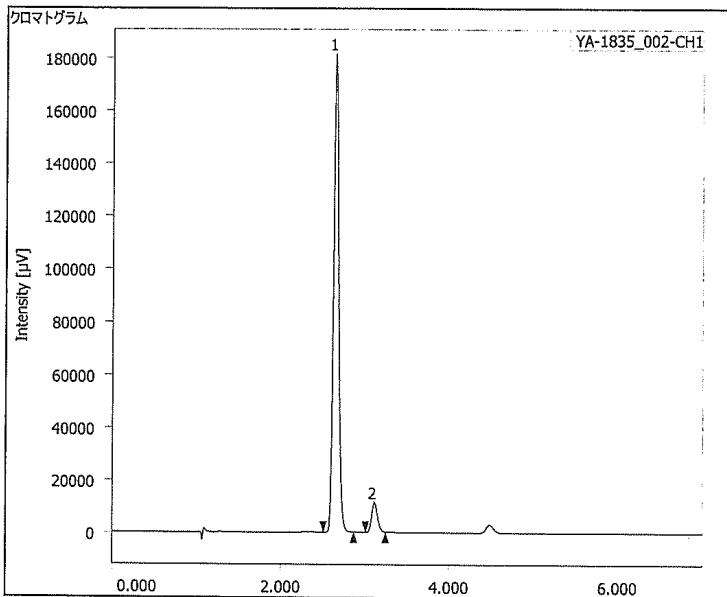
ピーク情報

#	ピーク名	tR [min]	面積 [ $\mu\text{V}\cdot\text{sec}$ ]	高さ [ $\mu\text{V}$ ]	面積%	高さ%	定量値	NTP	分離度
1	Unknown	3.140	559165	113951	96.427	96.640	N/A	9853	1.601
2	Unknown	3.350	20722	3962	3.573	3.360	N/A	9626	N/A



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SFC trace for 3Di

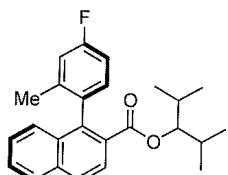


## クロマトグラム情報

ユーザー名 JASCO  
更新日時 2014/08/24 20:20:49  
コメント  
HPLC システム名 JASCO SFC  
測定日 2014/08/24 18:24:32  
注入量 1.00 [ $\mu$ L]  
サンプル# 13  
プロジェクト名 Akai  
取込時間 7.0 [min]  
測定シーケンス P2\_JPA5%\_8min\_220nm  
コントロールメソッド

## ピーク情報

#	ピーク名	tR [min]	面積 [ $\mu$ V·sec]	高さ [ $\mu$ V]	面積%	高さ%	定量値	NTP	分離度
1	Unknown	2.640	768743	181372	93.367	94.156	N/A	9310	3.973
2	Unknown	3.110	54613	11257	6.633	5.844	N/A	9474	N/A

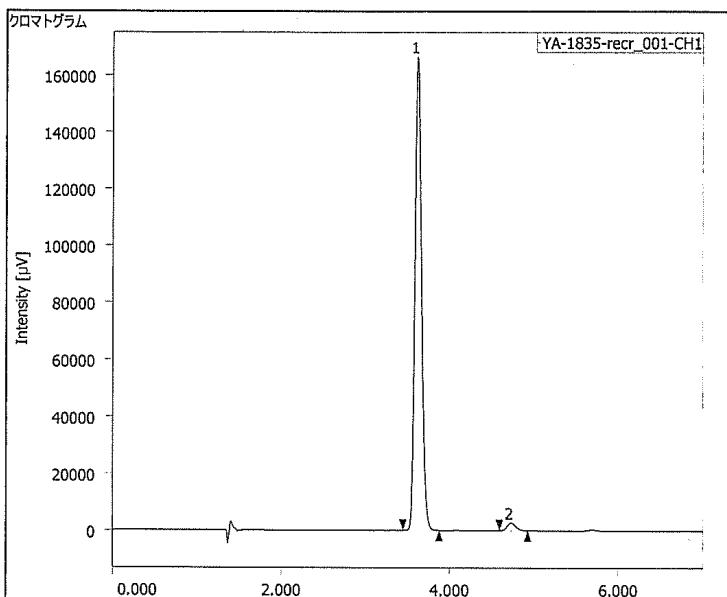


1 / 1

}

SFC trace for 3Dj

1007 YA-1835-recr\_001 2014/11/08 16:05:59

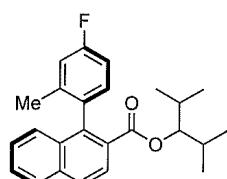


クロマトグラム情報

ユーザー名 JASCO  
更新日時 2014/10/07 18:22:03  
コメント  
HPLC システム名 JASCO SFC  
測定日 2014/10/07 14:44:59  
注入量 1.00 [ $\mu$ L]  
サンプル# 23  
プロジェクト名 Akai  
取込時間 7.0 [min]  
測定シーケンス P2\_JPA5%\_8min\_220nm  
コントロールメソッド

ピーク情報

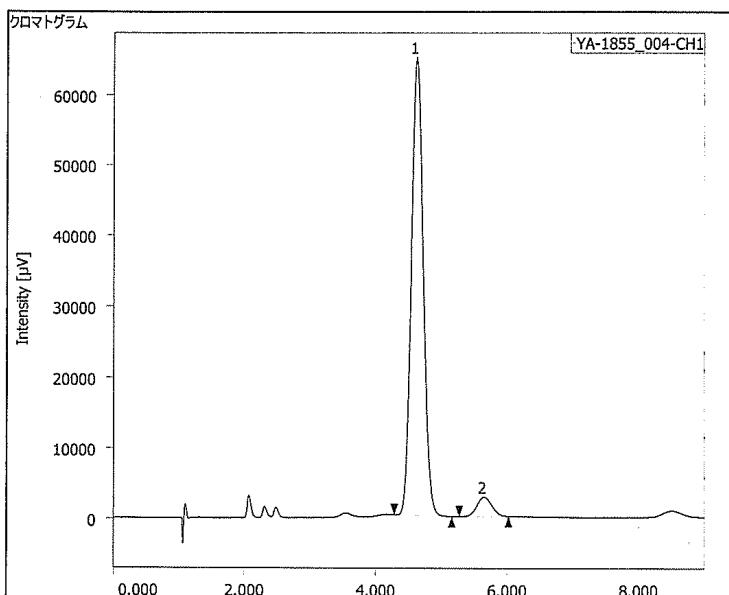
#	ピーク名	tR [min]	面積 [ $\mu$ V·sec]	高さ [mV]	面積%	高さ%	定量値	NTP	分離度
1	Unknown	3.617	910536	165423	97.956	98.428	N/A	10349	6.727
2	Unknown	4.727	19000	2657	2.044	1.572	N/A	10040	N/A



1 / 1

SFC trace for **3Dj** (after single recrystallization)

0903 YA-1855\_004 2014/11/08 16:00:10

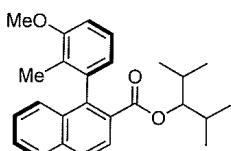


クロマトグラム情報

ユーザー名 JASCO  
更新日時 2014/09/03 12:48:19  
コメント  
HPLC システム名 JASCO SFC  
測定日 2014/09/03 11:47:48  
注入量 1.00 [ $\mu$ L]  
サンプル# 12  
プロジェクト名 Akai  
取込時間 9.0 [min]  
測定シーケンス P6\_IPA5%\_10min\_220nm  
コントロールメソッド

ピーク情報

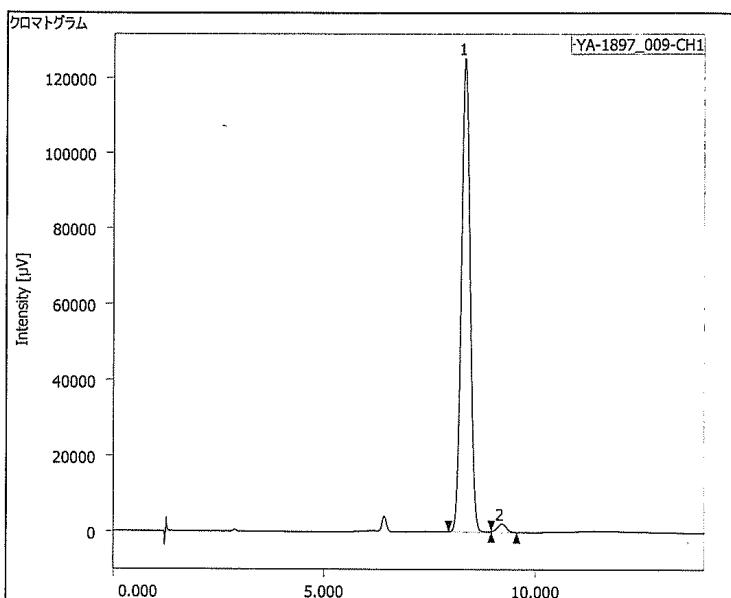
#	ピーカ名	tR [min]	面積 [ $\mu$ Vsec]	高さ [ $\mu$ V]	面積%	高さ%	定量値	NTP	分離度
1	Unknown	4.623	818450	64828	94.976	95.944	N/A	3085	2.742
2	Unknown	5.653	43293	2741	5.024	4.056	N/A	2893	N/A



1 / 1

SFC trace for 3Dk

1007 YA-1897\_009 2014/11/08 16:06:35

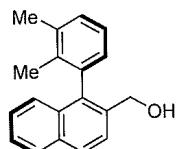


クロマトグラム情報

ユーザー名	JASCO
更新日時	2014/10/07 18:22:05
コメント	
HPLC システム名	JASCO SFC
測定日	2014/10/07 18:01:54
注入量	1.00 [ $\mu$ L]
サンプル#	25
プロジェクト名	Akai
取込時間	14.0 [min]
測定シーケンス	
コントロールメソッド	P4_IPA10%_15min_220nm

ピーク情報

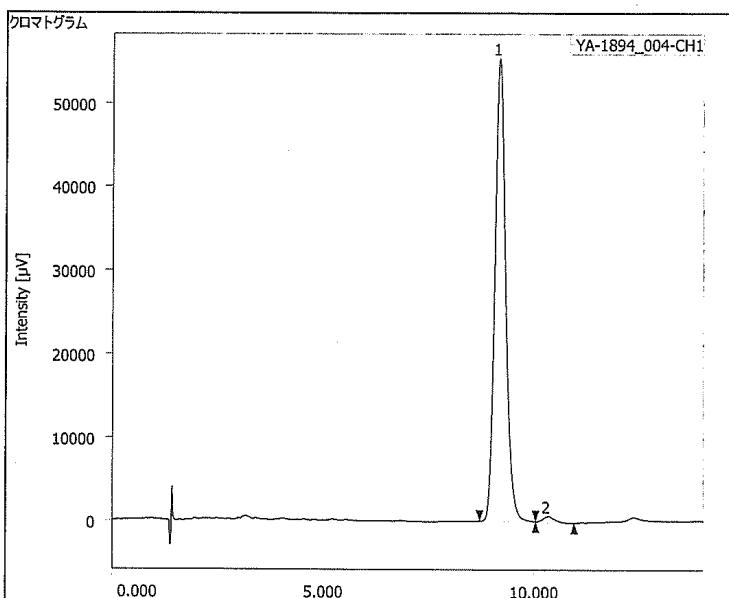
#	ピーク名	tR [min]	面積 [mV·sec]	高さ [ $\mu$ V]	面積%	高さ%	定量値	NTP	分離度
1	Unknown	8.337	1806464	125188	98.198	98.279	N/A	7651	2.223
2	Unknown	9.203	63150	2192	1.802	1.721	N/A	8447	N/A



1 / 1

SFC trace for **4Dg**

1006 YA-1894\_004 2014/11/08 16:07:10

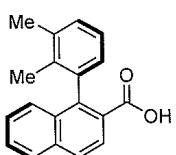


クロマトグラム情報

ユーザー名 JASCO  
更新日時 2014/10/06 12:03:20  
コメント  
HPLC システム名 JASCO SFC  
測定日 2014/10/06 11:45:15  
注入量 1.00 [ $\mu\text{L}$ ]  
サンプル# 24  
プロジェクト名 Akai  
取込時間 14.0 [min]  
測定シーケンス P2\_IPA10%\_15min\_220nm  
コントロールメソッド

ピーク情報

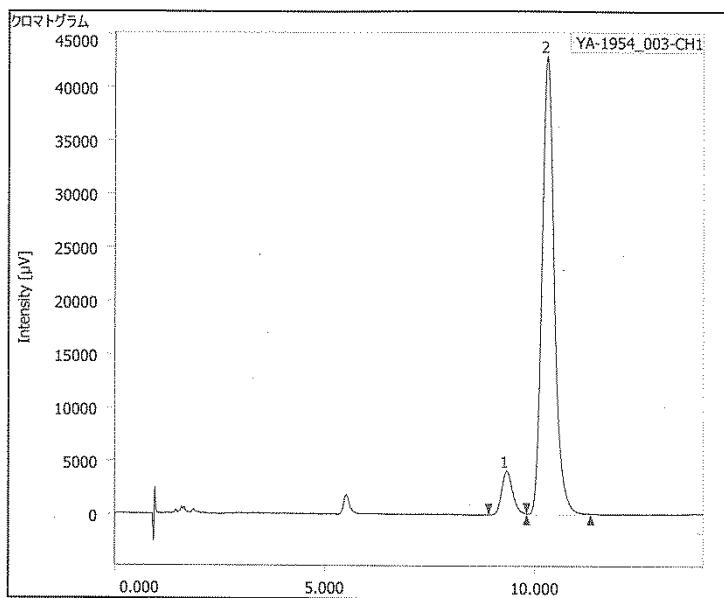
#	ピーク名	tR [min]	面積 [ $\mu\text{V}\cdot\text{sec}$ ]	高さ [ $\mu\text{V}$ ]	面積%	高さ%	定量値	NTP	分離度
1	Unknown	9.173	983210	55411	98.661	98.697	N/A	6539	2.428
2	Unknown	10.323	14352	731	1.439	1.303	N/A	6933	N/A



1 / 1

SFC trace for **5Dg**

0120 YA-1954\_003 2015/01/21 10:57:54

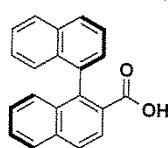


クロマトグラム情報

ユーザー名 JASCO  
更新日時 2015/01/20 19:41:52  
コメント  
HPLC システム名 JASCO SFC  
測定日 2015/01/20 19:25:48  
注入量 1.00 [ $\mu$ L]  
サンプル# 28  
プロジェクト名 Akai  
取込時間 14.0 [min]  
測定シーケンス P2\_IPA20%\_15min\_220nm  
コントロールメソッド

ピーク情報

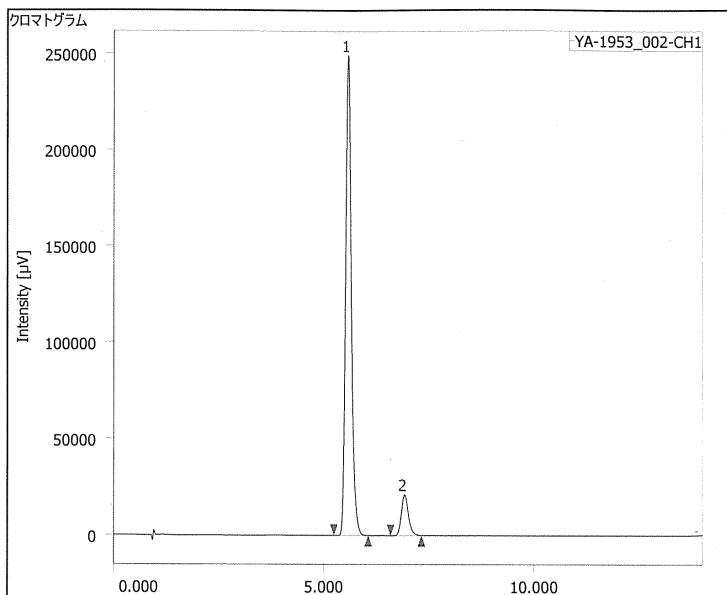
#	ピーク名	tR [min]	面積 [ $\mu$ V·sec]	高さ [ $\mu$ V]	面積%	高さ%	定置値	NTP	分離度
1	Unknown	9.327	3766	4089	7.831	8.719	N/A	6478	1.969
2	Unknown	10.287	868200	42808	92.169	91.281	N/A	6408	N/A



1/1

SFC trace for **S1**

0120 YA-1953\_002 2015/01/21 10:57:42

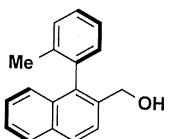


クロマトグラム情報

ユーザー名 JASCO  
更新日時 2015/01/20 19:41:51  
コメント  
HPLC システム名 JASCO SFC  
測定日 2015/01/20 19:08:49  
注入量 1.00 [ $\mu$ L]  
サンプル# 27  
プロジェクト名 Akai  
取込時間 14.0 [min]  
測定シーケンス P2\_IPA20%\_15min\_220nm  
コントロールメソッド

ピーク情報

#	ピーク名	tR [min]	面積 [ $\mu$ V sec]	高さ [ $\mu$ V]	面積%	高さ%	定量値	NTP	分離度
1	Unknown	5.577	2359878	249387	90.548	92.136	N/A	8632	5.041
2	Unknown	6.923	246353	21287	9.452	7.864	N/A	8774	N/A



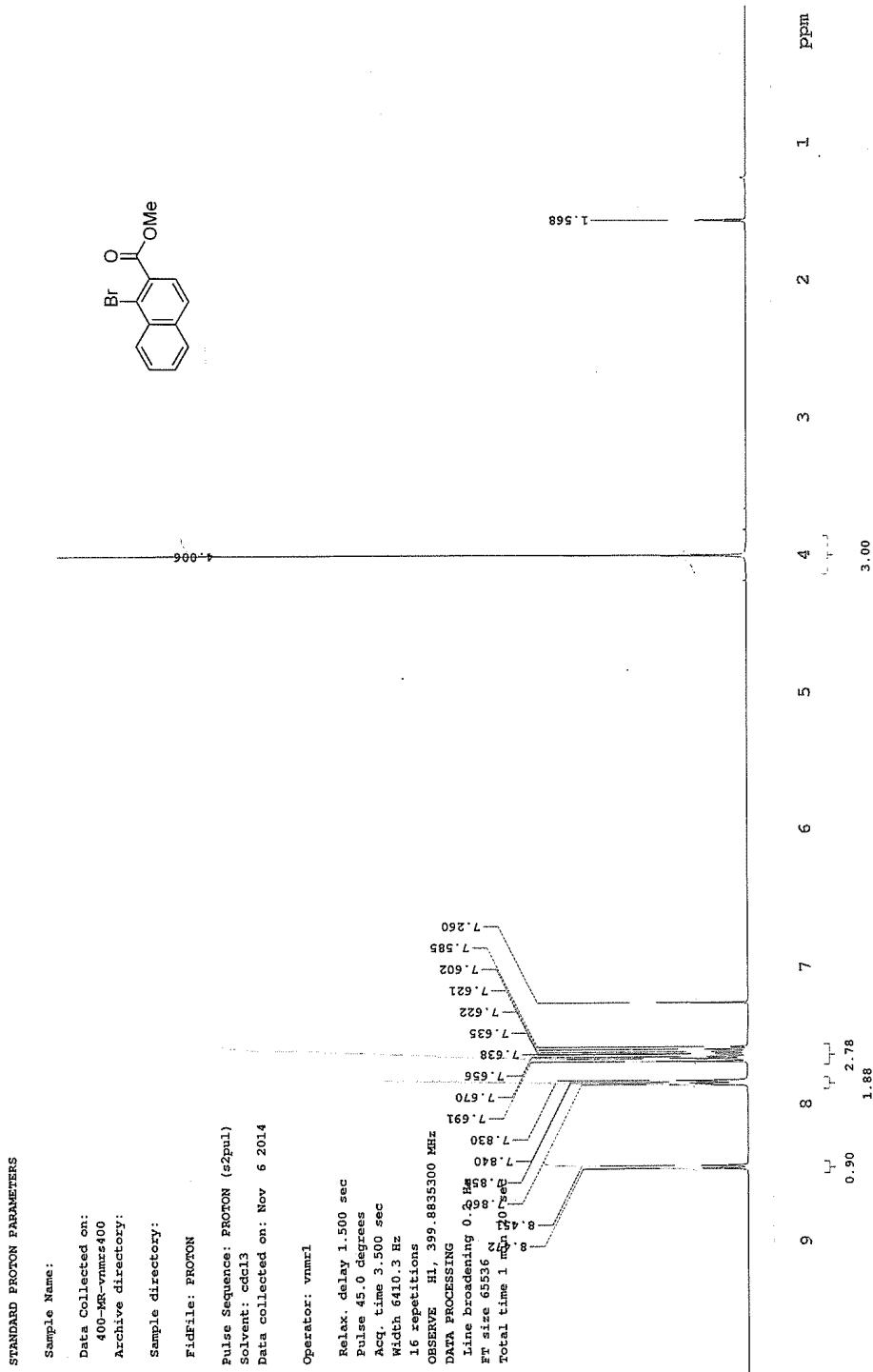
1 / 1

SFC trace for S2

#### **4. References**

- [S1] a) Yamamoto, T.; Akai, Y.; Nagata, Y.; Suginome, M. *Angew. Chem., Int. Ed.* **2011**, *50*, 8844.  
b) Yamamoto, T.; Akai, Y.; Suginome, M. *Angew. Chem., Int. Ed.* **2014**, *53*, 12785.
- [S2] a) Tang, W.; Patel, N. D.; Xu, G.; Xu, X.; Savoie, J.; Ma, S.; Hao, M.-H.; Keshipeddy, S.; Capacci, A. G.; Wei, X.; Zhang, Y.; Gao, J. J.; Li, W.; Rodriguez, S.; Lu, B. Z.; Yee, N. K.; Senanayake, C. H., *Org. Lett.* **2012**, *14*, 2258.  
b) Sun, L.; Dai, W.-M. *Tetrahedron* **2011**, *67*, 9072.

## 5. NMR Spectra and GPC Chart of New Compounds



STANDARD CARBON PARAMETERS

Sample Name:

Data Collected on:  
400-MR-TWINS400

Archive directory:

Sample directory:

FidFile: CARBON

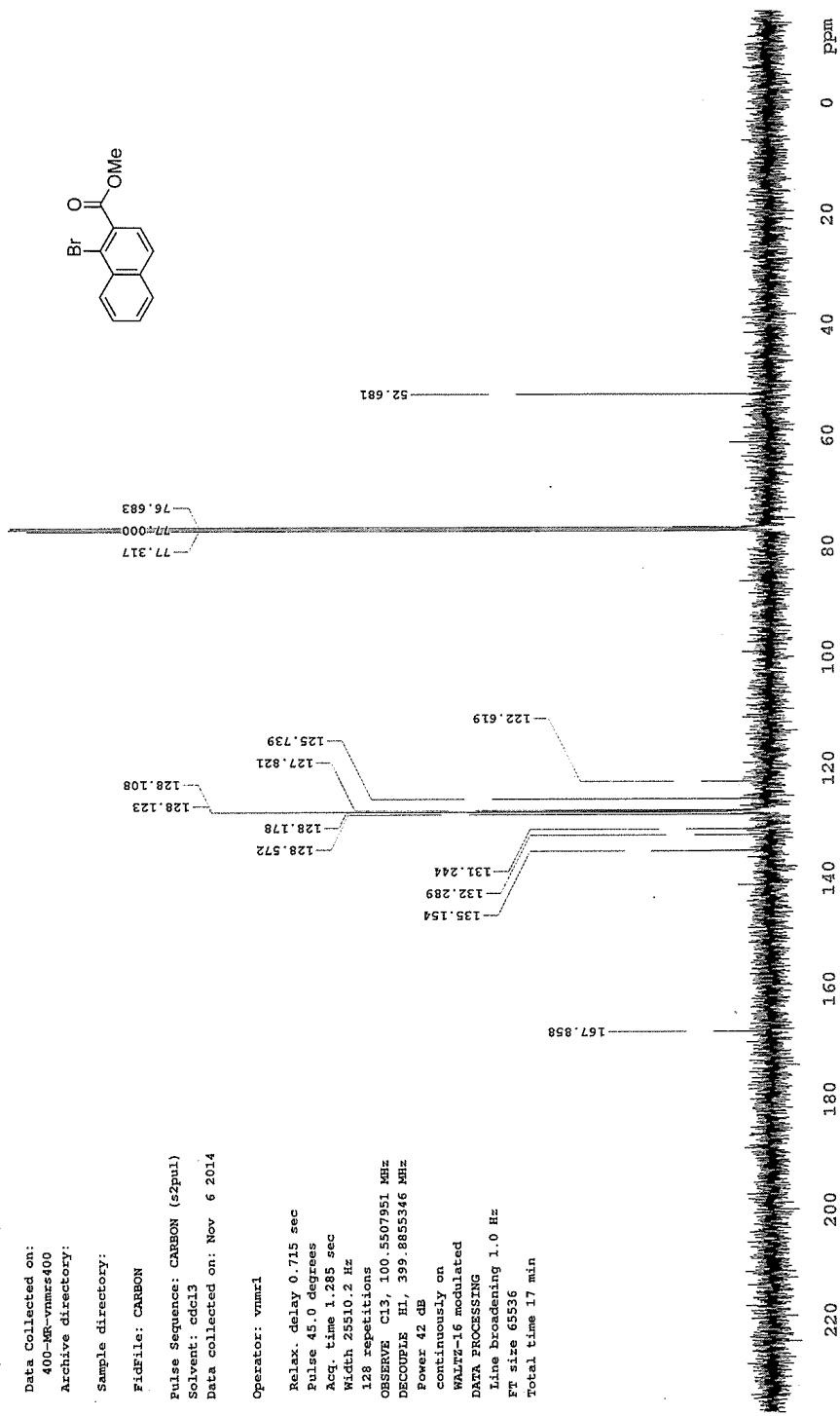
Pulse Sequence: CARBON (s2pul)

Solvent: cdcl<sub>3</sub>

Data collected on: Nov 6 2014

Operator: vnmrl

Relax. delay 0.715 sec  
Pulse 45.0 degrees  
Acc. time 1.285 sec  
Width 25510.2 Hz  
128 repetitions  
OBSERVE C13, 100.5507951 MHz  
DECOUPLE H1, 399.8855346 MHz  
Power 42 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 65536  
Total time 17 min



<sup>13</sup>C NMR of compound 1A

STANDARD PROTON PARAMETERS

Sample Name:

Data Collected on:  
400-MR-vnmrs400

Archive directory:

Sample directory:

FidFile: PROTON

Pulse Sequence: PROTON (sp2pul)

Solvent: cdcl3

Data collected on: Nov 6 2014

Operator: vnmrs1

Relax. delay 1.500 sec

Pulse 45.0 degrees

Acc. time 3.500 sec

Width 640.3 Hz

16 repetitions

OBSERVE H1, 399.8835302 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 65536

Total time 1 min 49.49

9.49

8.49

7.49

6.49

5.49

4.49

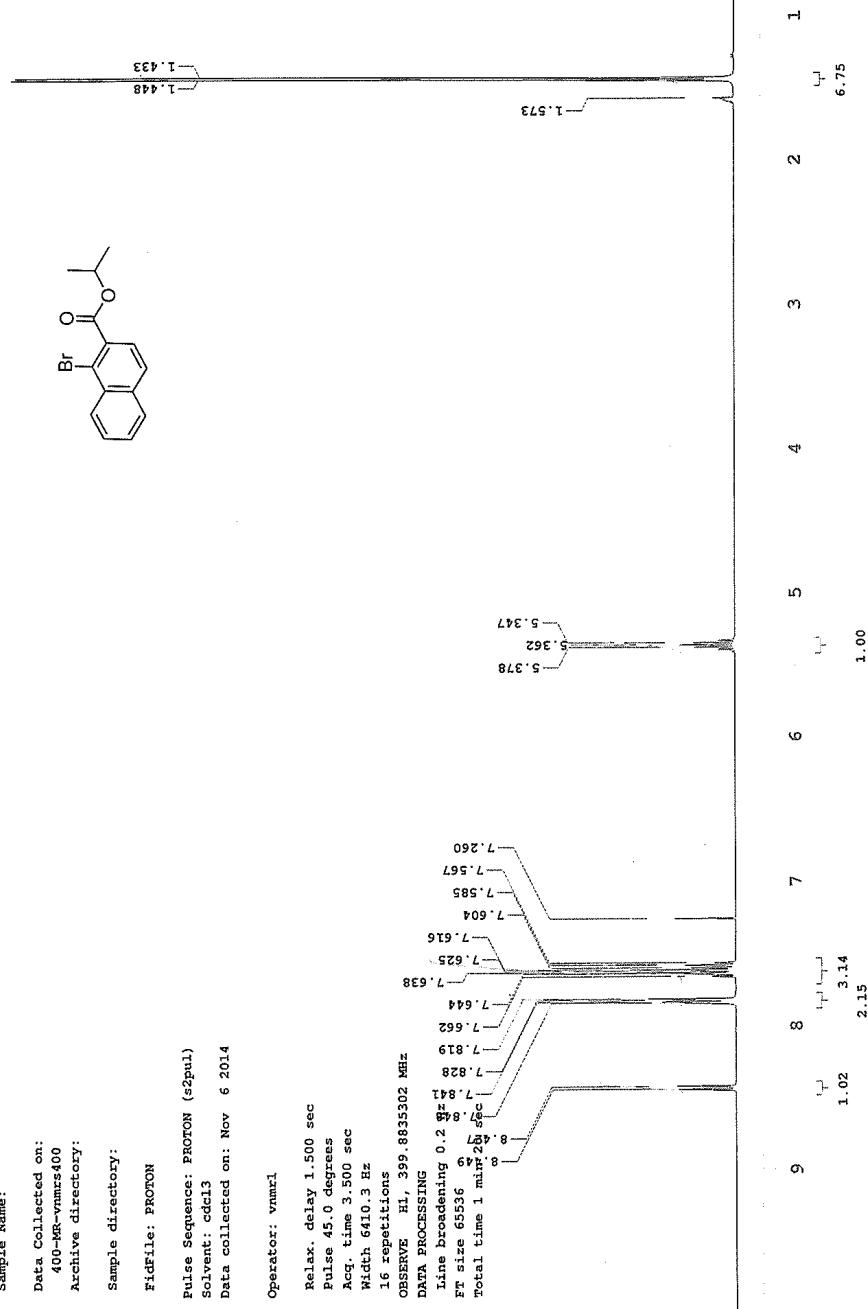
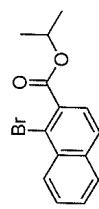
3.49

2.49

1.49

0.49

ppm



<sup>1</sup>H NMR of compound 1B

STANDARD CARBON PARAMETERS

Sample Name:

Data Collected on:  
400-MR-vnmr400

Archive directory:

Sample directory:

FidFile: CARBON

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl<sub>3</sub>

Data collected on: Nov 6 2014

Operator: vnmri

Relax. delay 0.715 sec

Pulse 45.0 degrees

Acq. time 1.225 sec

Width 25510.2 Hz

64 repetitions

OBSERVE C13, 100.5507951 MHz

DECOPPLE H1, 399.8855346 MHz

Power 42 dB

continuously on

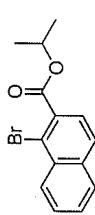
WALTZ-16 modulated

DATA PROCESSING

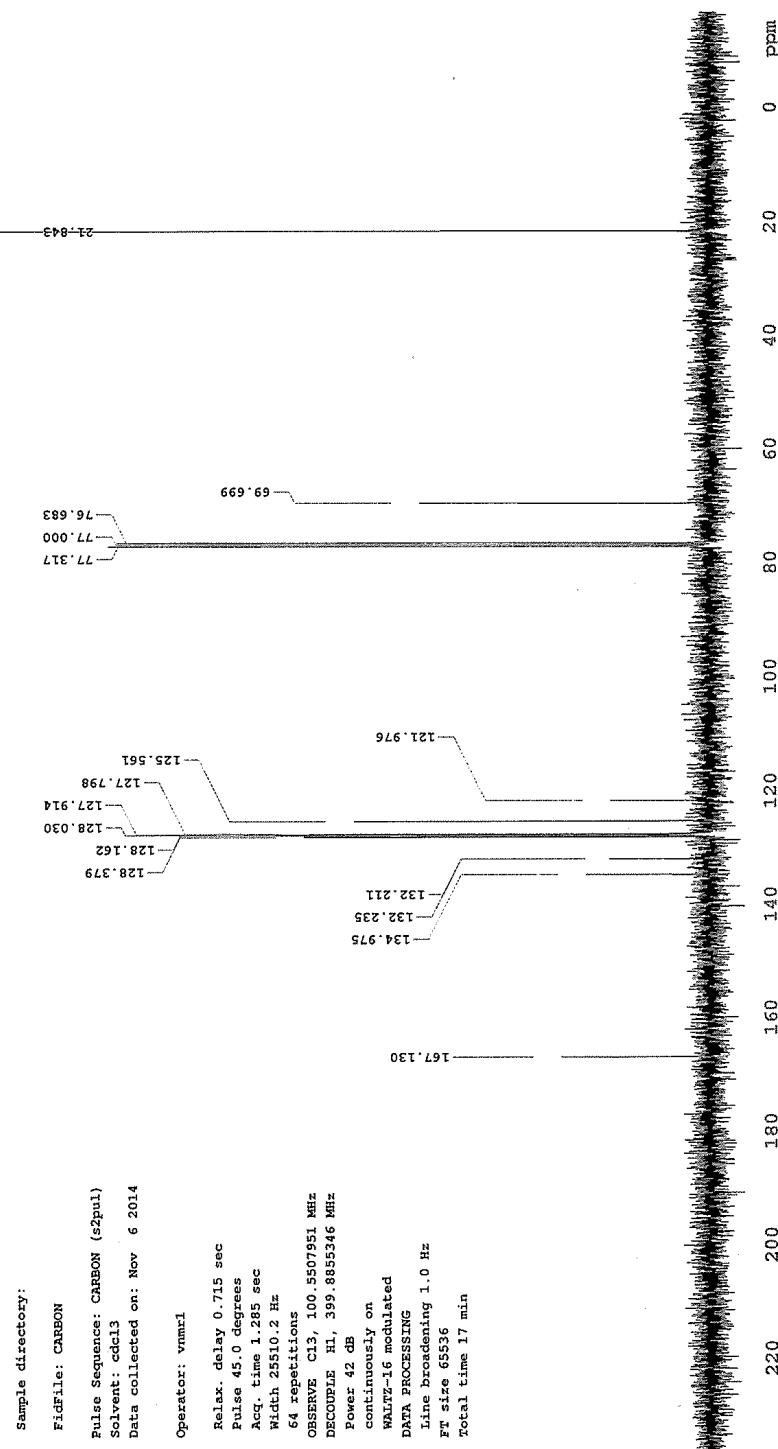
Line broadening 1.0 Hz

FT size 65536

Total Time 17 min



21-843



<sup>13</sup>C NMR of compound 1B

STANDARD PROTON PARAMETERS

Sample Name:

Data Collected on:  
400-MR-vnmrs400

Archive directory:

Sample directory:

FidFile: PROTON

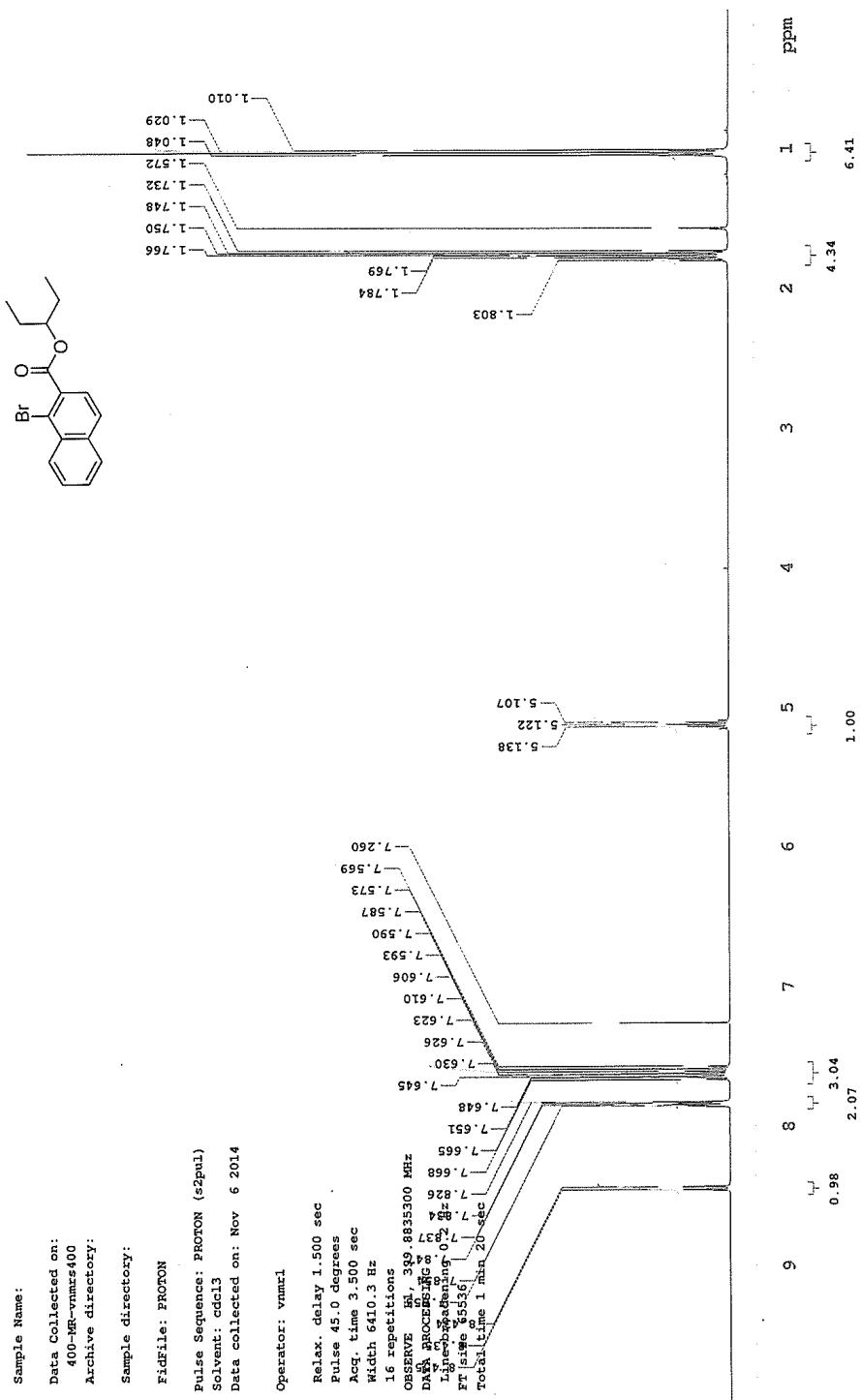
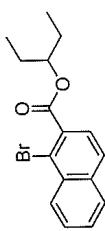
Pulse Sequence: PROTON (sp-pul)

Solvent: cdcl<sub>3</sub>

Data collected on: Nov 6 2014

Operator: vnmrl

Relax. delay 1.500 sec  
 Pulse 45.0 degrees  
 ACG. time 3.500 sec  
 Width 6410.3 Hz  
 16 repetitions  
 OBSERVE H1 399.8835300 MHz  
 DATA PROCESSING H1 399.8835300 MHz  
 1D  
 L1 Head-Sw1 0°  
 FT Size 2532  
 Window 0.500 sec  
 Total time 1 min 20 sec



<sup>1</sup>H NMR of compound 1C

STANDARD CARBON PARAMETERS

Sample Name:

Data Collected on:  
400-MR-vnmrs400

Archive directory:

Sample directory:

FidFile: CARBON

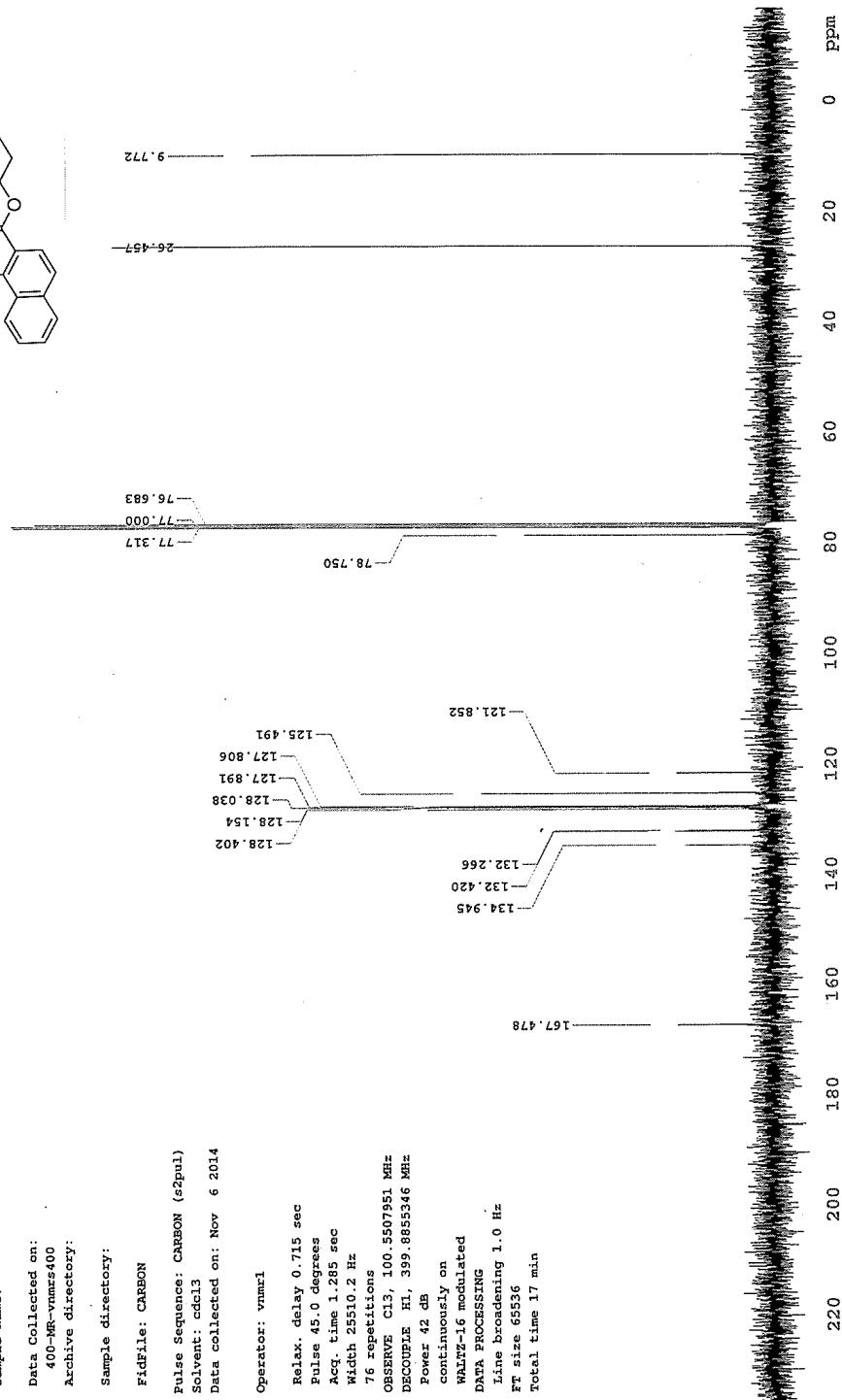
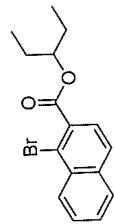
Pulse Sequence: CARBON (s2pul)

Solvent: cdd13

Data collected on: Nov 6 2014

Operator: vnmr1

Relax. delay 0.715 sec  
Pulse 45.0 degrees  
Acc. time 1.285 sec  
Width 25510.2 Hz  
76 repetitions  
OBSERVE CL3, 100.550951 MHz  
DECOUPLE H1, 399.8855346 MHz  
Power 42 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 65336  
Total time 17 min



<sup>13</sup>C NMR of compound 1C

## STANDARD PROTON PARAMETERS

Surname Name:

Data Collected on:  
400-MR-units400

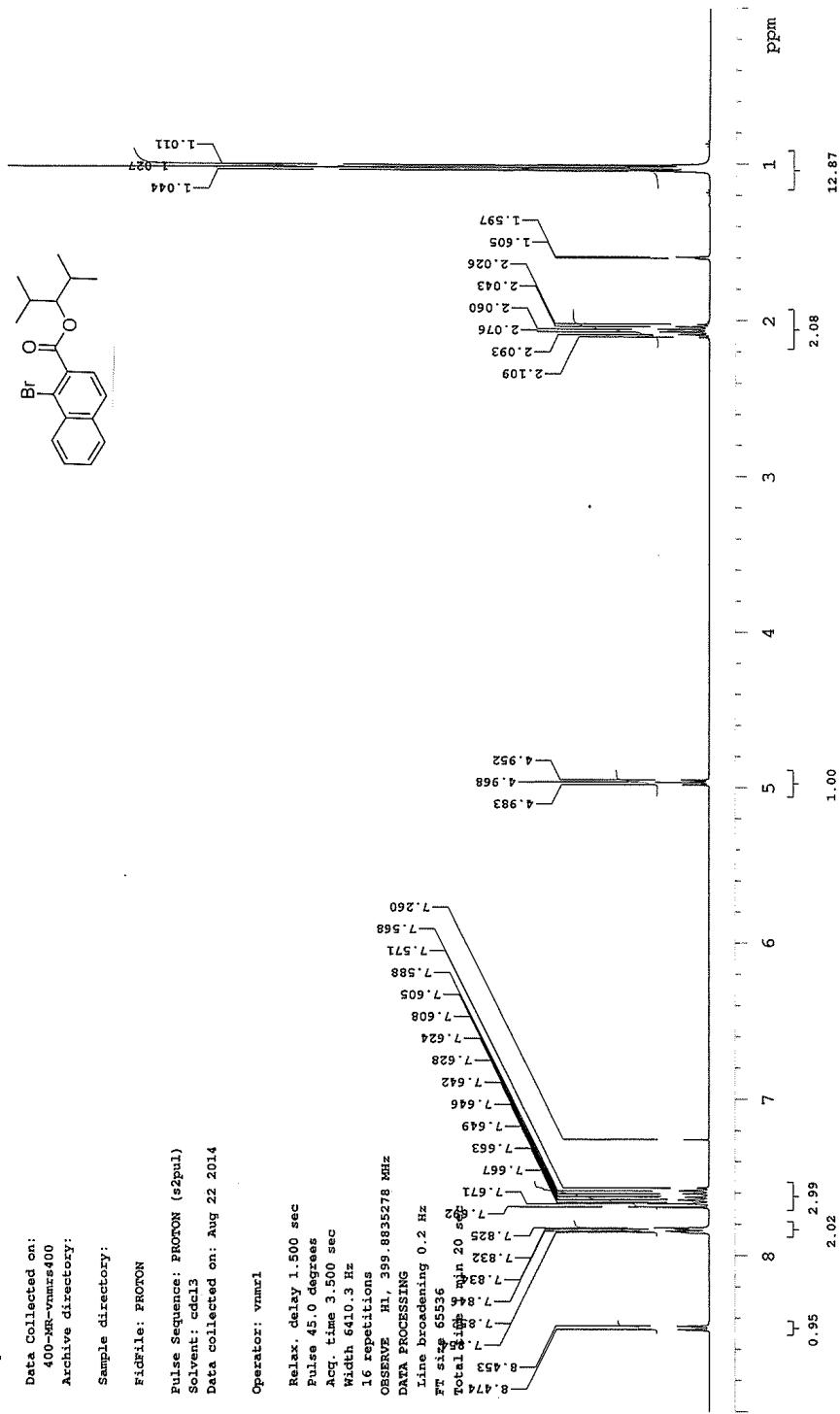
Sample directory:

Fidfile: PROTON

卷之三

Data collected on: Aug 22 2014

二三



### <sup>1</sup>H NMR of compound 1D

STANDARD CARBON PARAMETERS

Sample Name:

Data Collected on:  
400-MR-000-400

Archive directory:  
.....

Sample directory:

Fidfile: CARBON

Pulse Sequence: CARBON (32pul)

Solvent: cdcl<sub>3</sub>

Data collected on: Aug 22 2014

Operator: vmarl

Relax. delay 0.715 sec  
Pulse 45.0 degrees

Avg. time 1.205 sec  
Width 2550.2 Hz

64 repetitions

observe C13, 100.5507943 MHz

DECODEPL H1, 399.8855346 kHz

Power 42 dB

continuously on

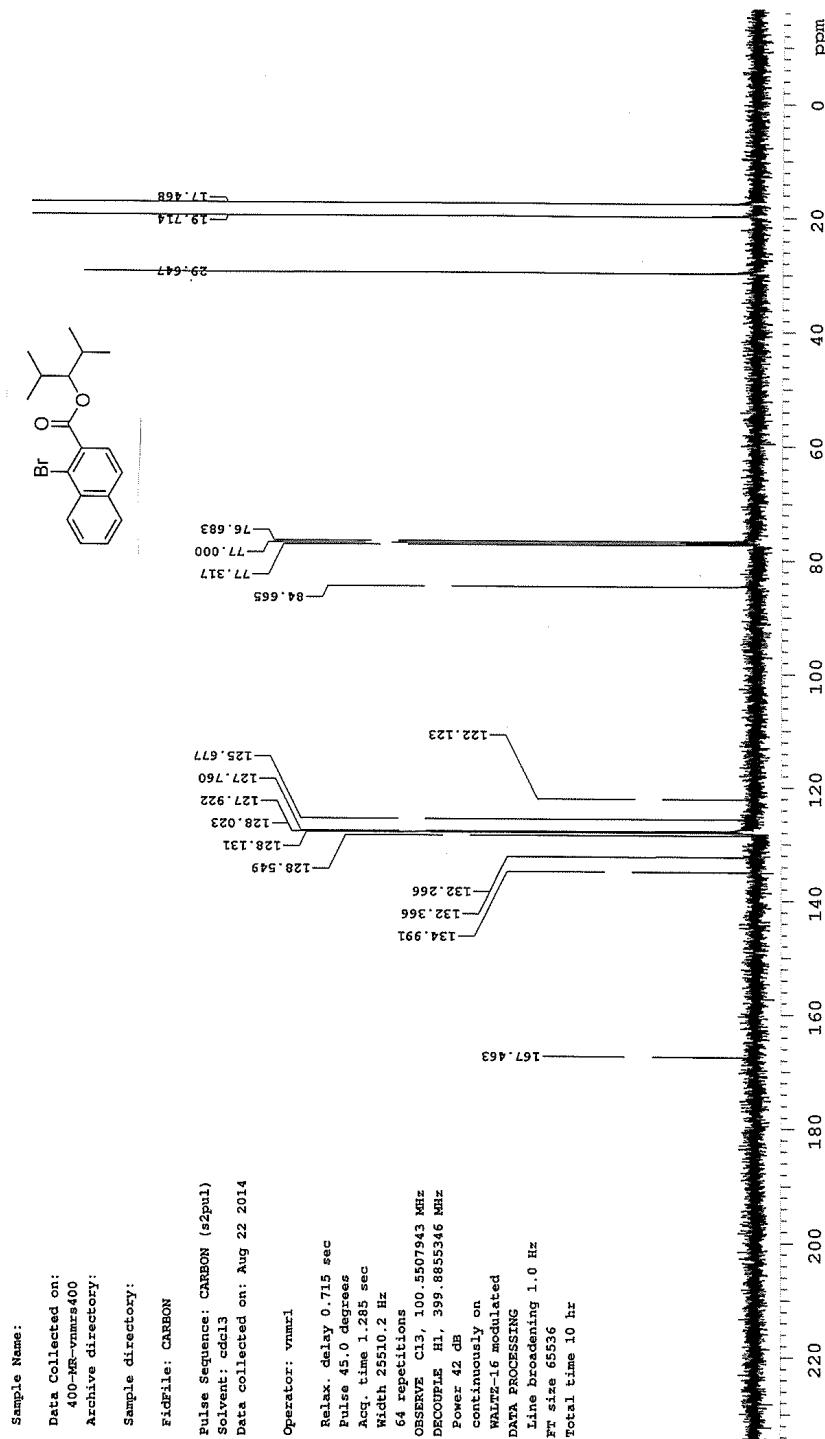
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 10 hr



<sup>13</sup>C NMR of compound 1D

## STANDARD PROTON PARAMETERS

Sample Name:

Data Collected on:  
400-MR-nmrs400

Archive directory:

Sample directory:

FidFile: PROTON

Pulse Sequence: PROTON

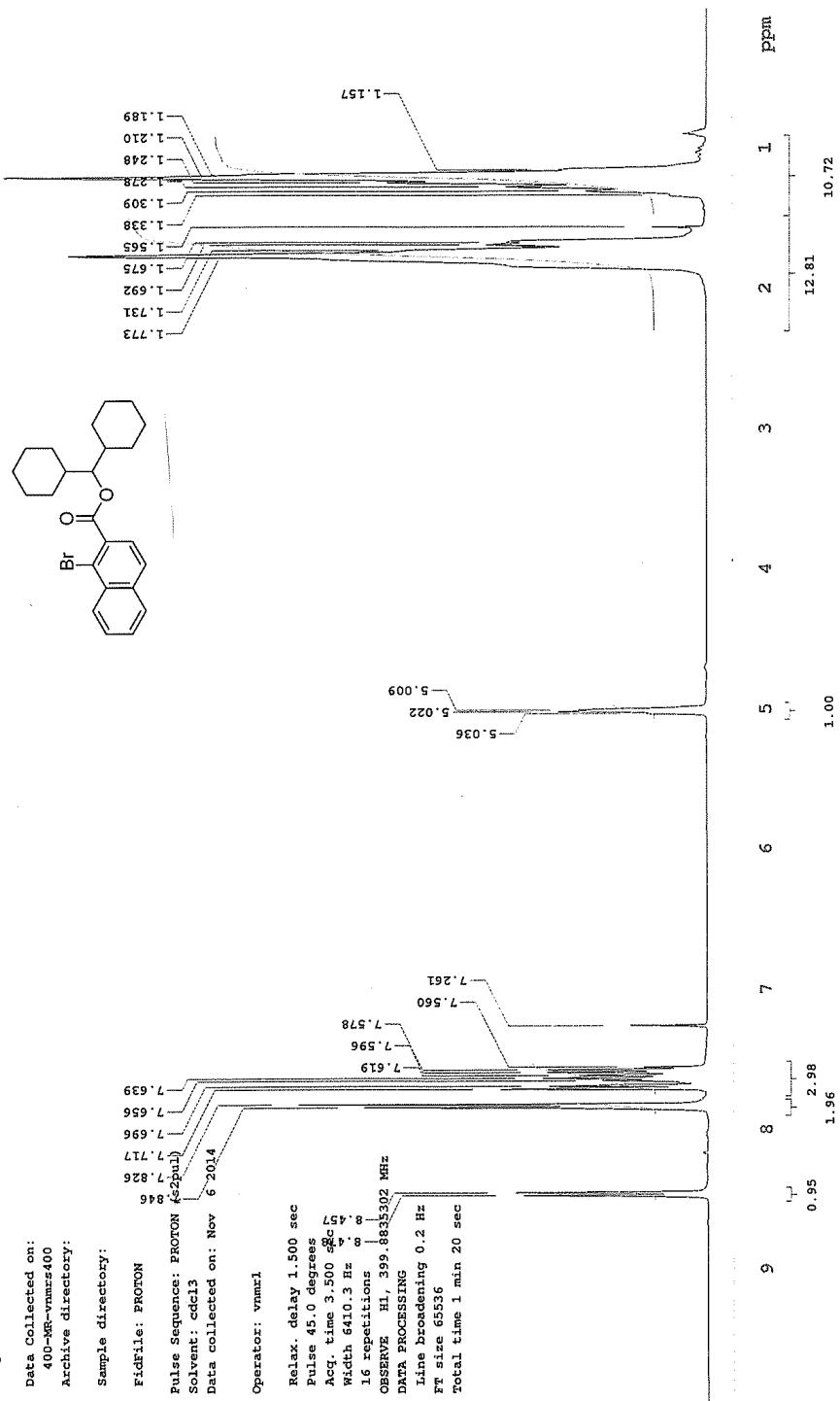
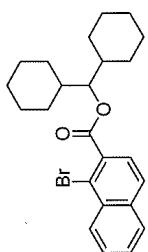
Solvent: dcd13

Data collected on: Nov

6 2014

Operator: vnmrl

Relax. delay 1.500 sec  
 Pulse 45.0 degrees  
 Acq. time 3.500 sec  
 Width 6410.3 Hz  
 16 repetitions  
 OBSERVE H1, 399.8935392 MHz  
 DATA PROCESSING  
 Line broadening 0.2 Hz  
 FT size 65536  
 Total time 1 min 20 sec



STANDARD CARBON PARAMETERS

Sample Name:

Data Collected on:  
400-MR-vnmr400

Archive directory:

Sample directory:

FidFile: CARBON

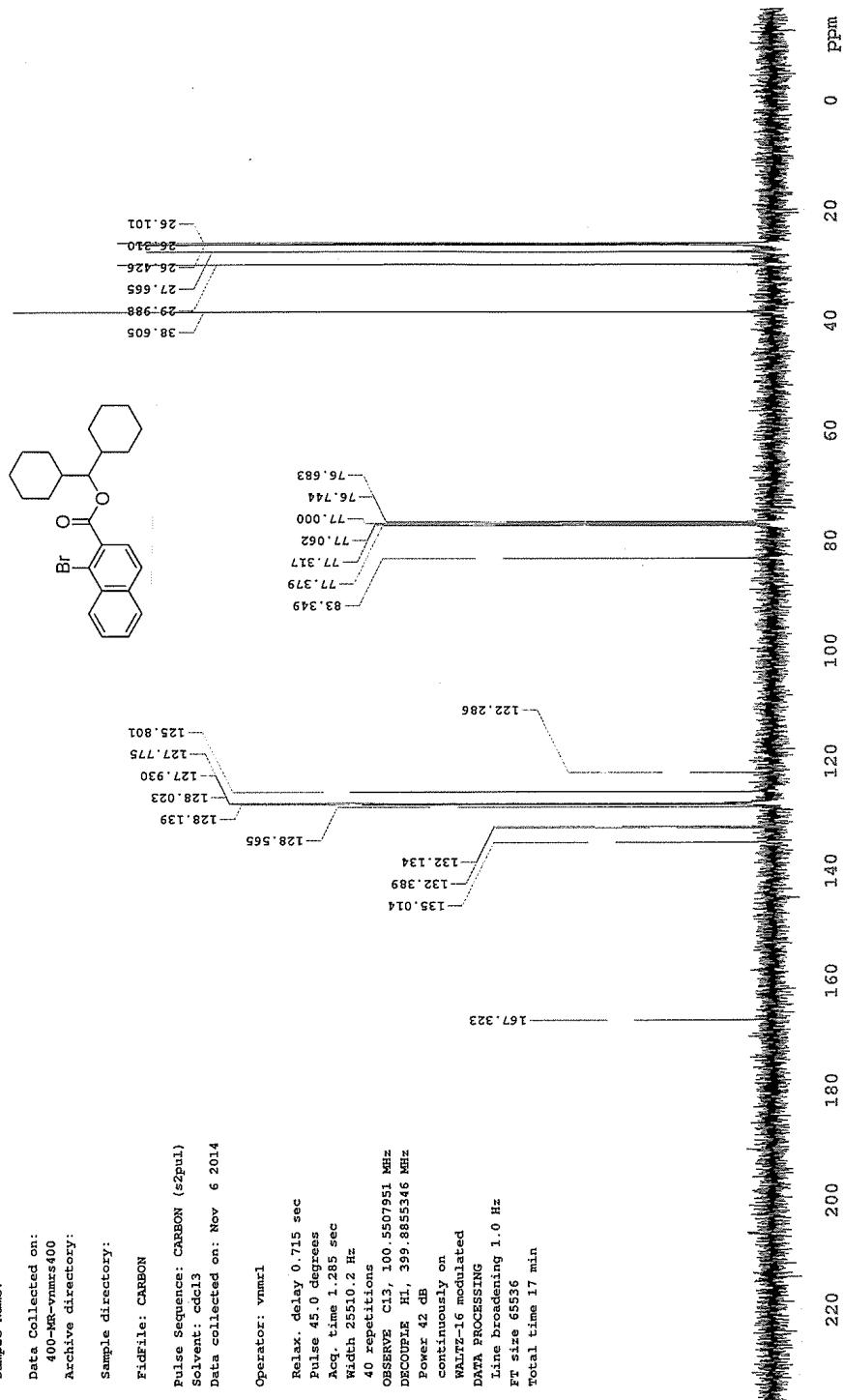
Pulse Sequence: CARBON (s2pul)

Solvent: c6d13

Data collected on: Nov 6 2014

Operator: vnmr1

Relax. delay 0.715 sec  
Pulse 45.0 degrees  
Acq. time 1.285 sec  
Width 25510.2 Hz  
40 repetitions  
OBSERVE C13, 100.5507051 MHz  
DECOPLEAR H1, 399.8855346 MHz  
Power 42 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 65536  
Total time 17 min



<sup>13</sup>C NMR of compound 1E

## STANDARD PROTON PARAMETERS

Sample Name :

Data Collected on:  
400-NS-vnmrs400

Archive directory:

Sample directory:

Pulse Sequenced Proton (<sup>1</sup>H)Solvent: CDCl<sub>3</sub>

Data collected on: Nov 6 2014

Operator: vnmrJ

Relax. delay 1.500 sec.

Pulse 45.0 degrees

Acq. time 3.500 sec

Width 6410.3 Hz

16 repetitions

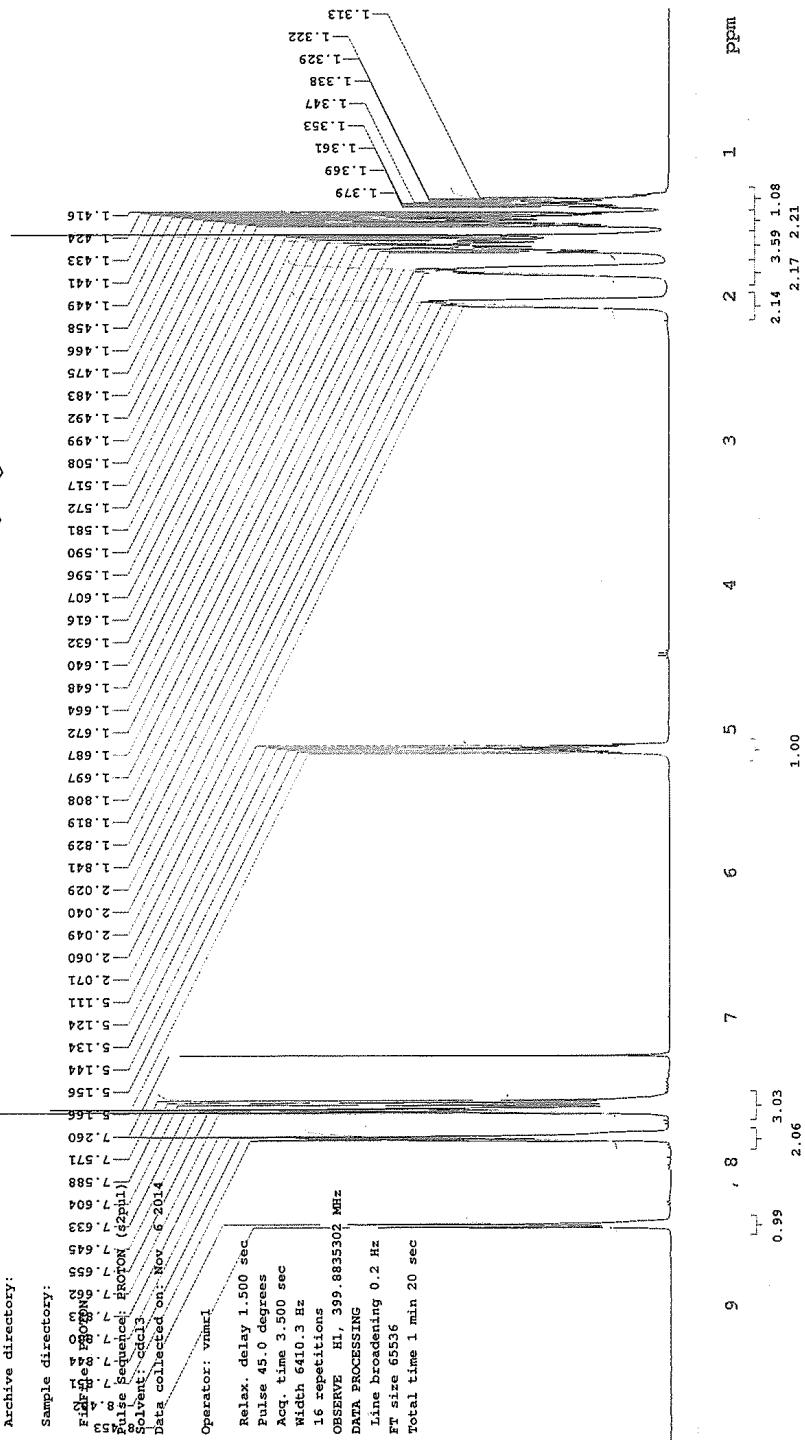
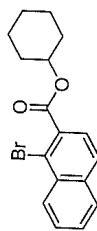
OBSERVE H1, 399.883532 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 65536

Total time 1 min 20 sec



STANDARD CARBON PARAMETERS

Sample Name:

Data Collected on:  
400-MR-NMR400

Archive directory:

Sample directory:

FidFile: CARBON

Pulse Sequence: CARBON (sp2ul)

Solvent: cdcl3

Data collected on: Nov 6 2014

Operator: vnmrl

Relax. delay 0.715 sec

Pulse 45.0 degrees

Avg. time 1.285 sec

Width 25510.2 Hz

128 repetitions

OBSERVE C13, 100.5507949 MHz

DECOUPLE H1, 399.8855346 MHz

Power 42 dB

continuously on

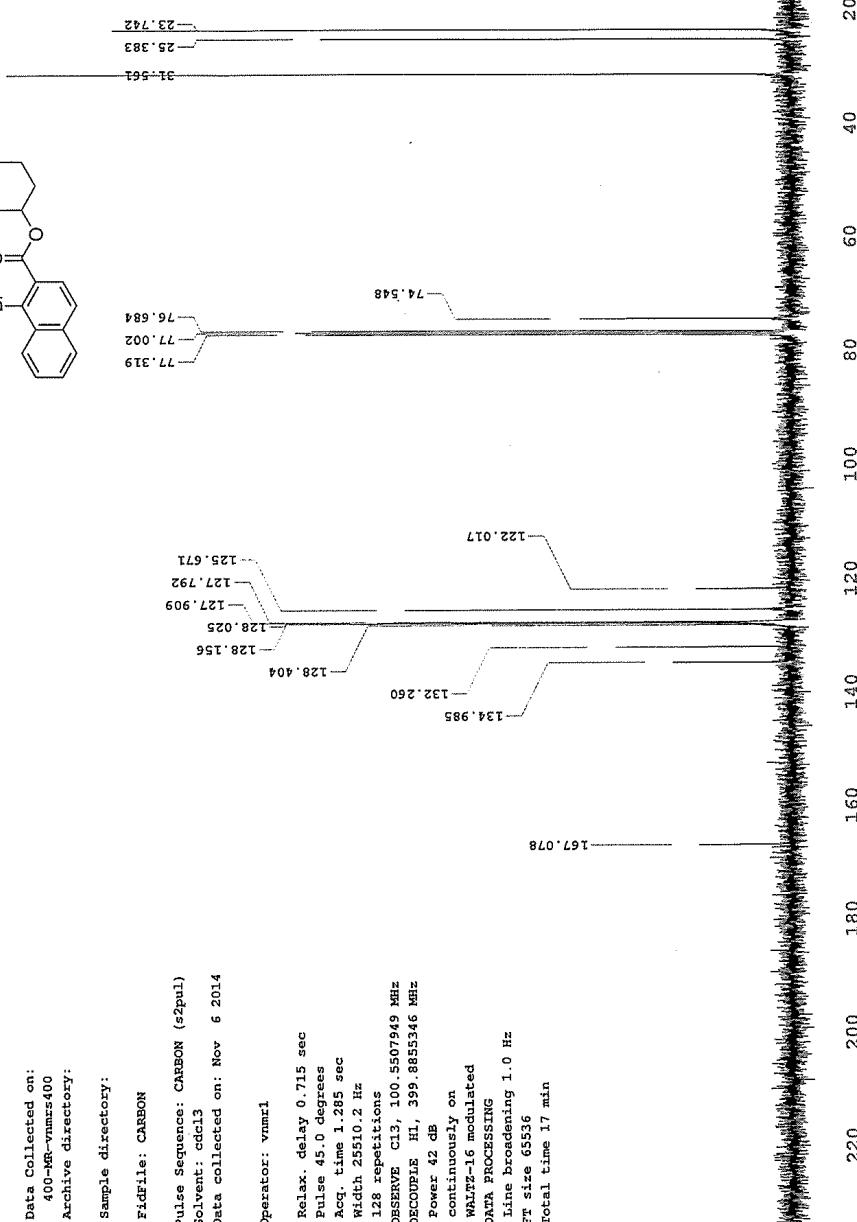
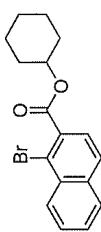
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65336

Total time 17 min



<sup>13</sup>C NMR of compound 1F

## STANDARD PROTON PARAMETERS

Sample Name:

Data Collected on:  
400-MR-vnmrs400

Archive directory:

Sample directory:

FidFile: PROTON

Pulse Sequence: PROTON (ss)

Solvent: cdcl<sub>3</sub>

Data collected on: NMR

Operator: vnmrs400

Relax. delay 1.300 sec

Pulse 45.0 degrees

Aqc. time 3.500 sec

Width 6410.3 Hz

16 repetitions

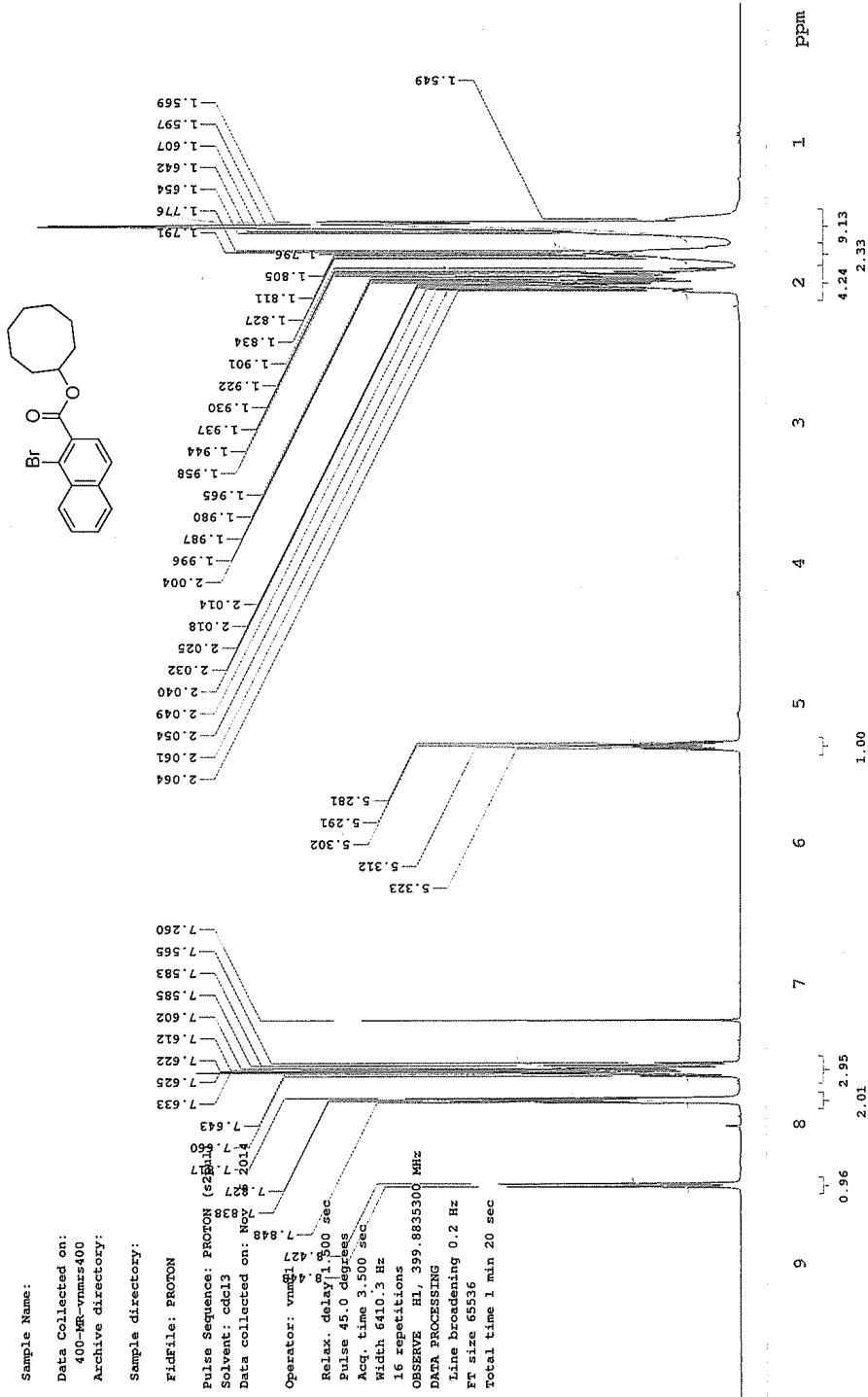
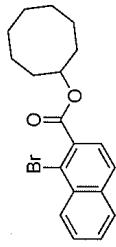
OBSERVE H1 399.8835300 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 65536

Total time 1 min 20 sec

<sup>1</sup>H NMR of compound 1G

## STANDARD CARBON PARAMETERS

Sample Name:

Data Collected on:  
400-MR-vnmrs400

Archive directory:

Sample directory:

FidFile: CARBON

Pulse Sequence: CARBON (sp2pul)

Solvent: cdcl<sub>3</sub>

Data collected on: Nov 6 2014

Operator: vnmrs1

Relax. delay 0.715 sec

Pulse 45.0 degrees

Acc. time 1.285 sec

Width 25510.2 Hz

96 repetitions

OBSERVE C13, 100.5507951 MHz

DECOUPLE H1, 399.8855346 MHz

Power 42 dB

continuously on

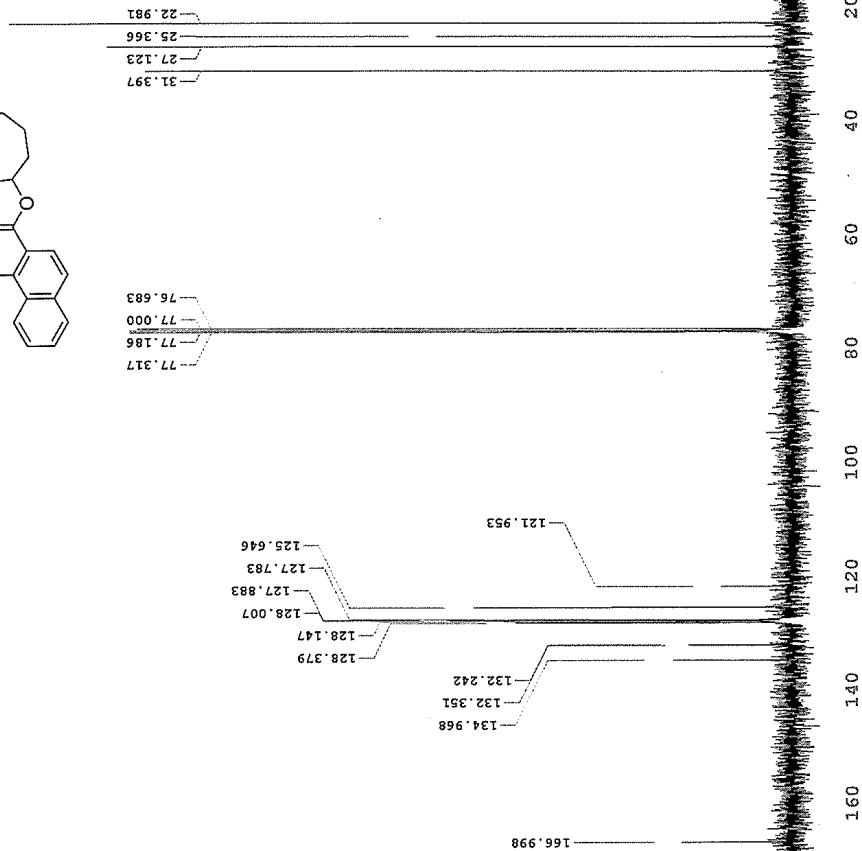
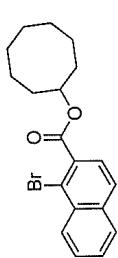
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 17 min



STANDARD PROTON PARAMETERS

Sample Name:

Data Collected on:  
400-MR-vnmrs400

Archive directory:

### Sample directory:

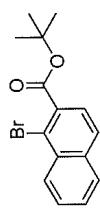
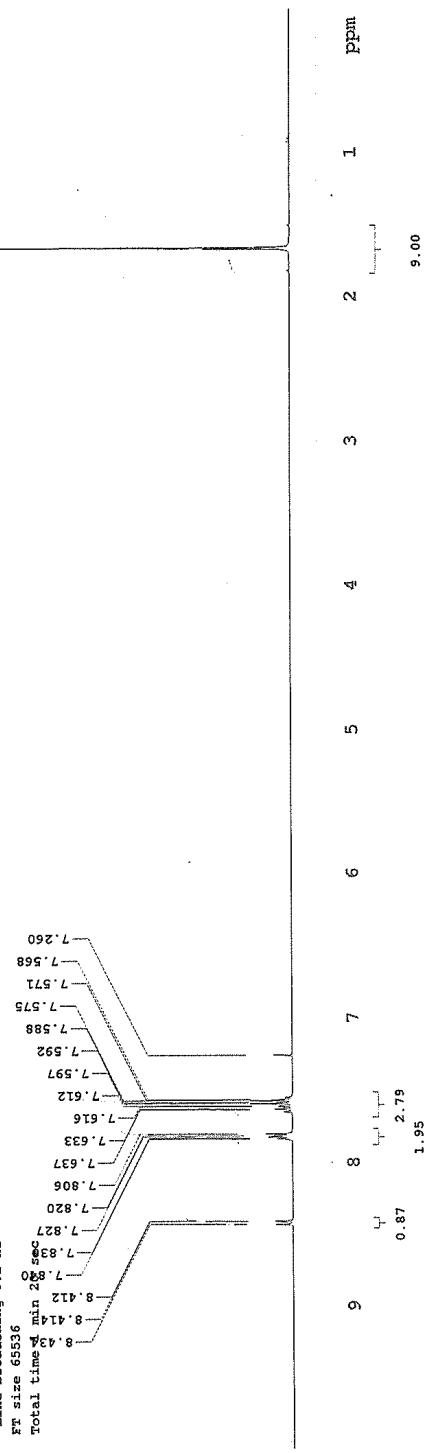
### **FidFile: PROTON**

rule sequence: PATTON (5281)

COLLUM: 2003

ISSUE INDEX

Relax. delay 1.500 sec  
Pulse 45.0 degrees  
A.C.Q. time 3.500 sec  
Width 6410.3 Hz  
16 repetitions  
DESPERATE H1, 339.8825300 MHz  
DATA PROCESSING  
Line broadening 0.2 Hz  
Total time 65536 min 20 sec.  
FFT size 65536



### <sup>1</sup>H NMR of compound **1H**

## STANDARD CARBON PARAMETERS

Sample Name:

Data Collected on:  
400-MR-vnmrs 400  
Archive directory:

Sample directory:

FidFile: CARBON

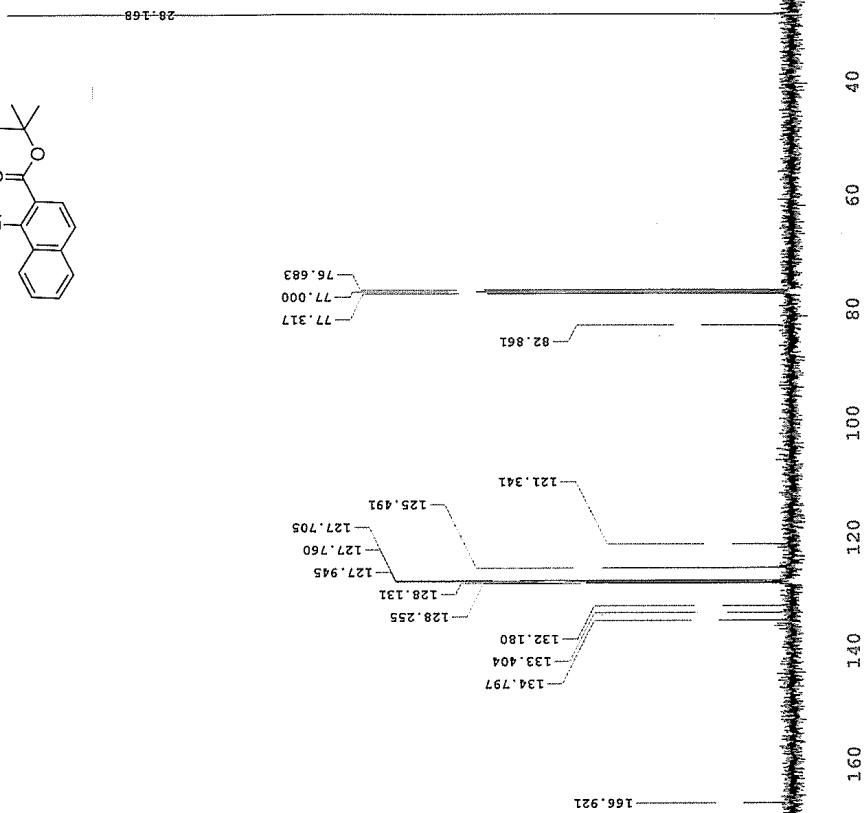
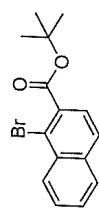
Pulse Sequence: CARBON (s2pul)

Solvent: cdd13

Data collected on: Nov 6 2014

Operator: vnmr1

Relax. delay 0.715 sec  
Pulse 45.0 degrees  
Acq. time 1.285 sec  
Width 25510.2 Hz  
48 repetitions  
OBSERVE C13, 100.5507959 MHz  
DECOPPLE H1, 399.8855346 MHz  
Power 42 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 65536  
Total time 17 min

<sup>13</sup>C NMR of compound 1H

## STANDARD PROTON PARAMETERS

Sample Name:

Data Collected on:  
400-MR-vnmrs400  
Archive directory:

Sample directory:

FidFile: PROTON

Pulse Sequence: PROTON

Solvent: cddc13

Data collected on: Nov 6 2014

Operator: VNMRS

N N 105

order: 1,0,0

delay 1.500 sec

pulses 480

degrees

Acc. time .500 sec

Width 6410.3 Hz

16 repetitions

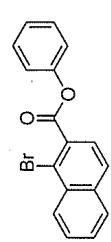
OBSERVE H1, 399.8855300 MHz

DATA PROCESSING

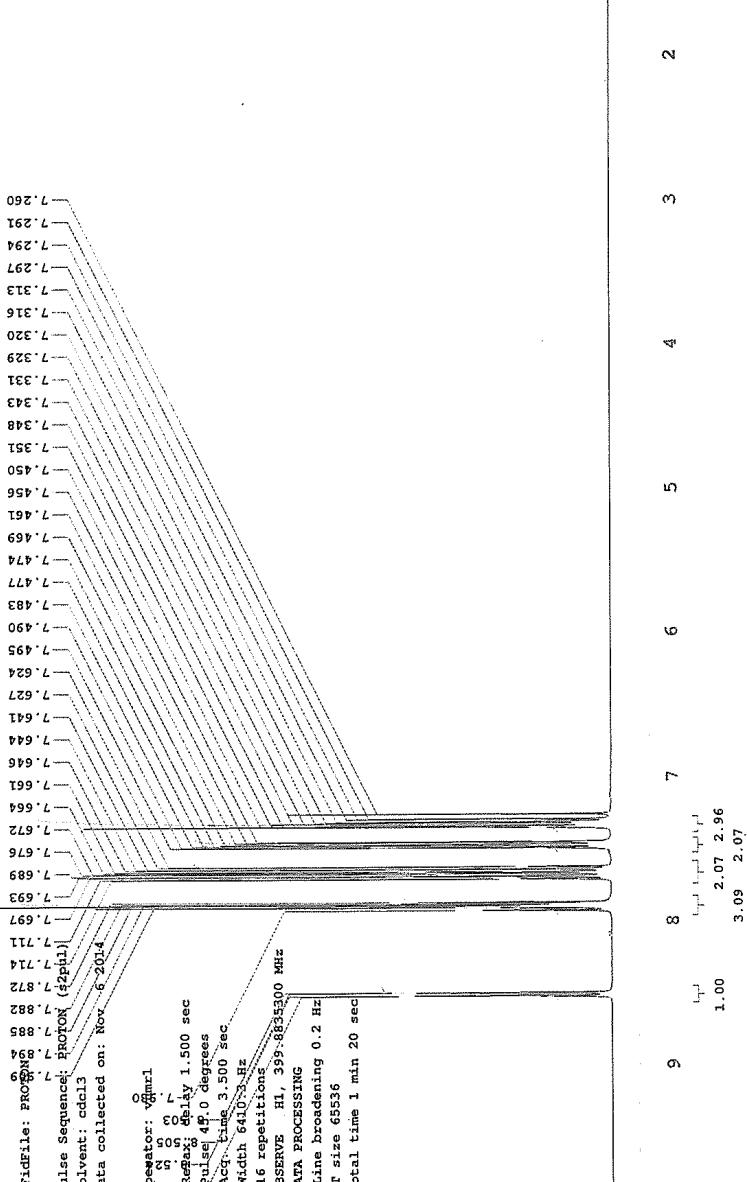
Line broadening 0.2 Hz

FT size 65536

Total time 1 min 20 sec



1.554

<sup>1</sup>H NMR of compound II

STANDARD CARBON PARAMETERS

Sample Name:

Data Collected on:  
400-MR-vnmrs400  
Archive directory:

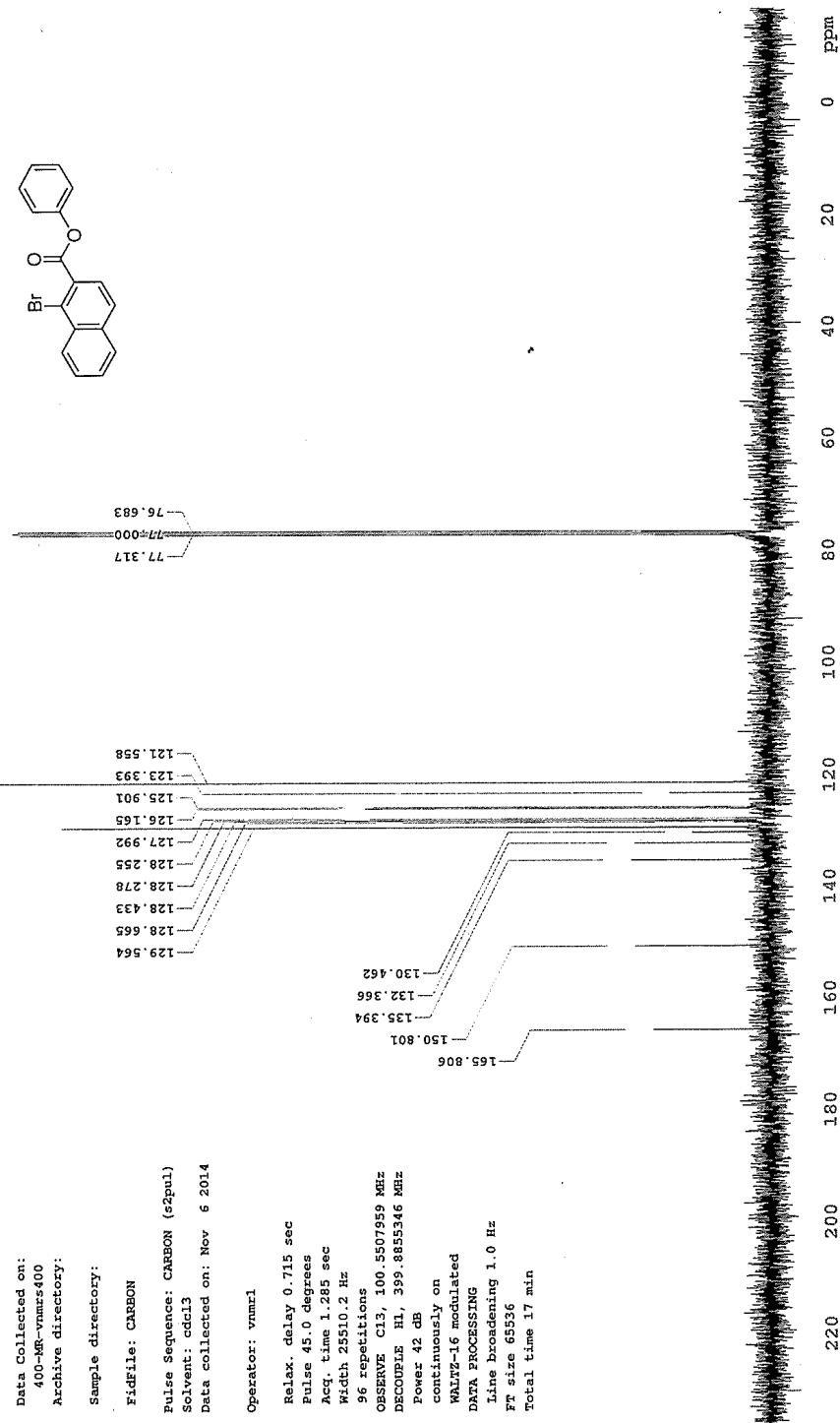
Sample directory:

FidFile: CARBON

Pulse Sequence: CARBON (g2pul)  
Solvent: cdd13  
Data collected on: Nov 6 2014

Operator: vnmrl

Relax. delay 0.715 sec  
Pulse 45.0 degrees  
Acc. time 1.285 sec  
Width 25510.2 Hz  
96 repetitions  
OBSERVE C13, 100.5507959 MHz  
DECUPLE H1, 399.8855346 MHz  
Power 42 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 65536  
Total time 17 min



<sup>13</sup>C NMR of compound II

## STANDARD PROTON PARAMETERS

Sample Name:

Data Collected on:  
400-MR-vnmrs400

Archive directory:

Sample directory:

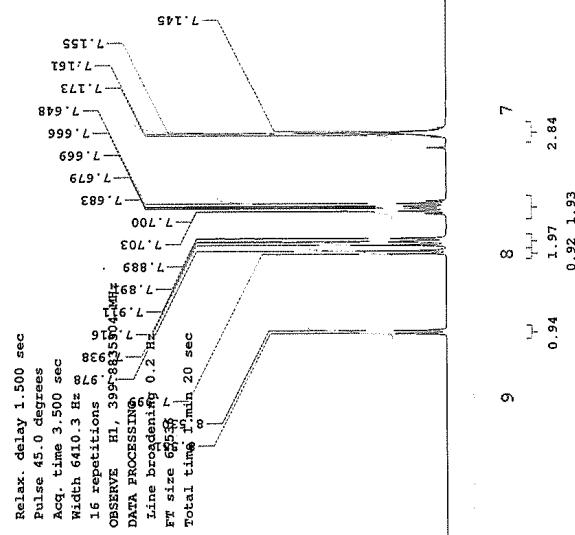
FidFile: PROTON

Pulse Sequence: PROTON (s2pul)

Solvent: cdd013

Data collected on: Nov 6 2014

Operator: vnmrl

<sup>1</sup>H NMR of compound 1J

STANDARD CARBON PARAMETERS

Sample Name:

Data Collected on:  
400-ER-Vnmrs400

Archive directory:

Sample directory:

FidFile: CARBON

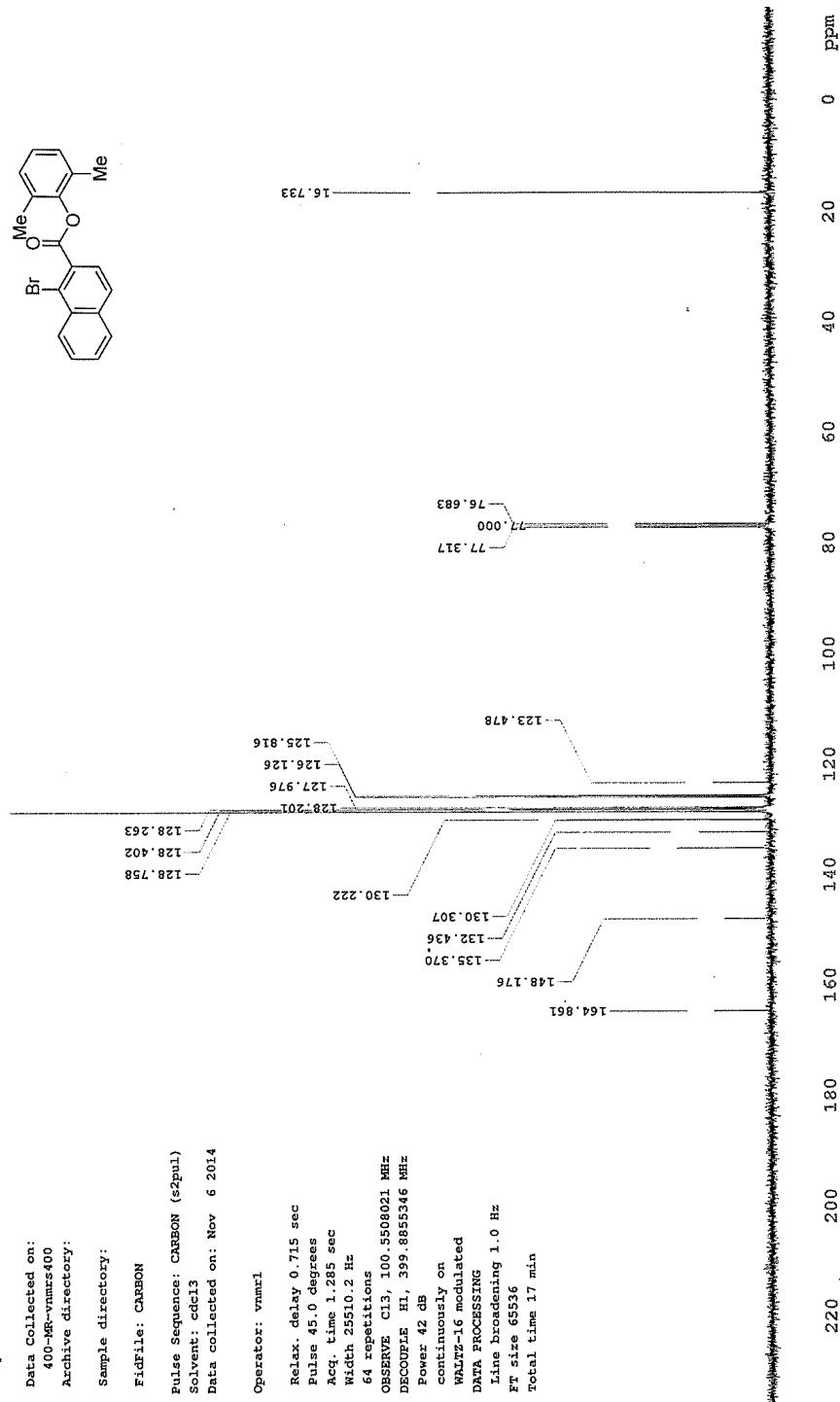
Pulse Sequence: CARBON (sp2ul)

Solvent: cdcl3

Data collected on: Nov 6 2014

Operator: vnmrl

Relax. delay 0.715 sec  
Pulse 45.0 degrees  
Acq. time 1.285 sec  
Width 25510.2 Hz  
64 repetitions  
OBSERVE C13, 100.5508021 MHz  
DECOUPLE H1, 399.8855346 MHz  
Power 42 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 6536  
Total time 17 min



<sup>13</sup>C NMR of compound 1J

## STANDARD PROTON PARAMETERS

Sample Name:

Data Collected on:  
400-MR-vnmrs400

Archive directory:

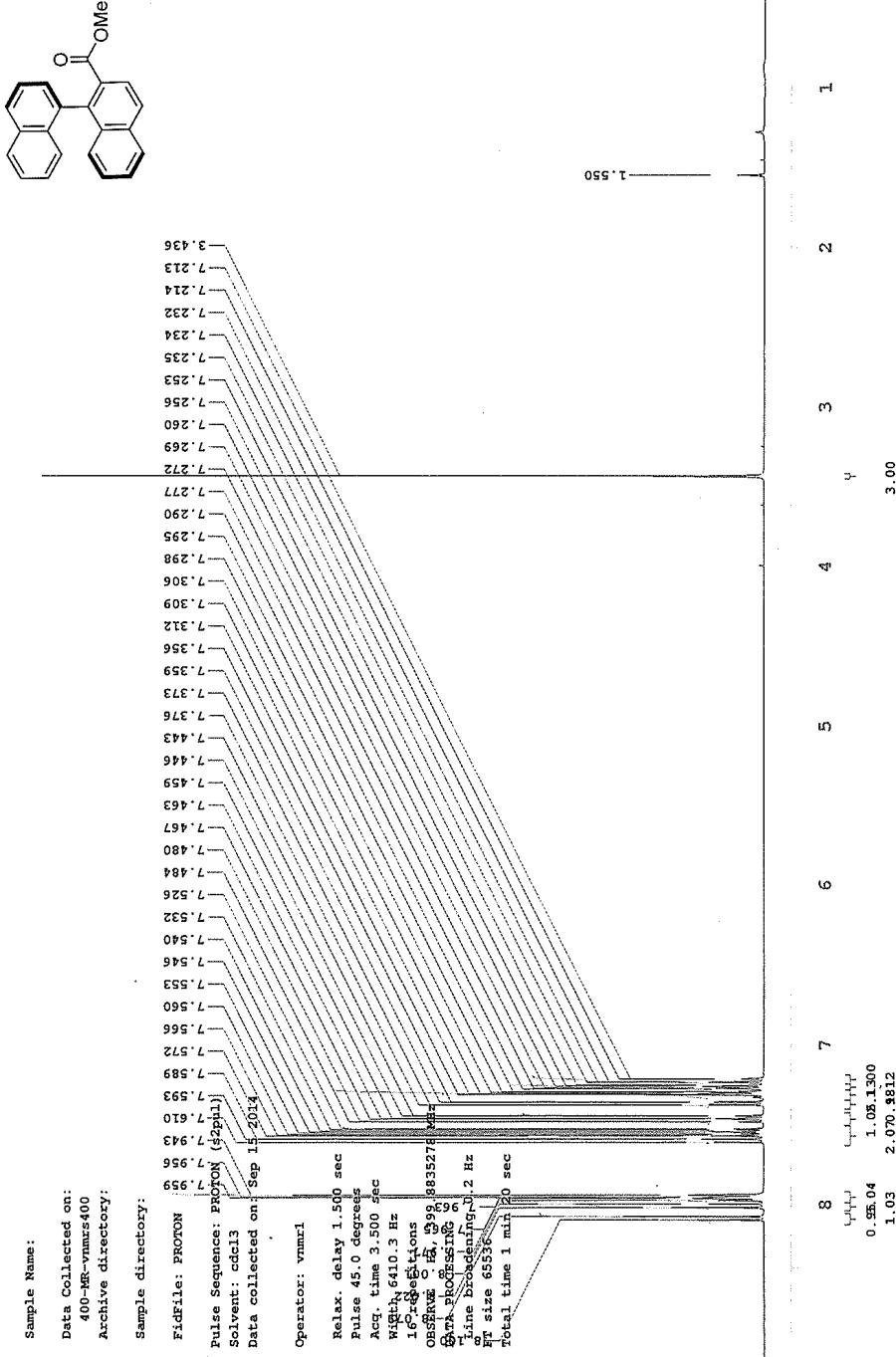
Sample directory:

Fidfile: PROTON  
Pulse Sequence: PRQFTNSolvent: cdcl3  
Data collected on Sep 15 2014

Operator: vnmrl

Relax. delay 1.500 sec  
 Pulse 45.0 degrees  
 Acc. time 3.500 sec  
 WIDTH 6410.3 Hz  
 1.6-ppm ref. buttons  
 OBSERVE C 13, 138.35278

BIN phasing 96.0 Hz  
 Line broadening 1.2 Hz  
 FID size 65536  
 Total time 1 min 20 sec

<sup>1</sup>H NMR of compound 3Aa

## STANDARD CARBON PARAMETERS

Sample Name:

Data Collected on:  
400-MR-vnmrs400

Archive directory:

Sample directory:

FidFile: CARBON

Pulse Sequence: CARBON (s2pul)

Solvent: ccd13

Data collected on: Sep 15 2014  
Operator: vnmrs1

Relax. delay 0.715 sec

Pulse 45.0 degrees

Acc. time 1.285 sec

Width 28510.2 Hz

64 repetitions

OBSERVE C13, 100.5507966 MHz

DECOUPLE H1, 399.8855346 MHz

Power 42 dB

continuously on

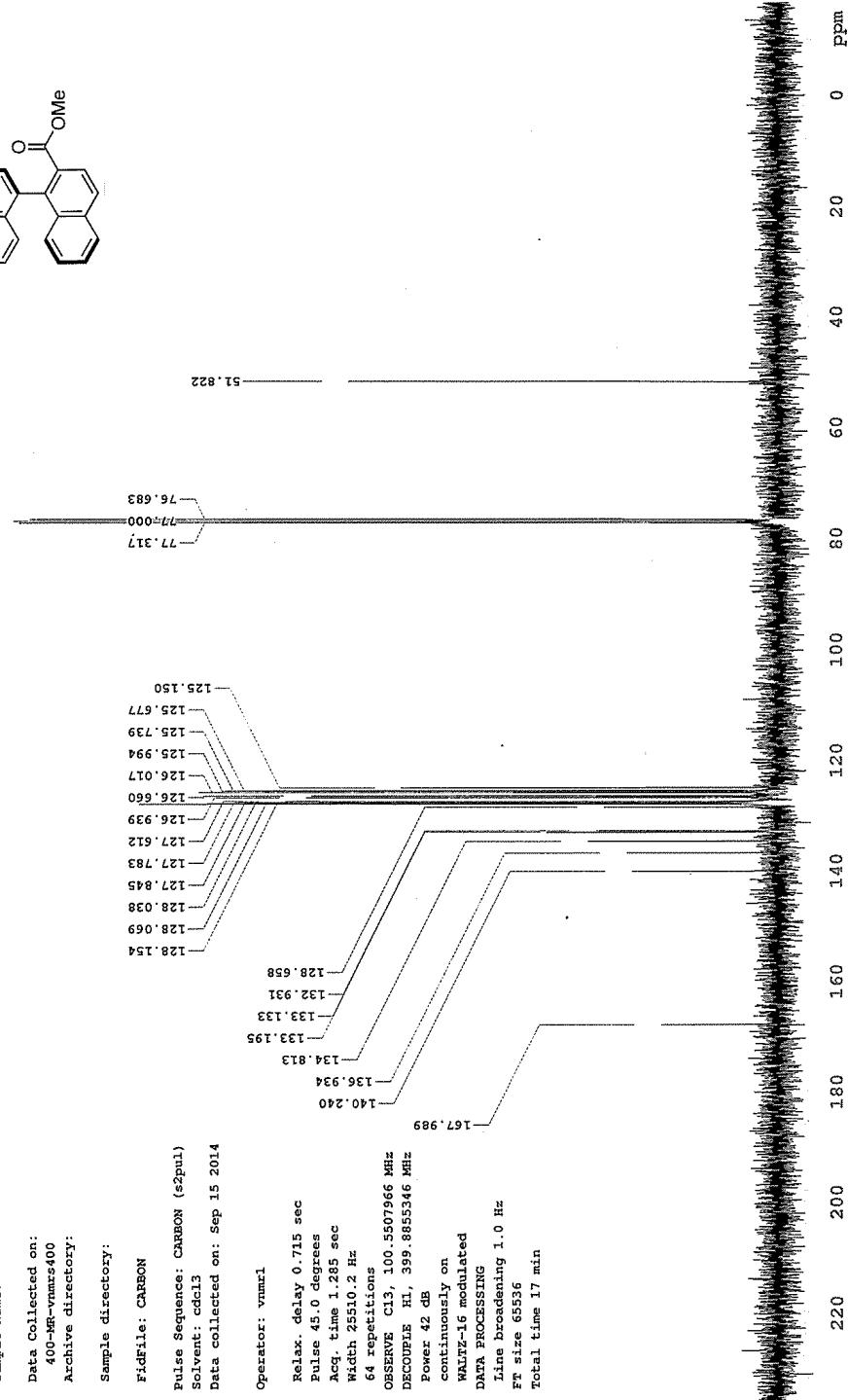
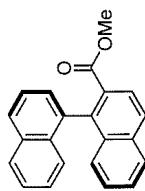
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 17 min

<sup>13</sup>C NMR of compound 3Aa



STANDARD CARBON PARAMETERS

Sample Name:

Data Collected on:  
400-MR-vnmrs400

Archive directory:

Sample directory:

Operator: vnmrs1

FidFile: CARBON

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: Sep 15 2014

Relax: delay 0.715 sec

Pulse 45.0 degrees

Acc. time 1.285 sec

Width 25510.2 Hz

120 repetitions

OBSERVE C13, 100.5507931 MHz  
DECUPLE H1, 399.8855346 MHz

continuously on

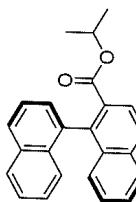
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 0 min 0 sec



20.821

20.751

67.934

76.675

76.992

77.310

125.119

125.677

125.847

126.389

126.575

127.117

127.117

127.419

127.520

127.868

127.930

127.961

128.000

128.961

129.796

130.110

133.257

133.326

134.728

137.345

139.288

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## STANDARD PROTON PARAMETERS

Sample Name:

Data Collected on:  
400-MR-vnmrs400

Archive directory:

Sample directory:

Fidfile: PROTON

Pulse Sequence: 16384

Solvent: cdcl3

S1 Selected on: Sep

Date: 16/09/08

Operator: vnmr1

Relax. delay: 1.00 sec

Pulse 45.0 degrees

Acq. time: 3.500 sec

Width: 6410.3 Hz

16 repetitions

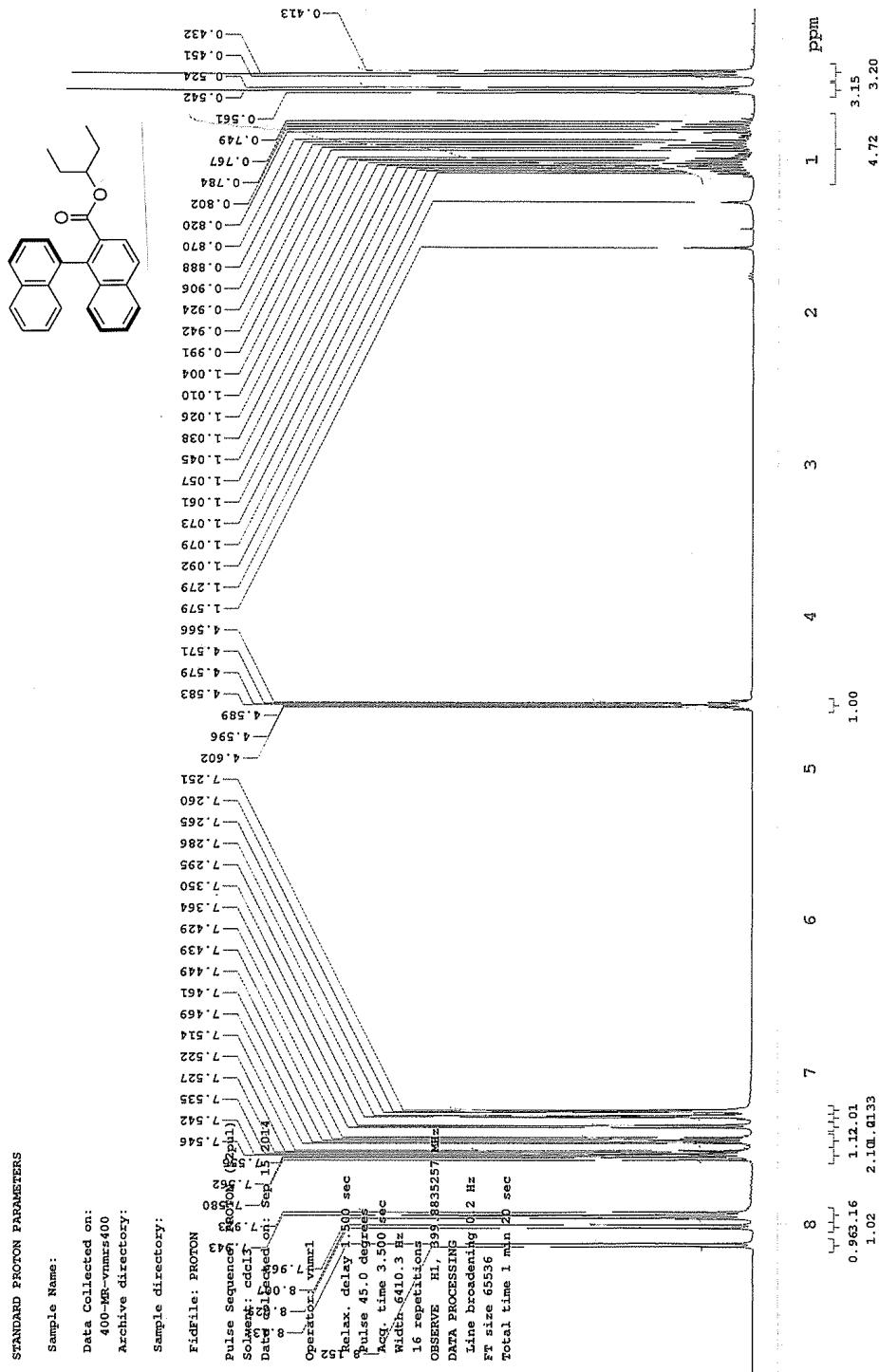
OBSERVE H1, B9, 3835277 MHz

DATA PROCESSING

Line broadening: 0.2 Hz

FT size: 65536

Total time: 1 min 20 sec



**STANDARD CARBON PARAMETERS**

Sample Name:

Data Collected on:  
400-MR-vnms400

Archive directory:

Sample directory:

FidFile: CARBON

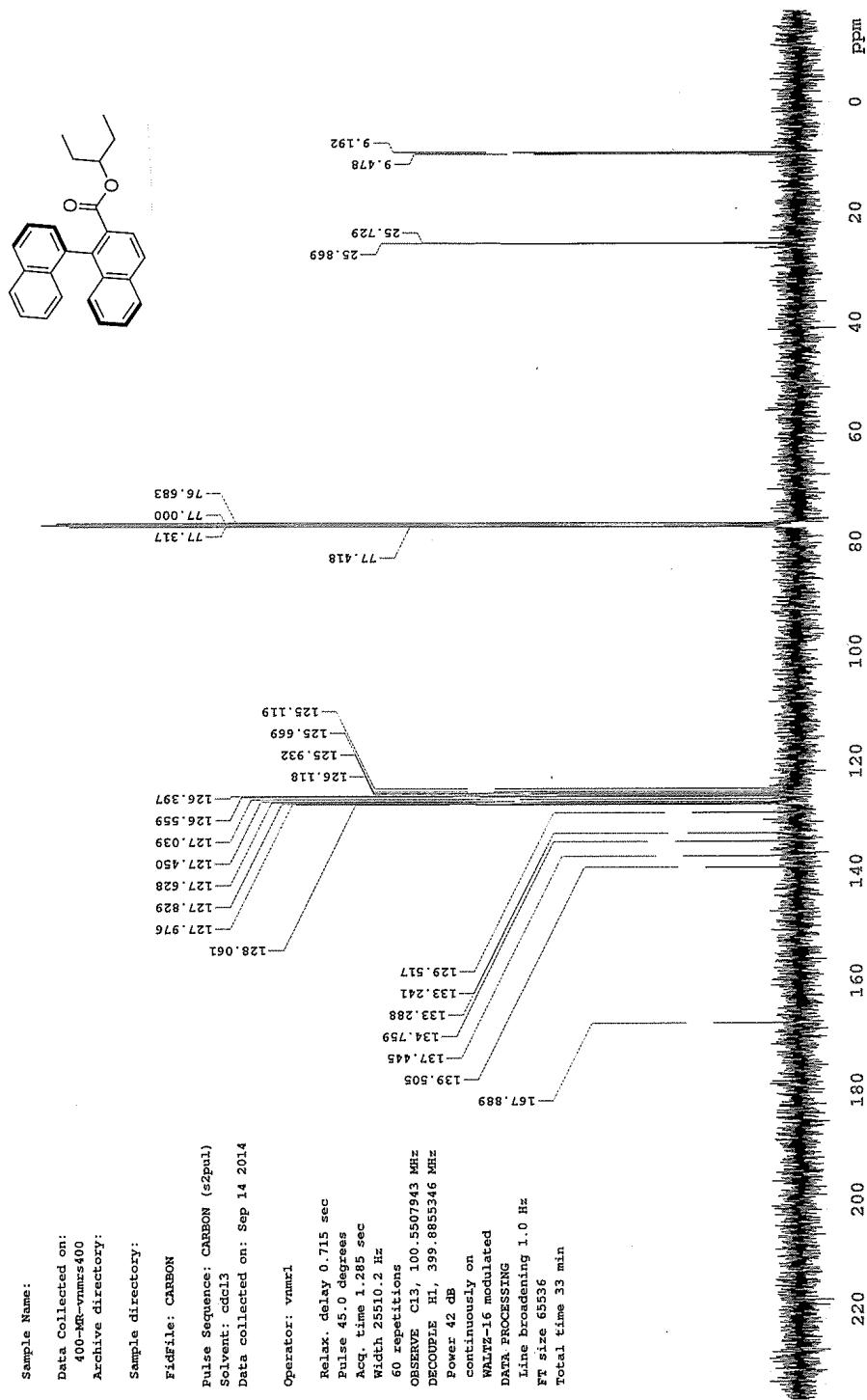
Pulse Sequence: CARBON (s2p1)

Solvent: cdc13

Data collected on: Sep 14 2014

Operator: vnmri

Relax. delay 0.715 sec  
Pulse 45.0 degrees  
Acq. time 1.205 sec  
Width 25510.2 Hz  
60 repetitions  
OBSERVE C13, 100.5507943 MHz  
DECODED H1, 399.8855346 MHz  
Power 42 dB  
continuously on.  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 65536  
Total time 33 min



<sup>13</sup>C NMR of compound 3Ca

STANDARD PROTON PARAMETERS

Sample Name: Data Collected on:  
400-MR-vnmrs400  
Archive directory:

<sup>1</sup>H NMR of compound **3Da**

## STANDARD CARBON PARAMETERS

Sample Name:

Data Collected on:  
400-MR-vnmrs400

Archive directory:

Sample directory:

FidFile: CARBON (62pul)

Pulse Sequence: CARBON (62pul)

Solvent: cdcl3  
Data collected on: Sep 14 2014

Operator: vnmrl

Relax. delay 0.715 sec  
Pulse 45.0 degreesAcq. time 1.285 sec  
Width 25510.2 Hz

96 repetitions

OBSERVE C13, 100.5507949 MHz  
DECOPPLER H1, 399.8855346 MHz

Power 42 dB

continuously on

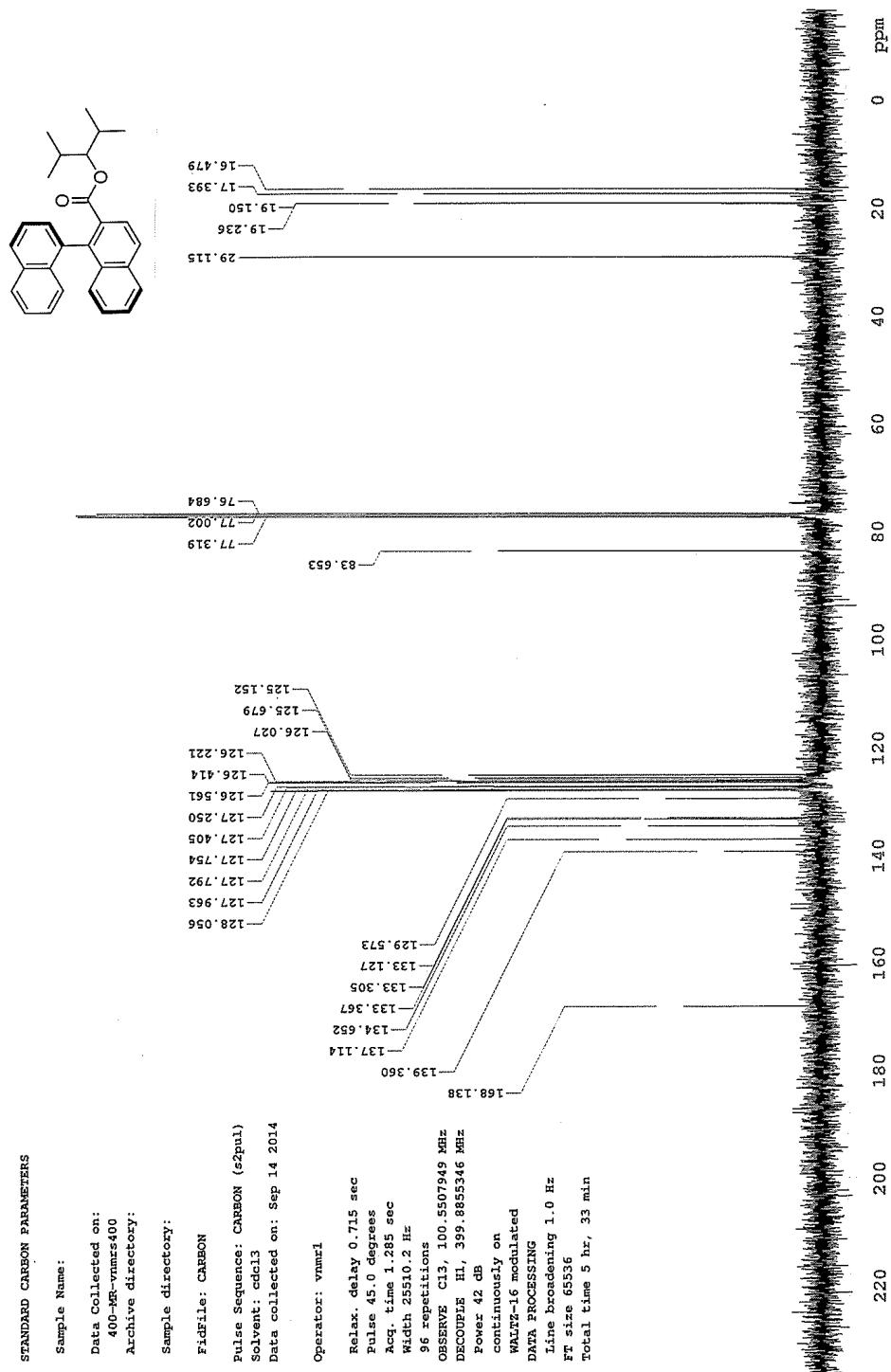
WALTZ-16 modulated

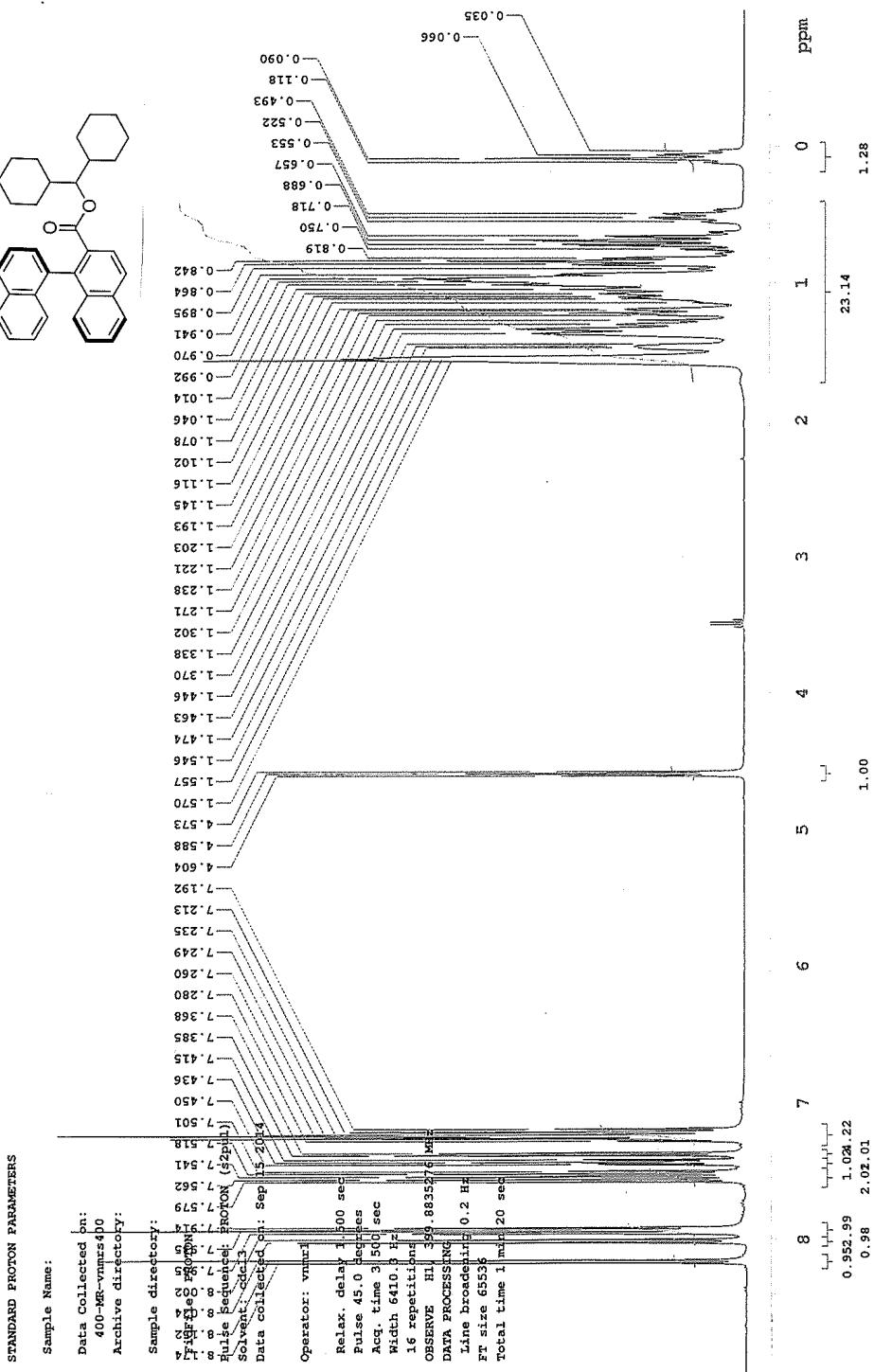
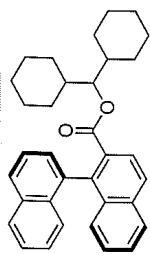
DATA PROCESSING

Line broadening 1.0 Hz

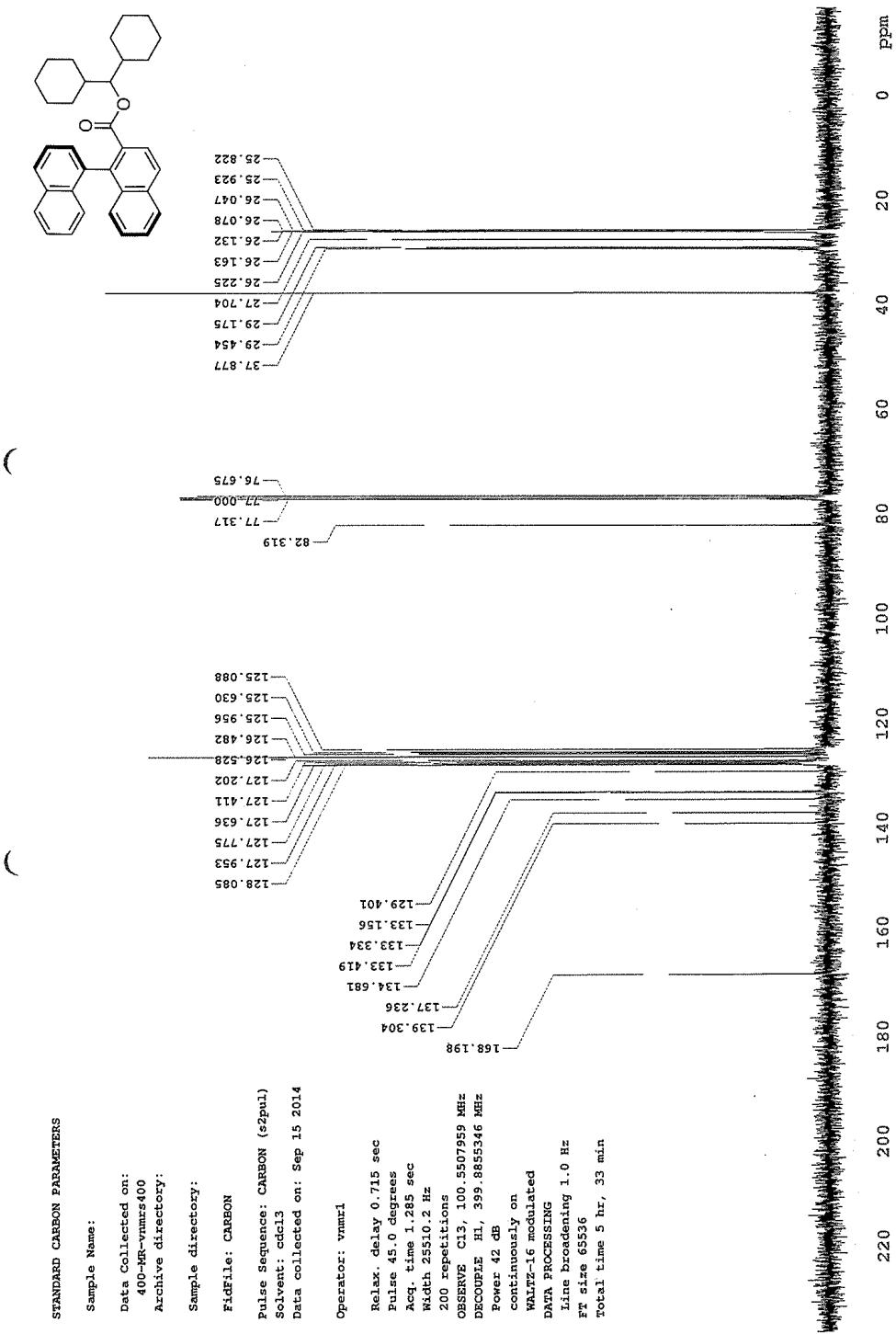
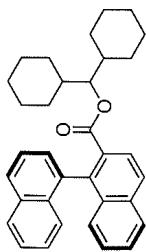
FT size 65536

Total time 5 hr, 33 min

<sup>13</sup>C NMR of compound 3Da



### <sup>1</sup>H NMR of compound 3Ea



### <sup>13</sup>C NMR of compound 3Ea



**STANDARD CARBON PARAMETERS**

Sample Name:

Data Collected on:  
400-MR-vnmrs400

Archive directory:

Sample directory:

Fidfile: CARBON

Pulse Sequence: CARBON (5-pul)

Solvent: cdcl<sub>3</sub>

Data collected on: Sep 15 2014

Operator: vnmr1

Relax. delay 0.715 sec

Pulse 45.0 degrees

Acc. time 1.285 sec

Width 25510.2 Hz

96 repetitions

OBSERVE C13, 100.5507959 MHz

DECUPLE H1, 399.8855346 MHz

Power 42 dB

continuously on

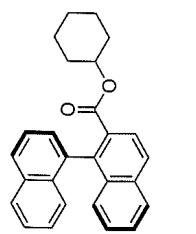
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 5 hr, 33 min



30.708  
77.317  
127.667  
127.969  
127.380  
127.078  
126.552  
126.366  
125.932  
125.677  
125.150  
125.121  
77.000  
76.683  
73.121  
23.391  
23.438  
25.118

220 200 180 160 140 120 100 80 60 40 20 0 ppm

<sup>13</sup>C NMR of compound 3Fa

STANDARD PROTON PARAMETERS

Sample Name:

Data Collected on:  
400-MR-vnmrs400

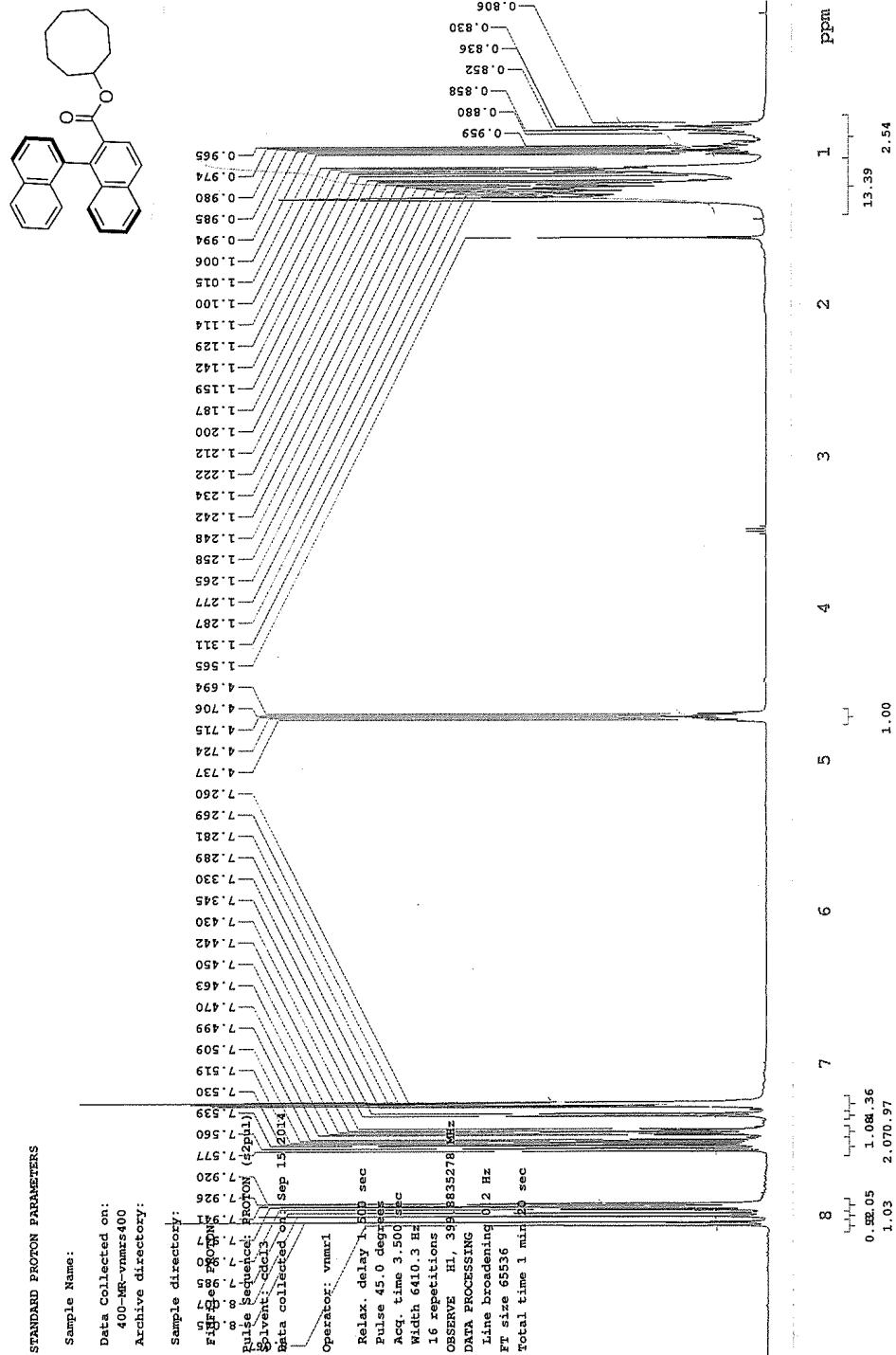
Archive directory:

Sample directory:

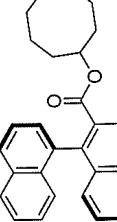
Pulse Sequence: PROTON (4.7 sec)  
Solvent: CDCl<sub>3</sub>  
Data collected on: Sep 15 2014  
Operator: vnmri

Relax. delay 1.500 sec  
Pulse 45.0 degrees  
Acq. time 3.500 sec  
Width 640.3 Hz  
16 repetitions  
OBSERVE H1, 339.8835278 MHz

DATA PROCESSING  
Line broadening 0.2 Hz  
FID size 65536  
Total time 1 min 20 sec



<sup>1</sup>H NMR of compound 3Ga



STANDARD CARBON PARAMETERS

Sample Name:

Data Collected on: 1000-VIS-1000

Archive directory:

1

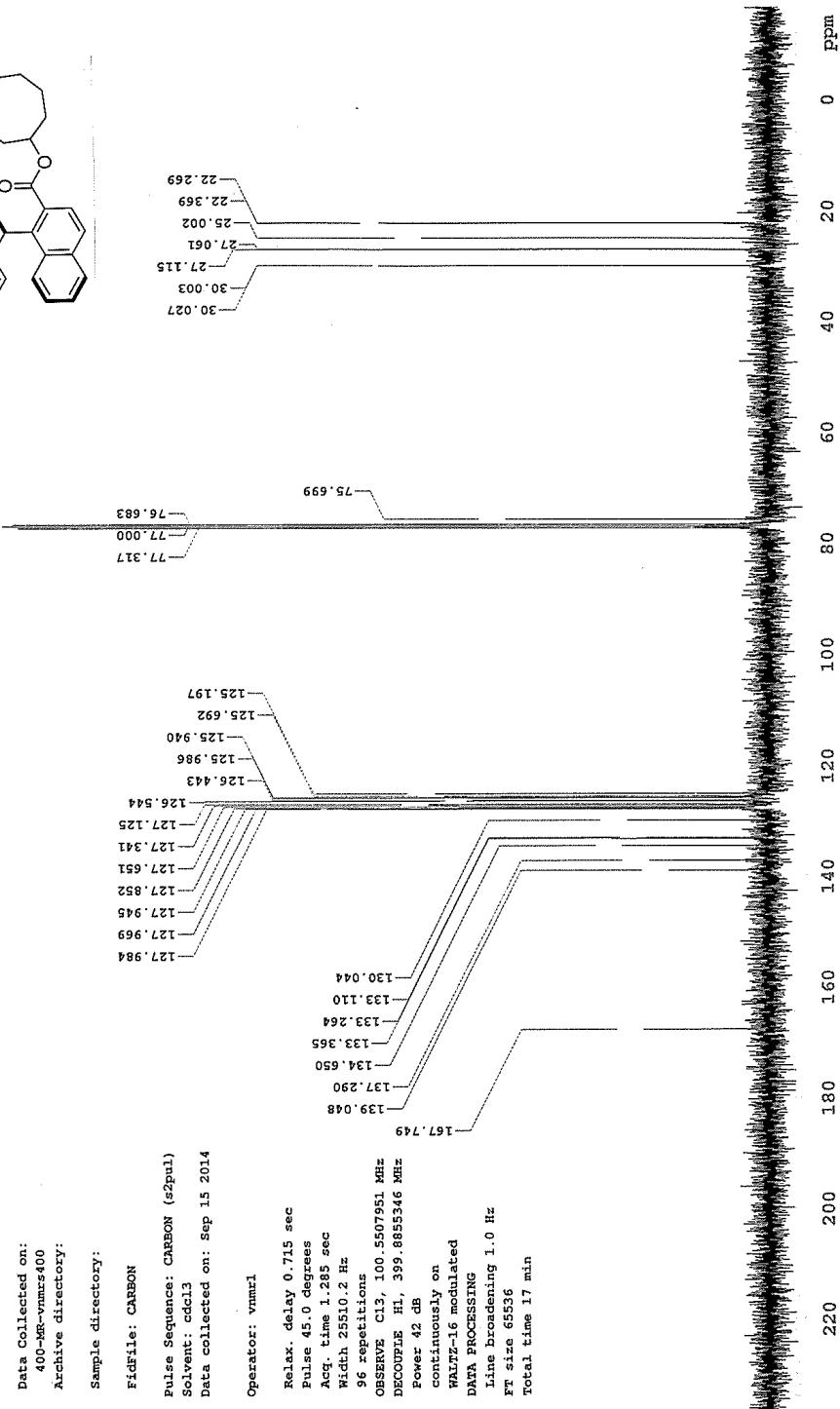
## Pulse Sequence: CARBON (s2p1)

Solvent:  $\text{cdcl}_3$

Data collected on: Sep 15 2014

卷之三

Relax. delay 0.715 sec  
 Pulses 45.0 degrees  
 Acq. time 1.285 sec  
 Width 25.510.2 Hz  
 96 repetitions  
 OBSERVE C13, 100.5507951 MHz  
 DECOUPLE H1, 359.88553346 MHz  
 Power -42 dB  
 continuously on  
 WAIT=16 modulated  
 DATA PROCESSING  
 Line broadening 1.0 Hz  
 FT size 56336  
 Total time 17 min



### <sup>13</sup>C NMR of compound 3Ga

STANDARD PROTON PARAMETERS

Sample Name:

Data Collected on:  
400-MR-vnmrs400

Archive directory:

Sample directory:

FidFile: PROTON

Pulse Sequence: PROTON (sPul)

Solvent: cdcl<sub>3</sub>

Data collected on: Sep 16 2014

Operator: vnmrs1

Relax. delay 1.500 sec

Pulse 45.0 degrees

Acc. time 3.500 sec

Width 6610.3 Hz

16 repetitions

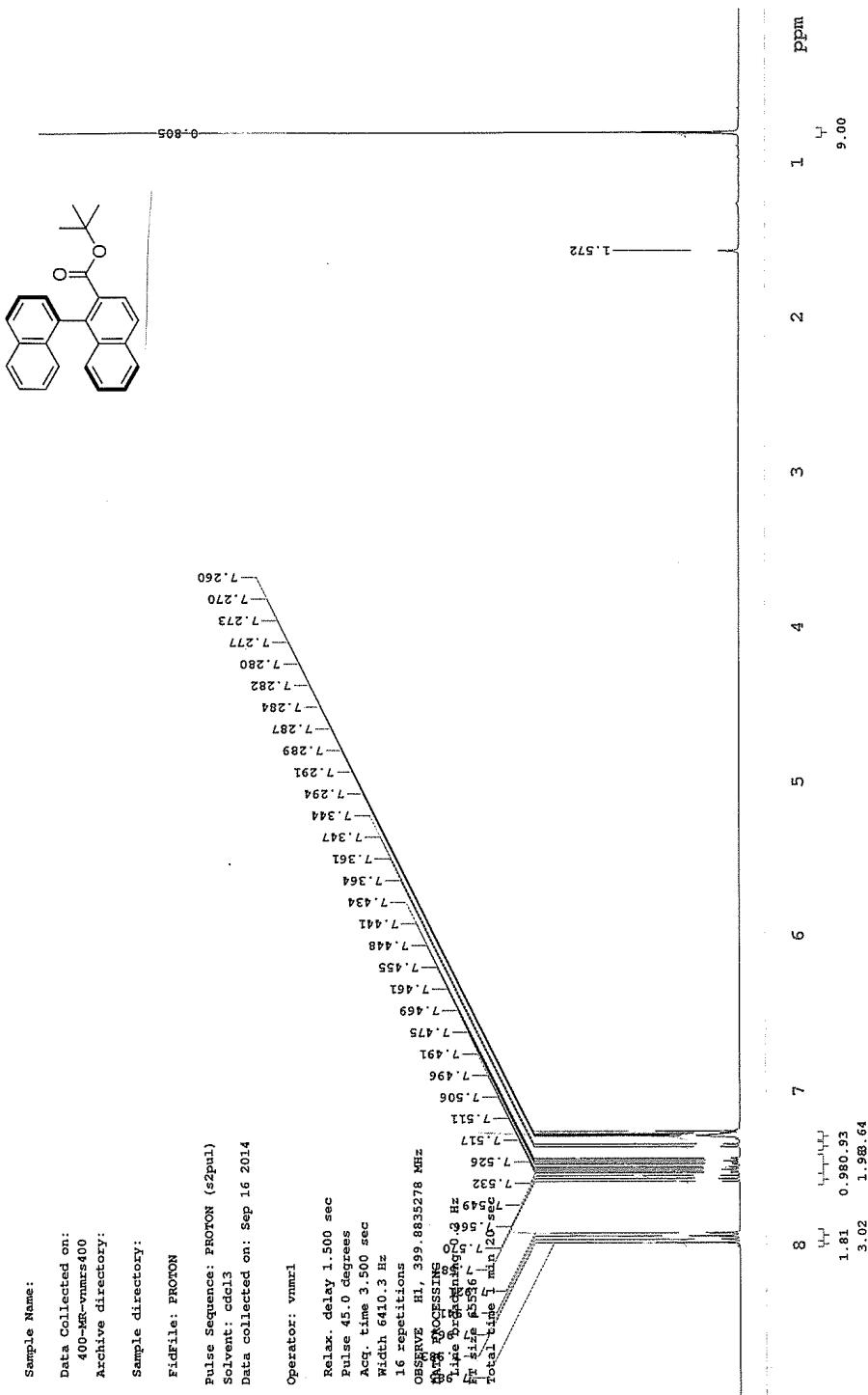
OBSERVE: H1, 399.8835278 MHz

DATA PROCESSING: 32

Line Brg: 653.6 Hz

Line Siz: 653.6 Hz

Total time: 7 min 12.0 sec



<sup>1</sup>H NMR of compound 3Ha

## STANDARD CARBON PARAMETERS

Sample Name:

Data Collected on:  
400-MR-mmrs400

Archive directory:

Sample directory:

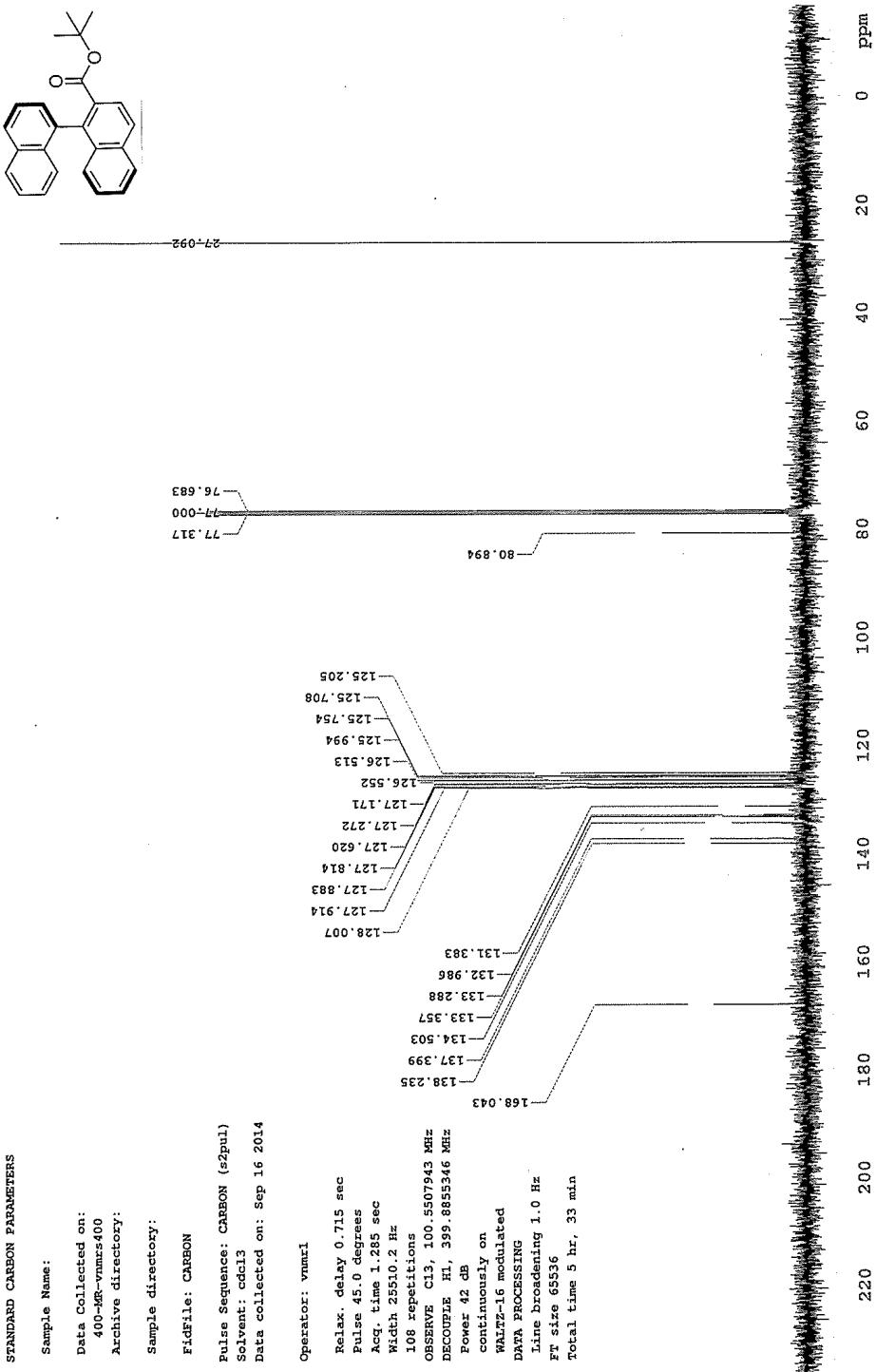
FidFile: CARBON

Pulse Sequence: CARBON (s2pul)

Solvent: ccdl3  
Data collected on: Sep 16 2014

Operator: vnumrl

Relax.: delay 0.715 sec  
 Pulse 45.0 degrees  
 Acq. time 1.285 sec  
 Width 25510.2 Hz  
 108 repetitions  
 OBSERVE C13, 100.5507943 MHz  
 DECOPUE 1H, 399.8855346 MHz  
 Power 42 dB  
 continuously on  
 WALTZ-16 modulated  
 DATA PROCESSING  
 Line broadening 1.0 Hz  
 FT size 65536  
 Total time 5 hr, 33 min

<sup>13</sup>C NMR of compound 3Ha

## STANDARD PROTON PARAMETERS

Sample Name:

Data Collected on:  
400-MR-vnms400

Archive directory:

Sample directory:

Julie Bedard

Spectrometer:

Solvent:

Data collected on:

Operator: vms1

Relax. delay 1.500 sec

Pulse 90.0 deg deg

Aq. time 3.500 sec

Width 6410.3 Hz

16 repetitions

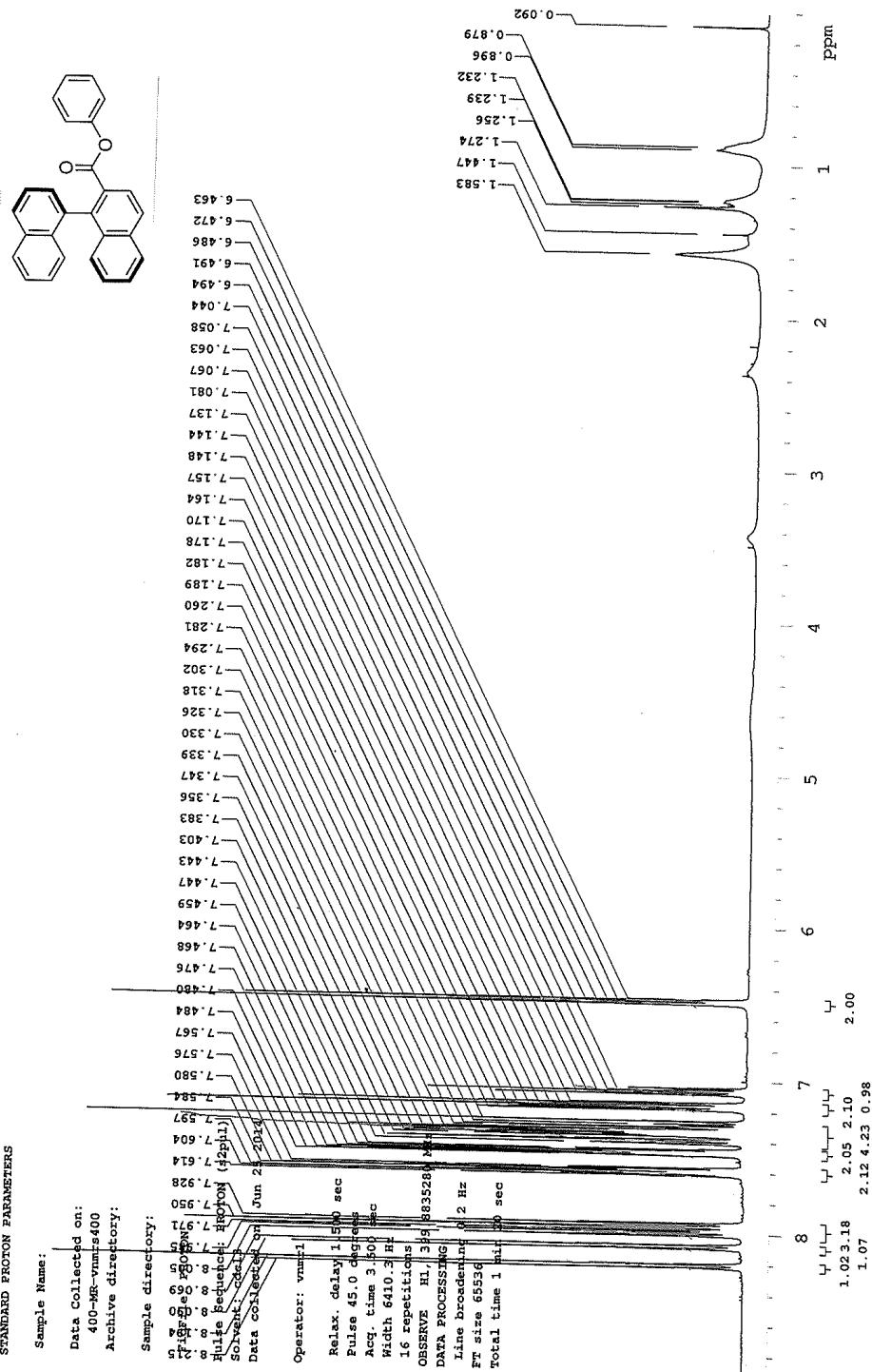
OBSERVE H1

DATA PROCESSING

Line broadening 0.2 Hz

FT size 65536

Total time 1 min 30 sec

<sup>1</sup>H NMR of compound 3Ia



STANDARD PROTON PARAMETERS

Sample Name: LiKOS\_FineQ

Data Collected on:

400-MR-vnmrs400  
Archive directory:

Sample directory:

FidFile: PROTON

Data Collected on: Jun 26 2014

Operator: vnmrl

Pulse Sequence: PROTON

Solvent: cdd13

Relax. delay 1.500 sec

Pulse 45.0 Degrees

Acq. time 3.500 sec

Width 6410.3 Hz

16 repetitions

OBSERVE H1, 399.833528 Hz

DATA PROCESSING

Time 0.00 sec

Time 0.2 sec

Time 0.4 sec

Time 0.6 sec

Time 0.8 sec

Time 1.0 sec

Time 1.2 sec

Time 1.4 sec

Time 1.6 sec

Time 1.8 sec

Time 2.0 sec

Time 2.2 sec

Time 2.4 sec

Time 2.6 sec

Time 2.8 sec

Time 3.0 sec

Time 3.2 sec

Time 3.4 sec

Time 3.6 sec

Time 3.8 sec

Time 4.0 sec

Time 4.2 sec

Time 4.4 sec

Time 4.6 sec

Time 4.8 sec

Time 5.0 sec

Time 5.2 sec

Time 5.4 sec

Time 5.6 sec

Time 5.8 sec

Time 6.0 sec

Time 6.2 sec

Time 6.4 sec

Time 6.6 sec

Time 6.8 sec

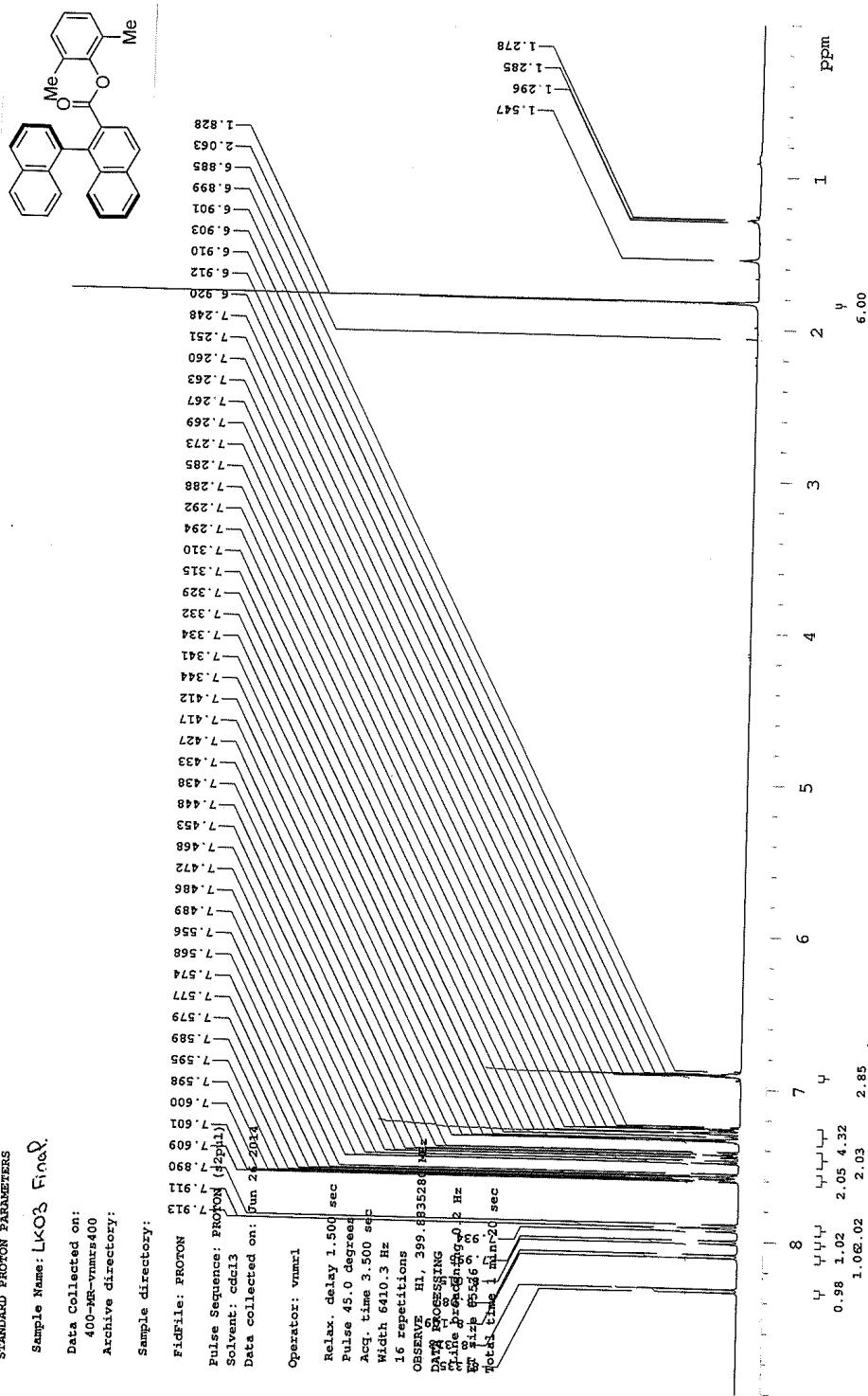
Time 7.0 sec

Time 7.2 sec

Time 7.4 sec

Time 7.6 sec

Time 7.8 sec



STANDARD CARBON PARAMETERS

Sample Name: Lx03 .C1d0

Data Collected on:  
400-MR-vnmrs400

Archive directory:

Sample directory:

Fidfile: CARBON

Pulse Sequence: CARBON

Solvent: ccd13

Data collected on: Jul 14 2014

Operator: vnmr1

Relax. delay 0.715 sec

Pulse 45.0 deg 90°

Acq. time 1.283 sec

Width 2350.0-1.0 Hz

1000 Apw 16.0 sec

133.00000000000001 Hz

OBSERVE: CH3; 1H; 399.038553346 MHz

DECODE: CH3; 1H; 399.038553346 MHz

POWER: 12 dB

SPININHOUSET: on

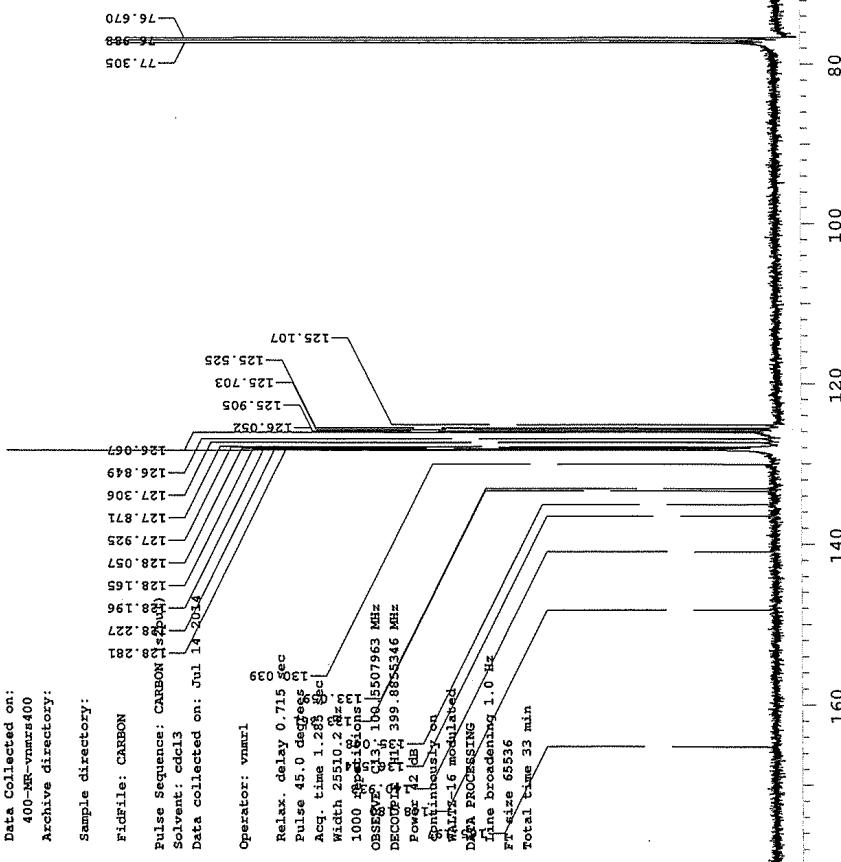
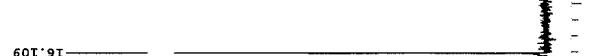
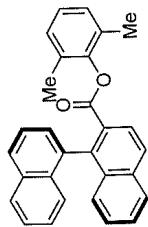
SWRES16: modulated

DIGA PROCESSING

FRIZE: 65536

LINE BROADENING: 1.0 Hz

FRIZE TIME: 33 min



<sup>13</sup>C NMR of compound 3Ja

STANDARD PROTON PARAMETERS

Sample Name: 1KOF-Final.

Data Collected on:  
400-MR-vnmrs400  
Archive directory:

Sample directory:

File#FILE: PROTON

Pulse Sequence: PROTON (#2414)

Solvent: ccdl3

Data collected on: Jul 3 2014

Operator: vnmr1

00

Rebax: 1.00 sec

Pulse: 45°

W1CG: 3.500 sec

Chidt: 6.01 sec

on repetitions

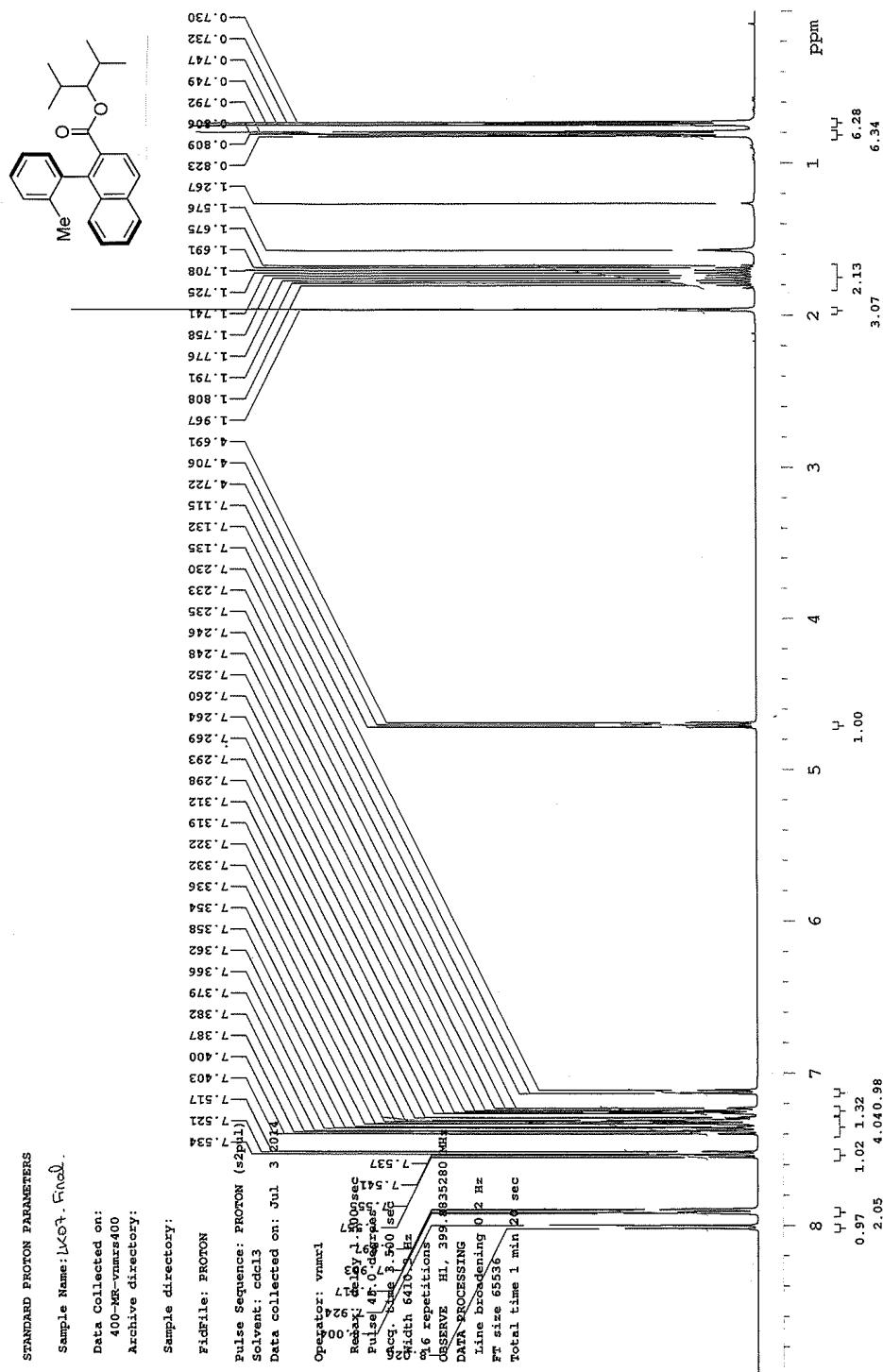
OBSERVE H1, 399.4835280

DATA PROCESSING

Line broadening 0.2 Hz

FT size 65536

Total time 1 min 20 sec



<sup>1</sup>H NMR of compound 3Db

**STANDARD CARBON PARAMETERS**

Sample Name:

Data Collected on:  
400-MR-vnmrs400

Archive directory:

Sample directory:

Operator: vnmrs1

Pulse Sequence: CARBON (sp Pul)

Solvent: cdcl3

Data collected on: Sep 15 2014

Pulse: CARBON

Relax. delay 0.715 sec

Pulse 45.0 degrees

Acc. time 1.285 sec

Width 25510.2 Hz

96 repetitions

OBSERVE Cl3, 100.5507951 MHz

DECOUPLE HI, 399.6895346 MHz

Power 42 dB

continuously on

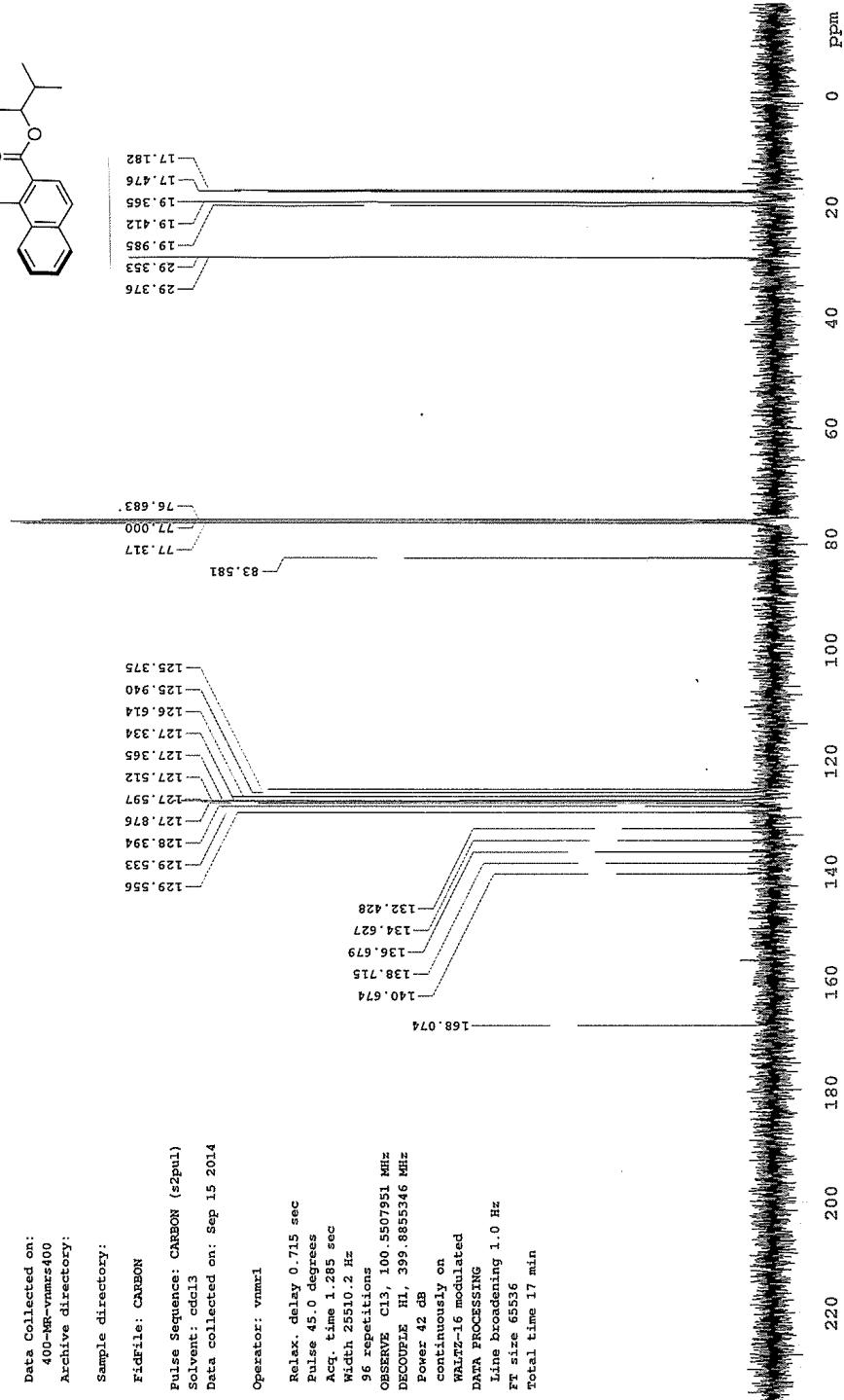
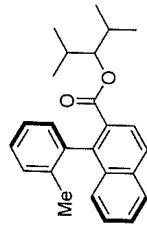
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 17 min



<sup>13</sup>C NMR of compound 3Db

## STANDARD PROTON PARAMETERS

Sample Name: L495 - Fmoc<sup>0</sup>

Data Collected on:

400-MR-varian400

Archive directory:

Sample directory:

Fidfile: PROTON

Pulse Sequence: PROTON (s2pal)

Solvent: cdd13

Date collected on: Jul 25 2011

Pulse: 45.0 sec

dgcg: 0.039 sec

Opfreq: 6.060 Hz

Relax: 1.0 sec

Pulse: 45.0 sec

dgcg: 0.039 sec

Opfreq: 6.060 Hz

Relax: 1.0 sec

Pulse: 45.0 sec

dgcg: 0.039 sec

Opfreq: 6.060 Hz

Relax: 1.0 sec

Pulse: 45.0 sec

dgcg: 0.039 sec

Opfreq: 6.060 Hz

Relax: 1.0 sec

Pulse: 45.0 sec

dgcg: 0.039 sec

Opfreq: 6.060 Hz

Relax: 1.0 sec

Pulse: 45.0 sec

dgcg: 0.039 sec

Opfreq: 6.060 Hz

Relax: 1.0 sec

Pulse: 45.0 sec

dgcg: 0.039 sec

Opfreq: 6.060 Hz

Relax: 1.0 sec

Pulse: 45.0 sec

dgcg: 0.039 sec

Opfreq: 6.060 Hz

Relax: 1.0 sec

Pulse: 45.0 sec

dgcg: 0.039 sec

Opfreq: 6.060 Hz

Relax: 1.0 sec

Pulse: 45.0 sec

dgcg: 0.039 sec

Opfreq: 6.060 Hz

Relax: 1.0 sec

Pulse: 45.0 sec

dgcg: 0.039 sec

Opfreq: 6.060 Hz

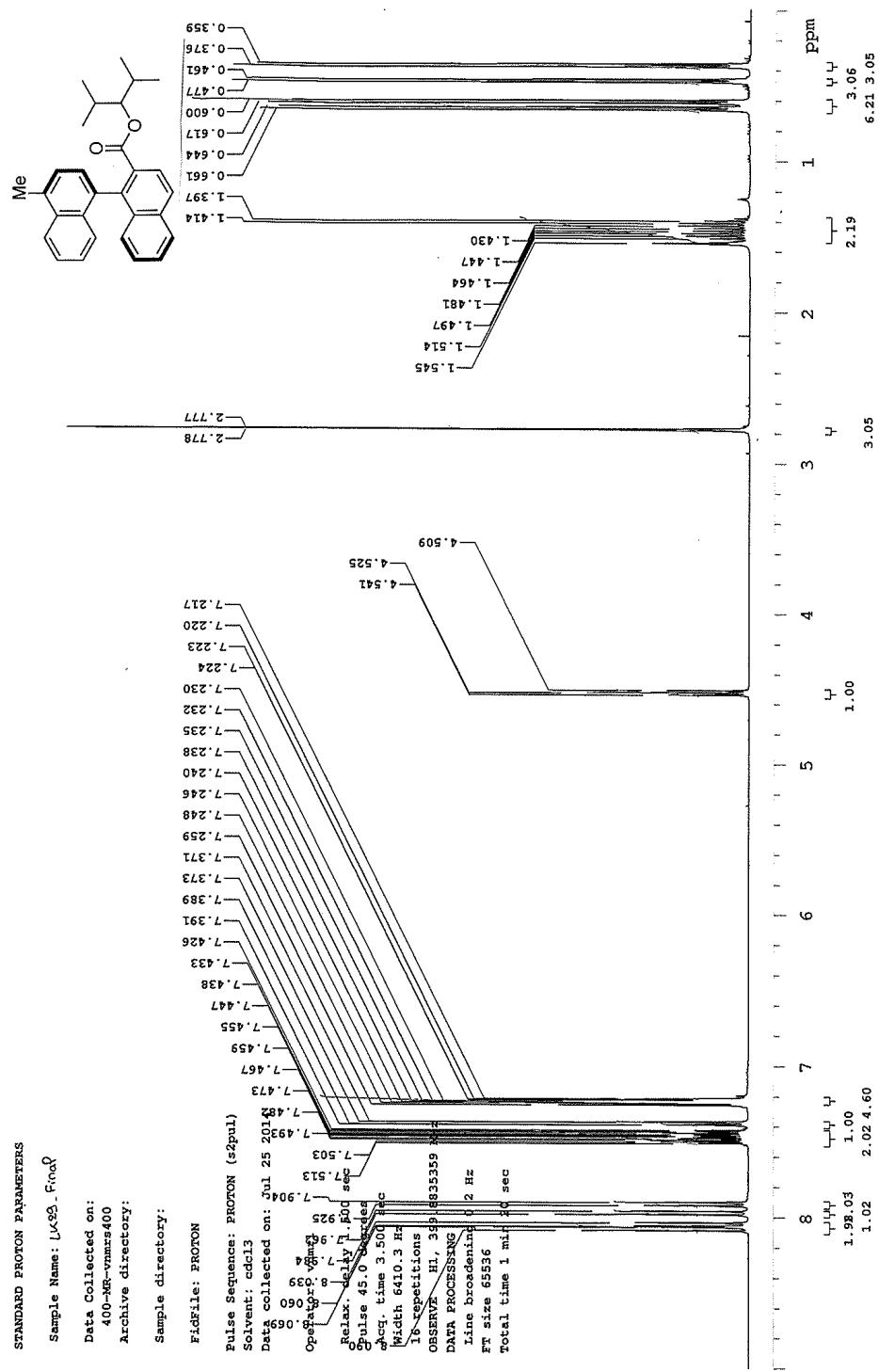
Relax: 1.0 sec

Pulse: 45.0 sec

dgcg: 0.039 sec

Opfreq: 6.060 Hz

Relax: 1.0 sec

<sup>1</sup>H NMR of compound 3Dc

STANDARD CARBON PARAMETERS

Sample Name: Lx3D

Data Collected on:  
400-MR-mars400

Archive directory:

Sample directory:

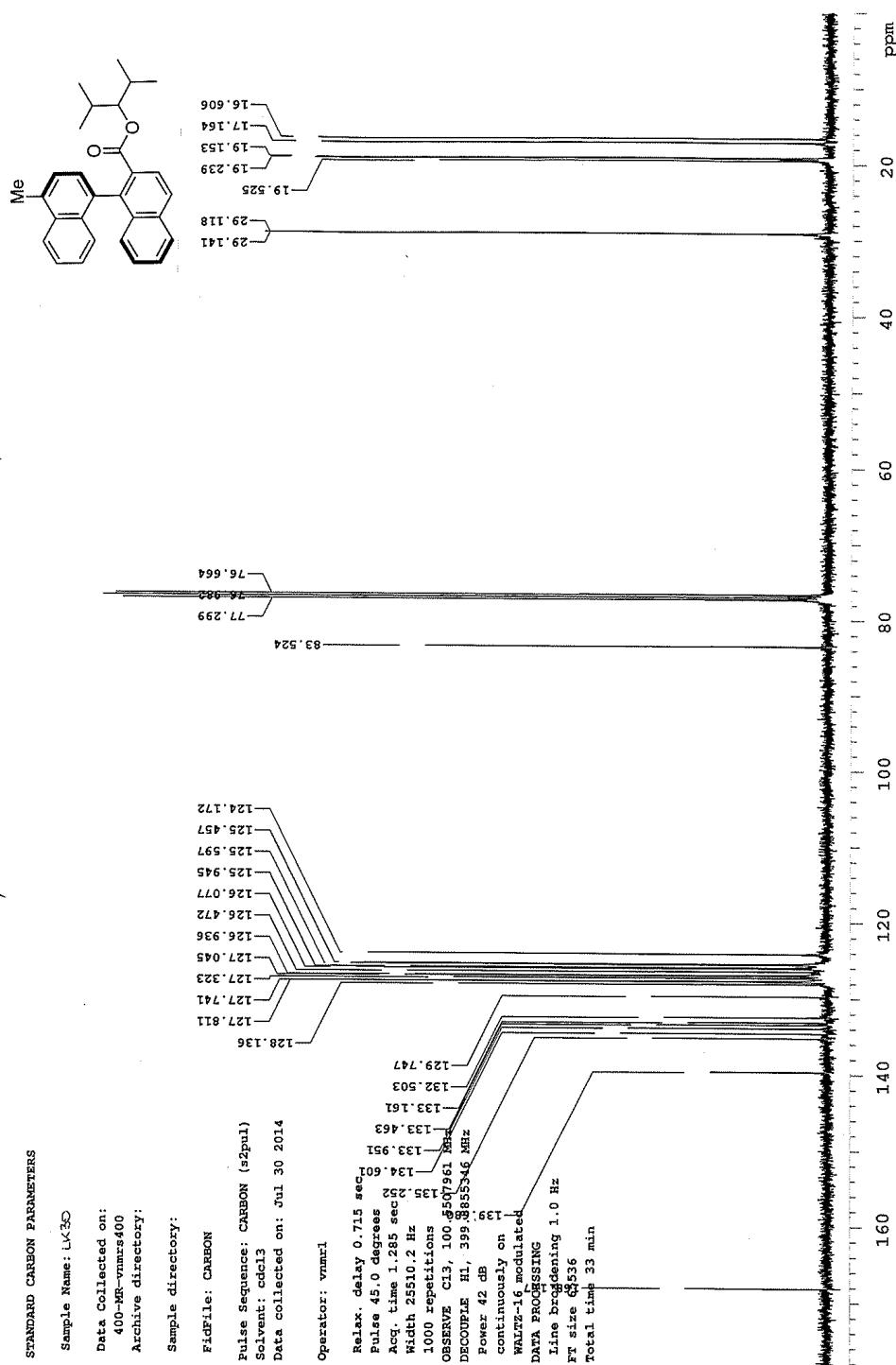
FidFile: CARBON

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl<sub>3</sub>  
Data collected on: Jul 30 2014

Operator: vmarl

Relax. delay 0.715 sec  
Pulse 45.0 degrees  
Acc. time 1.285 sec  
Width 25310.2 Hz  
1000 repetitions  
OBSERVE C13, 100.5507761  
DECODE H1, 299.9553461  
Power 42 dB  
continuously on  
WAL22-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 6536  
Total Time 33 min



<sup>13</sup>C NMR of compound 3Dc

STANDARD PROTON PARAMETERS

Sample Name:

Data Collected on:  
400-MR-vnmrs100

Archive directory:

Sample directory:

FIDFILE: PROTON

Pulse Sequence: PROTON (sep14)

Solvent: cdcl<sub>3</sub>

Data collected on: Sep 15 2014

Operator: vnmr1

Relax: delay 1.500 sec  
Pulse 45.0 degrees  
Acc. time 3.500 sec  
Width 6410.3 Hz  
OBSERVE 20 sec, 1399.883278 MHz  
DATA PROCESSING 20 sec, 1399.883278 MHz  
Line Broadening 2 Hz  
EW size 65536  
No. of FIDs 1 min 20 sec

16 repetitions

4.21

4.24

4.38

4.41

4.46

4.507

4.535

4.551

4.566

4.597

4.917

5.162

5.233

5.263

5.27

5.292

5.294

5.400

5.403

5.417

5.421

5.424

5.438

5.441

5.491

5.496

5.507

5.512

5.517

5.523

5.527

5.535

5.551

5.566

5.597

6.917

6.987

7.162

7.183

7.236

7.249

7.253

7.257

7.260

7.263

7.267

7.272

7.287

7.292

7.294

7.400

7.403

7.417

7.421

7.424

7.438

7.441

7.491

7.496

7.507

7.512

7.517

7.523

7.527

7.535

7.551

7.566

7.597

7.697

7.897

7.917

7.937

7.957

7.977

7.997

8.017

8.037

8.057

8.077

8.097

8.117

8.137

8.157

8.177

8.197

8.217

8.237

8.257

8.277

8.297

8.317

8.337

8.357

8.377

8.397

8.417

8.437

8.457

8.477

8.497

8.517

8.537

8.557

8.577

8.597

8.617

8.637

8.657

8.677

8.697

8.717

8.737

8.757

8.777

8.797

8.817

8.837

8.857

8.877

8.897

8.917

8.937

8.957

8.977

8.997

9.017

9.037

9.057

9.077

9.097

9.117

9.137

9.157

9.177

9.197

9.217

9.237

9.257

9.277

9.297

9.317

9.337

9.357

9.377

9.397

9.417

9.437

9.457

9.477

9.497

9.517

9.537

9.557

9.577

9.597

9.617

9.637

9.657

9.677

9.697

9.717

9.737

9.757

9.777

9.797

9.817

9.837

9.857

9.877

9.897

9.917

9.937

9.957

9.977

9.997

10.017

10.037

10.057

10.077

10.097

10.117

10.137

10.157

10.177

10.197

10.217

10.237

10.257

10.277

10.297

10.317

10.337

10.357

10.377

10.397

10.417

10.437

10.457

10.477

10.497

10.517

10.537

10.557

10.577

10.597

10.617

10.637

10.657

10.677

10.697

10.717

10.737

10.757

10.777

10.797

10.817

10.837

10.857

10.877

10.897

10.917

10.937

10.957

10.977

10.997

11.017

11.037

11.057

11.077

11.097

11.117

11.137

11.157

11.177

11.197

11.217

11.237

11.257

11.277

11.297

11.317

11.337

11.357

11.377

11.397

11.417

11.437

11.457

11.477

11.497

11.517

11.537

11.557

11.577

11.597

11.617

11.637

11.657

STANDARD CARBON PARAMETERS

Sample Name:

Data Collected on:  
400-MR-vnmrs400

Archive directory:

Sample directory:

FidFile: CARBON

Pulse Sequence: CARBON (sepul)

Solvent: cdcl<sub>3</sub>

Data collected on: Sep 15 2014

Operator: vnmrl

Relax. delay 0.715 sec

Pulse 45.0 degrees

Acc. time 1.285 sec

Width 2550.0 Hz

128 repetitions

OBSERVE C13, 100.5507943 MHz  
DECOUPLE H1, 399.8855346 MHz

Power 42 dB

continuously on

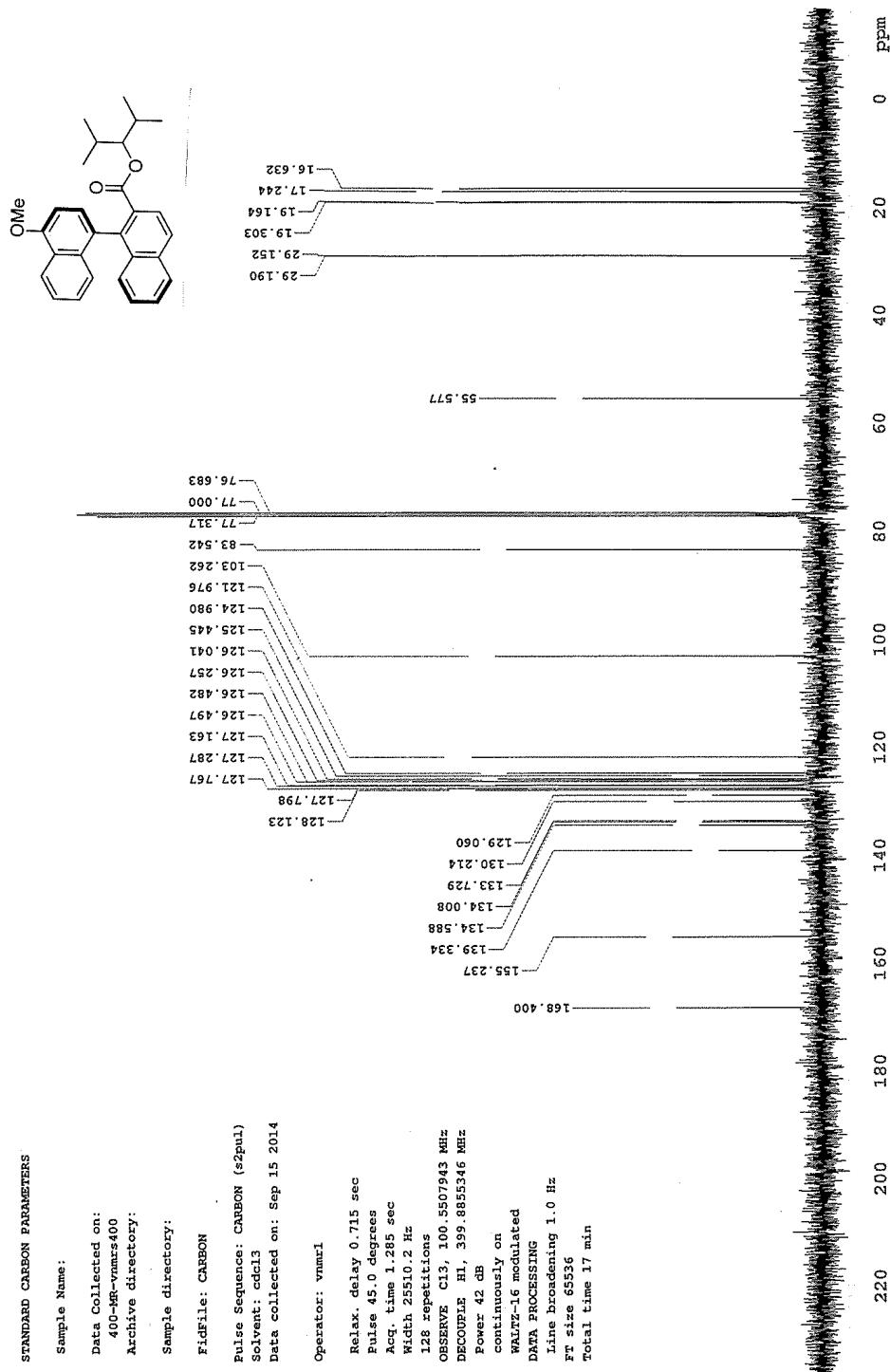
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 17 min



<sup>13</sup>C NMR of compound 3Dd

STANDARD PROTON PARAMETERS

Sample Name:

Data Collected on:  
400-MR-vnmrs400

Archive directory:

Sample directory:

Fidfile: PROTON

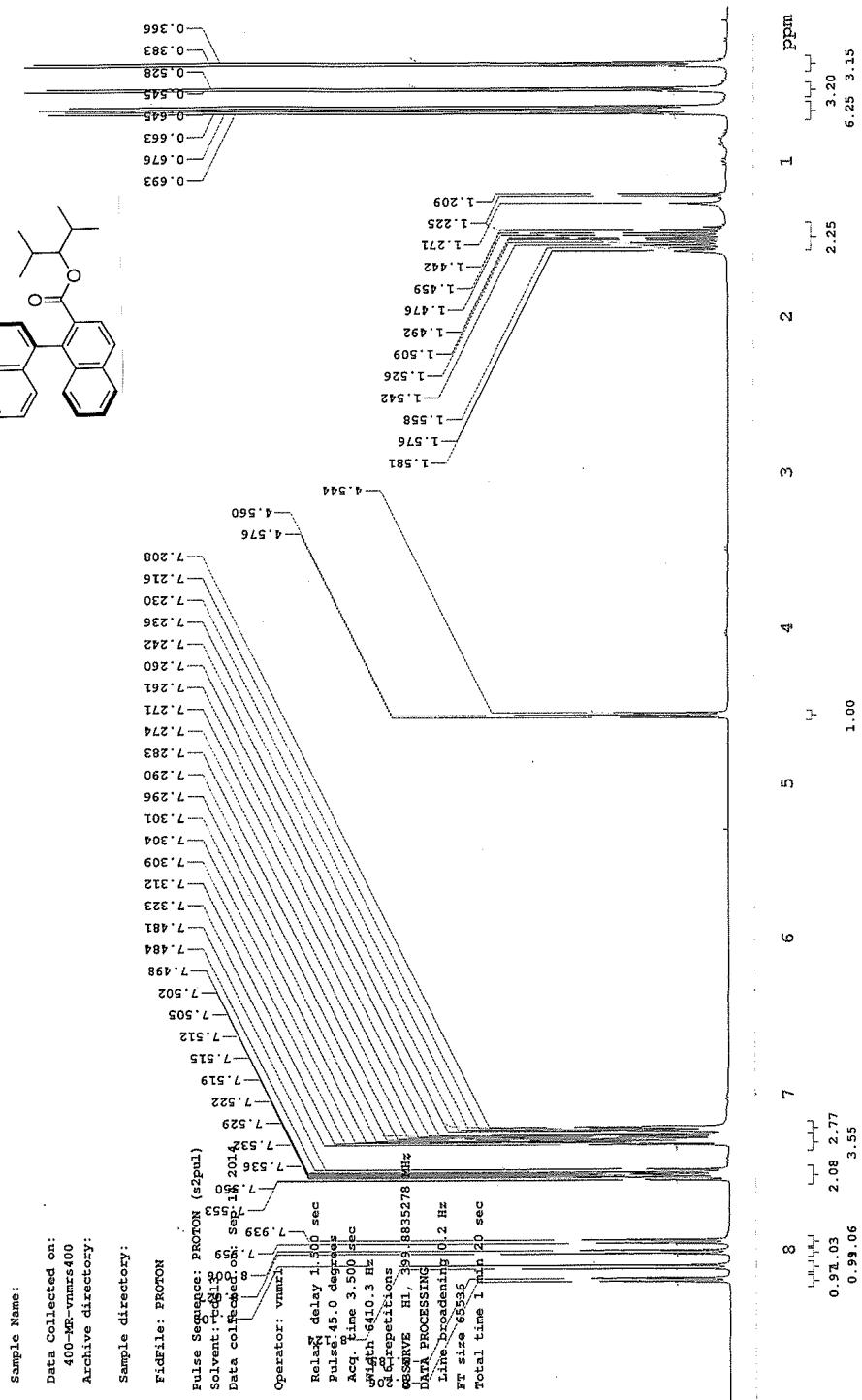
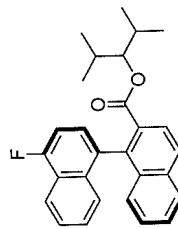
Pulse Sequence: PROTON (spdpul)

Solvent: <sup>1</sup>H-OH

Data collected on: 05/09/2014

Operator: vnmrs

Relax delay 1.500 sec  
Pulse<sup>1</sup> 45.0 degrees  
Acc. time 3.500 sec  
Width 610.3 Hz  
QF 2000000  
Aquisitions 1000000  
Observe H1 399.9835278 Hz  
DATA PROCESSING  
Line broadening 0.2 Hz  
FT size 65536  
Total time 1 min 20 sec



STANDARD CARBON PARAMETERS

Sample Name:

Data Collected on:  
400-MR-vnrmrs400

Archive directory:

Sample directory:

Fidfile: CARBON

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl<sub>3</sub>

Data collected on: Sep 14 2014

Operator: vnmr1

Relax. delay 0.715 sec

Pulse 45.0 degrees

Acr. time 1.385 sec

Width 25510.2 Hz

1.44 repetitions

OBSERVE C13, 100.5507951 MHz

DECORREL H1, 399.8855346 MHz

Power 42 dB

continuously on

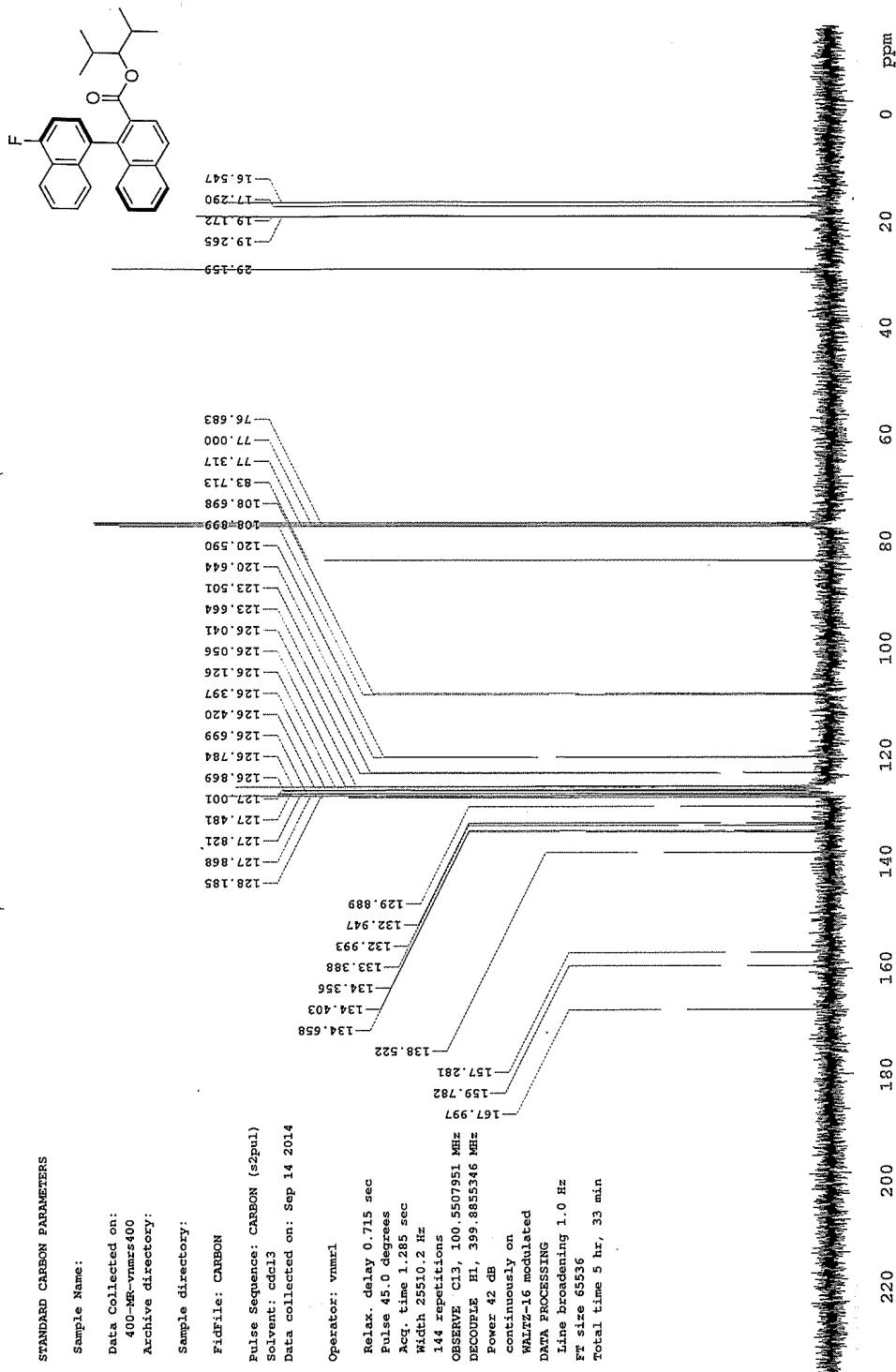
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 5 hr, 33 min



### STANDARD PROTON PARAMETERS

```

Sample Name: UX32-FWV.
Data Collected on: 1998-08-15 15:43:43
400-MHz-Vnmr400
Archive directory: UX32-FWV

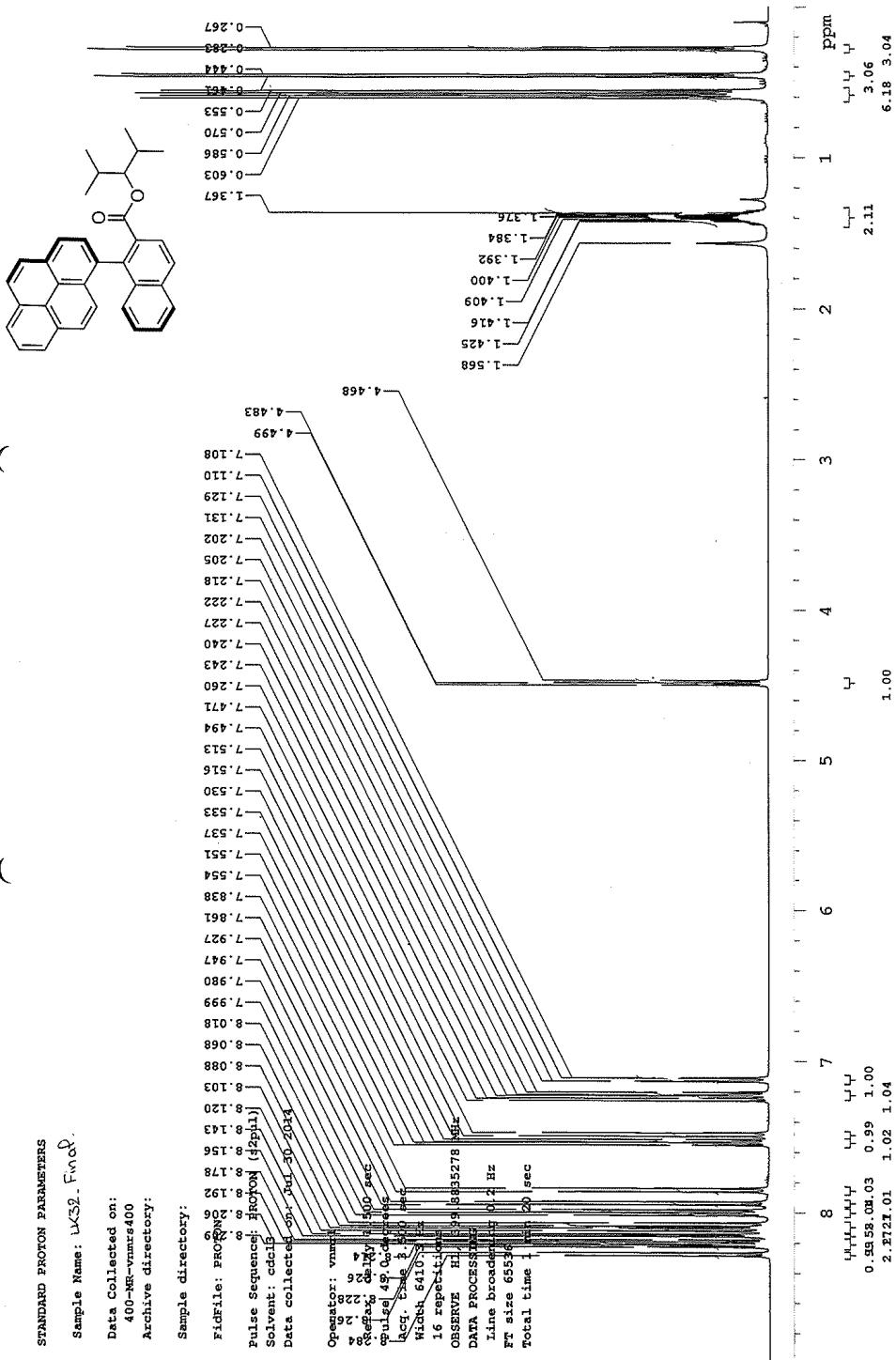
Sample directory: UX32-FWV
Fidfile: UX32-FWV.fid
Pulse Sequence: cdc135
Solvent: dcd135
Data collected on: 1998-08-15 15:43:43

```

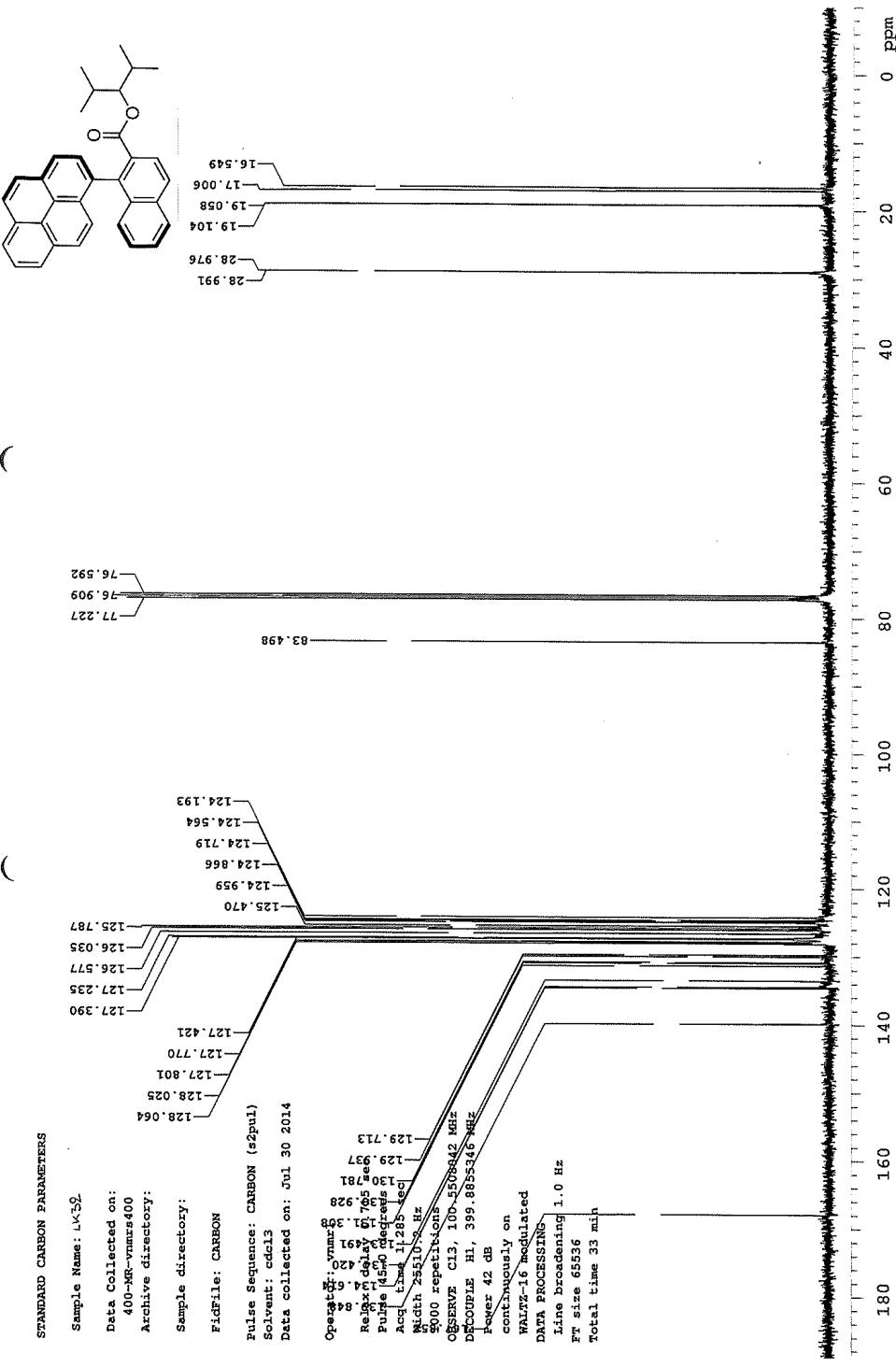
```

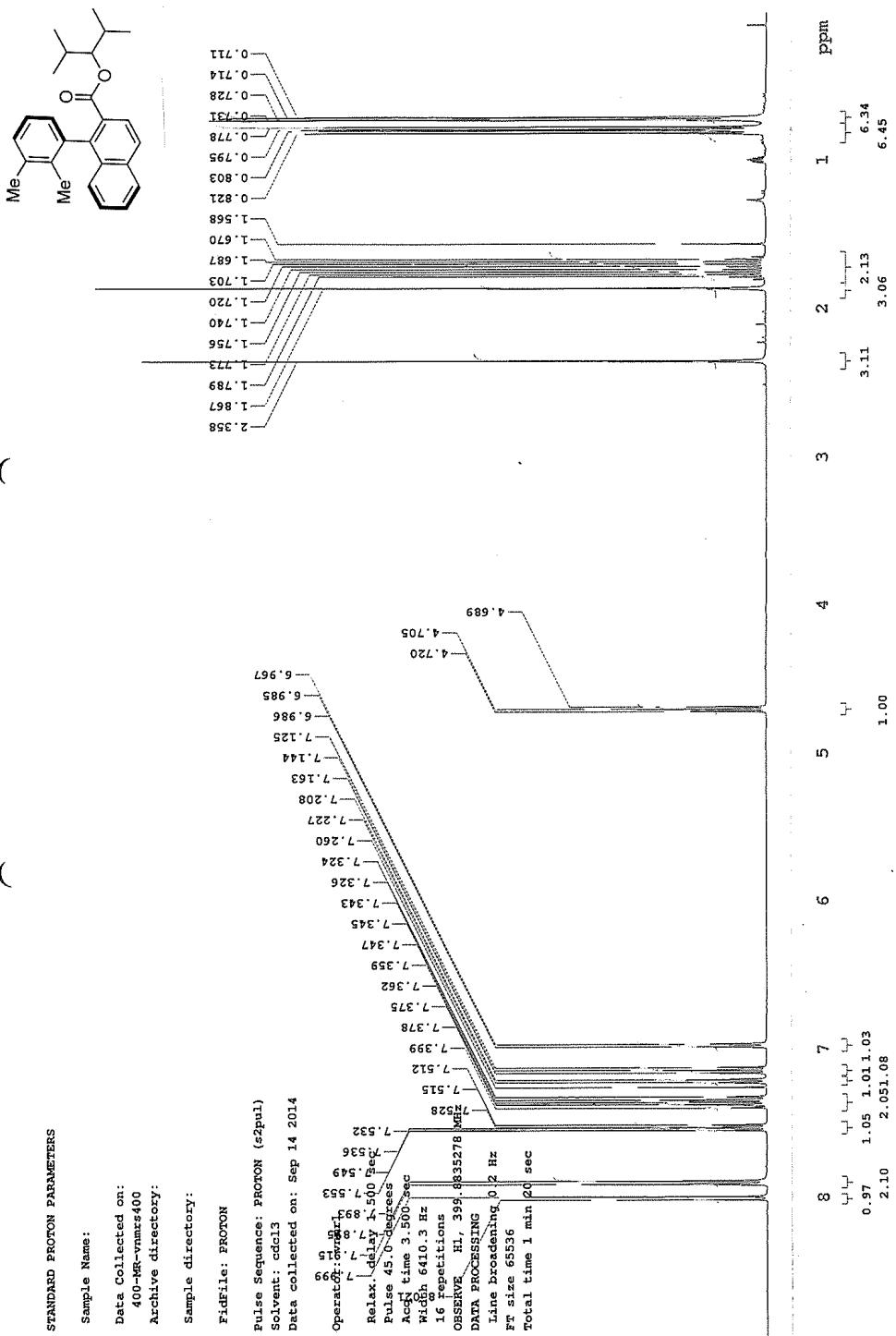
Operator: vnm
Run ID: 9
Run Type: 2
Run Date: 1992-07-22
Run Time: 10:50:00 sec
CPU time: 49.00 sec
Memory usage: 1.00 MB
Log file: log.1
Log file size: 6111 bytes
Number of 16 repetitions: 1
Number of observations: 1
Number of data processing steps: 1
Number of line broadcasts: 1
Line broadcast rate: 0.2 Hz
File size: 65536 bytes
Time taken: 20 sec

```



### <sup>1</sup>H NMR of compound 3Df





STANDARD CARBON PARAMETERS

Sample Name:

Data Collected on:  
400-MR-mmes100

Archive directory:

Sample directory:

Fidfile: CARBON

Pulse Sequences: CARBON (spdpul)

Solvent: cdcl3

Data collected on: Sep 14 2014

Operator: vnmrl

Relax. delay 0.715 sec

Pulse 45.0 degrees

Acq. time 1.285 sec

width 25510.2 Hz

100 repetitions

OBSERVE C13, 100.5507951 MHz

DECODE H1, 399.8855346 MHz

Power 42 dB

continuously on

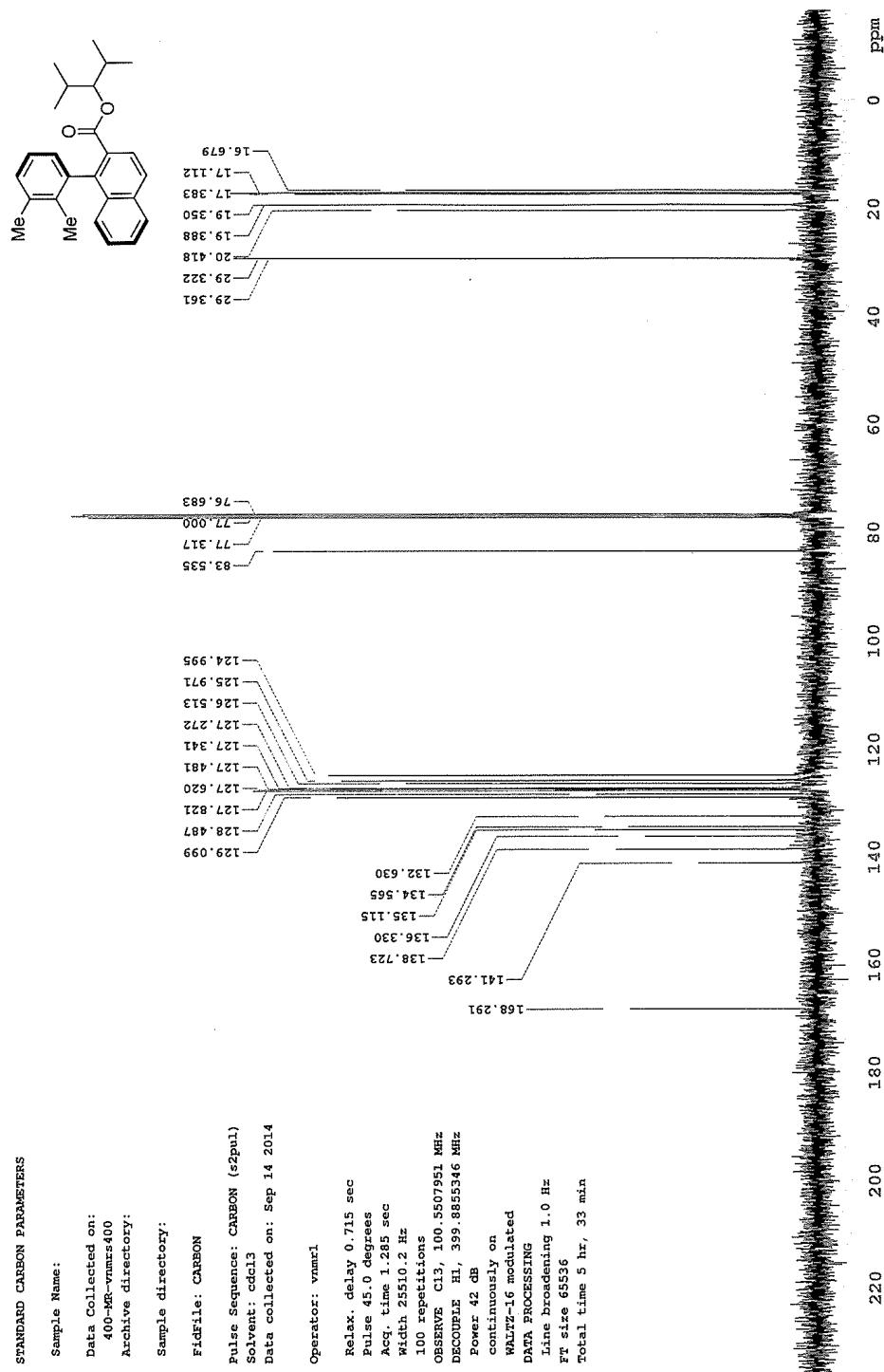
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 5 hr, 33 min



<sup>13</sup>C NMR of compound 3Dg

**STANDARD PROTON PARAMETERS**

Sample Name:

Data Collected on:  
400-MR-mars400

Archive directory:

Sample directory:

FidFile: PROTON

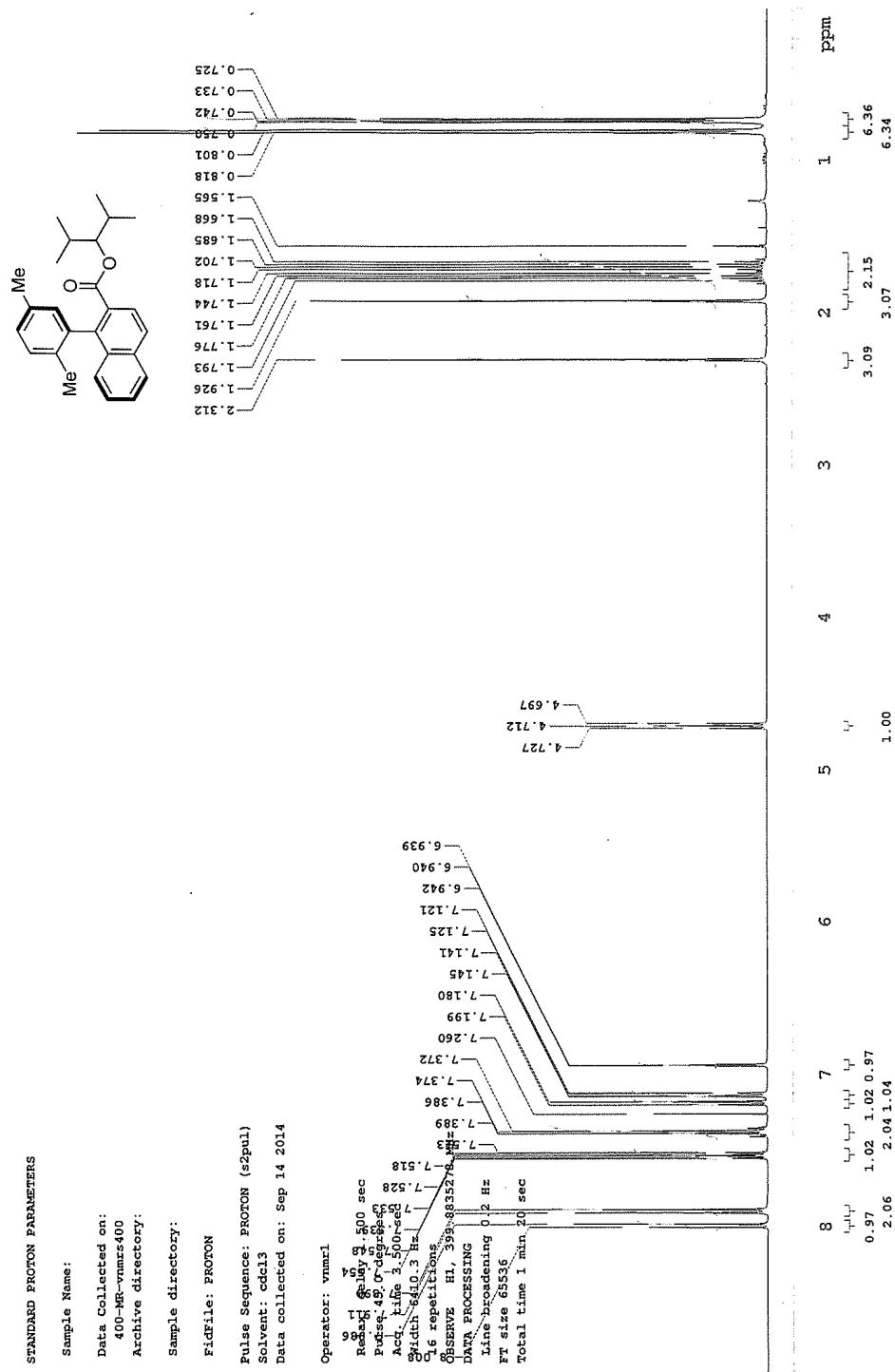
Pulse Sequence: PROTON (sp-pul)

Solvent: cdcl3

Data collected on: Sep 14 2014

Operator: vnmrl

9.5  
8.5  
7.5  
6.5  
5.5  
4.5  
3.5  
2.5  
1.5  
0.5  
0.0 sec  
Read 1 sec  
Gated 5 sec  
Pulse 45°, 0°, 90°, 180°  
Pulse 45°, 0°, 90°, 180°  
Acc. time 3.500 sec  
Grdth 6410.3 Hz  
1.6 repetitions  
QSPRVE H1 3398.888888888888 Hz  
DATA PROCESSING 0.2 Hz  
FT size 65336  
Line broadening 0.2 Hz  
total time 1 min. 20 sec



STANDARD CARBON PARAMETERS

Sample Name:

Data Collected on:  
400-MR-vnmrs400

Archive directory:

Sample directory:

FidFile: CARBON

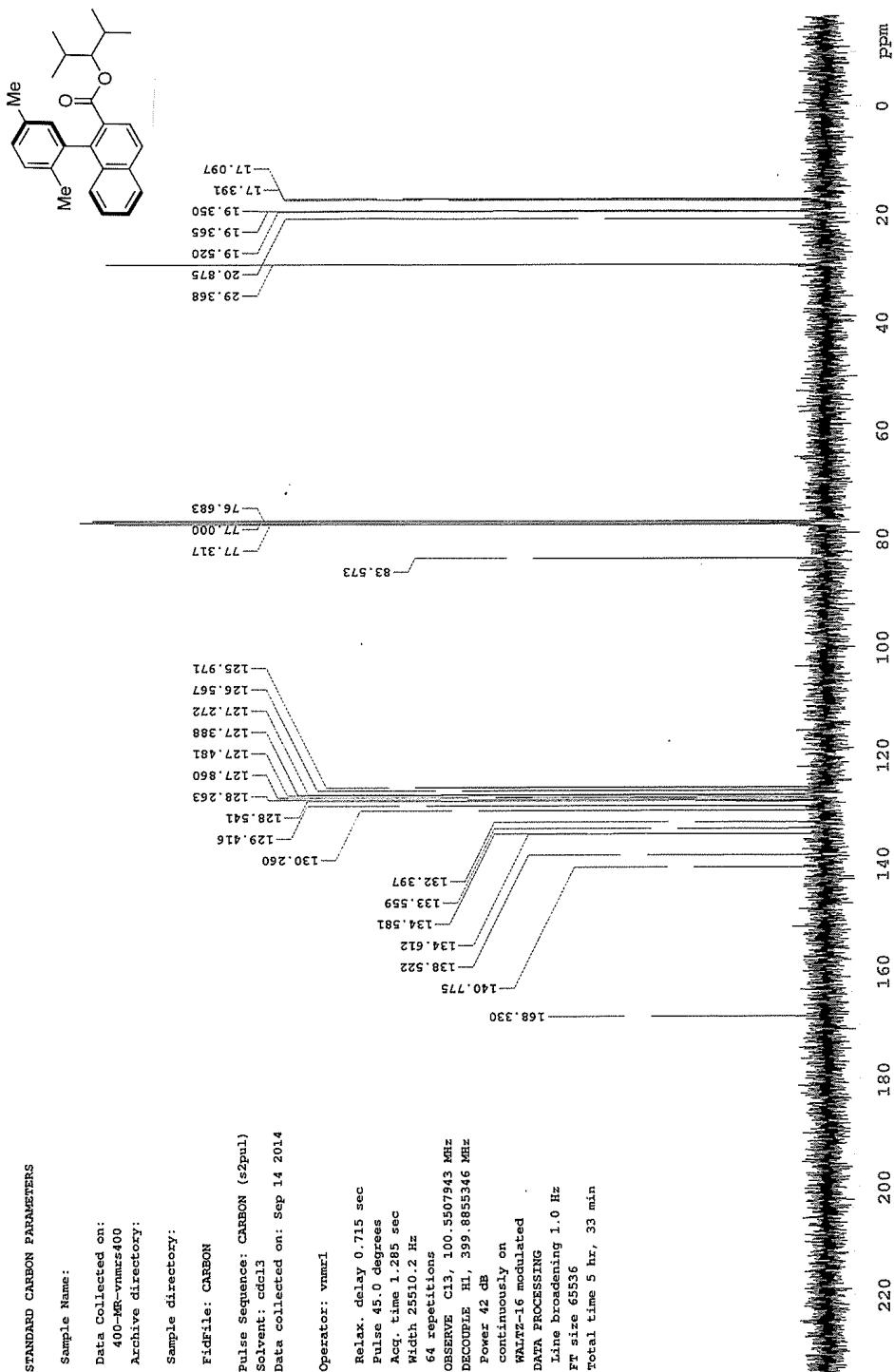
Pulse Sequence: CARBON (s2pul)

Solvent: cdcl<sub>3</sub>

Data collected on: Sep 14 2014

Operator: vnmrs1

Relax. delay 0.715 sec  
 Pulse 45.0 degrees  
 Acc. time 1.285 sec  
 Width 25510.2 Hz  
 64 repetitions  
 OBSERVE C13, 100.5507943 MHz  
 DECOPPLE H1, 399.8855346 MHz  
 Power 42 dB  
 Continuously on  
 WALTZ-16 modulated  
 DATA PROCESSING  
 Line broadening 1.0 Hz  
 FT size 65536  
 Total time 5 hr, 33 min



<sup>13</sup>C NMR of compound 3Dh

STANDARD PROTON PARAMETERS

Sample Name:

Data Collected on:  
400-MR-mmrs400

Archive directory:

Sample directory:

Fidfile: PROTON

Pulse Sequence: PROTON (s2pul)

Solvent: ccd13

Data collected on: Sep 14 2014

0.029

0.024

0.029

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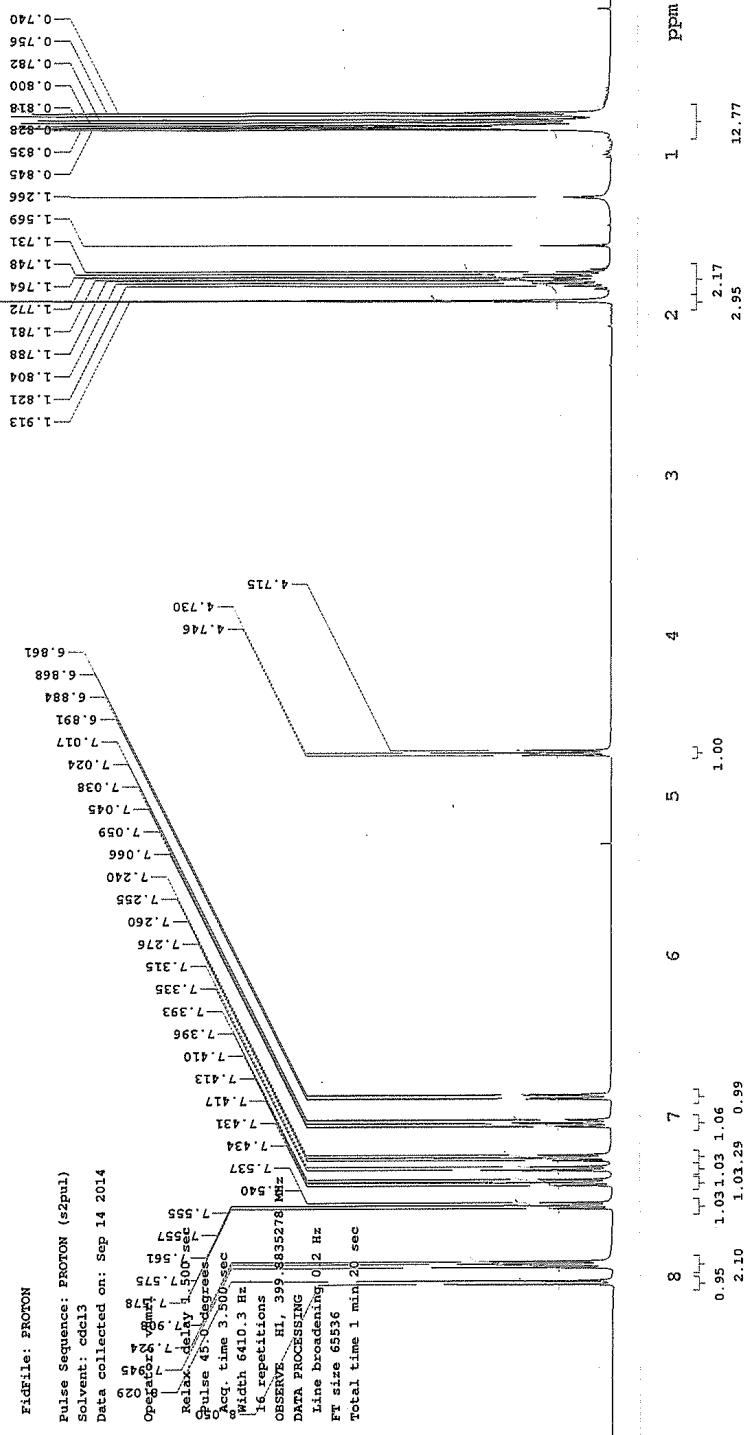
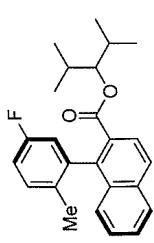
0.024

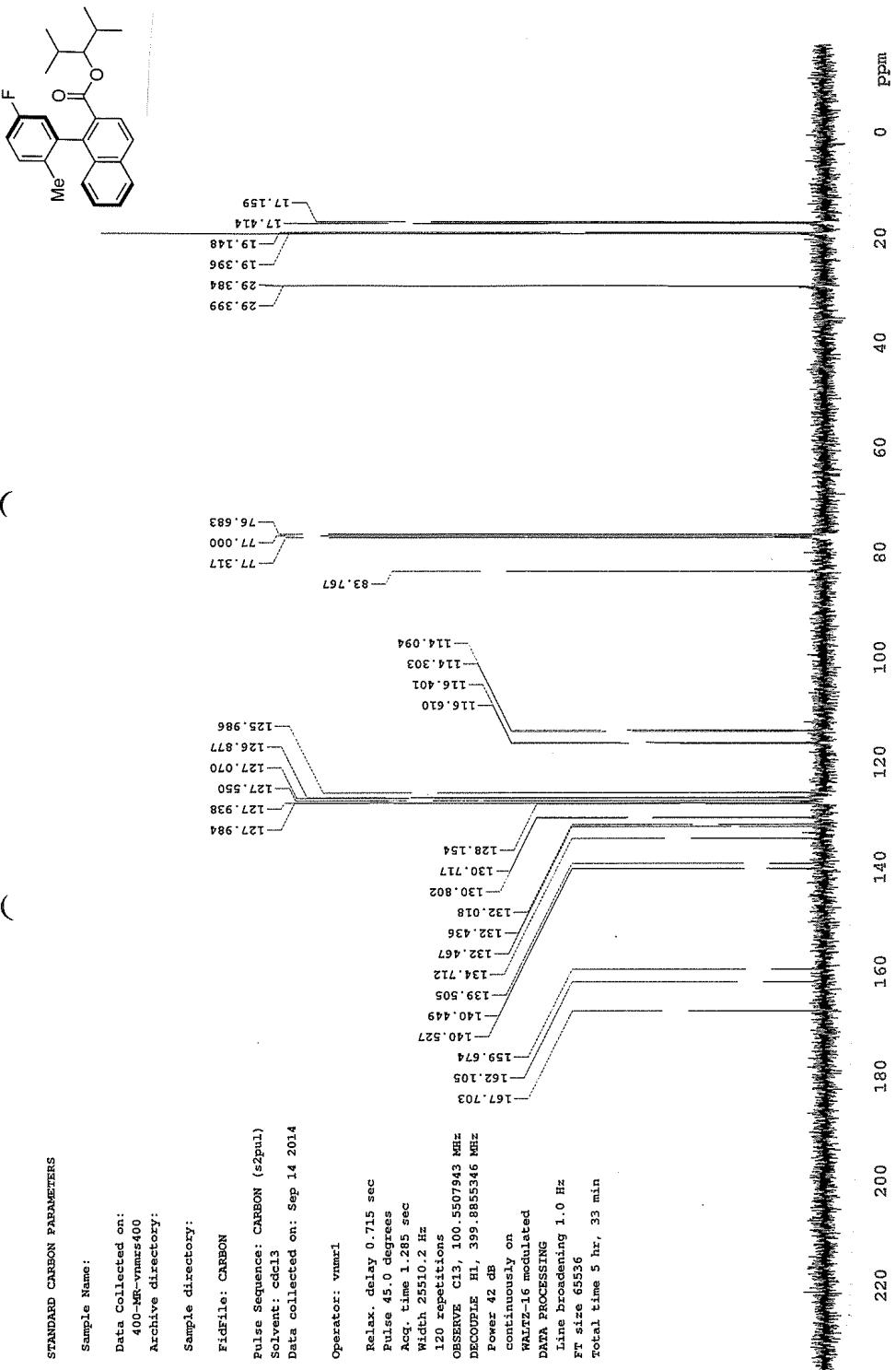
0.029

0.024

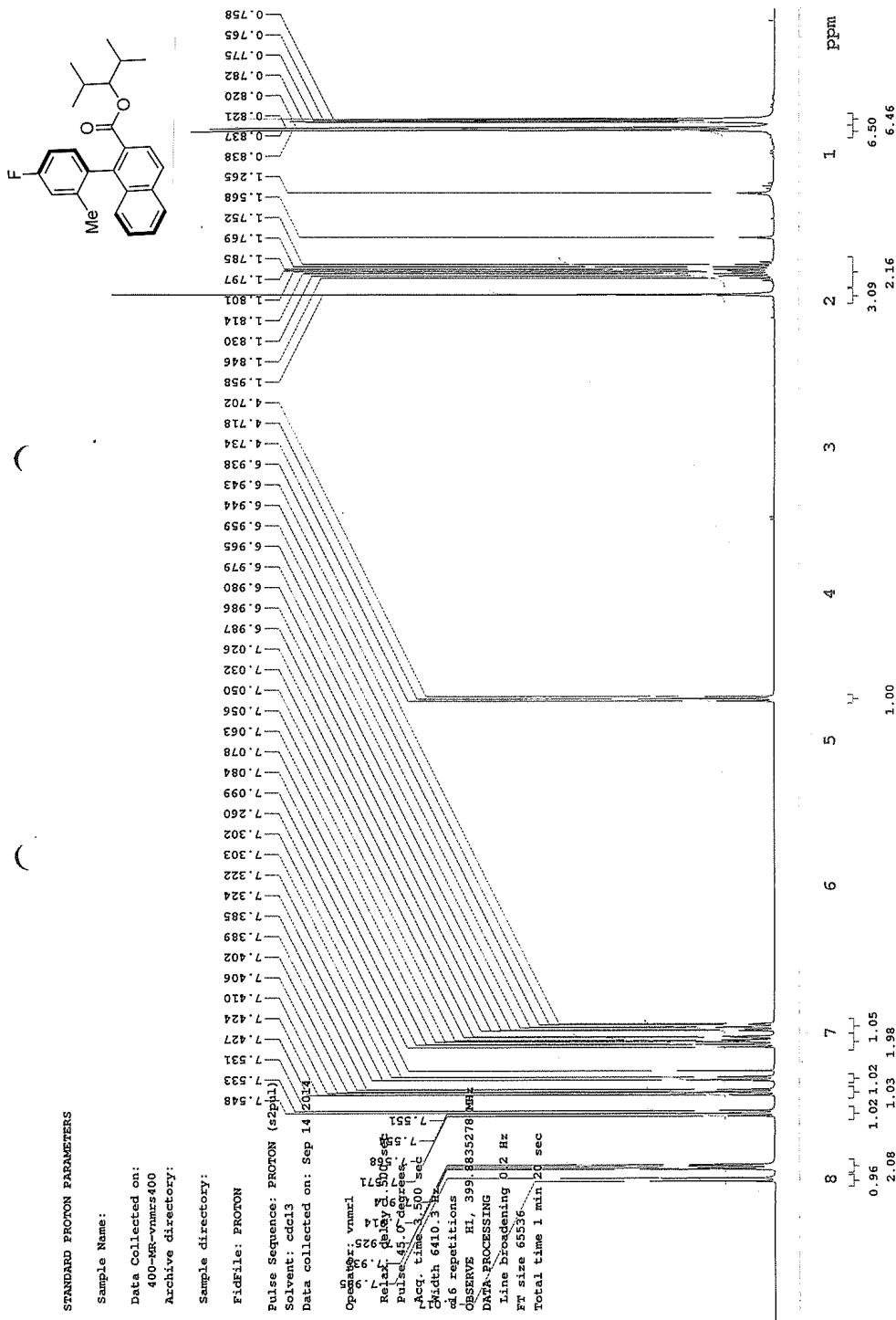
0.029

0.024





<sup>13</sup>C NMR of compound **3Di**



**<sup>1</sup>H NMR of compound 3Dj**

STANDARD CARBON PARAMETERS

Sample Name:

Data Collected on:  
400-MR-vnmr400

Archive directory:

Sample directory:

FidFile: CARBON

Pulse Sequence: CARBON (seqpl)

Solvent: cdcl<sub>3</sub>

Data collected on: Sep 14 2014

Operator: vnmrl

Relax. delay 0.715 sec

Pulse 95.0 degrees

Acq. time 1.285 sec

Width 25510.2 Hz

120 repetitions

OBSERVE CL3, 100.5507943 MHz

DECOUPLE HL, 399.8855346 MHz

Power 42 dB

continuously on

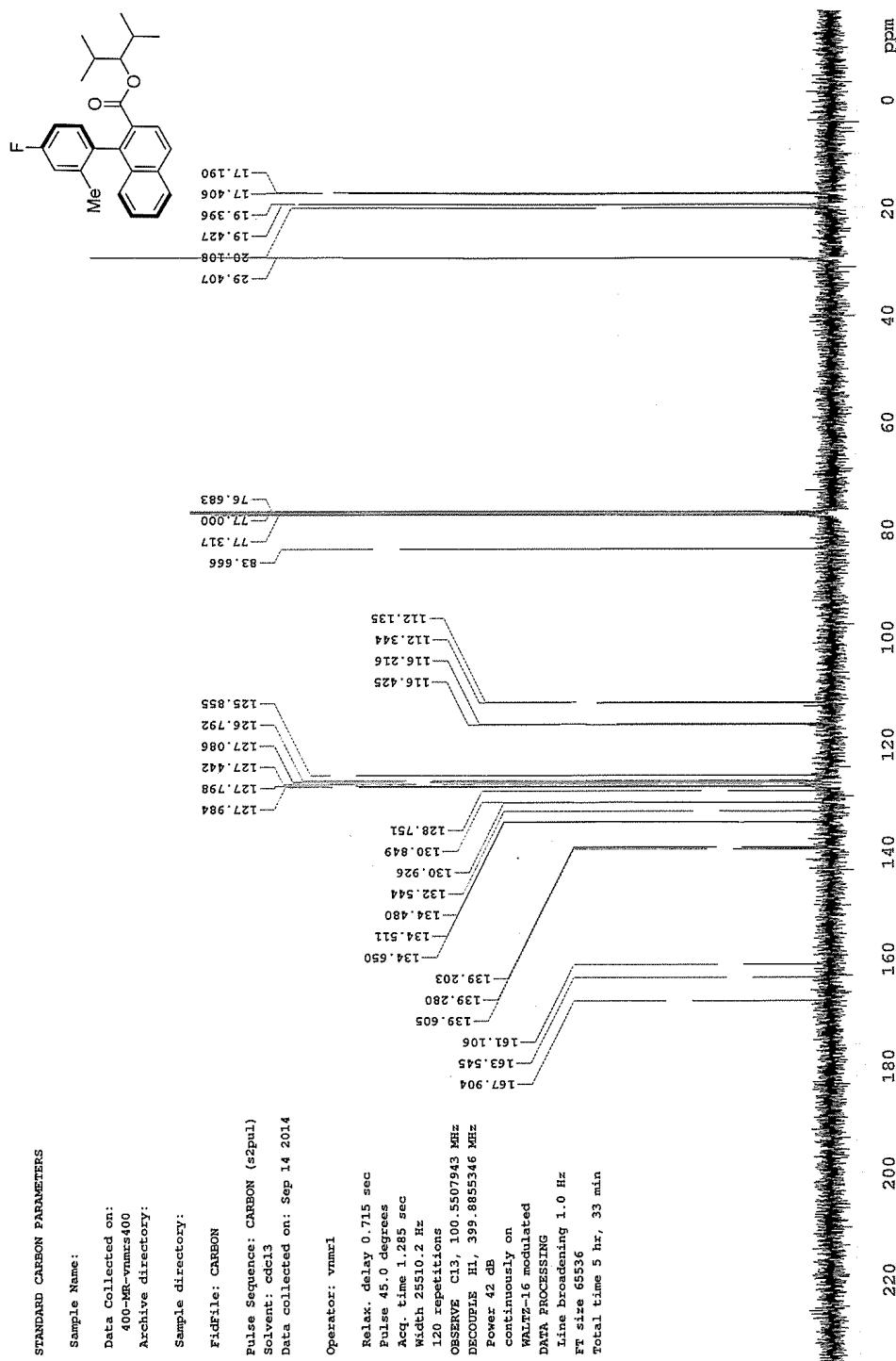
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65336

Total time 5 hr, 33 min



<sup>13</sup>C NMR of compound 3Dj

**STANDARD PROTON PARAMETERS**

Sample Name:

Data Collected on:  
400-MR-vnmrs400

Archive directory:

Sample directory:

Fidfile: PROTON

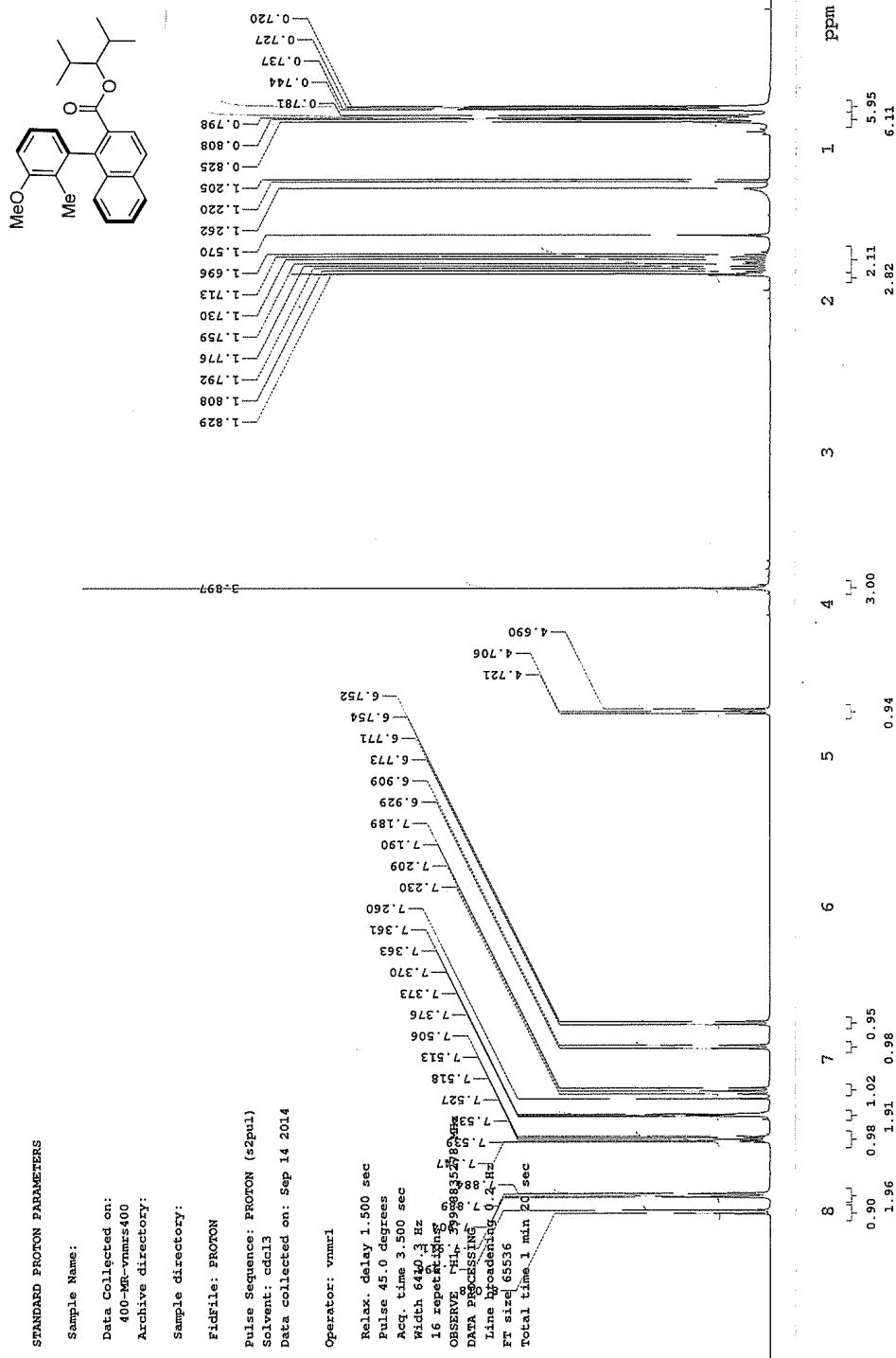
Pulse Sequence: PROTON (s2p1)

Solvent: ccdl3

Data collected on: Sep 14 2014

Operator: vnmr1

Relax. delay 1.500 sec  
Pulse 45.0 degrees  
Acc. time 3.500 sec  
Width 6410.3 Hz  
16 repeating  
OBSERVE <sup>1</sup>H, 3.500 sec  
DATA PROCESSING  
Line broadening 0.2-2 Hz  
FT size 65536  
Total time 1 min 20 sec



<sup>1</sup>H NMR of compound 3Dk

## STANDARD CARBON PARAMETERS

Sample Name:

Data Collected on:  
400-MR-nmrs400

Archive directory:

Sample directory:

FidFile: CARBON

Pulse Sequence: CARBON (sp3pul)

Solvent: cddc13  
Data collected on: Sep 14 2014

Operator: vnmrl

Relax. delay 0.715 sec

Pulse 45.0 degrees

Asg. time 1.285 sec

Width 25510.2 Hz

72 repetitions

OBSERVE C13, 100.5507943 MHz

DECOUPLE H1, 399.8855346 MHz

Power 42 dB

continuously on

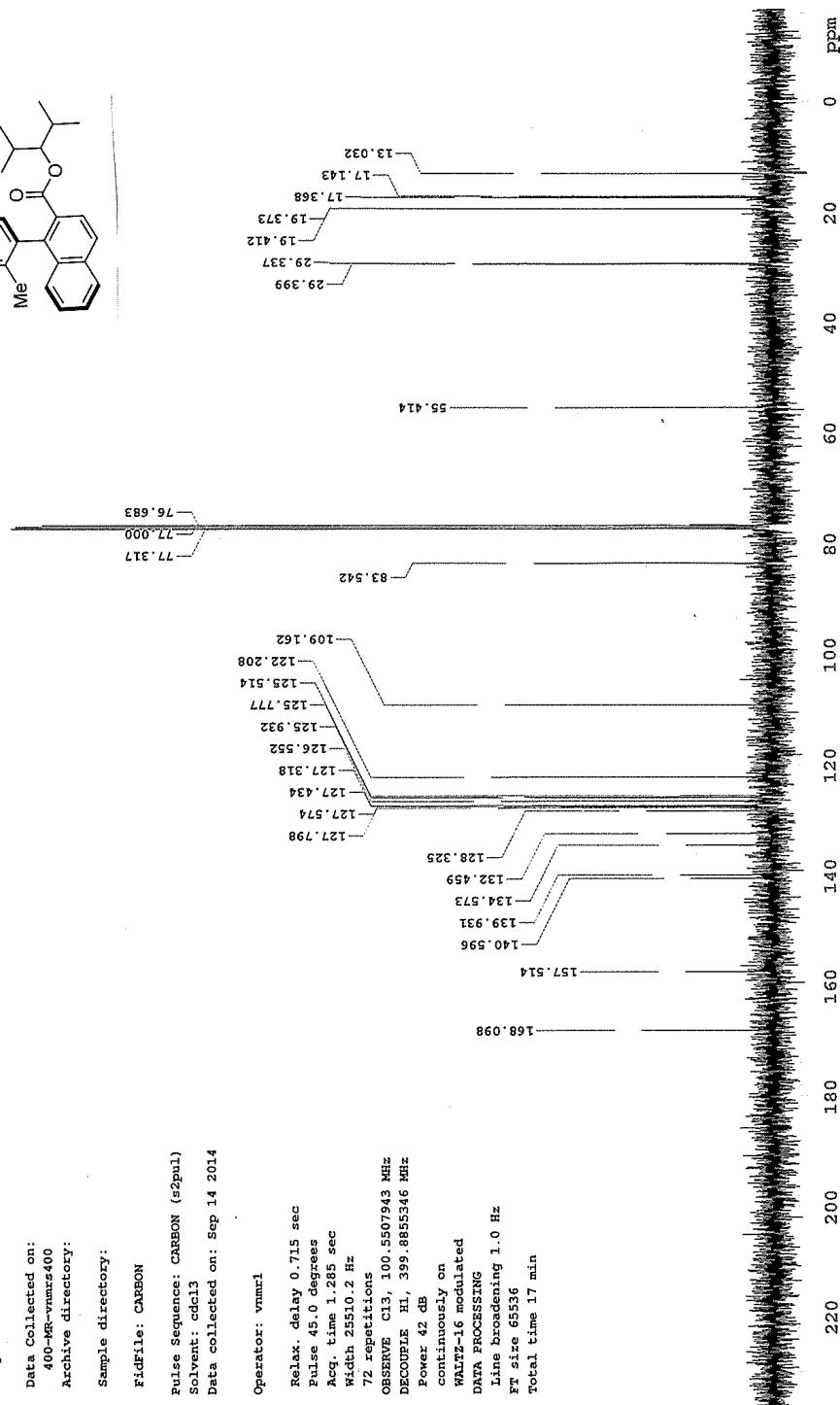
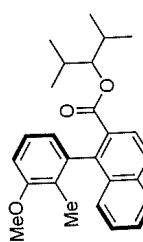
WALTZ-16 modulated

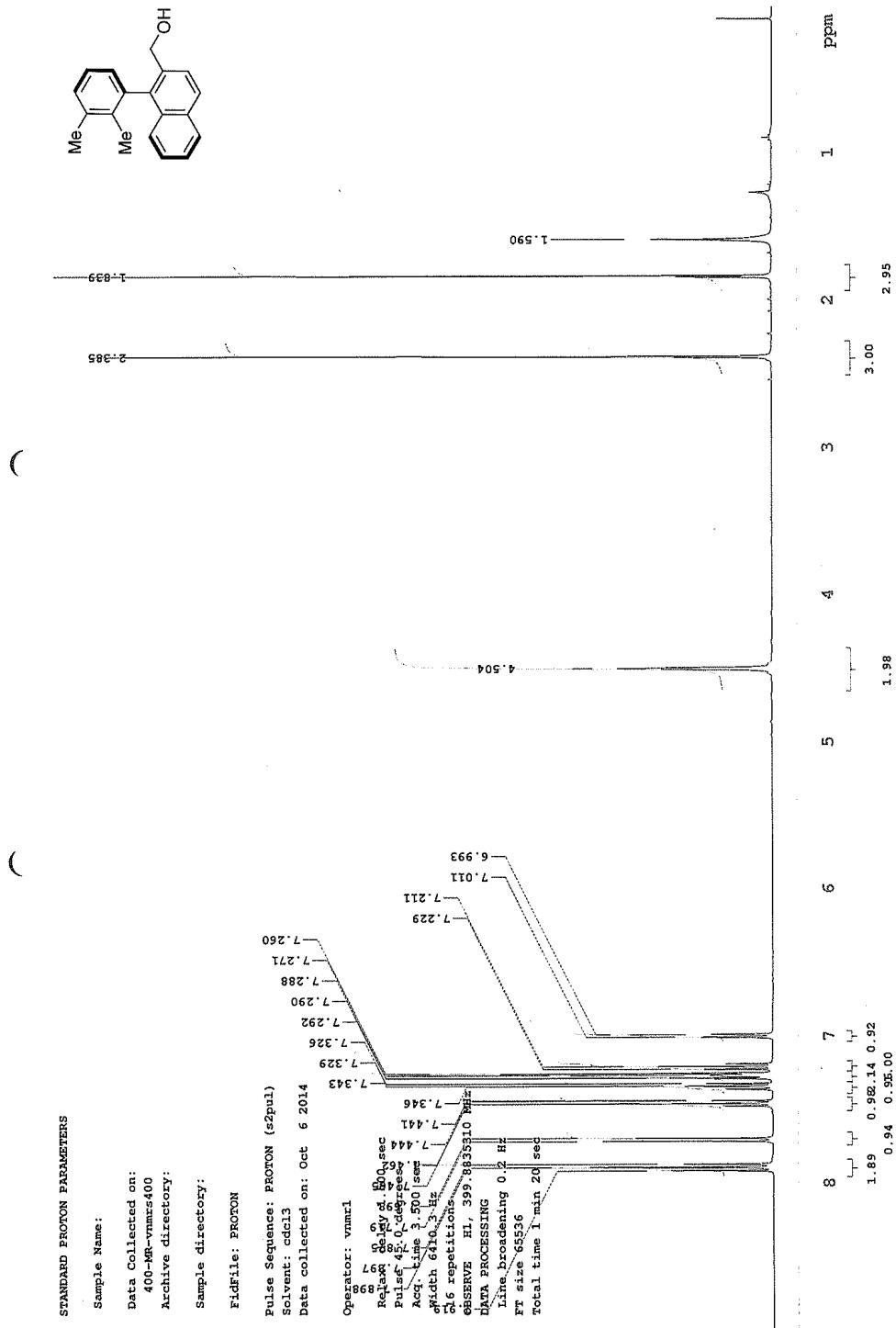
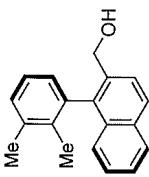
DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 17 min

<sup>13</sup>C NMR of compound 3Dk



<sup>1</sup>H NMR of compound **4Dg**

STANDARD CARBON PARAMETERS

Sample Name:

Data Collected on:  
400-MR-vnmrs400

Archive directory:

Sample directory:

FidFile: CARBON

Pulse Sequence: CARBON (spini)

Solvent: cdcl<sub>3</sub>

Data collected on: Oct 6 2014

Operator: vnmrl

Relax. delay 0.715 sec

Pulse 45.0 degrees

Avg. time 1.285 sec

Width 25510.2 Hz

96 repetitions

OBSERVE C13, 100.5507959 MHz

DECORRELATE H1, 399.8855346 MHz

Power 42 dB

Continuously on

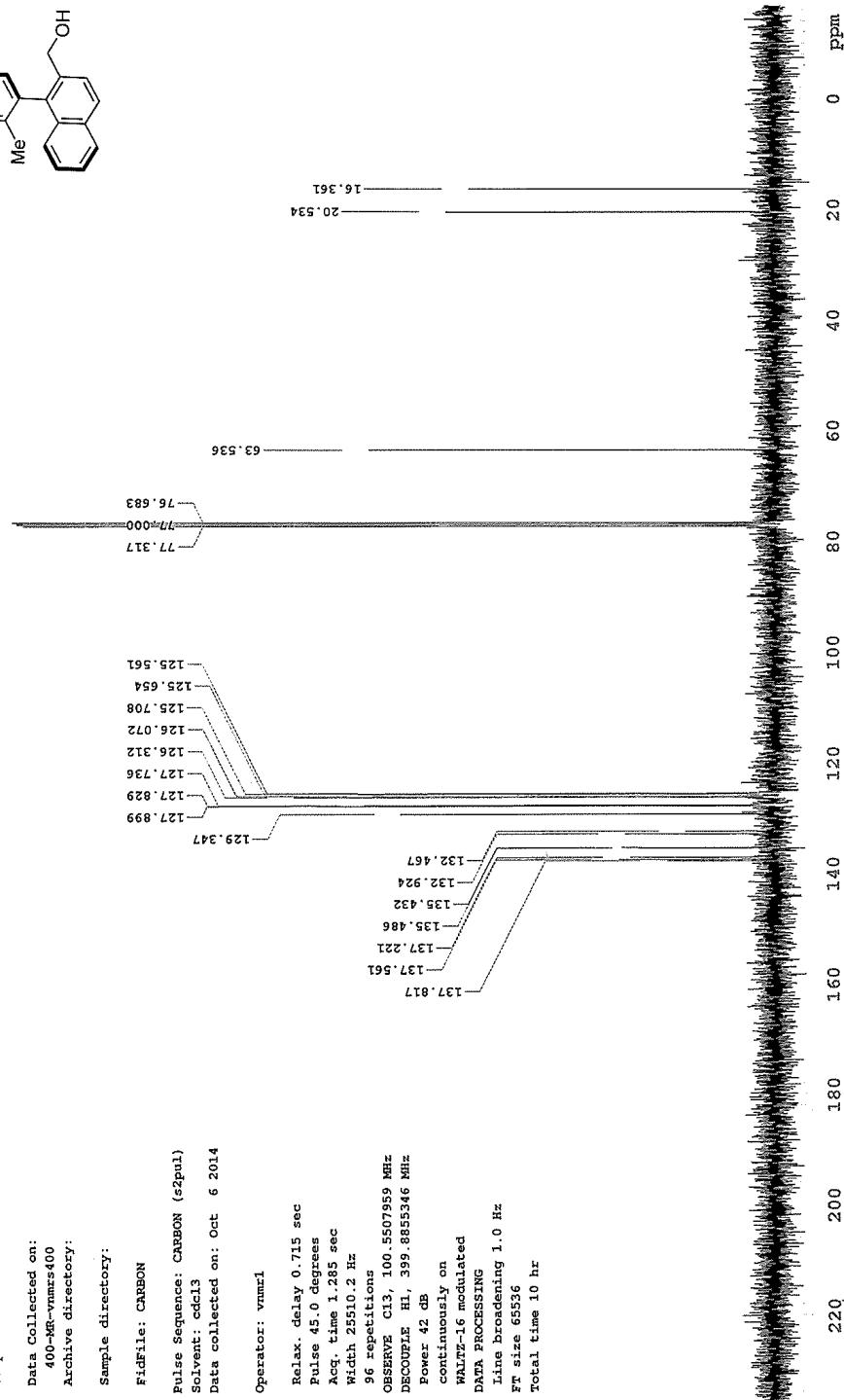
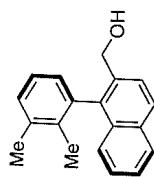
WALZ=16 modulated

DATA PROCESSING

Line Broadening 1.0 Hz

FT size 65536

Total time 10 hr



<sup>13</sup>C NMR of compound 4Dg

STANDARD PROTON PARAMETERS

Sample Name:

Data Collected on:  
400-MR-vnmrs400

Archive directory:

Sample directory:

Fidfile: PROTON

Pulse Sequence: PROTON (s2phi)

Solvent: cddc13

Data collected on: Oct 3 2014

Operator:  00000000000000000000000000000000

SN: 00000000000000000000000000000000

Relax: 1.0 sec

Delay 1.0 sec

Pulse: 45.0 degrees

Dec time 3.50 sec

Width 6410.3 Hz

16 repetitions

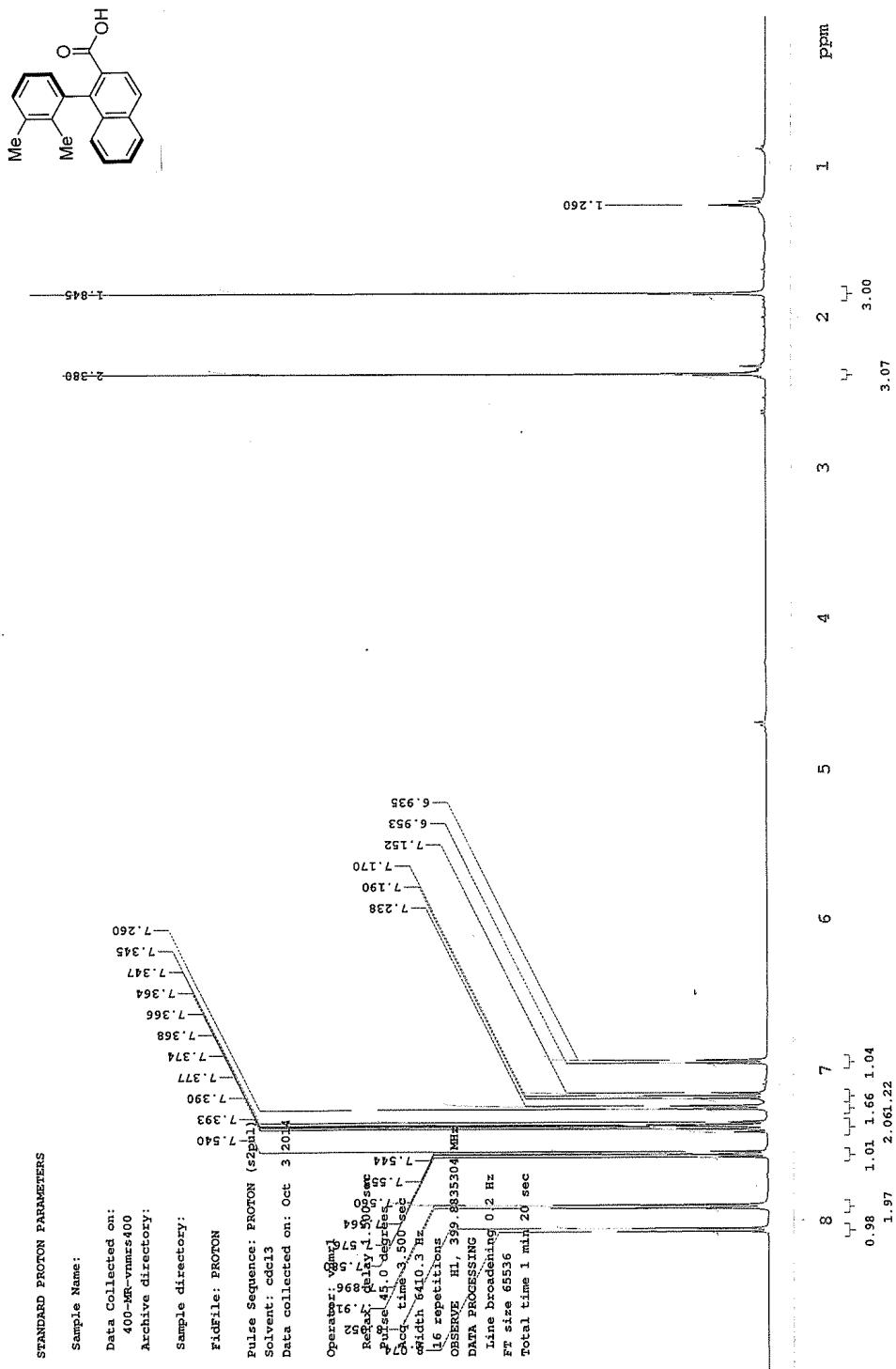
OBSERVE: H1, 3.99, 3035304

DATA PROCESSING

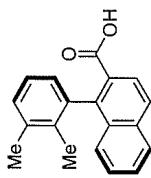
Line broadening 0.2 Hz

FID size 65536

Total time 1 min 20 sec



<sup>1</sup>H NMR of compound 5Dg



STANDARD CARBON PARAMETERS

Sample Name:

Data Collected on:  
400-MR-vnmrs400  
Archive directory:

Sample directory:

FidFile: CARBON

Pulse Sequence: CARBON (32pul)

Solvent: cdcl<sub>3</sub>

Data collected on: Oct 3 2014

Operator: vnmrl

Relax. delay 0.715 sec

Pulse 45.0 degrees

Acq. time 1.265 sec

Width 25510.2 Hz

220 repetitions

OBSERVE C13, 100.5507943 MHz

DECOUPLE H1, 399.8855346 MHz

Power 42 dB

continuously on

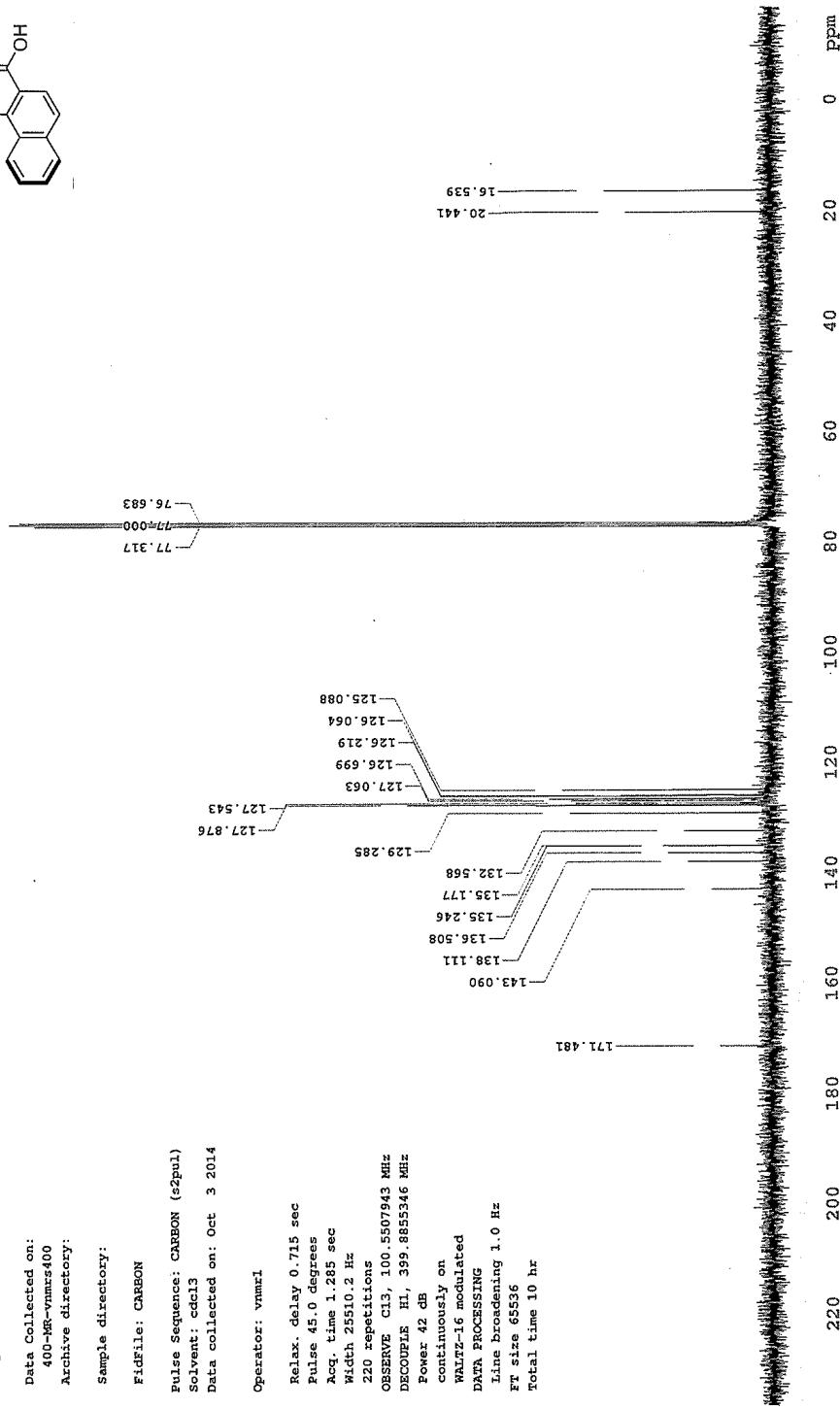
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 10 hr



<sup>13</sup>C NMR of compound 5Dg