

**Nickel-Catalyzed Cross Couplings of Benzylic Pivalates
with Arylboroxines: Stereospecific Formation of
Diarylalkanes and Triarylmethanes**

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General Information

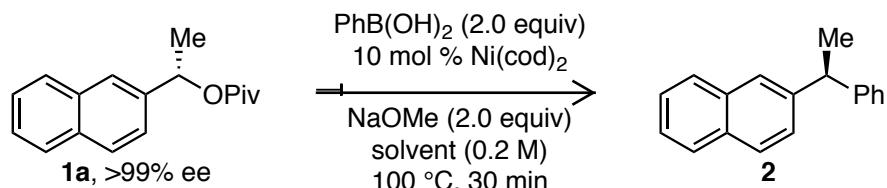
Reactions were performed either in a N₂-atmosphere glovebox in oven-dried 1-dram vials with Teflon-lined caps or in oven-dried round-bottomed flasks unless otherwise noted. Flasks were fitted with rubber septa, and reactions were conducted under a positive pressure of N₂. Stainless steel syringes or cannulae were used to transfer air- and moisture-sensitive liquids. Flash chromatography was performed on silica gel 60 (40-63 µm, 60Å). Thin layer chromatography (TLC) was performed on glass plates coated with silica gel 60 with F254 indicator. Commercial reagents were purchased from Sigma Aldrich, Acros, Fisher, Strem, TCI, Combi Blocks, Alfa Aesar, or Cambridge Isotopes Laboratories and used as received with the following exceptions: toluene, CH₂Cl₂, dioxane, and Et₂O were dried by passing through drying columns.¹ Toluene was then degassed by sparging with N₂ and stored over activated 4Å MS in a N₂-atmosphere glovebox. Anhydrous K₃PO₄ was purchased from Acros and used as received. Pivaloyl chloride was purchased from Sigma Aldrich, and used as received. Boronic acids were purchased from Combi Blocks and were converted to the boroxines according to literature procedure.² CDCl₃ was stored over oven-dried potassium carbonate. Proton nuclear magnetic resonance (¹H NMR) spectra and carbon nuclear magnetic resonance (¹³C NMR) spectra were recorded on both 400 MHz and 600 MHz spectrometers. Chemical shifts for protons are reported in parts per million downfield from tetramethylsilane and are referenced to residual protium in the NMR solvent (CHCl₃ = δ 7.28). Chemical shifts for carbon are reported in parts per million downfield from tetramethylsilane and are referenced to the carbon resonances of the solvent (CDCl₃ =

¹ Pangborn, A. B.; Giardello, M. A.; Grubbs, R. H.; Rosen, R. K.; Timmers, F. J. *Organometallics* **1996**, *15*, 1518.

² Xiao, Q.; Tian, L.; Tan, R.; Xia, Y.; Qiu, D.; Zhang, Y.; Wang, J. *Org. Lett.* **2012**, *14*, 4230.

δ 77.07). Data are represented as follows: chemical shift, multiplicity (br = broad, s = singlet, d = doublet, t = triplet, q = quartet, p = pentet, m = multiplet, dd = doublet of doublets, h = heptet), coupling constants in Hertz (Hz), integration. Infrared (IR) spectra were obtained using FTIR spectrophotometers with material loaded onto a NaCl plate. The mass spectral data were obtained at the University of Illinois, Urbana–Champaign, mass spectrometry facility and the University of Delaware mass spectrometry facility. Optical rotations were measured using a 2.5 mL cell with a 1 dm path length. Melting points were taken on a Stuart SMP10 instrument.

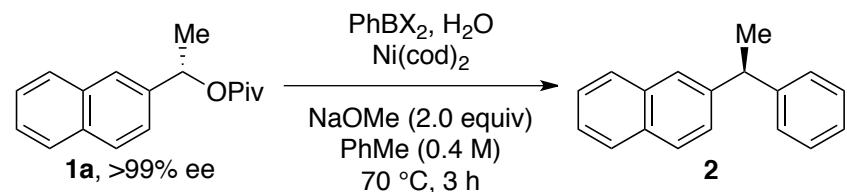
Effect of Solvent



entry	solvent	yield (%) ^a	ee (%) ^b
1	C ₆ H ₆	89	94
2	PhMe	87	94
3	THF/PhMe (1:1)	56	16
4	DME	24	10
5	Dioxane	63	7

^a Determined by ¹H NMR analysis using 1,3,5-trimethoxybenzene as internal standard. ^b Determined by chiral HPLC analysis.

Effect of Water

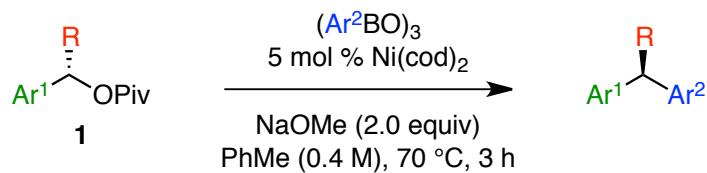


entry	mol % Ni	PhBX ₂ (equiv)	equiv H ₂ O	yield (%) ^a	ee (%) ^b
1	10	PhB(OH) ₂ (3.0)	0.5	80	98
2	10	PhB(OH) ₂ (3.0)	1.0	49	94
3	5	(PhBO) ₃ (0.83)	0.5	74	96
4	5	(PhBO) ₃ (0.83)	1.0	59	94
5	5	(PhBO) ₃ (0.83)	2.5	55	92

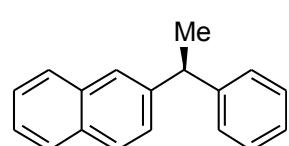
^a Determined by ¹H NMR analysis using 1,3,5-trimethoxybenzene as internal standard. ^b Determined by chiral HPLC analysis.

Stereospecific Cross Couplings of Benzyl Pivalates to Give Diarylalkanes and Triarylmethanes

General Procedure

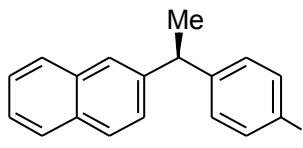


In a N_2 -atmosphere glovebox, $\text{Ni}(\text{cod})_2$ (2.8 mg, 0.01 mmol, 5 mol %) and NaOMe (21.6 mg, 0.4 mmol, 2.0 equiv) were weighed into a 1-dram vial. Benzylic pivalate (0.2 mmol, 1.0 equiv) and boroxine (0.167 mmol, 0.83 equiv) were added, followed by toluene (0.5 mL, 0.4 M). The vial was capped with a Teflon-lined cap and removed from the glovebox. The mixture was heated for 3 h at 70 °C. The reaction mixture was then diluted with Et_2O (1.5 mL) and filtered through a plug of silica gel, which was rinsed with Et_2O (10 mL). The filtrate was concentrated and purified by silica gel chromatography to give the diaryl- or triarylmethane product.



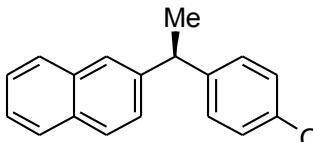
(S)-2-(1-Phenylethyl)naphthalene (2). Diarylethane **2** was prepared via the General Procedure using pivalate **1a** (prepared in >99% ee). The crude material was purified by silica gel chromatography (2–3% Et_2O /hexanes) to give compound **2** (run 1: 41.8 mg, 90%; run 2: 40.4 mg, 87%) as a colorless oil. The enantiomeric excess was determined to be 98% ee (run 1: 98% ee; run 2: 98% ee) by chiral HPLC analysis (CHIRACEL OD-H, 0.8 mL/min, 100% hexane, $\lambda=220$ nm); $t_{\text{R}}(\text{major})=21.40$ min, $t_{\text{R}}(\text{minor})=27.35$ min. $[\alpha]_{\text{D}}^{24}=$

+21.0° (c 1.00, CHCl₃): ¹H NMR (400 MHz, CDCl₃) δ 7.87 – 7.80 (m, 2H), 7.78 (d, *J* = 8.5 Hz, 1H), 7.75 – 7.72 (m, 1H), 7.55 – 7.41 (m, 2H), 7.37 – 7.27 (m, 5H), 7.26 – 7.19 (m, 1H), 4.36 (q, *J* = 7.2 Hz, 1H), 1.78 (d, *J* = 7.2 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 146.2, 143.8, 133.5, 132.1, 128.4, 127.9, 127.8, 127.7, 127.6, 126.9, 126.1, 125.9, 125.4, 125.3, 44.9, 21.8. The spectral data for this compound matches that reported in the literature.³



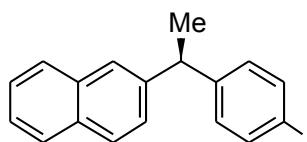
(S)-*N,N*-Dimethyl-4-(1-(naphthalen-2-yl)ethyl)aniline (3). Diarylethane **3** was prepared via the General Procedure using pivalate **1a** (prepared in >99% ee), except that 10 mol% Ni(cod)₂ was used and the reaction mixture was heated at 90 °C for 12 h. The crude material was purified by silica gel chromatography (5–6% Et₂O/hexanes) to give compound **3** (run 1: 35.2 mg, 64%; run 2: 34.1 mg, 62%) as a colorless oil. The enantiomeric excess was determined to be 86% ee (run 1: 86% ee; run 2: 86% ee) by chiral HPLC analysis (CHIRALPAK 1A, 1.0 mL/min, 0.3% *i*-PrOH/hexane, λ =254 nm); *t*_R(major)=10.87 min, *t*_R(minor)=9.35 min. [α]_D²⁴ = -62.5° (c 0.60, CHCl₃): ¹H NMR (400 MHz, CDCl₃) δ 7.82 – 7.76 (m, 2H), 7.73 (d, *J* = 8.5 Hz, 1H), 7.69 (d, *J* = 1.9 Hz, 1H), 7.48 – 7.38 (m, 2H), 7.32 (dd, *J* = 8.5, 1.8 Hz, 1H), 7.16 – 7.11 (m, 2H), 6.73 – 6.67 (m, 2H), 4.24 (q, *J* = 7.2 Hz, 1H), 2.91 (s, 6H), 1.70 (d, *J* = 7.2 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 149.1, 144.7, 134.4, 133.5, 132.1, 128.3, 127.8, 127.7, 127.5, 126.9, 125.8, 125.2, 125.1, 112.8, 43.9, 40.8, 21.9; FTIR (NaCl/thin film) 3051, 2962, 2925, 2852, 2799, 1614, 1520, 1479, 1445, 1348, 1163, 1126, 948, 855, 816 cm⁻¹; HRMS (ESI) [M]⁺ calculated for C₂₀H₂₂N: 276.1752, found: 276.1751.

³ López-Pérez, A.; Adrio, J.; Carretero, J. C. *Org. Lett.* **2009**, *11*, 5514.



(*S*)-2-(1-(4-Methoxyphenyl)ethyl)naphthalene (4).

Diarylethane **4** was prepared via the General Procedure using pivalate **1a** (prepared in >99% ee). The crude material was purified by silica gel chromatography (3% Et₂O/hexanes) to give compound **4** (run 1: 49.3 mg, 94%; run 2: 50.1 mg, 95%) as a colorless oil. The enantiomeric excess was determined to be 96% ee (run 1: 96% ee; run 2: 96% ee) by chiral HPLC analysis (CHIRALPAK 1A, 1.0 mL/min, 0.2% *i*-PrOH/hexane, λ =254 nm); t_R (major)=8.64 min, t_R (minor)=8.09 min. $[\alpha]_D^{24} = -14.0^\circ$ (c 1.00, CHCl₃): ¹H NMR (400 MHz, CDCl₃) δ 7.89 – 7.81 (m, 2H), 7.78 (d, J = 8.5 Hz, 1H), 7.73 (s, 1H), 7.54 – 7.43 (m, 2H), 7.34 (dd, J = 8.5, 1.8 Hz, 1H), 7.25 – 7.17 (m, 2H), 6.94 – 6.81 (m, 2H), 4.31 (q, J = 7.2 Hz, 1H), 3.82 (s, 3H), 1.75 (d, J = 7.2 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 157.9, 144.2, 138.4, 133.5, 132.1, 128.7, 127.9, 127.7, 127.6, 126.8, 125.9, 125.3, 125.2, 113.8, 55.3, 44.0, 22.0. The spectral data for this compound matches that reported in the literature.⁴



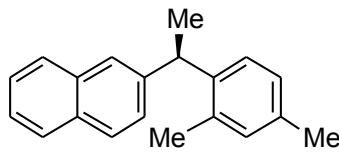
(*S*)-2-(1-(*p*-Tolyl)ethyl)naphthalene (5). Diarylethane **5**

was prepared via the General Procedure using pivalate **1a** (prepared in >99% ee). The crude material was purified by silica gel chromatography (2% Et₂O/hexanes) to give compound **5** (run 1: 43.1 mg, 87%; run 2: 41.4 mg, 84%) as a colorless oil. The enantiomeric excess was determined to be 97% ee (run 1: 97% ee; run 2: 97% ee) by chiral HPLC analysis (CHIRACEL OD-H, 0.8 mL/min, 100% hexane, λ =220 nm); t_R (major)=20.43 min, t_R (minor)=23.63 min. $[\alpha]_D^{24} =$

⁴ Noji, M.; Ohno, T.; Fuji, K.; Futaba, N.; Tajima, H.; Ishii, K. *J. Org. Chem.* **2003**, *68*, 9340.

+51.2° (c 0.80, CHCl₃): ¹H NMR (400 MHz, CDCl₃) δ 7.85 – 7.80 (m, 2H), 7.78 (d, 1H), 7.76 – 7.72 (m, 1H), 7.52 – 7.43 (m, 2H), 7.34 (dd, *J* = 8.5, 1.8 Hz, 1H), 7.22 – 7.11 (m, 4H), 4.32 (q, *J* = 7.2 Hz, 1H), 2.35 (s, 3H), 1.76 (d, *J* = 7.2 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 144.0, 143.3, 135.6, 133.5, 132.1, 129.1, 127.9, 127.7, 127.6, 127.6, 126.9, 125.9, 125.3, 125.3, 44.5, 21.9, 21.0. The spectral data for this compound matches that reported in the literature.⁵

The absolute configuration of this compound was assigned as *S* by comparison of the HPLC of this compound to an authentic sample previously prepared in our group via a different method.⁶



(*R*)-2-(1-(2,4-Dimethylphenyl)ethyl)naphthalene (6).

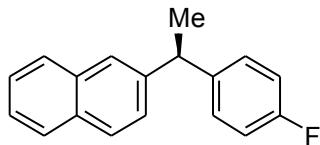
Diarylethane **6** was prepared via the General Procedure using pivalate **1a** (prepared in >99% ee). The crude material was purified by silica gel chromatography (3% Et₂O/hexanes) to give compound **6** (run 1: 49.1 mg, 93%; run 2: 49.3 mg, 96%) as a colorless oil. The enantiomeric excess was determined to be 96% ee (run 1: 96% ee; run 2: 96% ee) by chiral HPLC analysis (CHIRACEL OD-H, 0.8 mL/min, 100% hexane, λ =220 nm); *t*_R(major)=17.61 min, *t*_R(minor)=14.35 min. $[\alpha]_D^{24} = +11.0^\circ$ (c 1.00, CHCl₃): ¹H NMR (400 MHz, CDCl₃) ¹H NMR (400 MHz, CDCl₃) δ 7.84 – 7.79 (m, 2H), 7.76 (d, *J* = 8.5 Hz, 1H), 7.68 – 7.64 (m, 1H), 7.51 – 7.41 (m, 2H), 7.31 (dd, *J* = 8.5, 1.8 Hz, 1H), 7.22 (d, *J* = 7.8 Hz, 1H), 7.10 – 7.04 (m, 1H), 7.02 (s, 1H), 4.49 (q, *J* = 7.2 Hz, 1H), 2.35 (s, 3H), 2.29 (s, 3H), 1.72 (d, *J* = 7.1 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 143.9, 140.9, 136.0, 135.6, 133.5, 131.9, 131.3, 127.9, 127.7, 127.6, 126.9, 126.9, 126.7, 125.9, 125.4, 125.3, 40.8, 22.0, 20.9, 19.8;

⁵ Clark, P. D.; McKinnon, D. M. *Can. J. Chem.* **1981**, *59*, 1297.

⁶ Maity, P.; Shacklady-McAtee, D. M.; Yap, G. P. A.; Sirianni, E. R.; Watson, M. P. *J. Am. Chem. Soc.* **2012**, accepted.

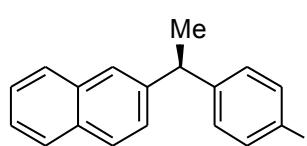
FTIR (NaCl/thin film) 3052, 3015, 2965, 2922, 1600, 1500, 1451, 1373, 1043, 855 cm⁻¹; HRMS (EI+) [M]⁺calculated for C₂₀H₂₀: 260.1565, found: 260.1573.

(S)-2-(1-(4-Fluorophenyl)ethyl)naphthalene (7).



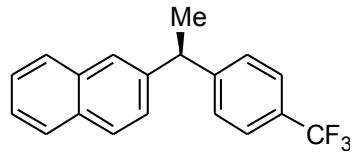
Diarylethane **7** was prepared via the General Procedure using pivalate **1a** (prepared in >99% ee). The crude material was purified by silica gel chromatography (2–2.5% Et₂O/hexanes) to give compound **7** (run 1: 44.2 mg, 88%; run 2: 42.8 mg, 85%) as a white solid (mp 57–61 °C). The enantiomeric excess was determined to be 96% ee (run 1: 97% ee; run 2: 95% ee) by chiral HPLC analysis (CHIRACEL OD-H, 0.8 mL/min, 100% hexane, $\lambda=220$ nm); t_R (major)=26.20 min, t_R (minor)=34.62 min. $[\alpha]_D^{24} = -31.0^\circ$ (c 1.60, CHCl₃): ¹H NMR (400 MHz, CDCl₃) δ 7.86 – 7.81 (m, 2H), 7.78 (d, $J = 8.5$ Hz, 1H), 7.72 – 7.68 (m, 1H), 7.53 – 7.44 (m, 2H), 7.31 (dd, $J = 8.4, 1.8$ Hz, 1H), 7.27 – 7.22 (m, 2H), 7.05 – 6.97 (m, 2H), 4.33 (q, $J = 7.2$ Hz, 1H), 1.75 (d, $J = 7.2$ Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 161.3 (d, $J_{C-F} = 245.4$ Hz), 143.6, 141.9 (d, $J_{C-F} = 3.0$ Hz), 133.5, 132.1, 129.1 (d, $J_{C-F} = 8.1$ Hz), 128.1, 127.7, 127.6, 126.7, 126.1, 125.5, 125.3, 115.1 (d, $J_{C-F} = 21.2$ Hz), 44.1, 22.0. The spectral data for this compound matches that reported in the literature.⁶

(S)-2-(1-(4-Chlorophenyl)ethyl)naphthalene (8).



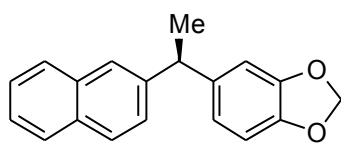
Diarylethane **8** was prepared via the General Procedure using pivalate **1a** (prepared in >99% ee) except that the reaction was done at 40 °C for 24 h. The crude material was purified by silica gel chromatography (100% petroleum ether) to give compound **8** (run 1: 34.5 mg, 65%; run 2: 32.5 mg, 61%) as a colorless oil. The enantiomeric excess was determined to be 94% ee (run 1: 94% ee;

run 2: 94% ee) by chiral HPLC analysis (CHIRACEL OD-H, 0.8 mL/min, 100% hexane, $\lambda=220$ nm); t_R (major)=25.82 min, t_R (minor)=34.31 min. $[\alpha]_D^{24} = -27.7^\circ$ (c 1.00, CHCl_3): ^1H NMR (400 MHz, CDCl_3) δ 7.82 (d, $J = 8.4$ Hz, 2H), 7.77 (d, $J = 8.5$ Hz, 1H), 7.69 (s, 1H), 7.51 – 7.44 (m, 2H), 7.31 – 7.26 (m, 3H), 7.23 – 7.18 (m, 2H), 4.31 (q, $J = 7.2$ Hz, 1H), 1.74 (d, $J = 7.2$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 144.7, 143.2, 133.5, 132.2, 131.8, 129.2, 128.5, 128.1, 127.7, 127.6, 126.7, 126.1, 125.6, 125.4, 44.3, 21.8; FTIR (NaCl/thin film) 3054, 2966, 2925, 2851, 1600, 1491, 1375, 1092, 1014, 856 cm^{-1} ; HRMS (EI+) $[\text{M}]^+$ calculated for $\text{C}_{18}\text{H}_{15}\text{Cl}$: 266.0862, found: 266.0855.



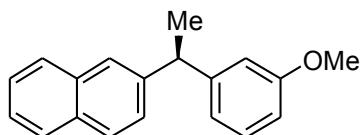
(S)-2-(1-(4-(Trifluoromethyl)phenyl)ethyl)naphthalene (9).

Diarylethane **9** was prepared via the General Procedure using pivalate **1a** (prepared in >99% ee). The crude material was purified by silica gel chromatography (3% $\text{Et}_2\text{O}/\text{hexanes}$) to give compound **9** (run 1: 59.4 mg, 99%; run 2: 60.0 mg, 100%) as a colorless oil. The enantiomeric excess was determined to be 97% ee (run 1: 96% ee; run 2: 97% ee) by chiral HPLC analysis (CHIRACEL OD-H, 0.8 mL/min, 100% hexane, $\lambda=220$ nm); t_R (major)=28.30 min, t_R (minor)=39.14 min. $[\alpha]_D^{24} = +36.4^\circ$ (c 1.60, CHCl_3): ^1H NMR (400 MHz, CDCl_3) δ 7.87 – 7.82 (m, 2H), 7.80 (d, $J = 8.5$ Hz, 1H), 7.75 – 7.72 (m, 1H), 7.58 (d, $J = 8.1$ Hz, 2H), 7.54 – 7.46 (m, 2H), 7.42 – 7.38 (m, 2H), 7.31 (dd, $J = 8.6, 1.8$ Hz, 1H), 4.41 (q, $J = 7.2$ Hz, 1H), 1.79 (d, $J = 7.2$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 150.3 (q, $J_{\text{C}-\text{F}} = 1.1$ Hz), 142.6, 133.5, 132.2, 128.4 (q, $J_{\text{C}-\text{F}} = 32.3$ Hz), 128.3 (q, $J_{\text{C}-\text{F}} = 1.0$ Hz), 128.1, 127.8, 127.6, 126.6, 126.2, 125.7, 125.5, 125.4 (q, $J_{\text{C}-\text{F}} = 4.0$ Hz), 124.8 (q, $J_{\text{C}-\text{F}} = 272.7$ Hz), 44.7, 21.6; FTIR (NaCl/thin film) 3056, 2970, 2932, 2876, 1919, 1618, 1600, 1416, 1326, 1164, 1118, 1071, 1017, 842 cm^{-1} ; HRMS (EI+) $[\text{M}]^+$ calculated for $\text{C}_{19}\text{H}_{15}\text{F}_3$: 300.1126, found: 300.1129.



(*R*)-5-(1-(Naphthalen-2-yl)ethyl)benzo[*d*][1,3]dioxole (10).

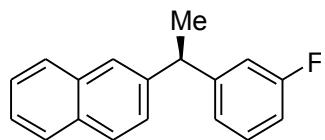
Diarylethane **10** was prepared via the General Procedure using pivalate **1a** (prepared in >99% ee). The crude material was purified by silica gel chromatography (2% Et₂O/hexanes) to give compound **10** (run 1: 48.2 mg, 88%; run 2: 47.1 mg, 85%) as a colorless oil. The enantiomeric excess was determined to be 97% ee (run 1: 97% ee; run 2: 96% ee) by chiral HPLC analysis (CHIRALPAK 1B, 1.0 mL/min, 0.3% *i*-PrOH/hexane, $\lambda=254$ nm); t_R (major)=9.30 min, t_R (minor)=10.14 min. $[\alpha]_D^{24} = -10.8^\circ$ (c 1.20, CHCl₃): ¹H NMR (400 MHz, CDCl₃) δ 7.84 – 7.77 (m, 2H), 7.75 (d, $J = 8.5$ Hz, 1H), 7.71 – 7.66 (m, 1H), 7.52 – 7.39 (m, 2H), 7.30 (dd, $J = 8.5, 1.8$ Hz, 1H), 6.78 – 7.66 (m, 3H), 5.91 (s, 2H), 4.24 (q, $J = 7.2$ Hz, 1H), 1.70 (d, $J = 7.2$ Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 147.7, 145.8, 143.8, 140.3, 133.5, 132.1, 127.9, 127.7, 127.6, 126.7, 125.9, 125.4, 125.2, 120.5, 108.4, 108.1, 100.9, 44.6, 21.9; FTIR (NaCl/thin film) 3065, 2964, 2919, 1726, 1603, 1507, 1484, 1435, 1370, 1231, 1036, 940, 861 cm⁻¹; HRMS (EI+) [M]⁺ calculated for C₁₉H₁₆O₂: 276.1150, found: 276.1154.



(*R*)-2-(1-(3-Methoxyphenyl)ethyl)naphthalene (11).

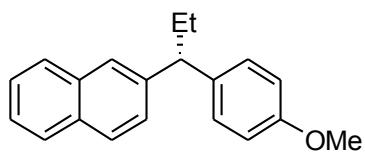
Diarylethane **11** was prepared via the General Procedure using pivalate **1a** (prepared in >99% ee). The crude material was purified by silica gel chromatography (2% Et₂O/hexanes) to give compound **11** (run 1: 49.5 mg, 94%; run 2: 51.2 mg, 97%) as a white solid (mp 88–91 °C). The enantiomeric excess was determined to be 98% ee (run 1: 97% ee; run 2: 98% ee) by chiral HPLC analysis (CHIRALPAK 1A, 1.0 mL/min, 0.2% *i*-PrOH/hexane, $\lambda=254$ nm); t_R (major)=7.54 min, t_R (minor)=8.69 min. $[\alpha]_D^{24} = -23.3^\circ$ (c 1.20, CHCl₃): ¹H NMR (400 MHz, CDCl₃) δ 7.86 – 7.80 (m, 2H), 7.77 (d, $J = 8.5$ Hz, 1H), 7.74 – 7.72 (m, 1H), 7.52 – 7.42 (m, 2H), 7.35 (dd, $J = 8.5, 1.8$ Hz,

1H), 7.24 (t, $J = 7.9$ Hz, 1H), 6.89 – 6.87 (m, 1H), 6.85 (t, $J = 2.1$ Hz, 1H), 6.77 (dd, 1H), 4.32 (q, $J = 7.2$ Hz, 1H), 3.79 (s, 3H), 1.76 (d, $J = 7.2$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 159.6, 147.9, 143.6, 133.5, 132.1, 129.4, 128.0, 127.8, 127.6, 126.8, 126.0, 125.4, 125.3, 120.3, 113.9, 111.0, 55.2, 44.9, 21.7; FTIR (NaCl/thin film) 3052, 2963, 2926, 1598, 1485, 1150, 1039, 818 cm^{-1} ; HRMS (EI+) [M] $^+$ calculated for $\text{C}_{19}\text{H}_{18}\text{O}$: 262.1358, found: 262.1366.



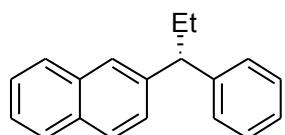
(R)-2-(1-(3-Fluorophenyl)ethyl)naphthalene (12).

Diarylethane **12** was prepared via the General Procedure using pivalate **1a** (prepared in >99% ee). The crude material was purified by silica gel chromatography (3% Et_2O /hexanes) to give compound **12** (run 1: 44.3 mg, 88%; run 2: 45.5 mg, 91%) as a colorless oil. The enantiomeric excess was determined to be 94% ee (run 1: 94% ee; run 2: 94% ee) by chiral HPLC analysis (CHIRACEL OD-H, 0.8 mL/min, 100% hexane, $\lambda=220$ nm); $t_{\text{R}}(\text{major})=24.61$ min, $t_{\text{R}}(\text{minor})=35.64$ min. $[\alpha]_D^{24} = +22.5^\circ$ (c 1.20, CHCl_3): ^1H NMR (400 MHz, CDCl_3) δ 7.87 – 7.81 (m, 2H), 7.79 (d, $J = 8.5$ Hz, 1H), 7.75 – 7.71 (m, 1H), 7.54 – 7.45 (m, 2H), 7.36 – 7.30 (m, 1H), 7.29 – 7.24 (m, 1H), 7.10 – 7.05 (m, 1H), 7.02 – 6.97 (m, 1H), 6.95 – 6.89 (m, 1H), 4.34 (q, $J = 7.2$ Hz, 1H), 1.76 (d, $J = 7.2$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 163.0 (d, $J_{\text{C}-\text{F}} = 246.4$ Hz), 148.9 (d, $J_{\text{C}-\text{F}} = 6.1$ Hz), 143.0, 133.5, 132.2, 129.8 (d, $J_{\text{C}-\text{F}} = 8.1$ Hz), 128.1, 127.8, 127.6, 126.6, 126.1, 125.6, 125.4, 123.5 (d, $J_{\text{C}-\text{F}} = 3.0$ Hz), 114.6 (d, $J_{\text{C}-\text{F}} = 22.2$ Hz), 113.0 (d, $J_{\text{C}-\text{F}} = 21.2$ Hz), 44.6, 21.6; FTIR (NaCl/thin film) 3055, 2967, 2928, 2873, 1726, 1613, 1559, 1487, 912, 818 cm^{-1} ; HRMS (EI+) [M] $^+$ calculated for $\text{C}_{18}\text{H}_{15}\text{F}$: 250.1158, found: 250.1164.



(*R*)-2-(1-(4-methoxyphenyl)propyl)naphthalene (13).

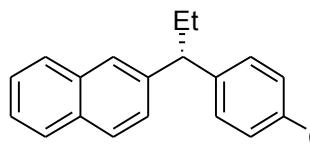
Diarylpropane **13** was prepared via the General Procedure using pivalate **1b** (prepared in >99% ee) except that 10 mol % $\text{Ni}(\text{cod})_2$ and 1.2 equiv of boroxine were used, and the reaction mixture was heated at 80 °C. The crude material was purified by silica gel chromatography (2.5% Et_2O /hexanes) to give compound **13** (run 1: 38.1 mg, 69%; run 2: 40.2 mg, 73%) as a colorless oil. The enantiomeric excess was determined to be 94% ee (run 1: 94% ee; run 2: 94% ee) by chiral HPLC analysis (CHIRACEL OD-H, 0.8 mL/min, 0.1% *i*-PrOH/hexane, $\lambda=254$ nm); $t_{\text{R}}(\text{major})=14.00$ min, $t_{\text{R}}(\text{minor})=13.23$ min. $[\alpha]_D^{24} = -19.0^\circ$ (c 1.00, CHCl_3): ^1H NMR (400 MHz, CDCl_3) δ 7.87 – 7.69 (m, 4H), 7.55 – 7.39 (m, 2H), 7.34 (dd, $J = 8.5, 1.8$ Hz, 1H), 7.26 – 7.18 (m, 2H), 6.91 – 6.79 (m, 2H), 3.93 (t, $J = 7.7$ Hz, 1H), 3.79 (s, 3H), 2.30 – 2.05 (m, 2H), 0.95 (t, $J = 7.3$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 157.8, 142.9, 137.2, 133.5, 132.1, 128.9, 127.9, 127.7, 127.6, 126.8, 125.9, 125.8, 125.3, 113.7, 55.2, 52.4, 28.5, 12.9; FTIR (NaCl/thin film) 3050, 2959, 2929, 2872, 2834, 1726, 1609, 1510, 1462, 1249, 1178, 1038, 815 cm^{-1} ; HRMS (EI+) [M] $^+$ calculated for $\text{C}_{20}\text{H}_{20}\text{O}$: 276.1514, found: 276.1505.



(*R*)-2-(1-Phenylpropyl)naphthalene (14). Diarylpropane **14**

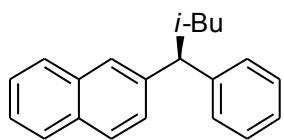
was prepared via the General Procedure using pivalate **1b** (prepared in >99% ee) except that 1.2 equiv of boroxine was used and the reaction mixture was heated at 80 °C. The crude material was purified by silica gel chromatography (3% Et_2O /hexanes) to give compound **14** (run 1: 44.6 mg, 91%; run 2: 47.2 mg, 94%) as a colorless oil. The enantiomeric excess was determined to be 97% ee (run 1: 96% ee; run 2: 98% ee) by chiral HPLC analysis (CHIRACEL OD-H, 1.0 mL/min, 100% hexanes, $\lambda=220$ nm); $t_{\text{R}}(\text{major})=19.18$ min, $t_{\text{R}}(\text{minor})=14.40$ min. $[\alpha]_D^{24} = -32.5^\circ$ (c 0.80, CHCl_3): ^1H NMR (400 MHz, CDCl_3) δ 7.87 – 7.72 (m, 4H), 7.54 – 7.41 (m, 3H), 7.37 (dd, $J = 8.5, 1.8$ Hz, 1H), 7.35 – 7.29 (m, 3H), 7.26 – 7.16 (m, 1H),

4.00 (t, $J = 7.7$ Hz, 1H), 2.28 – 2.15 (m, 2H), 0.98 (t, $J = 7.3$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 145.0, 142.6, 133.5, 132.1, 128.4, 128.1, 128.0, 127.7, 127.6, 126.9, 126.1, 125.9, 125.9, 125.3, 53.3, 28.4, 12.9; FTIR (NaCl/thin film) 3056, 3025, 2962, 2928, 2872, 1727, 1599, 1506, 1493, 1377, 1155, 855 cm^{-1} ; HRMS (EI+) [M] $^+$ calculated for $\text{C}_{19}\text{H}_{18}$: 246.1409, found: 246.1414.



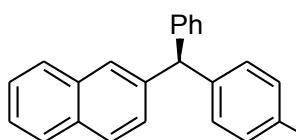
(*R*)-2-(1-(4-(trifluoromethyl)phenyl)propyl)naphthalene (15).

Diarylpropane **15** was prepared via the General Procedure using pivalate **1b** (prepared in >99% ee) except that 1.2 equiv of boroxine was used and the reaction mixture was heated at 80 °C. The crude material was purified by silica gel chromatography (3% Et_2O /hexanes) to give compound **15** (run 1: 58.4 mg, 93%; run 2: 59.2 mg, 94%) as a colorless oil. The enantiomeric excess was determined to be 96% ee (run 1: 96% ee; run 2: 96% ee) by chiral HPLC analysis (CHIRACEL OD-H, 0.8 mL/min, 100% hexanes, $\lambda=220$ nm); $t_{\text{R}}(\text{major})=31.94$ min, $t_{\text{R}}(\text{minor})=19.93$ min. $[\alpha]_D^{24} = -78.3^\circ$ (c 0.60, CHCl_3): ^1H NMR (400 MHz, CDCl_3) δ 7.87 – 7.81 (m, 2H), 7.80 (d, $J = 8.5$ Hz, 1H), 7.76 – 7.73 (m, 1H), 7.57 (d, $J = 8.1$ Hz, 2H), 7.54 – 7.45 (m, 2H), 7.43 (d, $J = 8.0$ Hz, 2H), 7.34 (dd, $J = 8.5, 1.8$ Hz, 1H), 4.06 (t, $J = 7.7$ Hz, 1H), 2.35 – 2.11 (m, 2H), 0.99 (t, $J = 7.3$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 149.1 (q, $J_{\text{C}-\text{F}} = 1.1$ Hz), 141.5, 133.5, 133.2, 128.4 (q, $J_{\text{C}-\text{F}} = 32.3$ Hz), 128.4 (q, $J_{\text{C}-\text{F}} = 1.0$ Hz), 128.3, 127.8, 127.6, 126.7, 125.2, 125.1, 125.6, 125.4 (q, $J_{\text{C}-\text{F}} = 4.0$ Hz), 124.3 (q, $J_{\text{C}-\text{F}} = 272.7$ Hz), 53.1, 28.2, 12.7; FTIR (NaCl/thin film) 3056, 2965, 2932, 2875, 1921, 1766, 1618, 1417, 1326, 1164, 1120, 1068, 1018, 815 cm^{-1} ; HRMS (EI+) [M] $^+$ calculated for $\text{C}_{20}\text{H}_{17}\text{F}_3$: 314.1282, found: 314.1287.



(S)-2-(3-Methyl-1-phenylbutyl)naphthalene (16).

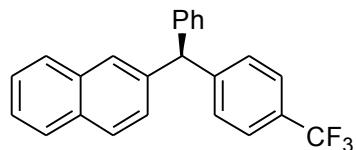
Diarylmethane **16** was prepared via the General Procedure using pivalate **1c** (prepared in 93% ee) except that 1.3 equiv of boroxine was used and the reaction time was 12 h. The crude material was purified by silica gel chromatography (1–2% Et₂O/hexanes) to give compound **16** (run 1: 40.2 mg, 73%; run 2: 39.0 mg, 71%) as a colorless oil. The enantiomeric excess was determined to be 91% ee (run 1: 90% ee; run 2: 91% ee) by chiral HPLC analysis (CHIRACEL OD-H, 0.8 mL/min, 100% hexanes, λ =220 nm); t_R (major)=15.47 min, t_R (minor)=25.37 min. $[\alpha]_D^{24} = -32.8^\circ$ (c 0.60, CHCl₃): ¹H NMR (400 MHz, CDCl₃) δ 7.71 (dd, J = 12.7, 8.0 Hz, 2H), 7.66 (d, J = 8.5 Hz, 1H), 7.64 – 7.62 (m, 1H), 7.39 – 7.31 (m, 2H), 7.28 (dd, J = 8.6, 1.8 Hz, 1H), 7.24 – 7.17 (m, 4H), 7.11 – 7.07 (m, 1H), 4.11 (t, J = 8.0 Hz, 1H), 2.02 – 1.88 (m, 2H), 1.46 – 1.37 (m, 1H), 0.87 (d, J = 2.4 Hz, 3H), 0.87 (d, J = 2.4 Hz, 3H); ¹³C NMR (151 MHz, CDCl₃) δ 145.1, 142.7, 133.5, 132.1, 128.4, 128.01, 128.0, 127.6, 127.5, 126.8, 126.07, 125.8, 125.3, 48.9, 44.7, 25.5, 22.7, 22.6; FTIR (NaCl/thin film) 3056, 2965, 2932, 2875, 1921, 1766, 1618, 1417, 1326, 1164, 1120, 1068, 1018, 815 cm⁻¹; HRMS (EI+) [M]⁺ calculated for C₂₁H₂₂: 274.1722, found: 274.1717.



(S)-2-((4-Methoxyphenyl)(phenyl)methyl)naphthalene

Triarylmethane **17** was prepared via the General Procedure using pivalate **1d** (prepared in >99% ee) except that 10 mol % of Ni(cod)₂ and 1.0 equiv of boroxine were used and the reaction mixture was heated at 90 °C. The crude material was purified by silica gel chromatography (5% Et₂O/hexanes) to give compound **17** (run 1: 55.2 mg, 85%; run 2: 54.3 mg, 84%) as a colorless oil. The enantiomeric excess was determined to be 86% ee (run 1: 86% ee; run 2: 86% ee) by chiral HPLC analysis (CHIRACEL OD-H, 0.8 mL/min, 0.1% *i*-PrOH/hexane, λ =254 nm);

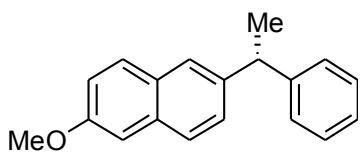
t_R (major)=24.00 min, t_R (minor)=22.03 min. $[\alpha]_D^{24} = +3.7^\circ$ (c 1.60, CHCl_3): ^1H NMR (400 MHz, CDCl_3) δ 7.85 – 7.81 (m, 1H), 7.79 (d, $J = 8.5$ Hz, 1H), 7.76 – 7.71 (m, 1H), 7.50 – 7.44 (m, 3H), 7.35 – 7.30 (m, 3H), 7.28 – 7.23 (m, 1H), 7.21 – 7.16 (m, 2H), 7.13 – 7.08 (m, 2H), 6.90 – 6.84 (m, 2H), 5.69 (s, 1H), 3.82 (s, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 158.1, 144.0, 141.9, 135.9, 133.4, 132.1, 130.5, 129.5, 128.4, 128.1, 127.9, 127.9, 127.7, 127.6, 126.3, 125.9, 125.6, 113.7, 56.1, 55.3. The spectral data for this compound matches that reported in the literature.⁷



(S)-2-(Phenyl(4-(trifluoromethyl)phenyl)methyl)naphthalene (18).

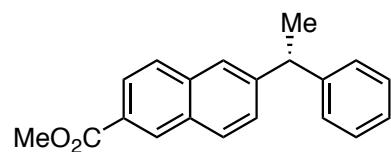
Triarylmethane **18** was prepared via the General Procedure using pivalate **1d** (prepared in >99% ee) except that 10 mol % of $\text{Ni}(\text{cod})_2$ and 1.0 equiv of boroxine were used and the reaction mixture was heated at 90 °C. The crude material was purified by silica gel chromatography (1% $\text{Et}_2\text{O}/\text{hexanes}$) to give compound **18** (70.4 mg, 96%) as a colorless oil. The enantiomeric excess was determined to be 80% ee by chiral HPLC analysis (CHIRALPAK 1B, 0.6 mL/min, 100% hexane, $\lambda=254$ nm); t_R (major)= 21.44 min, t_R (minor)= 23.27 min. $[\alpha]_D^{24} = -8.8^\circ$ (c 1.92, CHCl_3): ^1H NMR (400 MHz, CDCl_3) δ 7.76 – 7.72 (m, 1H), 7.71 (d, $J = 8.5$ Hz, 1H), 7.66 – 7.62 (m, 1H), 7.48 (d, $J = 8.1$ Hz, 2H), 7.40 – 7.36 (m, 3H), 7.27 – 7.22 (m, 2H), 7.22 – 7.16 (m, 4H), 7.07 (dd, $J = 7.2, 1.7$ Hz, 2H), 5.68 (s, 1H); ^{13}C NMR (151 MHz, CDCl_3) δ 147.8, 142.8, 140.5, 133.4, 132.3, 129.9, 129.0, 128.8 (q, $J_{\text{C}-\text{F}} = 32.1$ Hz), 128.6, 128.5, 128.2, 128.9 (q, $J_{\text{C}-\text{F}} = 1.5$ Hz), 127.8, 127.6, 126.8, 126.2, 125.9, 125.4 (q, $J_{\text{C}-\text{F}} = 3.9$ Hz), 124.3 (q, $J_{\text{C}-\text{F}} = 273.5$), 56.8; FTIR (NaCl/thin film) 3058, 3027, 2926, 1924, 1600, 1494, 1416, 1326, 1165, 1068, 814 cm^{-1} ; HRMS (EI+) $[\text{M}]^+$ calculated for $\text{C}_{24}\text{H}_{17}\text{F}_3$: 362.1824, found: 362.1276.

⁷ Taylor, B. L. H.; Harris, R. M.; Jarvo, E. R. *Angew. Chem. Int. Ed.* **2012**, 24, 7910.



(*R*)-2-Methoxy-6-(1-phenylethyl)naphthalene (19).

Diarylethane **19** was prepared via the General Procedure using pivalate **1e** (prepared in >99% ee) except that 1.0 equiv of boroxine was used and the reaction mixture was heated at 50 °C. The crude material was purified by silica gel chromatography (1–2% Et₂O/hexanes) to give compound **19** (run 1: 45.6 mg, 87%; run 2: 45.5 mg, 87%) as a white solid (mp 97–101 °C). The enantiomeric excess was determined to be 93% ee (run 1: 93% ee; run 2: 93% ee) by chiral HPLC analysis (CHIRALPAK 1B, 0.6 mL/min, 1.0% *i*-PrOH/hexane, λ =254 nm); t_R (major)=11.29 min, t_R (minor)=9.83 min. $[\alpha]_D^{24} = -28.2^\circ$ (c 1.00, CHCl₃): ¹H NMR (400 MHz, CDCl₃) δ 7.73 (d, J = 8.9 Hz, 1H), 7.69 (s, 1H), 7.68 – 7.66 (m, 1H), 7.36 – 7.28 (m, 5H), 7.26 – 7.20 (m, 1H), 7.17 (dd, J = 8.8, 2.6 Hz, 1H), 7.15 – 7.12 (m, 1H), 4.32 (q, J = 7.2 Hz, 1H), 3.94 (s, 3H), 1.76 (d, J = 7.2 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 157.3, 146.5, 141.5, 133.1, 129.2, 128.9, 128.4, 127.8, 127.4, 126.9, 126.1, 125.2, 118.7, 105.6, 55.3, 44.7, 21.9; FTIR (NaCl/thin film) 3026, 2965, 2935, 2871, 1605, 1489, 1451, 1266, 1166, 1034, 851 cm⁻¹; HRMS (EI+) [M]⁺ calculated for C₁₉H₁₈O: 262.1358, found: 262.1353. The spectral data for this compound matches that reported in the literature.⁸

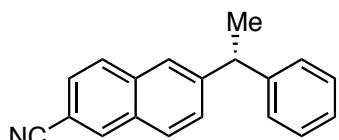


(*R*)-Methyl 6-(1-phenylethyl)-2-naphthoate (20).

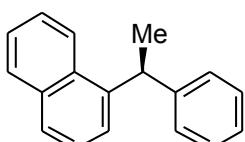
Diarylethane **20** was prepared via the General Procedure using pivalate **1f** (prepared in 98% ee). The crude material was purified by silica gel chromatography (5–6% Et₂O/hexanes) to give compound **20** (run 1: 49.2 mg, 85%; run 2: 47.4 mg, 82%) as viscous colorless oil. The enantiomeric excess was determined to be 94% ee (run 1: 93% ee; run 2: 94% ee) by chiral HPLC analysis (CHIRALPAK 1B, 1.0 mL/min, 2.0% *i*-PrOH/hexane, λ =254 nm);

⁸ Gayral, M.; Brown, J. M. *Synlett.* **2007**, 18, 2823.

t_R (minor)=6.75 min, t_R (major)=7.40 min. $[\alpha]_D^{24} = -31.8^\circ$ (c 1.92, CHCl_3): ^1H NMR (600 MHz, CDCl_3) δ 8.55 (s, 1H), 8.04 (dd, $J = 8.6, 1.7$ Hz, 1H), 7.84 (dd, $J = 8.5, 5.4$ Hz, 2H), 7.73 (s, 1H), 7.38 (dd, $J = 8.5, 1.8$ Hz, 1H), 7.33 – 7.24 (m, 4H), 7.23 – 7.18 (m, 1H), 4.33 (q, $J = 7.2$ Hz, 1H), 3.97 (s, 3H), 1.74 (d, $J = 7.2$ Hz, 3H); ^{13}C NMR (151 MHz, CDCl_3) δ 167.4, 146.6, 145.7, 135.7, 131.1, 130.8, 129.4, 128.5, 128.0, 127.8, 127.7, 126.9, 126.3, 125.4, 125.2, 52.2, 45.0, 21.7; FTIR (NaCl/thin film) 2966, 2929, 1719, 1632, 1494, 1437, 1289, 1237, 1201, 1097, 912 cm^{-1} ; HRMS (CI+) $[\text{M}+\text{H}]^+$ calculated for $\text{C}_{15}\text{H}_{19}\text{N}_2\text{O}_4$: 291.1385, found: 291.1387.

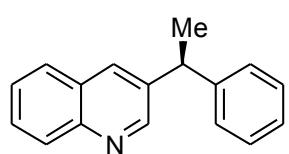


(*R*)-6-(1-Phenylethyl)-2-naphthonitrile (21). Diarylethane **21** was prepared via the General Procedure using pivalate **1g** (prepared in 95% ee). The crude material was purified by silica gel chromatography (5–10% EtOAc/hexanes) to give compound **21** (run 1: 23.6 mg, 46%; run 2: 22.1 mg, 43%) as viscous colorless oil. The enantiomeric excess was determined to be 90% ee (run 1: 89% ee; run 2: 89% ee) by chiral HPLC analysis (CHIRALPAK 1A, 1.0 mL/min, 0.5% *i*-PrOH/hexane, $\lambda=254$ nm); t_R (minor)=34.2 min, t_R (major)=37.2 min. $[\alpha]_D^{24} = -75.0^\circ$ (c 0.40, CHCl_3): ^1H NMR (600 MHz, CDCl_3) δ 8.17 (s, 1H), 7.86 (d, $J = 8.5$ Hz, 1H), 7.79 (d, $J = 8.5$ Hz, 1H), 7.73 (s, 1H), 7.58 (dd, $J = 8.5, 1.6$ Hz, 1H), 7.44 (dd, $J = 8.6, 1.7$ Hz, 1H), 7.33 – 7.28 (m, 2H), 7.25 – 7.20 (m, 3H), 4.34 (q, $J = 7.2$ Hz, 1H), 1.74 (d, $J = 7.2$ Hz, 3H); ^{13}C NMR (151 MHz, CDCl_3) δ 147.6, 145.3, 134.9, 133.8, 131.0, 129.0, 128.7, 128.6, 128.5, 127.7, 126.6, 126.5, 125.5, 119.4, 108.8, 45.0, 21.6; FTIR (NaCl/thin film) 3025, 2967, 2929, 2226, 1629, 1600, 1493, 1451, 1377, 1158, 894 cm^{-1} ; HRMS (CI+) $[\text{M}+\text{H}]^+$ calculated for $\text{C}_{16}\text{H}_{18}\text{O}_3$: 258.1283, found: 258.1277.



(*S*)-1-(1-phenylethyl)naphthalene (22). Diarylethane **22** was prepared via the General Procedure using pivalate **1h** (prepared in

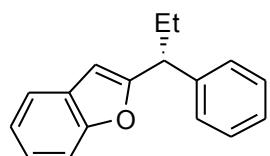
86% ee). The crude material was purified by silica gel chromatography (3% Et₂O/hexanes) to give compound **22** (run 1: 34.3 mg, 74%; run 2: 32.9 mg, 71%) as a colorless oil. The enantiomeric excess was determined to be 73% ee (run 1: 73% ee; run 2: 72% ee) by chiral HPLC analysis (CHIRALPAK 1B, 0.8 mL/min, 100% hexanes, $\lambda=220$ nm); t_R (major)=19.13 min, t_R (minor)=26.13 min. $[\alpha]_D^{24} = -23.3^\circ$ (c 0.60, CHCl₃): ¹H NMR (400 MHz, CDCl₃) δ 7.99 – 7.94 (m, 1H), 7.78 – 7.75 (m, 1H), 7.66 (dd, J = 7.8, 1.3 Hz, 1H), 7.41 – 7.31 (m, 4H), 7.20 – 7.14 (m, 4H), 7.10 – 7.06 (m, 1H), 4.85 (q, J = 7.1 Hz, 1H), 1.69 (d, J = 7.1 Hz, 3H); ¹³C NMR (151 MHz, CDCl₃) δ 146.7, 141.6, 134.0, 131.7, 128.8, 128.4, 127.6, 127.0, 126.0, 125.9, 125.5, 125.3, 124.4, 124.0, 40.6, 22.6; FTIR (NaCl/thin film) 3058, 2966, 2929, 2872, 1597, 1508, 1493, 1449, 1396, 1372, 1026, 800 cm⁻¹; HRMS (EI+) [M]⁺ calculated for C₁₈H₁₆: 232.1252, found: 232.1255. The spectral data for this compound matches that reported in the literature.⁹



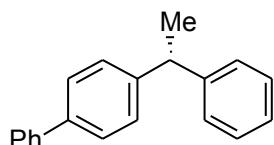
(S)-3-(1-Phenylethyl)quinoline (23). Diarylethane **23** was prepared via the General Procedure using pivalate **1i** (prepared in 82% ee). The crude material was purified by silica gel chromatography (20% EtOAc/hexanes) to give compound **23** (run 1: 43.2 mg, 93%; run 2: 44.3 mg, 94%) as pale yellow oil. The enantiomeric excess was determined to be 58% ee (run 1: 58% ee; run 2: 57% ee) by chiral HPLC analysis (CHIRALPAK 1C, 1.0 mL/min, 3.0% i-PrOH/hexane, $\lambda=210$ nm); t_R (minor)=14.95 min, t_R (major)=16.50 min. $[\alpha]_D^{24} = +12.1^\circ$ (c 0.98, CHCl₃): ¹H NMR (600 MHz, CDCl₃) δ 8.80 (d, J = 2.3 Hz, 1H), 8.07 (d, J = 8.5 Hz, 1H), 7.94 (d, J = 2.3 Hz, 1H), 7.77 (dd, J = 8.1, 1.6 Hz, 1H), 7.69 – 7.63 (m, 1H), 7.56 – 7.47 (m, 1H), 7.36 – 7.29 (m, 2H), 7.29 – 7.20 (m, 3H), 4.37 (q, J =

⁹ (a) Mazuela, J.; Verendel, J. J.; Coll, M.; Schaffner, B.; Borner, A.; Anderson, P. G.; Pamies, O.; Dieguez, M. *J. Am. Chem. Soc.* **2009**, *131*, 12344. (b) Gomez, E. D.; Albert, D.; Mattiza, J.; Duddeck, H.; Chojnowski, J.; Cypryk, M. *Tetrahedron Asym.* **2006**, *17*, 1743.

7.2 Hz, 1H), 1.77 (d, J = 7.2 Hz, 3H); ^{13}C NMR (151 MHz, CDCl_3) δ 151.8, 146.8, 144.9, 138.9, 133.0, 129.1, 128.8, 128.7, 128.1, 127.7, 127.6, 126.6, 126.5, 42.6, 21.6; FTIR (NaCl/thin film) 3060, 3026, 2967, 2930, 2873, 1602, 1570, 1494, 1380, 1333, 1125, 1028, 961 cm^{-1} ; HRMS (EI+) [M] $^+$ calculated for $\text{C}_{17}\text{H}_{15}\text{N}$: 233.1204, found: 233.1198.



(R)-2-(1-Phenylpropyl)benzofuran (24). Diarylpropane **24** was prepared via the General Procedure using pivalate **1j** (prepared in 82% ee) except that 1.2 equiv of boroxine was used and the reaction mixture was heated at 32 °C for 24 h. The crude material was purified by silica gel chromatography (4% Et_2O /hexanes) to give compound **24** (run 1: 24.0 mg, 51%; run 2: 22.2 mg, 47%) as a colorless oil. The enantiomeric excess was determined to be 72% ee (run 1: 72% ee; run 2: 72% ee) by chiral HPLC analysis (CHIRALPAK 1B, 1.0 mL/min, 0.2% *i*-PrOH/hexane, λ =254 nm); $t_{\text{R}}(\text{major})=37.28$ min, $t_{\text{R}}(\text{minor})=34.31$ min. $[\alpha]_D^{24} = -38.1^\circ$ (c 0.80, CHCl_3): ^1H NMR (400 MHz, CDCl_3) δ 7.42 – 7.40 (m, 1H), 7.33 – 7.30 (m, 1H), 7.26 – 7.21 (m, 4H), 7.18 – 7.14 (m, 1H), 7.14 – 7.07 (m, 2H), 6.38 (s, 1H), 3.88 (t, J = 7.7 Hz, 1H), 2.24 – 2.14 (m, 1H), 1.99 – 1.88 (m, 1H), 0.88 (t, J = 7.4 Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 161.2, 154.8, 141.9, 128.7, 128.5, 128.0, 126.7, 123.3, 122.4, 120.4, 110.9, 102.4, 47.6, 27.6, 12.4; HRMS (EI+) [M] $^+$ calculated for $\text{C}_{17}\text{H}_{16}\text{O}$: 236.1201, found: 236.1195. The spectral data for this compound matches that reported in the literature.¹⁰



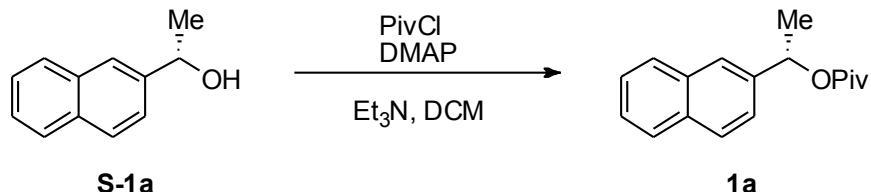
(S)-4-(1-Phenylethyl)biphenyl (25). Diarylethane **25** was prepared via the General Procedure using pivalate **1k** (prepared in 93% ee) except that 10 mol % $\text{Ni}(\text{cod})_2$ and 1.0 equiv of boroxine were used and the reaction mixture was heated at 100 °C for 24 h. The crude material was purified by silica gel chromatography (100% petroleum ether) to give

¹⁰ DeLuca, L.; Nieddu, G.; Giacomelli, G. *J. Org. Chem.* **2007**, 72, 3955.

compound **25** (run 1: 17.0 mg, 33%; run 2: 17.2 mg, 33%) as a white solid (mp 37–41 °C). The enantiomeric excess was determined to be 84% ee (run 1: 84% ee; run 2: 84% ee) by chiral HPLC analysis (CHIRALPAK 1B, 1.0 mL/min, 100% hexane, $\lambda=254$ nm); $t_R(\text{minor})=15.34$ min, $t_R(\text{major})=24.22$ min. $[\alpha]_D^{24} = -51.6^\circ$ (c 0.60, CHCl_3): ^1H NMR (600 MHz, CDCl_3) δ 7.63 – 7.57 (m, 2H), 7.57 – 7.52 (m, 2H), 7.47 – 7.43 (m, 2H), 7.38 – 7.29 (m, 7H), 7.25 – 7.12 (m, 1H), 4.23 (q, $J = 7.3$ Hz, 1H), 1.71 (d, $J = 7.3$ Hz, 3H); ^{13}C NMR (151 MHz, CDCl_3) δ 146.3, 145.5, 141.0, 139.0, 128.7, 128.4, 128.0, 127.6, 127.11, 127.05, 127.0, 126.1, 44.5, 21.9. The spectral data for this compound matches that reported in the literature.³

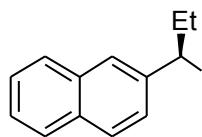
Preparation of Benzyl Pivalates

General Procedure for the Preparation of Benzyl Pivalates: Preparation of (*S*)-1-(Naphthalen-2-yl)ethyl Pivalate (**1a**)

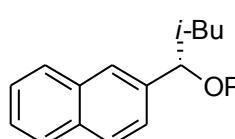


(*S*)-1-(Naphthalen-2-yl)ethanol (**S-1a**, 1.17 g, 6.8 mmol, 1.0 equiv) and DMAP (83 mg, 0.68 mmol, 0.10 equiv) were dissolved in CH_2Cl_2 (10 mL, 0.68 M). Et_3N (1.94 mL, 1.35 mmol, 2.0 equiv) and pivaloyl chloride (1.02 mL, 8.15 mmol, 1.2 equiv) were then added. The reaction mixture was then stirred for 12 h at room temperature, before H_2O (20 mL) was added. The organic layer was extracted with CH_2Cl_2 (2 x 20 mL). The combined organic layers were washed with 10% aq. KOH (20 mL), dried (MgSO_4), filtered and concentrated. The resulting residue was purified by silica gel chromatography (5% Et_2O /hexanes) to give compound **1a** (1.58 g, 91%) as a white solid. The enantiomeric excess was determined to be >99% ee by chiral HPLC analysis.

(CHIRALPAK IA, 0.6 mL/min, 1.0% *i*-PrOH/hexane, $\lambda=254$ nm); t_R (minor)=8.23 min, t_R (major)=10.31 min. $[\alpha]_D^{24} = -82.1^\circ$ (c 0.80, CHCl₃): ¹H NMR (600 MHz, CDCl₃) δ 7.85 – 7.81 (m, 3H), 7.80 – 7.77 (m, 1H), 7.50 – 7.45 (m, 3H), 6.02 (q, $J = 6.6$ Hz, 1H), 1.60 (d, $J = 6.6$ Hz, 3H), 1.23 (s, 9H); ¹³C NMR (151 MHz, CDCl₃) δ 177.7, 139.5, 133.2, 132.9, 128.3, 128.0, 127.7, 126.2, 126.0, 124.7, 124.0, 72.1, 38.8, 27.2, 22.3; HRMS (EI+) [M]⁺ calculated for C₁₇H₂₀O₂: 256.1463, found: 256.1469. The spectral data for this compound matches that reported in the literature for the racemic compound.¹¹



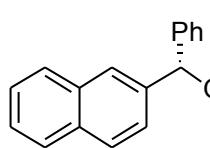
(R)-1-(Naphthalen-2-yl)propyl pivalate (1b). Prepared according to the General Procedure on a 4.03 mmol scale to give **1b** (900 mg, 83%) as a colorless oil. The enantiomeric excess was determined to be >99% ee by chiral HPLC analysis (CHIRALPAK IA, 0.6 mL/min, 1.0% *i*-PrOH/hexane, $\lambda=254$ nm); t_R (major)=8.26 min, t_R (minor)=10.45 min. $[\alpha]_D^{24} = +160.9^\circ$ (c 4.20, CHCl₃): ¹H NMR (600 MHz, CDCl₃) δ 7.88 – 7.75 (m, 4H), 7.54 – 7.44 (m, 3H), 5.83 (t, $J = 7.0$ Hz, 1H), 2.07 – 1.87 (m, 2H), 1.27 (s, 9H), 0.95 (t, $J = 7.2$ Hz, 3H); ¹³C NMR (151 MHz, CDCl₃) δ 177.7, 138.4, 133.2, 133.0, 128.2, 128.0, 127.7, 126.1, 125.9, 125.4, 124.2, 38.9, 29.5, 27.2, 26.5, 10.0; FTIR (NaCl/thin film) 3057, 2971, 2934, 2875, 1728, 1479, 1396, 1153, 817 cm⁻¹; HRMS (EI+) [M]⁺ calculated for C₁₈H₂₂O₂: 270.1620, found: 270.1626.



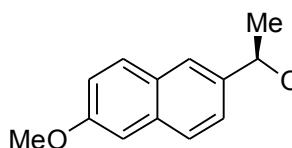
(S)-3-Methyl-1-(naphthalen-2-yl)butyl pivalate (1c). Prepared according to the General Procedure on a 1.17 mmol scale to give **1c** (302 mg, 87%) as a colorless oil. The enantiomeric excess was determined to be 93% ee by chiral HPLC analysis (CHIRALPAK 1A, 0.8 mL/min, 1.0% *i*-PrOH/hexane, $\lambda=254$ nm); t_R (major)=8.22 min, t_R (minor)=10.31 min. $[\alpha]_D^{24} = -77.8^\circ$ (c 0.84, CHCl₃): ¹H NMR (600 MHz, CDCl₃) δ 7.84 – 7.79 (m, 3H), 7.78 – 7.75 (m, 1H),

¹¹ Katsumata, H.; Yamada, S. *J. Org. Chem.* **2007**, 72, 3955.

7.50 – 7.43 (m, 3H), 5.93 (m, 1H), 1.95 (dd, J = 8.9, 7.8 Hz, 1H), 1.68 – 1.61 (m, 2H), 1.21 (s, 9H), 0.98 (d, J = 6.6 Hz, 3H), 0.95 (d, J = 6.0 Hz, 3H); ^{13}C NMR (151 MHz, CDCl_3) δ 177.7, 138.9, 133.2, 132.9, 128.3, 128.0, 127.7, 126.1, 125.9, 125.4, 124.2, 74.4, 45.7, 38.2, 27.1, 24.8, 22.9, 22.3; FTIR (NaCl/thin film) 2958, 2360, 1728, 1652, 1558, 1506, 1457, 1282, 1164 cm^{-1} ; HRMS (EI+) [M] $^+$ calculated for $\text{C}_{20}\text{H}_{26}\text{O}_2$: 298.1933, found: 298.1938.

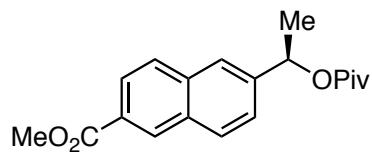


(S)-naphthalen-2-yl(phenyl)methyl pivalate (1d). Prepared according to the General Procedure on a 6.40 mmol scale to give **1d** (1.18 g, 88%) as a colorless oil. The enantiomeric excess was determined to be >99% ee by chiral HPLC analysis (CHIRALPAK 1B, 0.8 mL/min, 1.0% *i*-PrOH/hexane, λ =254 nm); $t_{\text{R}}(\text{major})$ =7.06 min, $t_{\text{R}}(\text{minor})$ =6.43 min. $[\alpha]_D^{24} = -46.2^\circ$ (c 0.80, CHCl_3): ^1H NMR (600 MHz, CDCl_3) δ 7.87 – 7.80 (m, 4H), 7.53 – 7.48 (m, 2H), 7.45 (dd, J = 8.6, 1.7 Hz, 1H), 7.43 – 7.34 (m, 4H), 7.33 – 7.30 (m, 1H), 7.02 (s, 1H), 1.30 (s, 9H); ^{13}C NMR (151 MHz, CDCl_3) δ 177.3, 140.4, 137.9, 133.1, 132.9, 128.5, 128.4, 128.1, 127.8, 127.7, 126.9, 126.3, 126.2, 126.0, 124.9, 77.2, 38.9, 27.2; FTIR (NaCl/thin film) 2971, 1727, 1478, 1278, 1147, 1123, 1031, 818 cm^{-1} ; HRMS (EI+) [M] $^+$ calculated for $\text{C}_{22}\text{H}_{22}\text{O}_2$: 318.1620, found: 318.1617.



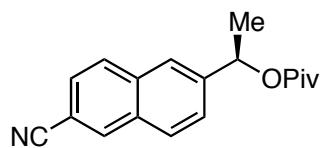
(R)-1-(6-Methoxynaphthalen-2-yl)ethyl pivalate (1e). Prepared according to the General Procedure on a 1.89 mmol scale to give **1e** (460 mg, 85%) as a white solid (mp 78–82°C). The enantiomeric excess was determined to be >99% ee by chiral HPLC analysis (CHIRALPAK IA, 0.8 mL/min, 1.0% *i*-PrOH/hexane, λ =254 nm); $t_{\text{R}}(\text{major})$ =6.88 min, $t_{\text{R}}(\text{minor})$ =6.02 min. $[\alpha]_D^{24} = +81.2^\circ$ (c 0.80, CHCl_3): ^1H NMR (600 MHz, CDCl_3) δ 7.75 – 7.69 (m, 3H), 7.43 (dd, J = 8.5, 1.7 Hz, 1H), 7.17 – 7.11 (m, 2H), 5.99 (q, J = 6.6 Hz, 1H), 3.92 (s, 3H), 1.59 (d, J = 6.6 Hz, 3H), 1.22 (s, 9H); ^{13}C NMR

(151 MHz, CDCl₃) δ 177.7, 157.8, 137.2, 134.1, 129.5, 128.6, 127.1, 124.7, 124.6, 118.9, 105.7, 72.1, 55.3, 38.8, 27.2, 22.2; FTIR (NaCl/thin film) 2979, 1724, 1608, 1506, 1487, 1391, 1284, 1237, 1166, 1031, 859 cm⁻¹; HRMS (EI+) [M]⁺ calculated for C₁₈H₂₂O₃: 286.1569, found: 286.1561.



(R)-Methyl 6-(1-(pivaloyloxy)ethyl)-2-naphthoate (1f).

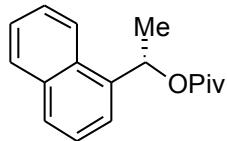
Prepared according to the General Procedure on a 1.84 mmol scale to give **1f** (470 mg, 81%) as a white solid (mp 85–88°C). The enantiomeric excess was determined to be 98% ee by chiral HPLC analysis (CHIRALPAK IA, 1.0 mL/min, 2.0% *i*-PrOH/hexane, λ=254 nm); *t*_R(major)=8.99 min, *t*_R(minor)=13.21 min. [α]_D²⁴ = +40.9° (c 0.62, CHCl₃): ¹H NMR (600 MHz, CDCl₃) δ 8.59 (s, 1H), 8.06 (dd, *J* = 8.6, 1.7 Hz, 1H), 7.94 (d, *J* = 8.5 Hz, 1H), 7.87 (d, *J* = 8.6 Hz, 1H), 7.82 (s, 1H), 7.52 (dd, *J* = 8.5, 1.7 Hz, 1H), 6.01 (q, *J* = 6.6 Hz, 1H), 3.98 (s, 3H), 1.60 (d, *J* = 6.6 Hz, 3H), 1.24 (s, 9H); ¹³C NMR (151 MHz, CDCl₃) δ 177.7, 167.2, 142.2, 135.4, 132.0, 130.8, 129.8, 128.3, 127.5, 125.6, 124.7, 124.44, 71.9, 52.3, 38.8, 27.1, 22.3; FTIR (NaCl/thin film) 2977, 2872, 1724, 1635, 1480, 1436, 1281, 1156, 1097 cm⁻¹; HRMS (CI+) [M+H]⁺ calculated for C₁₉H₂₃O₄: 315.1596, found: 315.1593.



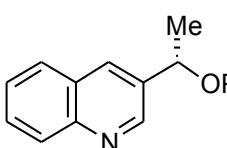
(R)-1-(6-Cyanonaphthalen-2-yl)ethyl pivalate (1g).

Prepared according to the General Procedure on a 0.89 mmol scale to give **1g** (170 mg, 67%) as a white solid (mp 74–76°C). The enantiomeric excess was determined to be 95% ee by chiral HPLC analysis (CHIRALPAK IA, 1.0 mL/min, 2.0% *i*-PrOH/hexane, λ=254 nm); *t*_R(major)=9.71 min, *t*_R(minor)=14.42 min. [α]_D²⁴ = +78.6° (c 0.90, CHCl₃): ¹H NMR (600 MHz, CDCl₃) δ 8.22 (s, 1H), 7.91 (d, *J* = 4.0 Hz, 1H), 7.89 (d, *J* = 4.1 Hz, 1H), 7.83 (s, 1H), 7.63 – 7.57 (m, 2H), 6.00 (q, *J* = 6.6 Hz, 1H), 1.60 (d, *J* = 6.6 Hz, 3H), 1.23 (s, 9H); ¹³C NMR (151 MHz, CDCl₃) δ 177.6, 143.1, 134.6, 133.9, 131.7, 129.3, 128.9, 126.8, 125.7, 124.7,

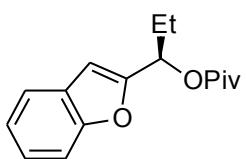
119.2, 109.4, 71.7, 38.8, 27.1, 22.3; FTIR (NaCl/thin film) 3059, 2977, 2227, 1727, 1634, 1470, 1281, 1157, 1133, 1064 cm⁻¹; HRMS (CI+) [M+H]⁺ calculated for C₁₈H₂₀NO₂: 282.1494, found: 282.1491.



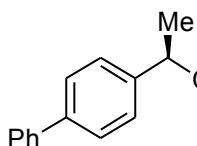
(S)-1-(Naphthalene-1-yl)ethyl pivalate (1h). Prepared according to the General Procedure on a 2.33 mmol scale to give **1h** (580 mg, 97%) as a colorless oil. The enantiomeric excess was determined to be 86% ee by chiral HPLC analysis (CHIRALPAK IA, 0.6 mL/min, 1.0% *i*-PrOH/hexane, $\lambda=254$ nm); t_R (major)=9.52 min, t_R (minor)=8.00 min. $[\alpha]_D^{24} = -39.2^\circ$ (c 3.80, CHCl₃): ¹H NMR (600 MHz, CDCl₃) δ 8.08 (d, $J = 8.6$ Hz, 1H), 7.87 (d, $J = 8.3$ Hz, 1H), 7.79 (d, $J = 8.2$ Hz, 1H), 7.59 (d, $J = 7.0$ Hz, 1H), 7.55 – 7.46 (m, 3H), 6.61 (q, $J = 6.6$ Hz, 1H), 1.68 (d, $J = 6.6$ Hz, 3H), 1.24 (s, 9H); ¹³C NMR (151 MHz, CDCl₃) δ 177.7, 137.9, 133.8, 130.2, 128.9, 128.3, 126.2, 125.6, 125.4, 123.3, 123.0, 69.3, 38.9, 27.2, 21.8; FTIR (NaCl/thin film) 2977, 2932, 1728, 1479, 1458, 1397, 1368, 1283, 1157, 1068, 1045 cm⁻¹; HRMS (EI+) [M]⁺ calculated for C₁₇H₂₀O₂: 256.1463, found: 256.1467.



(S)-1-(Quinolin-3-yl)ethyl pivalate (1i). Prepared according to the General Procedure on a 1.60 mmol scale to give **1i** (132.7 mg, 30%) as pale yellow oil. The enantiomeric excess was determined to be 82% ee by chiral HPLC analysis (CHIRALPAK IA, 1.0 mL/min, 1.0% *i*-PrOH/hexane, $\lambda=254$ nm); t_R (minor)=29.83 min, t_R (major)=33.26 min. $[\alpha]_D^{24} = -96.8^\circ$ (c 0.96, CHCl₃): ¹H NMR (600 MHz, CDCl₃) δ 8.96 (d, $J = 2.2$ Hz, 1H), 8.13 (d, $J = 8.5$ Hz, 1H), 8.11 (d, $J = 2.1$ Hz, 1H), 7.85 (d, $J = 8.1$ Hz, 1H), 7.63 – 7.53 (m, 1H), 6.09 (q, $J = 6.6$ Hz, 1H), 1.68 (d, $J = 6.7$ Hz, 3H), 1.26 (s, 9H); ¹³C NMR (151 MHz, CDCl₃) δ 177.6, 149.2, 147.8, 134.6, 132.9, 129.5, 129.3, 127.9, 127.7, 126.9, 70.1, 38.8, 27.1, 22.0; FTIR (NaCl/thin film) 2977, 2933, 2872, 1728, 1497, 1480, 1281, 1154, 1126, 1067, 909 cm⁻¹; HRMS (EI+) [M]⁺ calculated for C₁₆H₁₉O₂N: 257.1416, found: 257.1411.

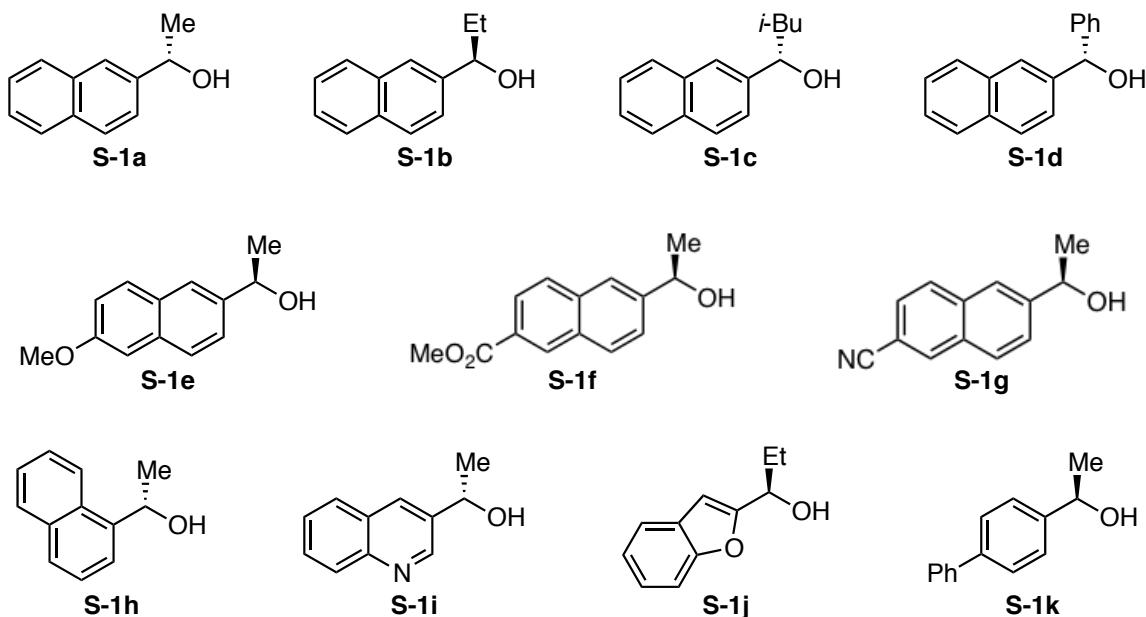


(*R*)-1-(Benzofuran-2-yl)propyl pivalate (1j**).** Prepared according to the General Procedure on a 1.66 mmol scale to give **1j** (320 mg, 74%) as a colorless oil. The enantiomeric excess was determined to be 82% ee by chiral HPLC analysis (CHIRALPAK IC, 1.0 mL/min, 0.3% *i*-PrOH/hexane, λ =254 nm); t_R (major)=9.25 min, t_R (minor)=8.49 min. $[\alpha]_D^{24} = +172.7^\circ$ (c 1.30, CHCl_3): ^1H NMR (600 MHz, CDCl_3) δ 7.58 – 7.54 (m, 1H), 7.51 – 7.46 (m, 1H), 7.33 – 7.21 (m, 2H), 6.67 (s, 1H), 5.91 (t, J = 12.0 Hz, 1H), 2.13 – 2.03 (m, 2H), 1.25 (s, 9H), 0.99 (t, J = 7.4 Hz, 3H); ^{13}C NMR (151 MHz, CDCl_3) δ 177.7, 155.7, 154.8, 127.9, 124.2, 122.7, 121.1, 111.3, 104.2, 70.2, 38.9, 27.1, 26.0, 9.6; FTIR (NaCl/thin film) 2793, 2936, 2877, 1732, 1454, 1396, 1279, 1151, 1083, 1031, 950, 807 cm^{-1} ; HRMS (EI+) [M] $^+$ calculated for $\text{C}_{16}\text{H}_{20}\text{O}_3$: 260.1413, found: 260.1411.



(*R*)-1-([1,1'-Biphenyl]-4-yl)ethyl pivalate (1k**).** Prepared according to the General Procedure on a 1.5 mmol scale to give **1k** (295 mg, 70%) as a white solid (mp 78–82°C). The enantiomeric excess was determined to be 93% ee by chiral HPLC analysis (CHIRALPAK IA, 1.0 mL/min, 0.3% *i*-PrOH/hexane, λ =254 nm); t_R (major)=9.02 min, t_R (minor)=10.74 min. $[\alpha]_D^{24} = +68.7^\circ$ (c 0.82, CHCl_3): ^1H NMR (600 MHz, CDCl_3) δ 7.64 – 7.57 (m, 4H), 7.50 – 7.42 (m, 4H), 7.41 – 7.35 (m, 1H), 5.92 (q, J = 6.6 Hz, 1H), 1.58 (d, J = 6.6 Hz, 3H), 1.26 (s, 9H); ^{13}C NMR (151 MHz, CDCl_3) δ 177.7, 141.2, 140.8, 140.6, 128.8, 127.3, 127.2, 127.1, 126.2, 71.7, 38.8, 27.2, 22.4; FTIR (NaCl/thin film) 3029, 2981, 1720, 1486, 1367, 1284, 1176, 1071, 1009, 836 cm^{-1} ; HRMS (EI+) [M] $^+$ calculated for $\text{C}_{19}\text{H}_{22}\text{O}_2$: 282.1620, found: 282.1629.

Preparation of Benzyl Alcohols



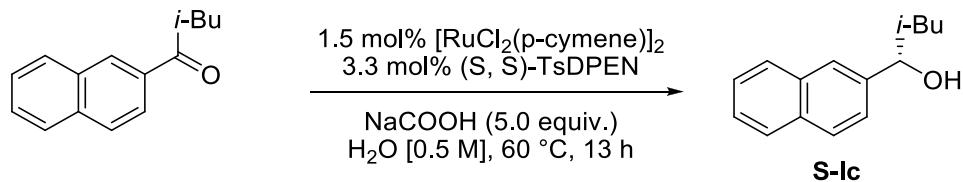
(*S*)-1-(Naphthalen-2-yl)ethanol (**S-1a**) was purchased from Sigma Aldrich. Alcohols **S-1b**,¹² **S-1d**,⁷ and **S-1j**¹² were prepared according to literature procedures using asymmetric addition of diethyl zinc to the aldehydes. The enantiomeric excesses of crystalline compounds **S-1b** and **S-1d** were then increased via recrystallization from hexanes. Alcohols **S-1e**, **S-1f**, **S-1g**, and **S-1k** were prepared via Corey-Bakshi-Shibata reduction of ketones.¹³ The enantiomeric excess of known alcohol **S-1e**¹³ was then increased via trituration with 5% Et₂O/hexanes. Alcohols **S-1c**, **S-1h**,¹⁴ and **S-1i** were prepared by asymmetric transfer hydrogenation of ketones.¹⁴ Because alcohols **S-1c**, **S-1f**, **S-1g**, **S-1i**,

¹² Taylor, B. L. H.; Swift, E. C.; Waetzig, J. D.; Jarvo, E. R. *J. Am. Chem. Soc.* **2011**, *133*, 389.

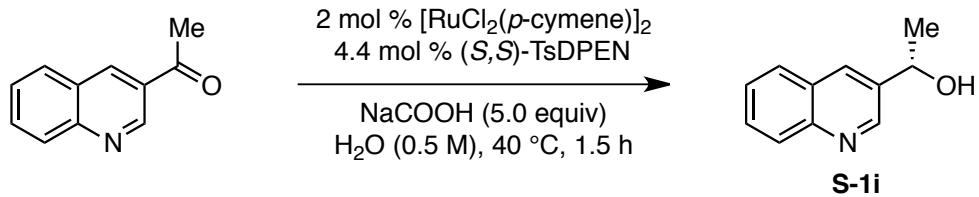
¹³ Bakshi, R. K.; Shibata, S.; Chen, C.; Singh, V. K.; Corey, E. J. *J. Am. Chem. Soc.* **1987**, *109*, 7925.

¹⁴ Wu, X.-F.; Li, X.-G.; Hems, W.; King, F.; Xiao, J.-L. *Org. Biomol. Chem.* **2004**, *2*, 1818.

and **S-1k** have not been prepared via these methods previously, we have included experimentals for our procedures below.

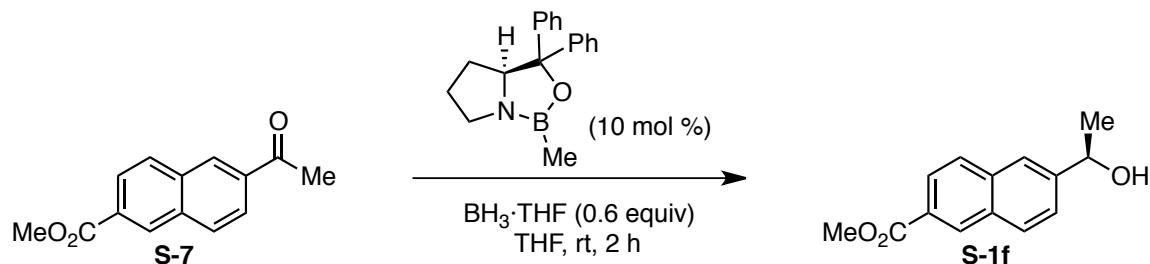


(S)-3-Methyl-1-(naphthalene-2-yl)butan-1-ol (S-1c). The following procedure is adapted from the literature.¹⁴ In a N₂-atmosphere glovebox, [RuCl₂(*p*-cymene)]₂ (21.9 mg, 0.0358 mmol, 1.5 mol %) and (S,S)-TsDPEN (28.8 mg, 0.0787 mmol, 3.3 mol %) were weighed into a vial. The vial was then capped and removed from glovebox. H₂O (degassed by sparging with N₂, 6.0 mL) was added, and the resulting mixture was stirred at 40 °C for 1 h. 3-Methyl-1-(naphthalene-2-yl)butan-1-one (387.0 mg, 2.39 mmol, 1.0 equiv) and NaCOOH (811.4 mg, 11.9 mmol, 5.0 equiv) were added. The vial was evacuated and refilled with N₂ three times and then heated at 60 °C for 13 h. The reaction mixture was diluted with Et₂O (10 mL) and filtered through silica gel, which was then rinsed with additional Et₂O (10 mL x 2). The combined organic layers were concentrated. The resulting residue was purified by silica gel chromatography (15% Et₂O/hexanes) and then recrystallized (hexanes) to give compound **S-1c** (238.3 mg, 61%, 93% ee) as a white solid. The spectral data for this compound matches that reported in literature.¹⁵



¹⁵ Da, C.; Wang, J.; Yin, X.; Fan, X.; Liu, Y.; Yu, S. *Org. Lett.* **2009**, 24, 5578.

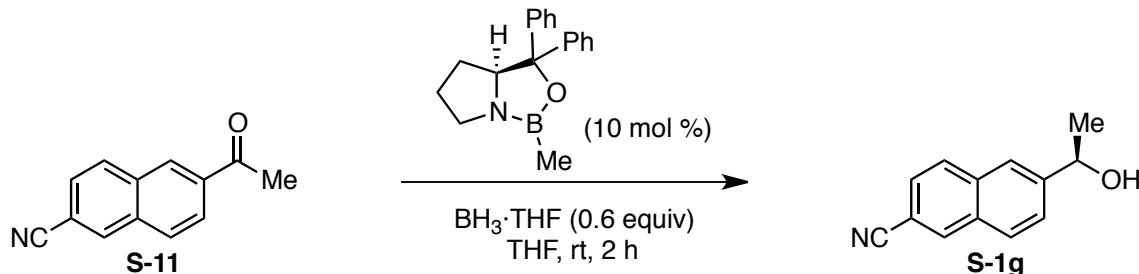
(S)-1-(Quinolin-3-yl)ethanol (S-1i**).** The following procedure is adapted from the literature.¹⁴ In a N₂-atmosphere glovebox, [RuCl₂(*p*-cymene)]₂ (3.58 mg, 0.0058 mmol, 2.0 mol %) and (S,S)-TsDPEN (4.71 mg, 0.0129 mmol, 4.4 mol %) were weighed into a vial. The vial was then capped and removed from glovebox. H₂O (degassed by sparging with N₂, 0.6 mL) was added, and the resulting mixture was stirred at 40 °C for 1 h. 3-Acetylquinoline (50.0 mg, 0.292 mmol, 1.0 equiv) and NaCOOH (99.3 mg, 1.46 mmol, 5.0 equiv) were added. The vial was evacuated and refilled with N₂ three times and then heated at 40 °C for 1.5 h. The reaction mixture was diluted with Et₂O (1 mL) and filtered through silica gel, which was then rinsed with additional Et₂O (1 mL x 2). The combined organic layers were concentrated. The resulting residue was purified by silica gel chromatography (50–90% EtOAc/hexanes) to give compound **S-1i** (45.7 mg, 90%, 85% ee) as a light brown oil. The spectral data for this compound matches to that reported in literature.¹⁶



(R)-Methyl 6-(1-hydroxyethyl)-2-naphthoate (S-1f**).** The following procedure is adapted from the literature.¹³ A solution of CBS catalyst (83.2 mg, 0.30 mmol, 10 mol %) and THF (5.0 mL) was slowly added to a solution of ketone **S-7** (685 mg, 3.0 mmol, 1.0 equiv), BH₃·THF (1 M in THF, 1.8 mL, 1.8 mmol, 0.60 equiv), and THF (55 mL). After stirring at room temperature for 2 h, H₂O (30 mL) was added. The mixture was extracted

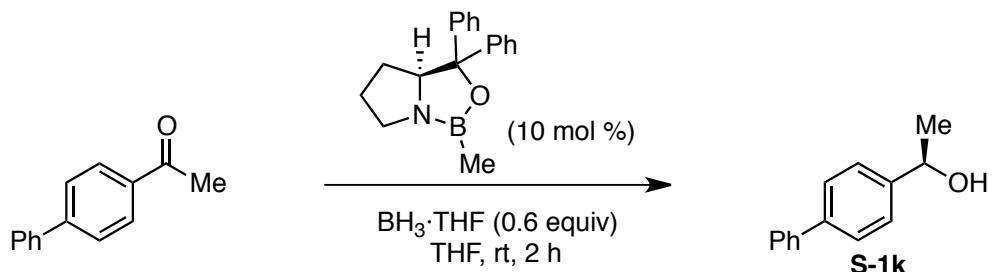
¹⁶ Legros, J.; Toffano, M.; Drayton, S. K.; Rivard, M.; Fiaud, J. *Tetrahedron Lett.* **1997**, 38, 1915.

with Et₂O (30 mL x 3). The combined organic layers were then dried (MgSO₄), filtered, concentrated. The resulting residue was purified by silica gel chromatography (40% EtOAc/hexanes) to give alcohol **S-1f** (460 mg, 67%) as a white solid (mp 74–77 °C): ¹H NMR (600 MHz, CDCl₃) δ 8.58 (s, 1H), 8.05 (dd, *J* = 8.6, 1.7 Hz, 1H), 7.93 (d, *J* = 8.5 Hz, 1H), 7.87 (s, 1H), 7.85 (s, 1H), 7.56 (dd, *J* = 8.5, 1.7 Hz, 1H), 5.16 – 5.06 (m, 1H), 3.97 (s, 3H), 1.96 (d, *J* = 3.5 Hz, 1H), 1.58 (d, *J* = 6.5 Hz, 3H); ¹³C NMR (151 MHz, CDCl₃) δ 167.3, 145.8, 135.5, 131.9, 130.8, 129.8, 128.2, 127.3, 125.6, 124.7, 123.6, 70.4, 52.3, 25.3; FTIR (NaCl/thin film) 3279, 2974, 2887, 1715, 1632, 1437, 1290, 1202, 1130, 1073, 898 cm⁻¹; HRMS (CI+) [M+H]⁺ calculated for C₁₄H₁₅O₃: 231.1021, found: 231.1019.



(R)-6-(1-Hydroxyethyl)-2-naphthonitrile (S-1g). The following procedure is adapted from the literature.¹³ A solution of CBS catalyst (30.7 mg, 0.11 mmol, 10 mol %) and THF (2.0 mL) was slowly added to a solution of ketone **S-11** (216.3 mg, 1.1 mmol, 1.0 equiv), BH₃·THF (1 M in THF, 0.66 mL, 0.66 mmol, 0.60 equiv), and THF (20 mL). After stirring at room temperature for 2 h, H₂O (20 mL) was added. The mixture was extracted with Et₂O (15 mL x 3). The combined organic layers were then dried (MgSO₄), filtered, concentrated. The resulting residue was purified by silica gel chromatography (60% EtOAc/hexanes) to give alcohol **S-1g** (202 mg, 93%) as a white solid (mp 75–77 °C): ¹H NMR (400 MHz, CDCl₃) δ 8.18 (s, 1H), 7.93 – 7.88 (m, 3H), 7.67 – 7.60 (m, 2H), 5.09 (q, *J* = 6.5 Hz, 1H), 2.10 (s, 1H), 1.57 (d, *J* = 6.5 Hz, 3H); ¹³C NMR (100 MHz,

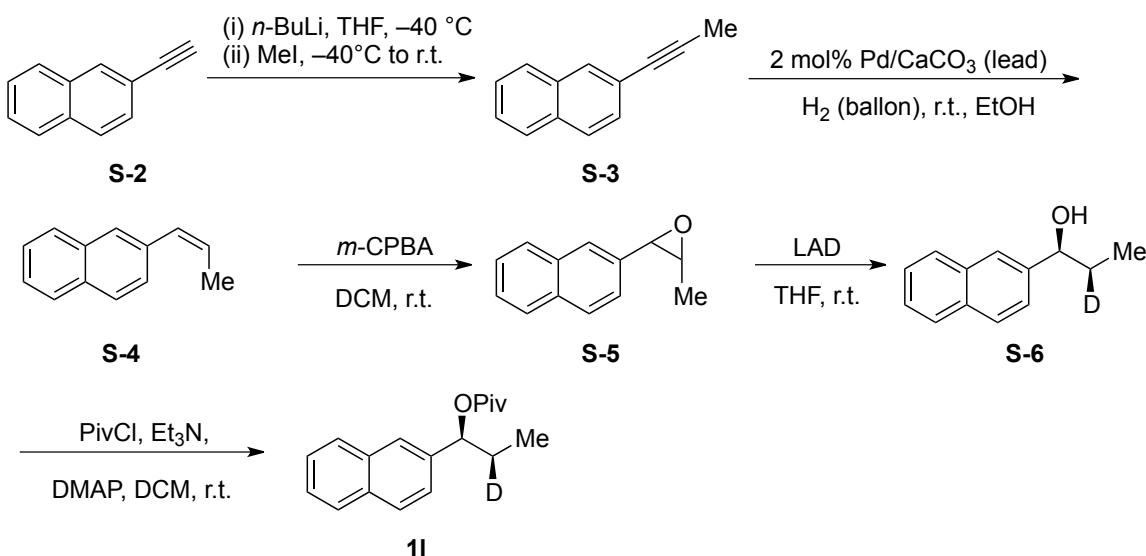
CDCl_3) δ 146.8, 134.7, 133.9, 131.7, 129.2, 128.8, 126.7, 125.8, 123.8, 119.3, 109.1, 70.2, 25.4; FTIR (NaCl/thin film) 3445, 2973, 2926, 2872, 2227, 1632, 1369, 1272, 1159, 1088, 895 cm^{-1} ; HRMS (CI+) $[\text{M}+\text{H}]^+$ calculated for $\text{C}_{13}\text{H}_{12}$ NO: 198.0919, found: 198.0917.



(R)-1-(Biphenyl-4-yl)ethanol (S-1k). The following procedure is adapted from the literature.¹³ A solution of CBS catalyst (97.0 mg, 0.35 mmol, 10 mol %) and THF (5.0 mL) was slowly added to a solution of 4-acetyl biphenyl (687.0 mg, 3.50 mmol, 1.0 equiv), $\text{BH}_3 \cdot \text{THF}$ (1 M in THF, 2.1 mL, 2.1 mmol, 0.60 equiv), and THF (70 mL). After stirring at room temperature for 2 h, H_2O (50 mL) was added. The mixture was extracted with Et_2O (30 mL x 3). The combined organic layers were then dried (MgSO_4), filtered, concentrated. The resulting residue was purified by recrystallization (hexanes) to give compound S-1k (640.7 mg, 92%, 93% ee) as a white solid. The spectral data for this compound matches that reported in the literature for the racemic compound.¹⁷

¹⁷ Inagaki, T.; Phong, L.; Furuta, A.; Ito, J.; Nishiyama, H. *Chem Eur J.* **2010**, *16*, 3090.

Preparation of Pivalate **1I**



2-(Prop-1-ynyl)naphthalene (S-3**).** The following procedure is adapted from the literature.¹⁸ A solution of terminal alkyne **S-2** (507.8 mg, 3.38 mmol, 1.0 equiv) and THF (20 mL) was cooled to -40°C in a 50-mL round-bottomed flask. *n*-BuLi (2.5 M in Hexanes, 2.7 mL, 6.76 mmol, 2.0 equiv) was added. After stirring at -40°C for 1 h, a solution of iodomethane (0.44 mL, 7.10 mmol, 2.1 equiv) and THF (2.5 mL) was added. The mixture was then stirred for 1 h at room temperature. Sat. aq. NH_4Cl (2 mL) and then water (40 mL) was added, and the mixture was extracted with Et_2O (30 mL x 3). The combined organic layers were then dried (MgSO_4), filtered, and concentrated. The resulting residue was purified by silica gel chromatography (100% petroleum ether) to give compound **S-3** (457.0 mg, 81%) as a colorless oil. The spectral data for compound **S-3** matches that reported in literature.¹⁹

¹⁸ Stuart, D. R.; Bertrand-Laperle, M.; Burgess, K. M. N.; Fagnou, K. *J. Am. Chem. Soc.* **2008**, *130*, 16474.

¹⁹ An, D.-L.; Zhang, Z.-Y.; Orita, A.; Mineyama, H.; Otera, J. *Synlett* **2007**, *12*, 1909.

(Z)-2-(Prop-1-enyl)naphthalene (S-4). The following procedure is adapted from the literature.²⁰ In a 25-mL flask, Pd/CaCO₃ (5%, poisoned with lead, 80.4 mg, 0.0378 mmol 2 mol %) and ethanol (5.0 mL) were combined. The flask was evacuated and refilled with H₂ (balloon) three times, then a solution of alkyne S-3 (314.1 mg, 1.89 mmol, 1.0 equiv) and ethanol (4.5 mL) was added slowly. The reaction mixture was stirred at room temperature for 40 minutes. It was then filtered through Celite and concentrated. The resulting residue was purified by silica gel chromatography (100% petroleum ether) to give S-4 (282.3 mg, 89%) as colorless oil. The ratio of Z:E olefins was 19:1, as determined by ¹H NMR analysis.²¹ This mixture was directly used in next step.

Cis-2-methyl-3-(naphtha-2-yl)oxirane (S-5). The following procedure is adapted from the epoxidation of (E)-2-(prop-1-enyl)naphthalene in literature.²² In a 50-mL flask, olefin S-4 (247.8 mg, 1.34 mmol, 1.0 equiv) and CH₂Cl₂ (5.0 mL) were combined. A solution of *meta*-chloroperoxybenzoic acid (400.0 mg, 1.62 mmol, 1.2 equiv) and CH₂Cl₂ (10 mL) was added. After stirring at 35 °C for 30 min, sat. Na₂SO₃ (6 mL), water (10 mL) and then sat. NaCl were added. The organic layer was separated, dried (Na₂SO₄), filtered, concentrated. The resulting residue was purified by silica gel chromatography (5% Et₂O/hexanes) to give compound S-5 (182.9 mg, 74%) as a colorless oil. The ¹H NMR showed that the ratio of *cis:trans* was 17:1.²³ This mixture was directly used in next step.

²⁰ Brunet, J.-J.; Caubere, P. *J. Org. Chem.* **1984**, *49*, 4058.

²¹ The assignment of olefins was done according to the reported spectral data, see: Dong, D.-J.; Li, Y.; Wang, J.-Q.; Tian, S.-K. *Chem. Comm.* **2011**, *47*, 2158.

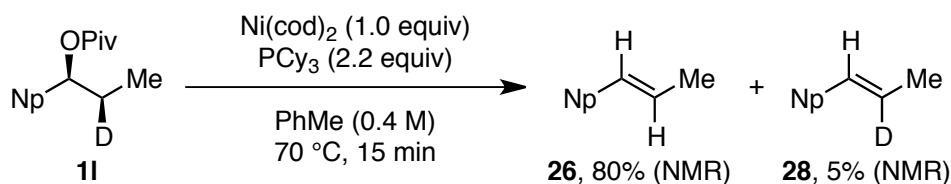
²² Kulasegaram, S.; Kulawiec, R. *J. J. Org. Chem.* **1997**, *62*, 6547.

²³ The assignment of epoxides was done according to the reported spectral data. For *cis*-2-methyl-3-(naphtha-2-yl)oxirane, see: Tian, H.-Q.; She, X.-G.; Shu, L.-H.; Yu, H.-W.; Shi, Y.-A. *J. Am. Soc. Chem.* **2000**, *122*, 11551. For *trans*-2-methyl-3-(naphtha-2-yl)oxirane, see reference 22.

1-(Naphthalene-2-yl)-2-deutero-propan-1-ol (S-6). In a 10-mL flask, lithium aluminum deuteride (105 mg, 2.5 mmol, 2.5 equiv) and THF (2 mL) were combined and cooled to 0 °C. A solution of **S-5** (183.0 mg, 1.0 mmol, 1.0 equiv) and THF (8 mL) was slowly added via addition funnel. After the addition was complete, the reaction mixture was warmed to room temperature and stirred for 2 hours. The reaction was then quenched by the addition of 1 N aq. HCl (5 mL). The organic layer was washed with sat. NaCl, dried (MgSO_4), filtered and concentrated. The resulting residue was purified by silica gel chromatography (17% Et_2O /hexanes) to give compound **S-6** (80.0 mg, 73%) as a colorless oil: ^1H NMR (600 MHz, CDCl_3) δ 7.89 – 7.84 (m, 3H), 7.81 (s, 1H), 7.54 – 7.47 (m, 3H), 4.80 (d, $J = 5.8$ Hz, 1H), 1.96 (s, 1H), 1.91 – 1.83 (m, 1H), 0.97 (d, $J = 7.4$ Hz, 3H); ^{13}C NMR (151 MHz, CDCl_3) δ 141.9, 133.3, 133.0, 128.3, 127.9, 127.7, 126.1, 125.8, 124.7, 124.2, 76.1, 31.4 (t, $J = 19.6$ Hz), 10.0; FTIR (NaCl/thin film) 3357 (br), 3055, 2962, 2931, 2874, 1602, 1508, 1457, 1376, 1270, 1124, 1059, 1017, 857 cm^{-1} ; HRMS (EI+) [M] $^+$ calculated for $\text{C}_{13}\text{H}_{13}\text{OD}$: 187.1106, found: 187.1114.

1-(Naphthalene-2-yl)-2-deutero-propyl pivalate (1l). Prepared according to the General Procedure for pivalate synthesis (see above) on a 0.176 mmol scale to give **1l** (38.2 mg, 80%) as a colorless oil: ^1H NMR (600 MHz, CDCl_3) δ 7.77 – 7.72 (m, 3H), 7.68 (s, 1H), 7.43 – 7.35 (m, 3H), 5.72 (d, $J = 6.0$ Hz, 1H), 1.81 (p, $J = 7.3$ Hz, 1H), 1.16 (s, 9H), 0.85 (d, $J = 7.4$ Hz, 3H); ^{13}C NMR (151 MHz, CDCl_3) δ 177.8, 138.4, 133.2, 133.0, 128.2, 128.0, 127.7, 126.1, 125.9, 125.4, 124.2, 77.2, 38.9, 29.2 (t, $J = 16.5$ Hz), 27.2, 9.9; FTIR (NaCl/thin film) 3057, 2971, 2934, 2874, 1728, 1479, 1459, 1283, 1154, 1125, 856 cm^{-1} ; HRMS (EI+) [M] $^+$ calculated for $\text{C}_{18}\text{H}_{21}\text{O}_2\text{D}$: 271.1683, found: 271.1675.

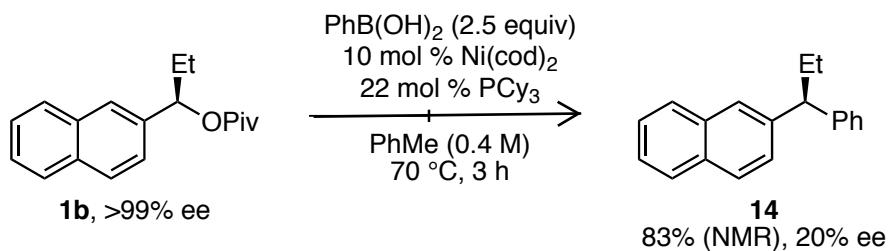
β -Hydride Elimination Experiment



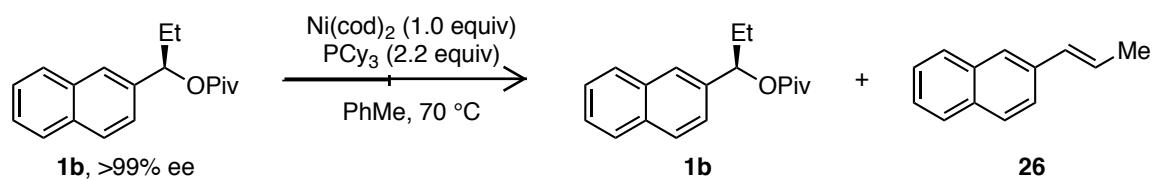
In a N_2 -atmosphere glovebox, $\text{Ni}(\text{cod})_2$ (45.7 mg, 0.166 mmol, 1.0 equiv) and PCy_3 (102.3 mg, 0.365 mmol, 2.2 equiv) were weighed into a 1-dram vial. Pivalate **11** (45.0 mg, 0.166 mmol, 1.0 equiv) was added, followed by toluene (0.41 mL, 0.4 M). The vial was capped with a Teflon-lined cap and removed from the glovebox. The mixture was heated for 15 minutes at 70 °C. The reaction mixture was then diluted with Et_2O (2.0 mL) and filtered through a plug of silica gel, which was rinsed with Et_2O (15 mL). The solution was concentrated. Then 1,3,5-trimethoxybenzene (27.9 mg, 0.166 mmol, 1.0 equiv) was added as an internal standard for ^1H NMR analysis. Based on the ^1H NMR spectrum, pivalate **11** underwent quantitative conversion to give products **26**²¹ (80%) and **28**²⁴ (5%).

Control Experiments for β -Hydride Elimination Experiment

Although the cross coupling proceeds with low levels of chirality transfer using a Ni/PCy_3 catalyst, we have determined that the starting material does not epimerize to a significant extent under the conditions used in the β -hydride elimination experiment.



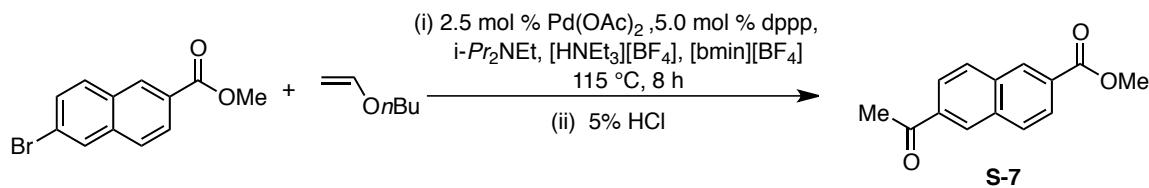
²⁴ Kulasegaram, S.; Kulawiec, R. J. *Tetrahedron* **1998**, *54*, 1361.



entry	time (min)	yield of 1b (%) ^a	ee of 1b (%) ^b	yield of 26 (%) ^a
1	15	0	n.d.	>95
2	1.5	19	95	81

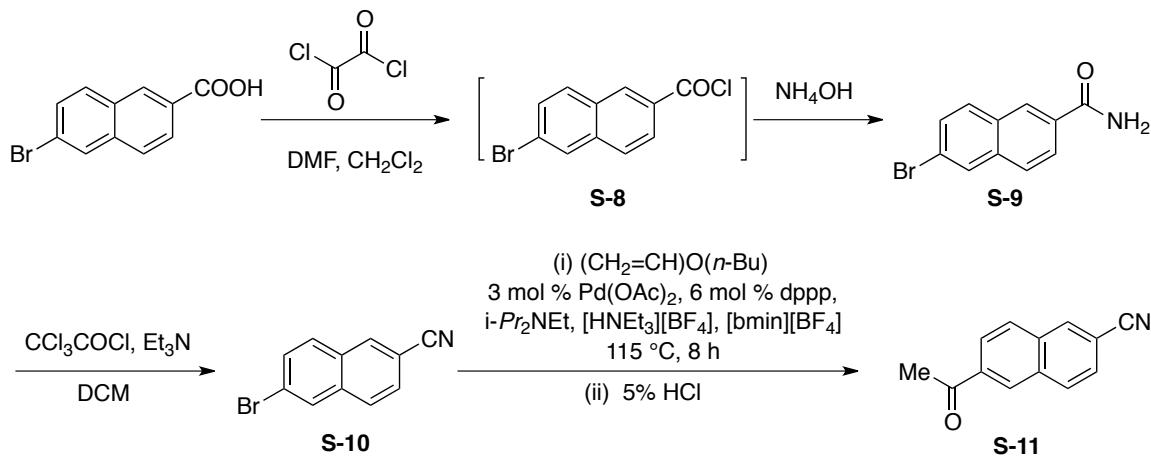
^a Determined by ¹H NMR analysis using 1,3,5-trimethoxybenzene as an internal standard. ^b Determined by chiral HPLC analysis.

Preparation of Ketones S-7 and S-11



Methyl 6-acetylene-2-naphthoate (S-7). The following procedure is adapted from the literature.²⁵ An oven-dried, two-necked round-bottomed flask was charged with methyl 6-bromo-2-naphthoate (1.25 g, 4.72 mmol, 1.0 equiv), Pd(OAc)₂ (26.4 mg, 0.12 mmol, 2.5 mol %), dppp (97.0 mg, 0.24 mmol, 5 mol %), [HNEt₃][BF₄] (1.38 g, 7.06 mmol, 1.5 equiv) and [bmin][BF₄] (10 mL). The flask was then evacuated and refilled with N₂ three times. 1-(Vinyloxy)butane (1.27 mL, 9.43 mmol, 2.0 equiv) and then *i*-Pr₂NEt (1.3 mL, 7.06 mmol, 1.5 equiv) were added via syringe. The reaction mixture heated at 115 °C. After 8 h, the mixture was cooled to room temperature. Aq. HCl (5%, 15 mL) was added, and the mixture was stirred at room temperature for 0.5 h. CH₂Cl₂ (30 mL) was then added, and the layers were separated. The aqueous layer was then extracted with CH₂Cl₂ (2 x 30 mL). The combined organic layers were washed with H₂O until neutral, dried (Na₂SO₄), filtered, and concentrated. The crude material was purified by silica gel chromatography (12% EtOAc/hexanes) to give compound S-7 (700.0 mg, 65%) as a white solid (mp 147–150 °C): ¹H NMR (600 MHz, CDCl₃) δ 8.62 (s, 1H), 8.48 (s, 1H), 8.13 (dd, *J* = 8.6, 1.6 Hz, 1H), 8.08 (dd, *J* = 8.6, 1.7 Hz, 1H), 8.01 (dd, *J* = 8.6, 2.5 Hz, 2H), 4.00 (s, 3H), 2.74 (s, 3H); ¹³C NMR (151 MHz, CDCl₃) δ 197.8, 166.8, 136.3, 134.7, 134.6, 130.6, 129.9, 129.8, 129.7, 129.6, 126.2, 124.7, 52.4, 26.8; FTIR (NaCl/thin film) 3058, 2952, 1711, 1608, 1438, 1360, 1338, 1291, 1264, 1180, 1097, 956 cm⁻¹; HRMS (CI+) [M+H]⁺ calculated for C₁₄H₁₃O₃: 229.0865, found: 229.0869.

²⁵ Mo, J.; Xiao, J.-L. *Angew. Chem. Int. Ed.* **2006**, *45*, 4152.



6-Acetyl 2-naphthonitrile (S-11). In a round-bottomed flask equipped with a nitrogen inlet and an oil bubbler to monitor gas flow, a solution of 6-bromo-2-naphthoic acid (4.0 g, 15.9 mmol, 1.0 equiv), DMF (0.25 mL, 3.18 mmol, 0.2 equiv) and CH_2Cl_2 (40.0 mL) was cooled to 0 °C. Oxalyl chloride (2.05 mL, 23.9 mmol, 1.5 equiv) was then added dropwise via syringe, and the immediate evolution of gas was observed by increased flow through the bubbler. The reaction mixture was stirred at 0 °C until gas flow slowed and was then stirred at room temperature for 1 hour. The resulting solution of acid chloride **S-8** was used in the subsequent step without purification.

After the oil bubbler was removed, the solution of acid chloride **S-8** was cooled to 0 °C. A solution of aq. NH_4OH (25%, 2.15 mL, 55.7 mmol, 3.5 equiv) was added dropwise via syringe. A precipitate immediately formed. The reaction mixture was then stirred at room temperature for 30 min. H_2O (40 mL) was added. The mixture was extracted with EtOAc (60 mL). The organic layer was washed with sat. NaCl (30 mL), dried (MgSO_4), filtered, and concentrated to give amide **S-9** (2.5g, 63%) as a white solid: $^1\text{H NMR}$ (600 MHz, CDCl_3) δ 8.35 – 8.28 (m, 1H), 8.06 (d, $J = 1.9$ Hz, 1H), 7.89 (d, $J = 1.7$ Hz, 1H), 7.87 (d, $J = 1.8$ Hz, 1H), 7.85 – 7.78 (m, 1H), 7.63 (dd, $J = 8.7, 2.0$ Hz, 1H),

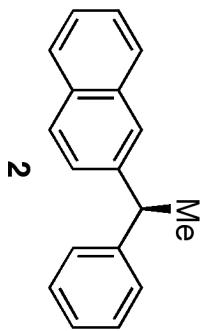
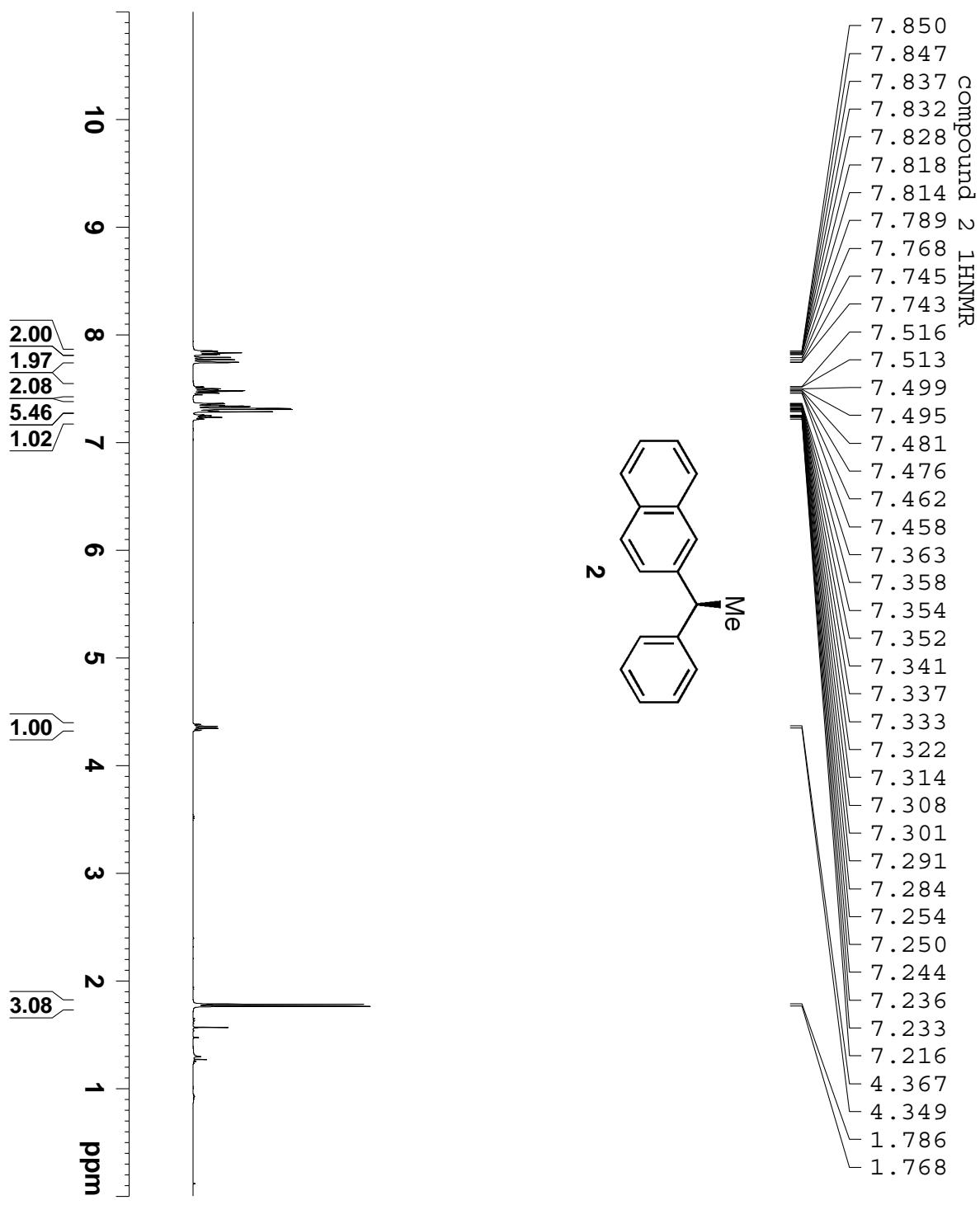
6.20 (s, 1H), 5.72 (s, 1H); ^{13}C NMR (151 MHz, CDCl_3) δ 168.8, 135.9, 131.0, 130.9, 130.6, 130.4, 129.9, 128.1, 127.7, 124.8, 122.3. Amide **S-9** was used in the subsequent step without further purification.

The following procedure is adapted from the literature.²⁶ A mixture of amide **S-9** (2.5 g, 10 mmol, 1.0 equiv), triethylamine (2.3 mL, 22 mmol, 2.2 equiv) and CH_2Cl_2 (30 mL) was cooled to 0 °C. A solution of trichloroacetyl chloride (2.3 mL, 20 mmol, 2.2 equiv) and CH_2Cl_2 (20 mL) was added, while the bath temperature was maintained between 0–5 °C. After the addition was finished, ice-cooled water (25 mL) was added and the layers were separated. The aqueous layer was extracted with CH_2Cl_2 (10 mL). The combined organic layers were then washed with aq. KOH (5%, 30 mL), and the resulting aqueous layer was extracted with CH_2Cl_2 (10 mL). The combined organic layers were then washed with aq. H_2SO_4 (5%, 30 mL), and the resulting aqueous layer was extracted with CH_2Cl_2 (10 mL). Finally, the combined organic layers were washed with H_2O (30 mL), and the resulting aqueous layer was extracted with CH_2Cl_2 (10 mL). The combined organic layers were dried (Na_2SO_4), filtered, and concentrated. The resulting residue was recrystallized (1:1 Et_2O /hexanes) to give **S-10** (1.81 g, 78%) as orange crystals: ^1H NMR (600 MHz, CDCl_3) δ 8.20 (s, 1H), 8.07 (d, J = 1.2 Hz, 1H), 7.83 (d, J = 8.5 Hz, 1H), 7.77 (d, J = 8.8 Hz, 1H), 7.69 (dd, J = 8.8, 1.9 Hz, 1H), 7.64 (dd, J = 8.5, 1.6 Hz, 1H); ^{13}C NMR (151 MHz, CDCl_3) δ 135.5, 134.0, 131.3, 130.7, 130.2, 129.9, 128.3, 127.5, 123.6, 118.8, 109.9.

The following procedure is adapted from the literature.²⁵ An oven-dried, two-necked round-bottomed flask was charged with aryl halide **S-10** (348.0 mg, 1.50 mmol, 1.0 equiv), $\text{Pd}(\text{OAc})_2$ (6.7 mg, 0.09 mmol, 3 mol %), dppp (74.2 mg, 0.18 mmol, 6 mol

²⁶Saednya, A. *Synthesis* **1985**, 184.

%), [HNEt₃][BF₄] (439 mg, 2.25 mmol, 1.5 equiv) and [bmin][BF₄] (2.5 mL). The flask was then evacuated and refilled with N₂ three times. 1-(Vinyloxy)butane (0.33 mL, 3.0 mmol, 2.0 equiv) and then *i*-Pr₂NEt (0.37 mL, 2.25 mmol, 1.5 equiv) were added via syringe. The reaction mixture was heated at 115 °C. After 8 h, the mixture was cooled to room temperature. Aq. HCl (5%, 5 mL) was added, and the mixture was stirred at room temperature for 0.5 h. CH₂Cl₂ (20 mL) was added, and the layers were separated. The aqueous layer was extracted with CH₂Cl₂ (2 x 20 mL). The combined organic layers were washed with H₂O until neutral, dried (Na₂SO₄), filtered, and concentrated. The crude material was purified by silica gel chromatography (20% Et₂O/hexanes) to give compound **S-11** (235.0 mg, 40%) as a yellow solid (mp 134–136 °C): ¹H NMR (600 MHz, CDCl₃) δ 8.49 (s, 1H), 8.27 (s, 1H), 8.15 (dd, *J* = 8.6, 1.6 Hz, 1H), 8.07 (d, *J* = 8.5 Hz, 1H), 7.98 (d, *J* = 8.6 Hz, 1H), 7.69 (dd, *J* = 8.4, 1.5 Hz, 1H), 2.75 (s, 3H); ¹³C NMR (151 MHz, CDCl₃) δ 197.4, 136.9, 134.3, 134.0, 133.8, 130.7, 129.7, 129.0, 127.3, 125.8, 118.7, 111.9, 26.8; FTIR (NaCl/thin film) 3057, 2225, 1686, 1476, 1374, 1368, 1273, 1238, 1189, 902 cm⁻¹; HRMS (Cl+) [M+H]⁺ calculated for C₁₃H₁₀NO: 196.0762, found: 196.0762.

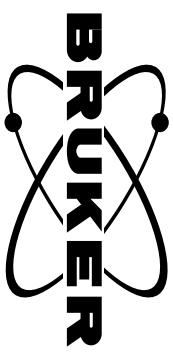


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PROCNO 1

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PULPROG zg30
TD 65536
SOLVENT CDCl₃
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 11.3
DW 60.400 usec
DE 6.00 usec
TE 298.2 K
D1 1.0000000 sec
TD0

===== CHANNEL f1 ======
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P1 15.00 usec
PL1 4.90 dB
PL1W 3.30822015 W
SF01 40.0.1324710 MHz

F2 - Processing parameters
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SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
PC 1.00

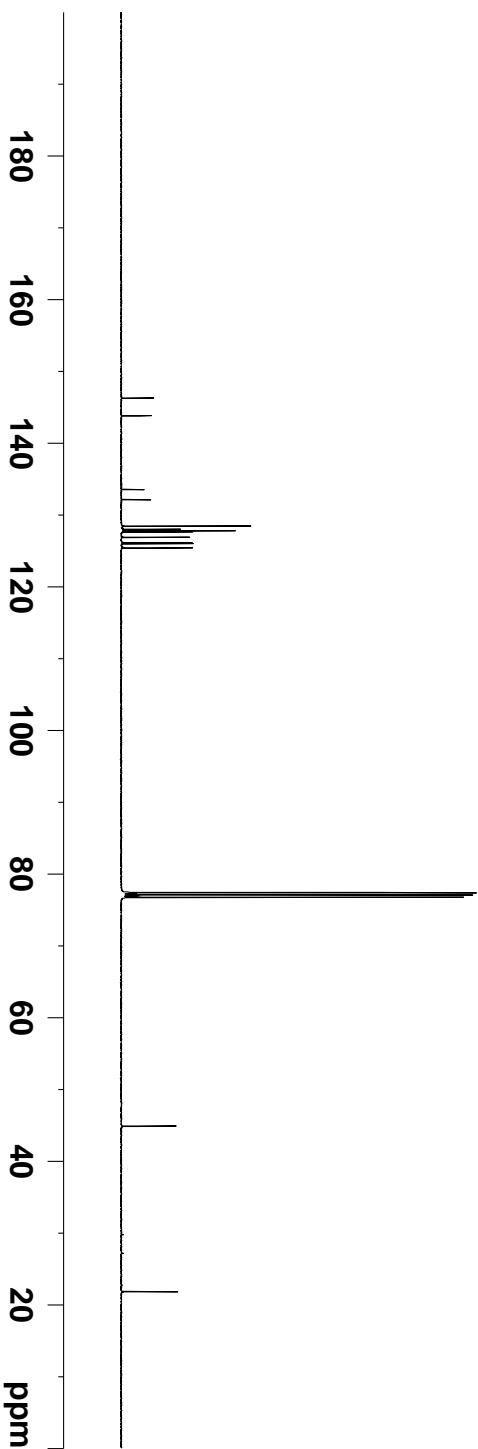
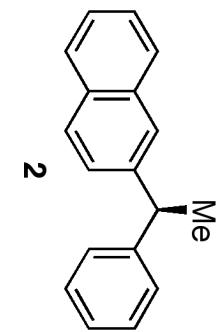


Compound 213CNMR

146.25
143.79
133.52
132.10
128.43
127.98
127.79
127.75
127.60
126.89
126.14
125.98
125.40
125.37

44.87

21.80



F2 - Acquisition Parameters
 Current Data Parameters
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 EXPNO 2
 PROCNO 1

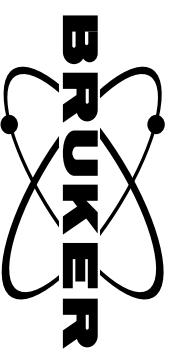
INSTRUM spect
 PROBHD 5 mm CPQNP 1H/
 PULPROG zpg30
 TD 65536
 SOLVENT CDCl3
 NS 256
 DS 4
 SWH 2.3980-814 Hz
 FIDRES 0.365918 Hz
 AQ 1.3664756 sec
 RG 512
 DW 20.850 usec
 DE 18.00 usec
 TE 298.1 K
 D1 2.0000000 sec
 D11 0.03000000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 9.25 usec
 PL1 0.55 dB
 PL1W 35.18820572 W
 SFO1 100.6228298 MHz

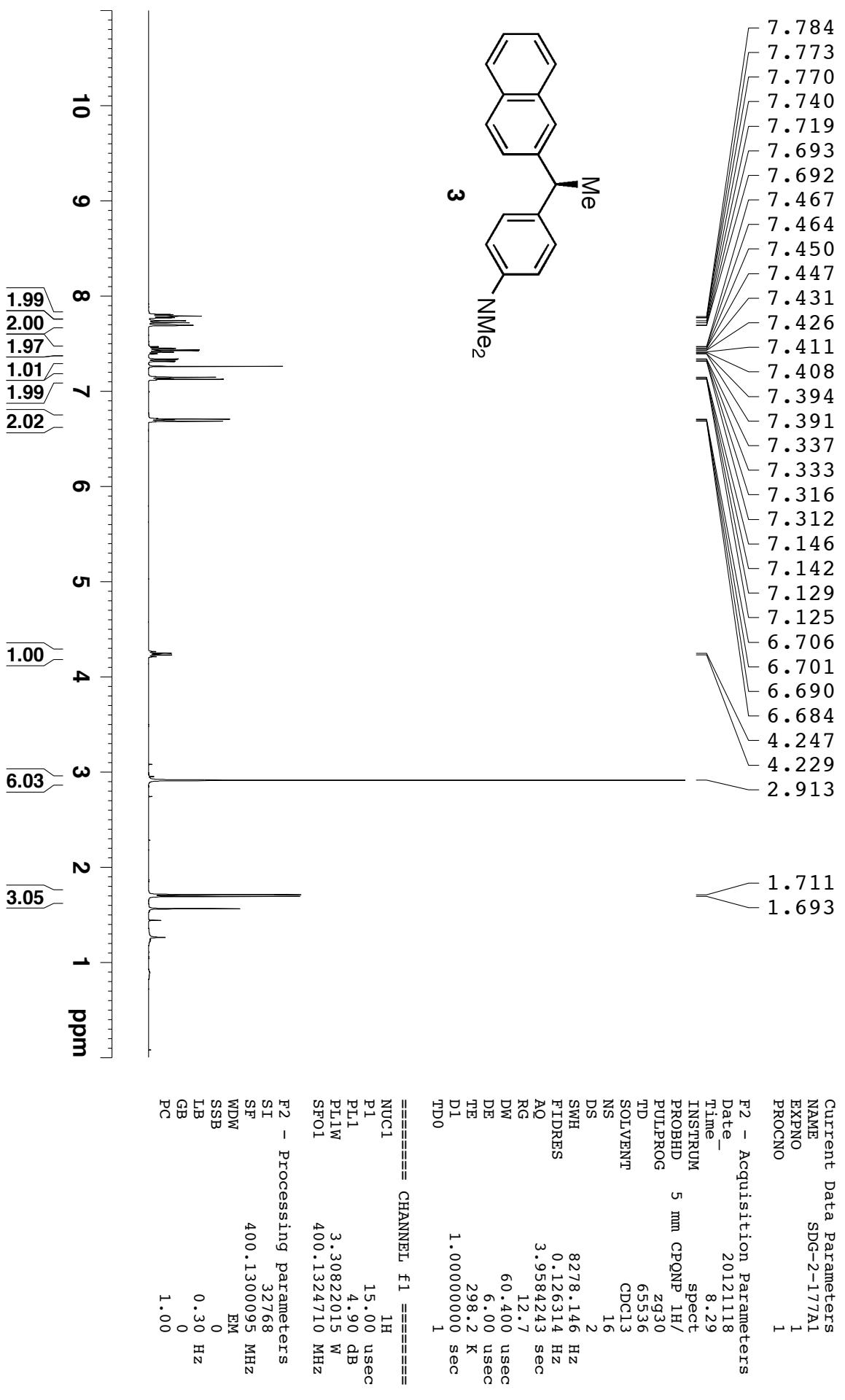
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 PCPD2 90.00 usec
 PL2 4.90 dB
 PL12 20.46 dB
 PL13 21.00 dB
 PL2W 3.30822015 W
 PL12W 0.09195905 W
 PL13W 0.08120718 W
 SFO2 400.1316005 MHz

F2 - Processing parameters
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 SP 100.6127690 MHz
 WDM EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Compound 3 1H NMR



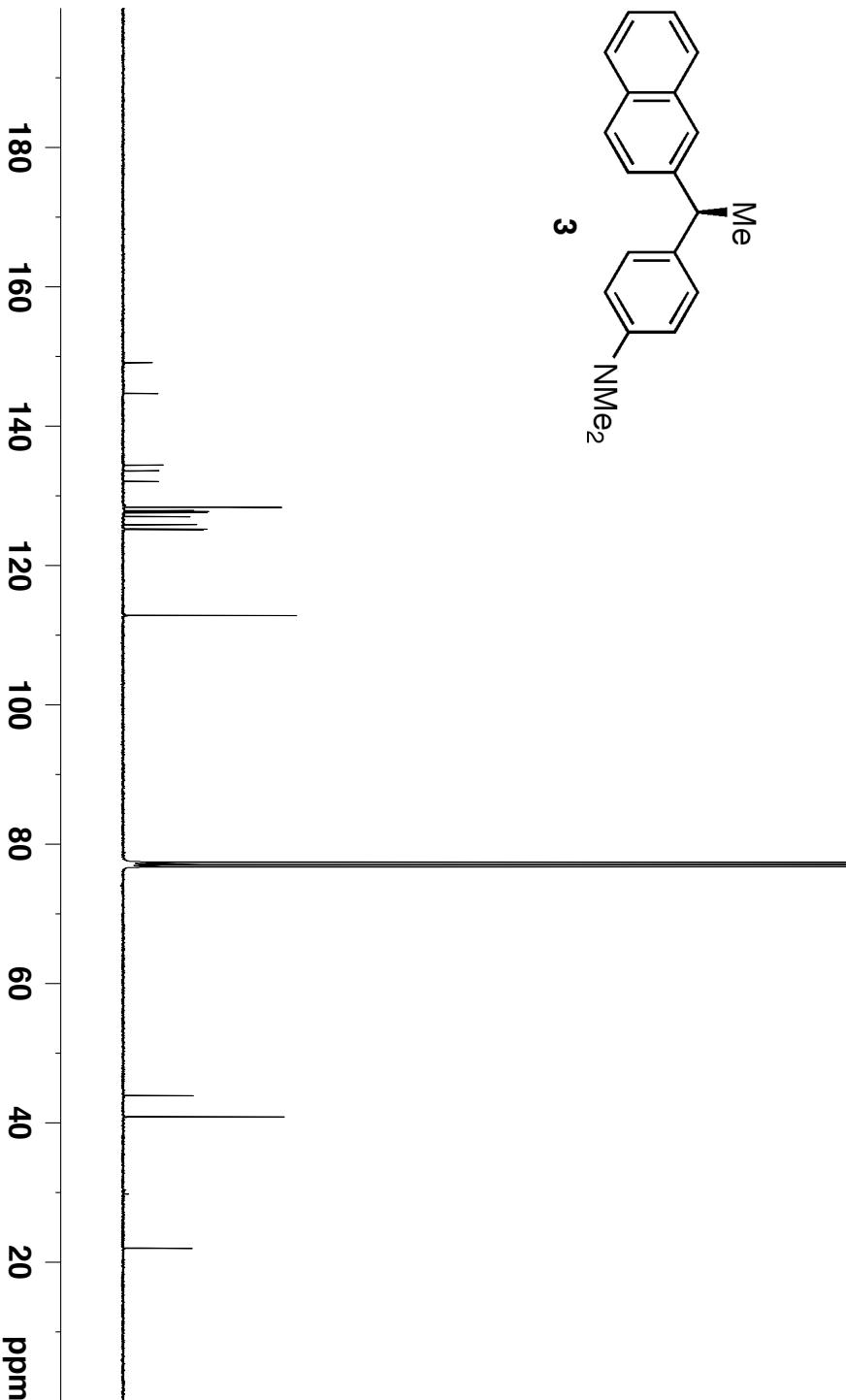
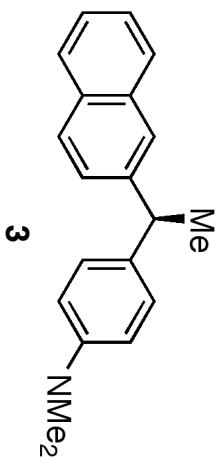
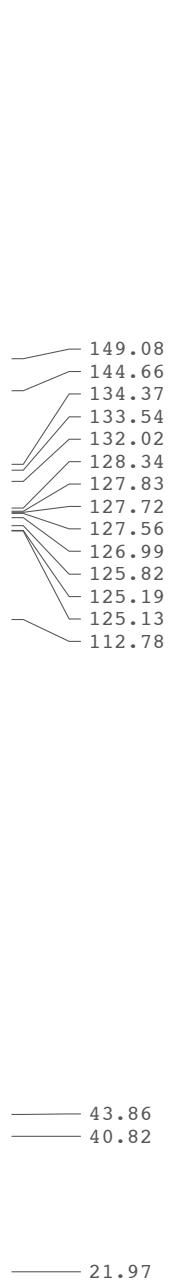
S43



Compound 3 13C NMR



S44



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P1	9.25 usec
PL1	0.55 dB
PL1W	35.18820572 W
SFO1	100.6228298 MHz

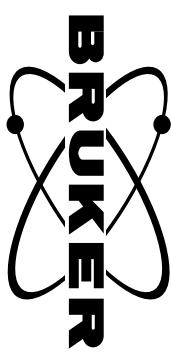
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NUC2	1H
PCPD2	90.00 usec
PL2	4.90 dB
PL12	20.46 dB
PL13	21.00 dB
PL2W	3.30822015 W
PL12W	0.09195905 W
PL13W	0.08120718 W
SFO2	400.1316005 MHz

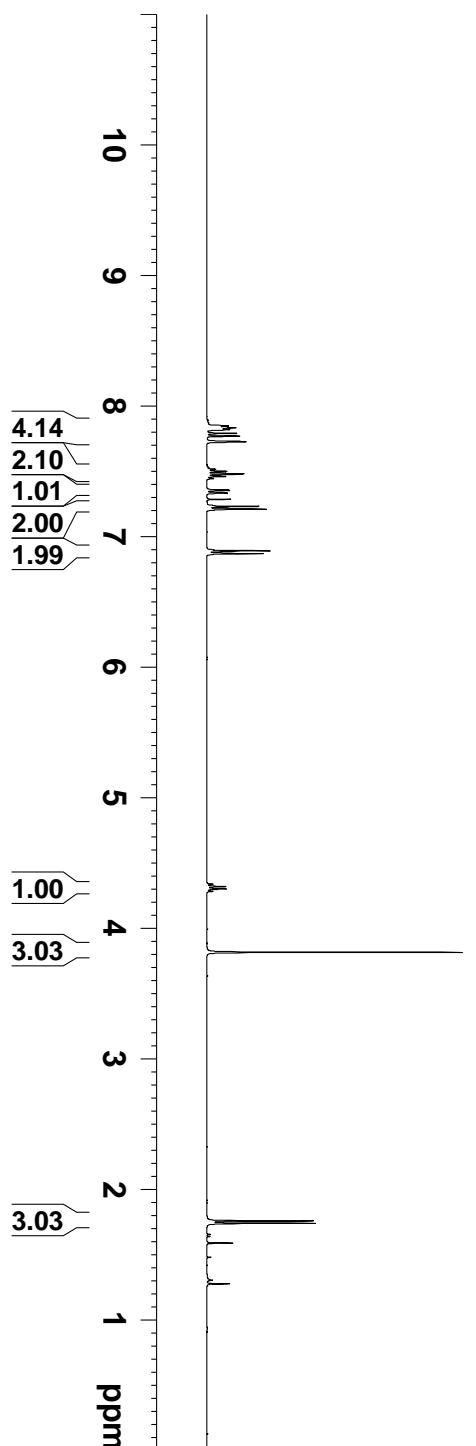
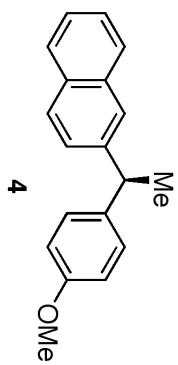
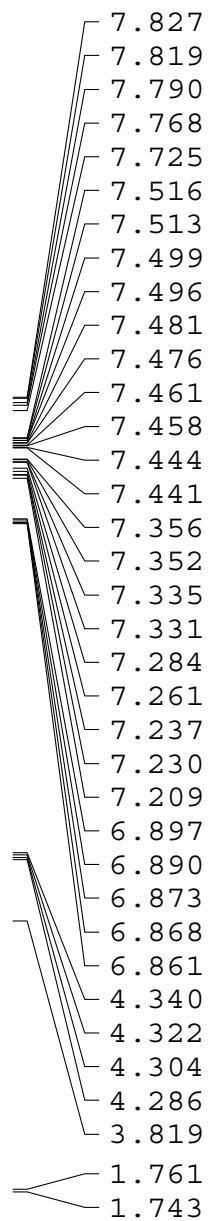
F2 - Processing parameters

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LB	1.00 Hz
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PC	1.40

Compound 4 1H NMR



S45



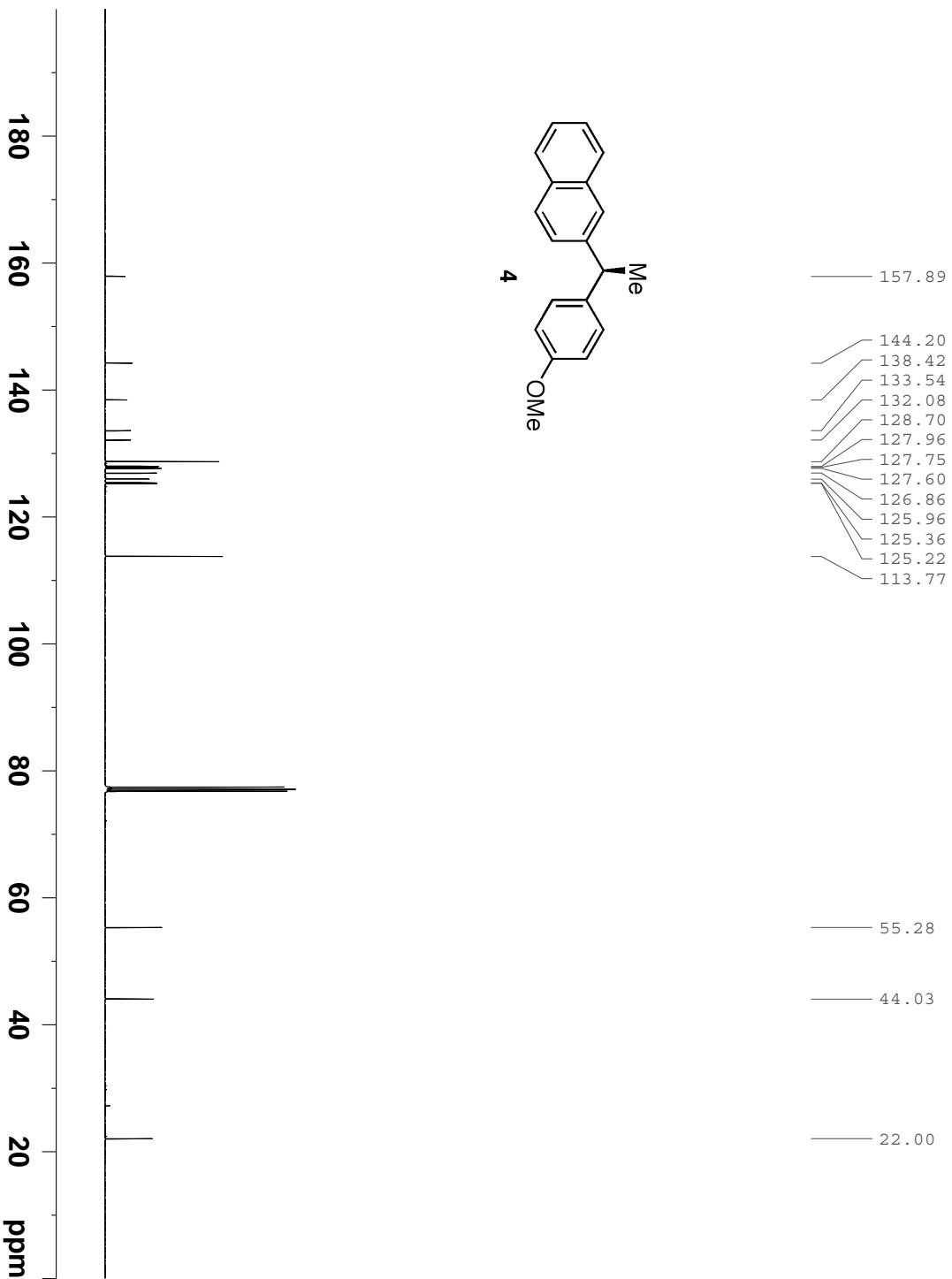
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 P1 15.00 usec
 PL1 4.90 dB
 PL1W 3.30822015 W
 SFO1 40.0.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 1.00
 PC

Compound 4 13C NMR



S46



```

Current Data Parameters
NAME HH-4-049A
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121115
Time 23.19
INSTRUM spect
PROBHD 5 mm CPQNP 1H/
PULPROG zpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 2.3980-814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 512
DW 20.850 usec
DE 18.000 usec
TE 298.1 K
D1 2.0000000 sec
D11 0.03000000 sec
TD0 1

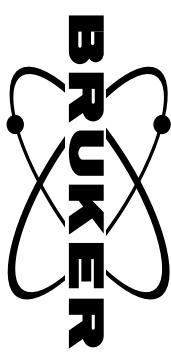
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NUC1 13C
P1 9.25 usec
PL1 0.55 dB
PL1W 35.18820572 W
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
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NUC2 1H
PCPD2 90.00 usec
PL2 4.90 dB
PL12 20.46 dB
PL13 21.00 dB
PL2W 3.30822015 W
PL12W 0.09195905 W
PL13W 0.08120718 W
SFO2 400.1316005 MHz

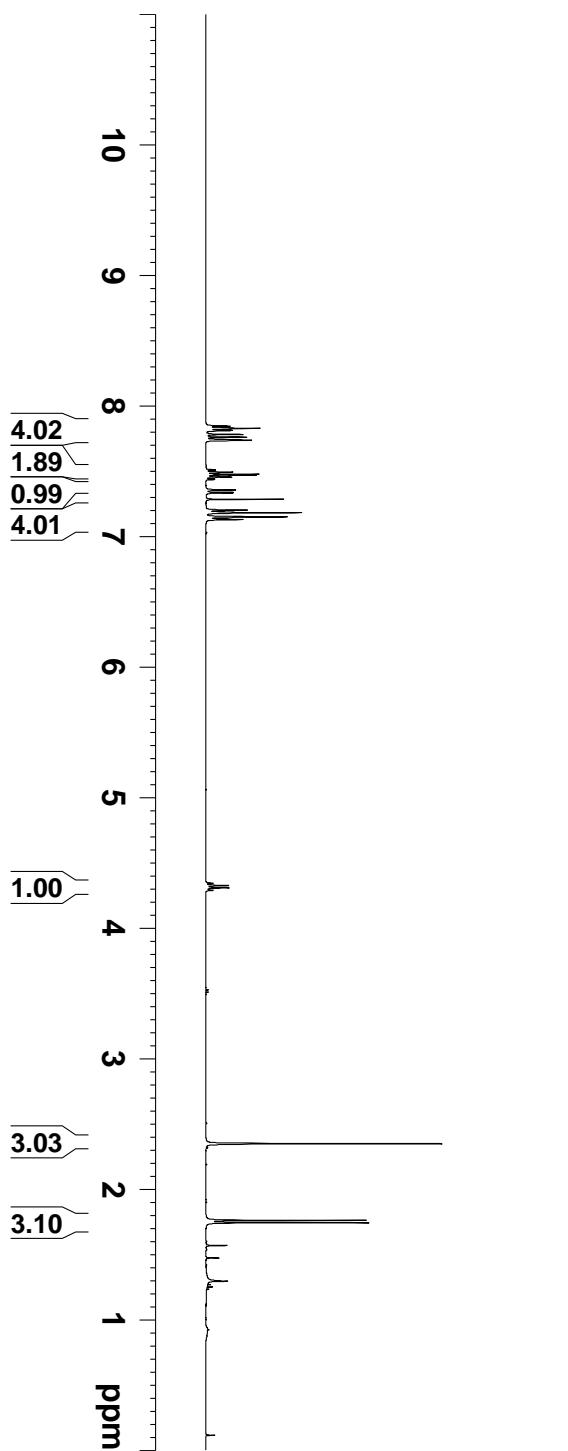
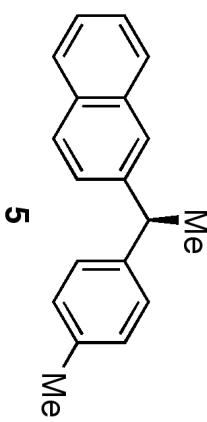
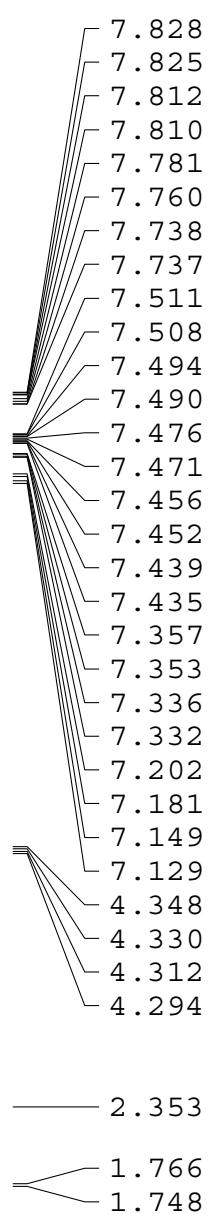
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

```

Compound 5 1H NMR



S47



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

NUC1	1H
P1	15.00 usec
PL1	4.90 dB
PL1W	3.30822015 W
SFO1	40.0.1324710 MHz

F2 - Processing parameters

SI	32768
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	1.00
PC	

Compound 5 13C NMR



S48



Current Data Parameters
NAME HH-4-047A
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121104
Time 20.59
INSTRUM spect
PROBHD 5 mm CPQNP 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 2.3980-814 Hz
FIDRES 0.366518 Hz
AQ 1.3664756 sec
RG 512
DW 20.850 usec
DE 18.000 usec
TE 298.2 K
D1 2.0000000 sec
D11 0.03000000 sec
TD0 1

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

NUC1 13C

P1 9.25 usec

PL1 0.55 dB

PL1W 35.18820572 W

SFO1 100.6228298 MHz

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

CPDPRG2 waltz16

NUC2 1H

PCPD2 90.00 usec

PL2 4.90 dB

PL12 20.46 dB

PL13 21.00 dB

PL2W 3.30822015 W

PL12W 0.09195905 W

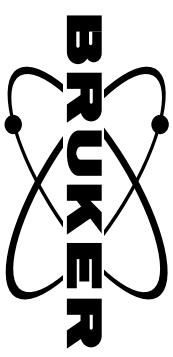
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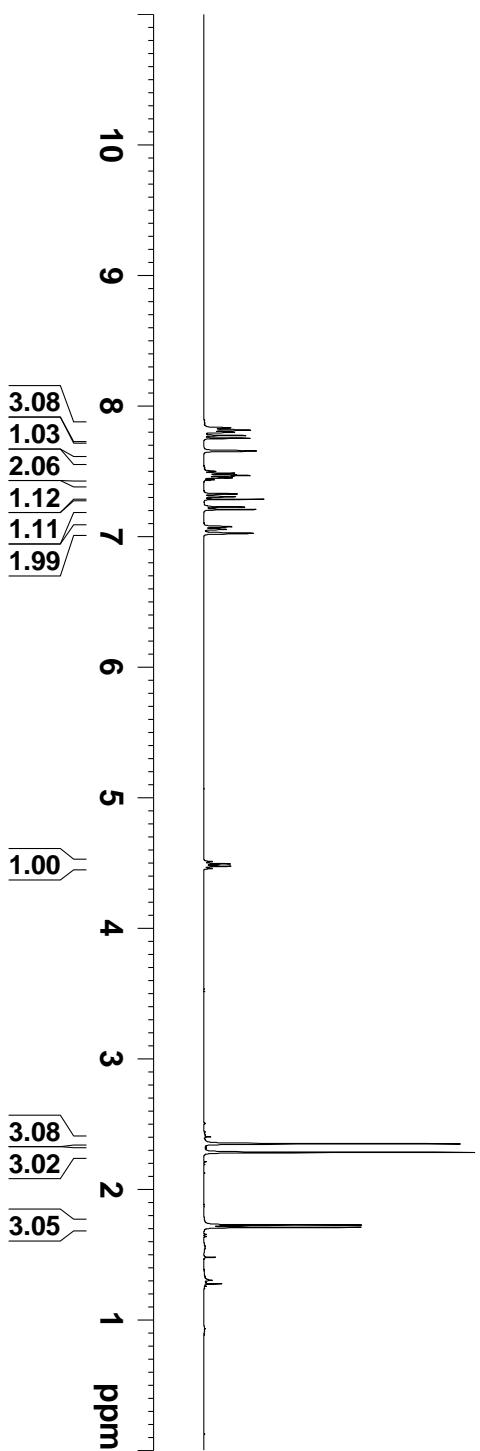
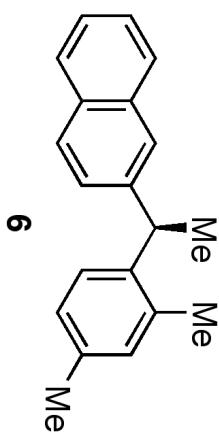
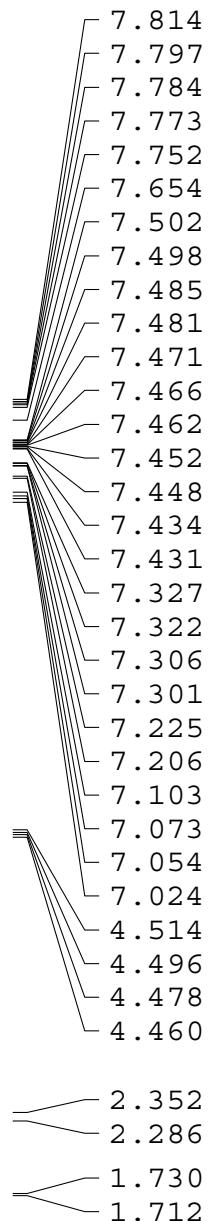
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160
140
120
100
80
60
40
20
ppm

F2 - Processing parameters
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WDW EM
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LB 1.00 Hz
GB 0
PC 1.40

Compound 6 1H NMR



S49



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

NUC1 1H
 P1 15.00 usec
 PL1 4.90 dB
 PL1W 3.30822015 W
 SFO1 40.0.1324710 MHz

F2 - Processing parameters

SI 32768
 SF 400.1300000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 1.00
 PC

Compound 6 13CNMR

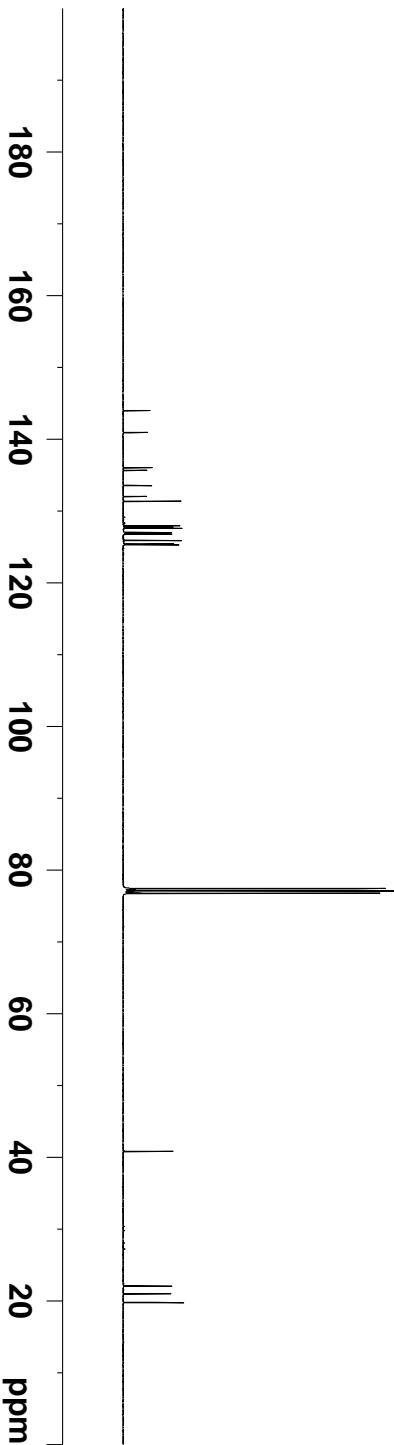
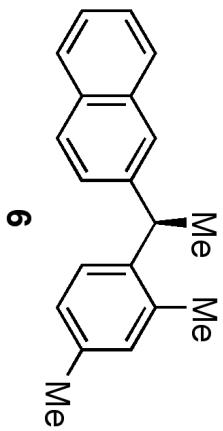


S50

143.95
140.89
136.00
135.63
133.53
132.00
131.33
127.92
127.71
127.58
126.98
126.88
126.74
125.88
125.37
125.27

22.04
20.97
19.75

40.80



Current Data Parameters
NAME QZ-3-060A
EXPNO 2
PROCNO 1

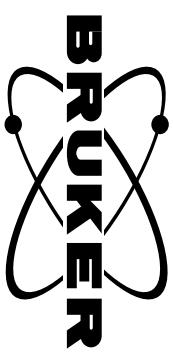
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PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 512
DW 20.850 usec
DE 18.000 usec
TE 298.2 K
D1 2.0000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 9.25 usec
PL1 0.55 dB
PL1W 35.18820572 W
SFO1 100.6228298 MHz

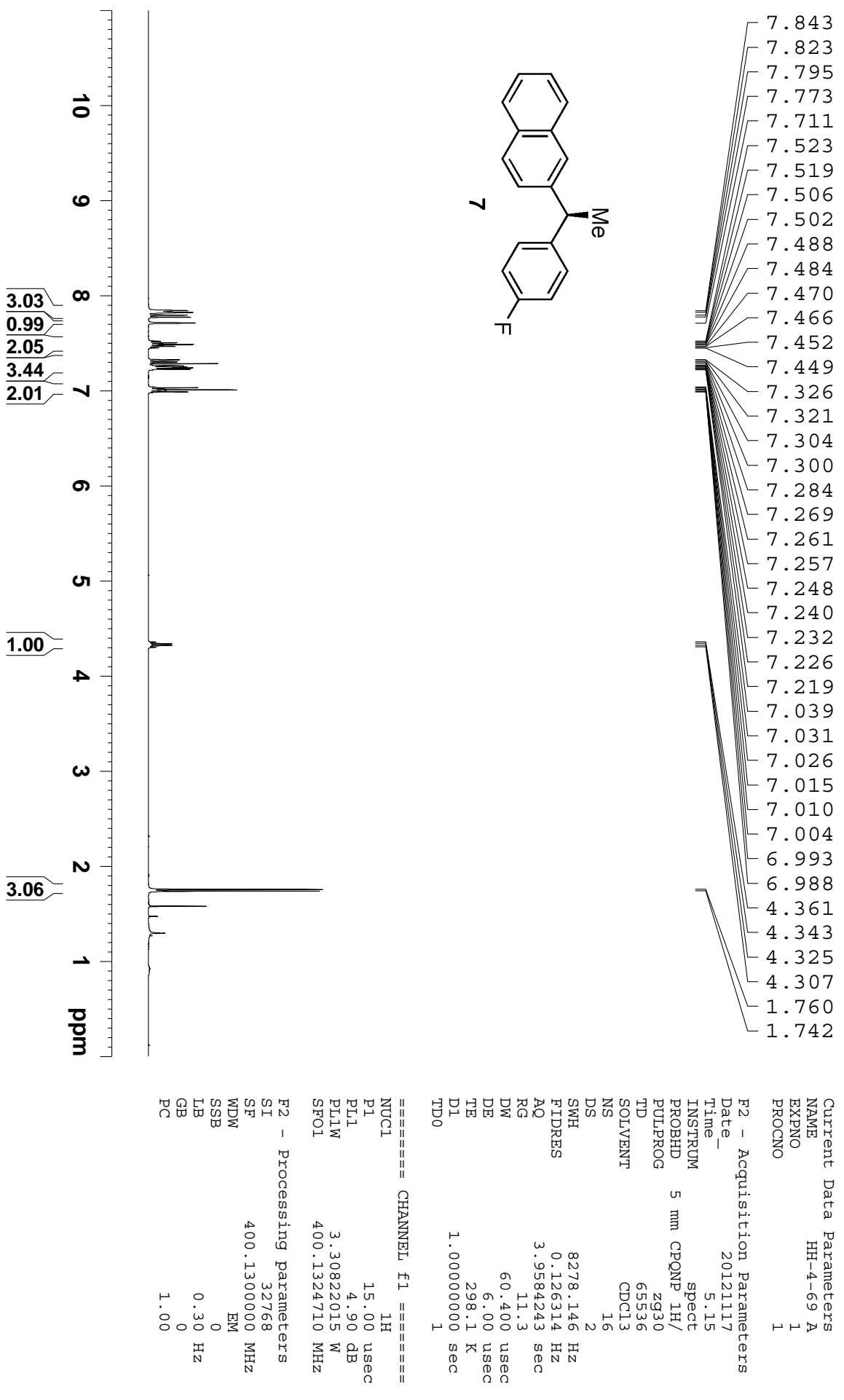
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PCPD2 90.00 usec
PL2 4.90 dB
PL12 20.46 dB
PL13 21.00 dB
PL2W 3.30822015 W
PL12W 0.09195905 W
PL13W 0.08120718 W
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Compound 7 1H NMR



S51



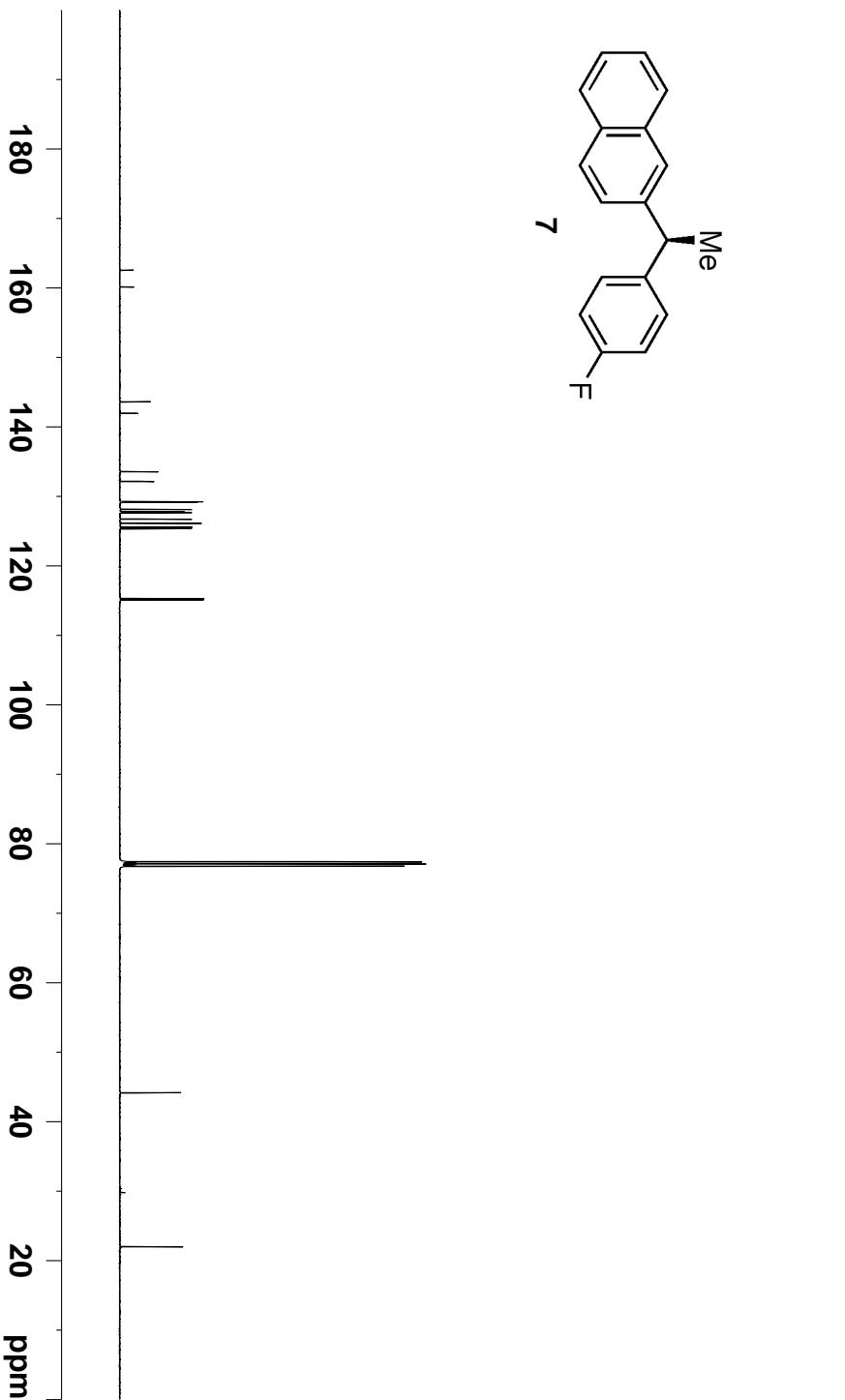
Compound 7 13CNMR



S52

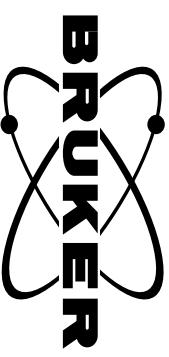
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 TD 65536
 SOLVENT CDCl3
 NS 256
 DS 4
 SWH 23.980-814 Hz
 FIDRES 0.365918 Hz
 AQ 1.3664756 sec
 RG 512
 DW 20.850 usec
 DE 18.00 usec
 TE 298.2 K
 D1 2.0000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 9.25 usec
 PL1 0.55 dB
 PL1W 35.18820572 W
 SFO1 100.6228298 MHz
 ===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 4.90 dB
 PL12 20.46 dB
 PL13 21.00 dB
 PL2W 3.30822015 W
 PL12W 0.09195905 W
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 SFO2 400.1316005 MHz

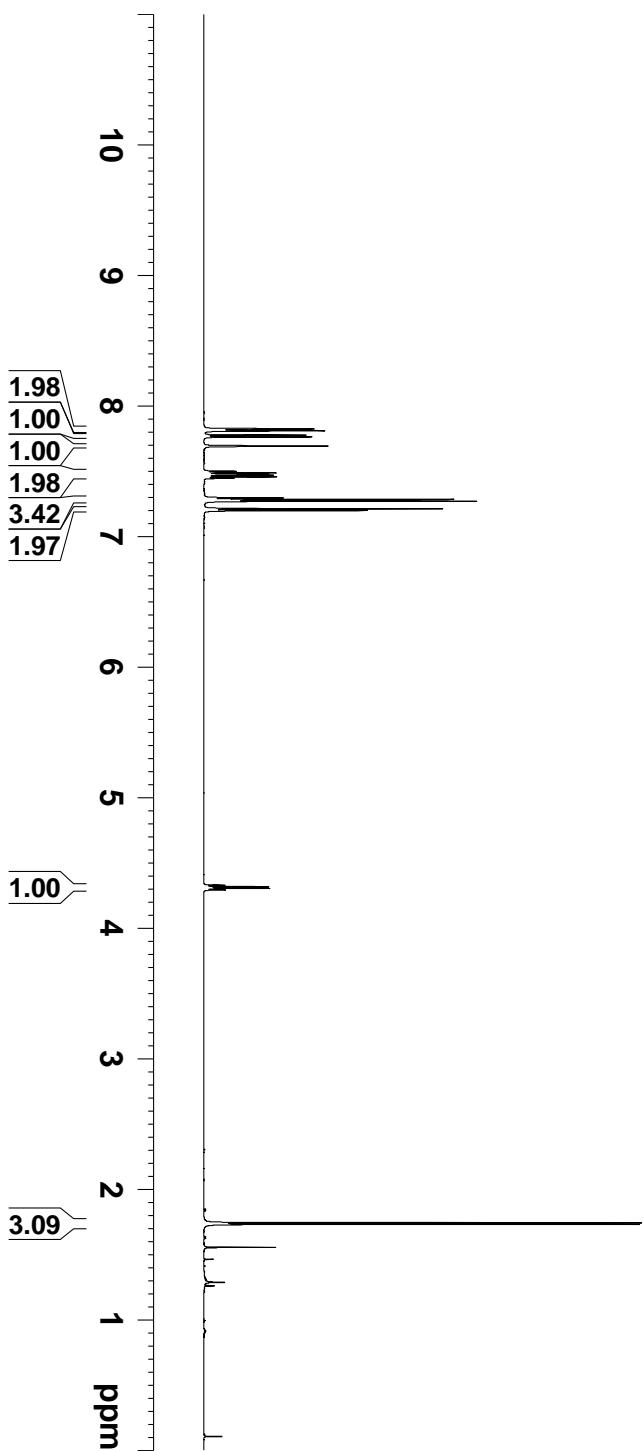
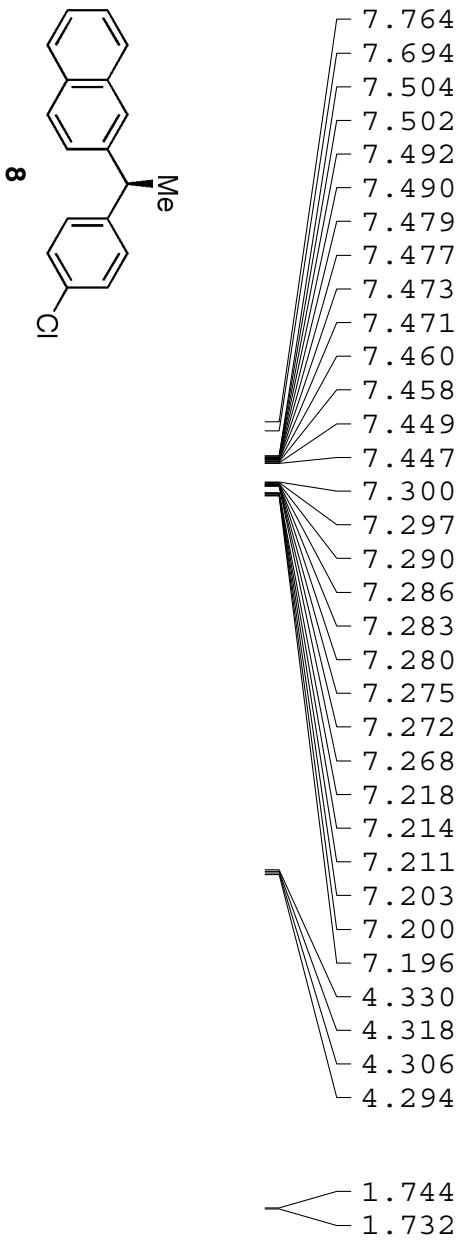


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 PC 1.40

Compound 8 1H NMR



S53

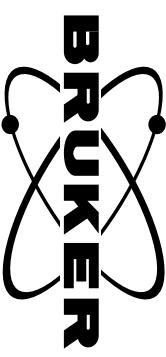


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PROCNO	1	DATE	20121209
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PULPROG	ZG30	PROBHD	
TD	65536	PULPROG	
SOLVENT	CDCl3	TD	
NS	8	SOLVENT	
DS	2	NS	
SWH	8403.361 Hz	DS	
FIDRES	0.128225 Hz	SWH	
AQ	3.8994420 sec	FIDRES	
RG	144	AQ	
DW	59.500 usec	RG	
DE	17.39 usec	DW	
TE	298.1 K	DE	
D1	1.0000000 sec	TE	
TD0		D1	
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NUC1	¹ H		
P1	10.77 usec		

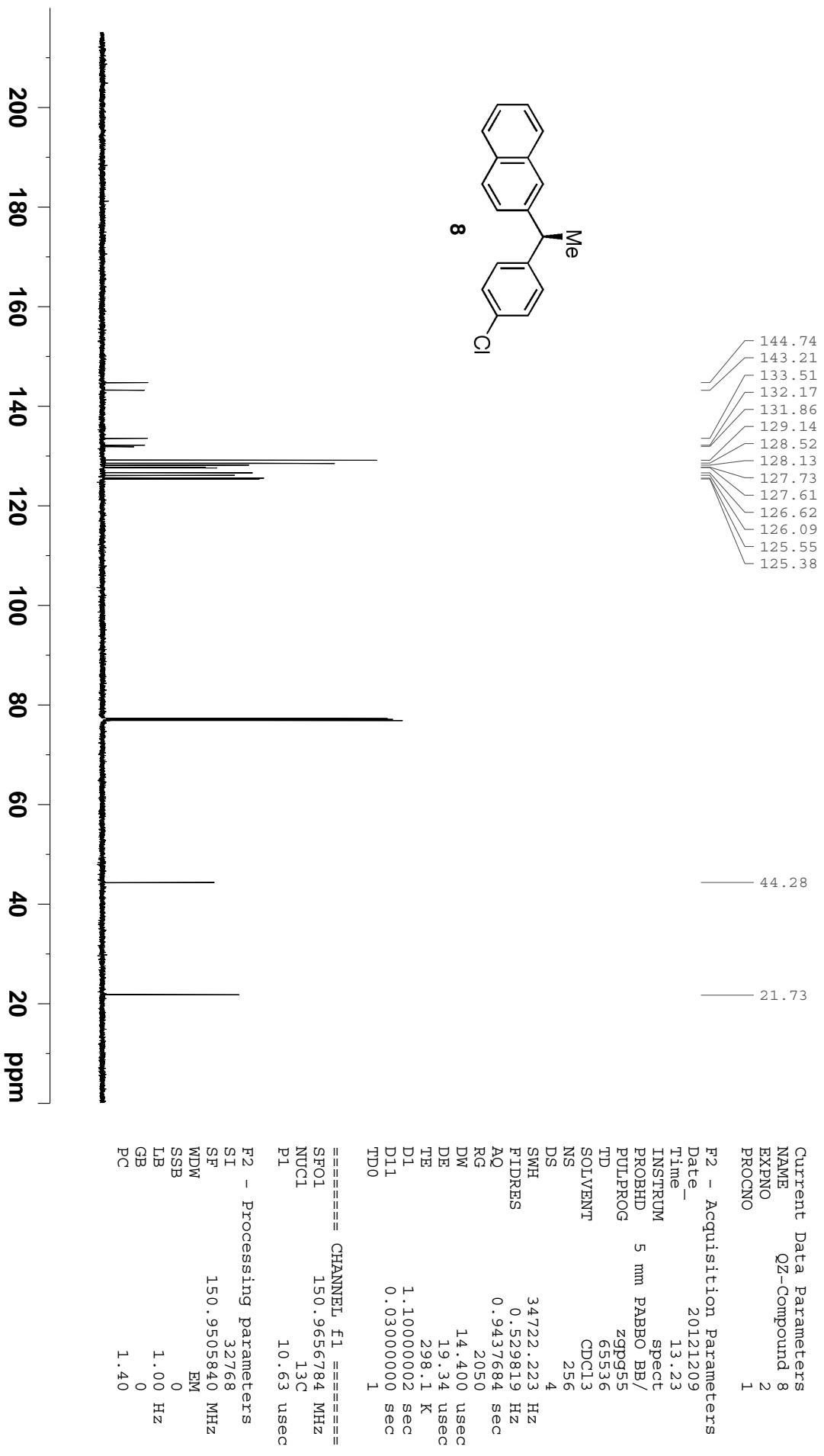
F2 - Processing parameters

SI	SF	WDW	SSB	LB	GB	PC
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Compound 8 13C NMR



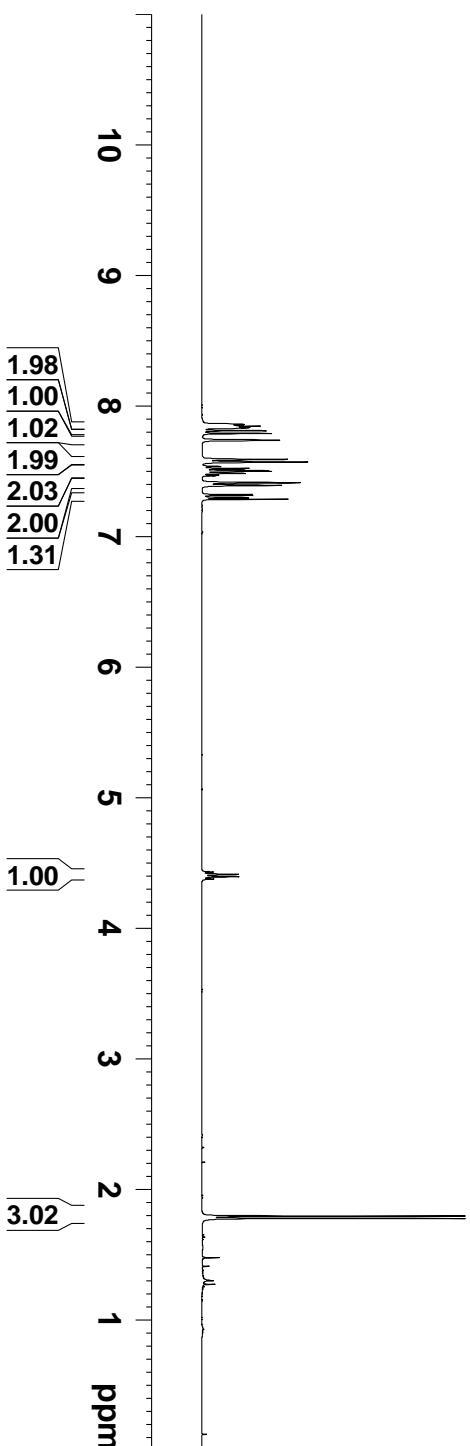
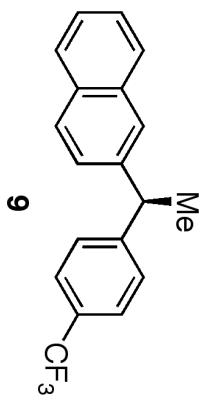
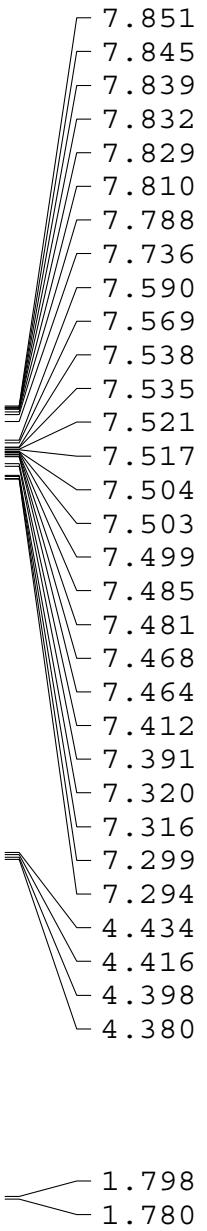
S54



Compound 9 1H NMR



S55



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

NUC1	1H
P1	15.00 usec
PL1	4.90 dB
PL1W	3.30822015 W
SFO1	40.0.1324710 MHz

F2 - Processing parameters

SI	32768
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
PC	1.00

Compound 9 13CNMR

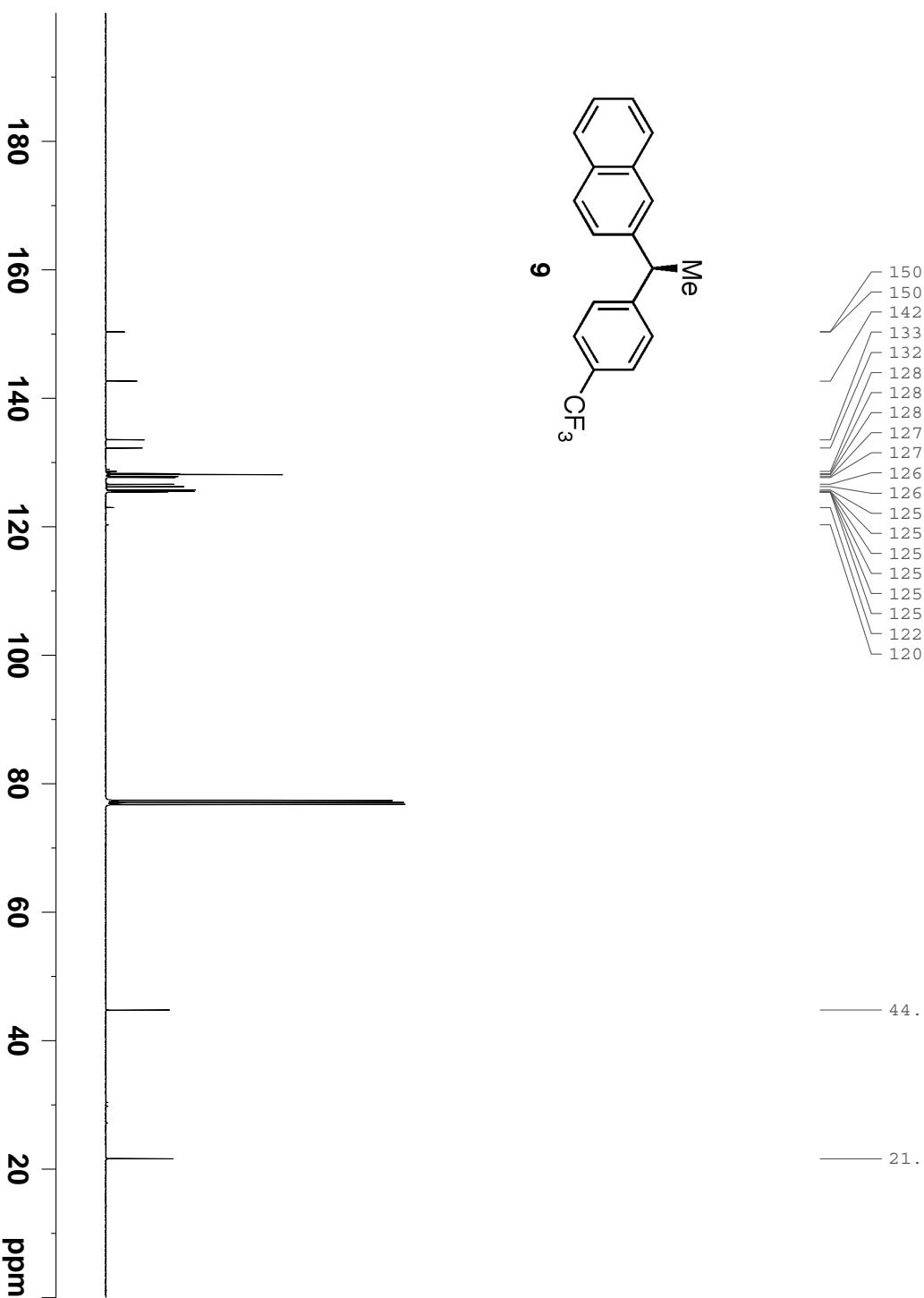
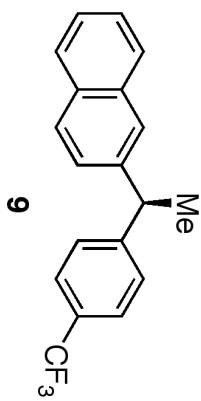


S56

150.33
150.32
142.65
133.50
132.22
128.59
128.26
128.11
127.76
127.65
126.58
126.20
125.69
125.51
125.45
125.41
125.37
125.34
122.96
120.26

44.75

21.59

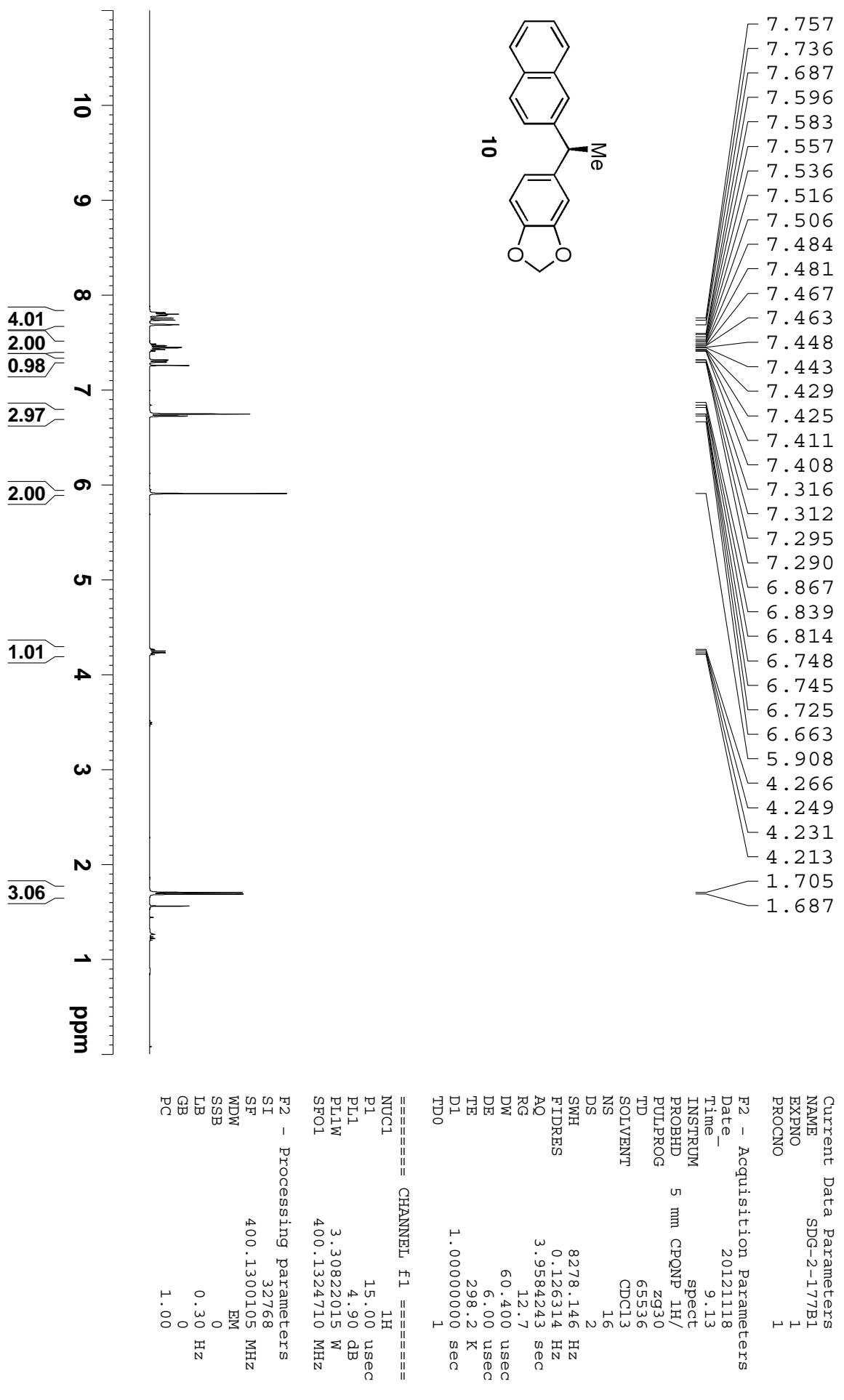
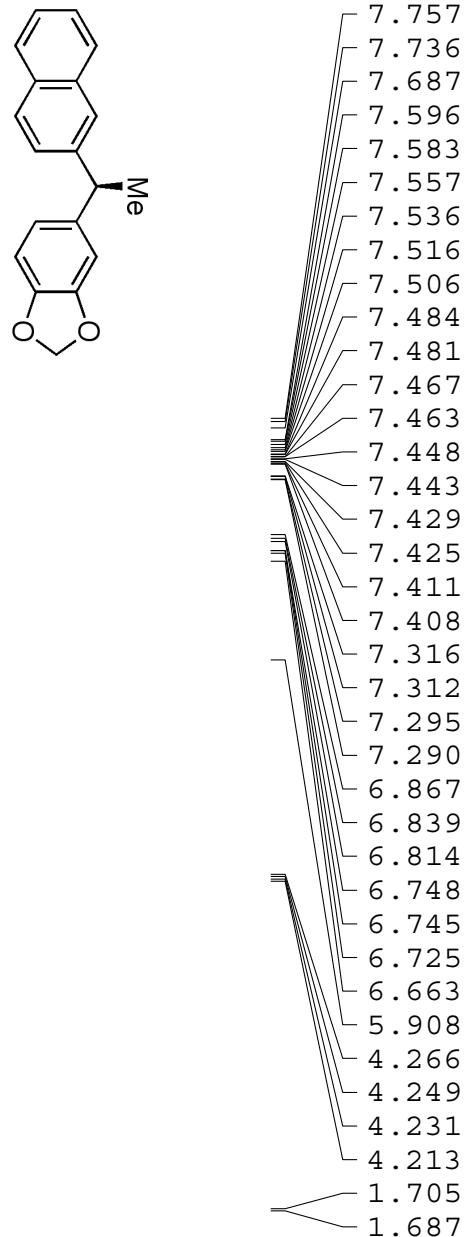


	Current	Data	Parameters
NAME	QZ-3-061A		
EXPNO	2		
PROCNO	1		
F2 - Acquisition Parameters			
INSTRUM	spect		
PROBHD	5 mm	CPQNP 1H/	
PULPROG	zgpg30		
TD	65536		
SOLVENT	CDCl ₃		
NS	256		
DS	4		
SWH	23.980-814 Hz		
FIDRES	0.365918 Hz		
AQ	1.3664756 sec		
RG	512		
DW	20.850 usec		
DE	18.000 usec		
TE	298.1 K		
D1	2.0000000 sec		
D11	0.03000000 sec		
TD0	1		
===== CHANNEL f1 =====			
NUC1	13C		
P1	9.25 usec		
PL1	0.55 dB		
PL1W	35.18820572 W		
SFO1	100.6228298 MHz		
===== CHANNEL f2 =====			
CPDPGR2	waltz16		
NUC2	1H		
PCPD2	90.00 usec		
PL2	4.90 dB		
PL12	20.46 dB		
PL13	21.00 dB		
PL2W	3.30822015 W		
PL12W	0.09195905 W		
PL13W	0.08120718 W		
SFO2	400.1316005 MHz		
F2 - Processing parameters			
SI	32768		
SP	100.6127690 MHz		
WDW	EM		
SSB	0		
LB	1.00 Hz		
GB	0		
PC	1.40		

Compound 10 1H NMR



S57



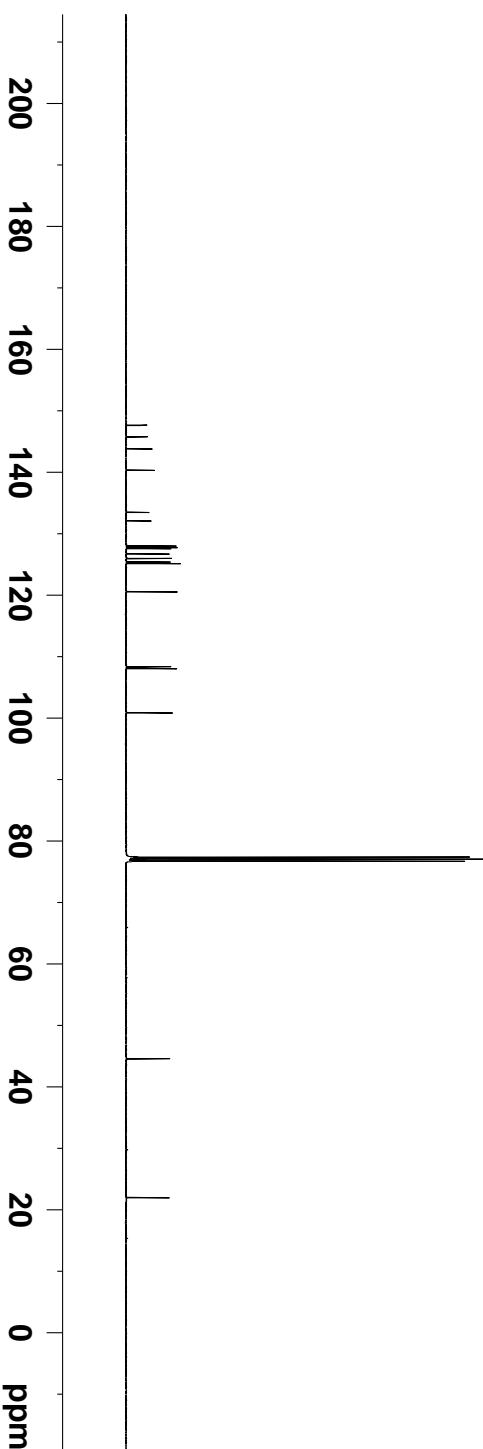
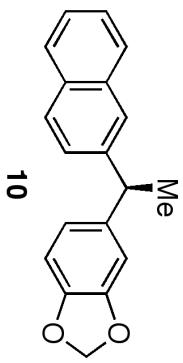
Compound 10 13C NMR



S⁵⁸

147.67
145.78
143.82
140.35
133.51
132.10
128.00
127.74
127.59
126.73
126.00
125.42
125.16
120.55
108.36
108.08
100.87

44.55
21.95



==== CHANNEL f1 =====
 NUC1 13C
 P1 9.25 usec
 PL1 0.55 dB
 PL1W 35.18820572 W
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 4.90 dB
 PL12 20.46 dB
 PL13 21.00 dB
 PL2W 3.30822015 W
 PL12W 0.09195905 W
 PL13W 0.08120718 W
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127690 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Current Data Parameters
 NAME SDG-2-177B1
 EXPNO 2
 PROCNO 1

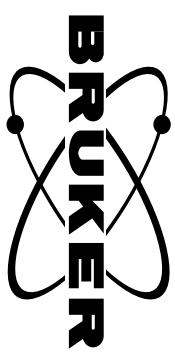
F2 - Acquisition Parameters
 Date 20121118
 Time 9.29
 INSTRUM spect
 PROBHD 5 mm CPQNP 1H/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 256
 DS 4
 SWH 23980.814 Hz
 FIDRES 0.365918 Hz
 AQ 1.3664756 sec
 RG 512
 DW 20.850 usec
 DE 18.00 usec
 TE 298.2 K
 D1 2.0000000 sec
 D11 0.03000000 sec
 TDO 1

==== CHANNEL f1 =====
 NUC1 13C
 P1 9.25 usec
 PL1 0.55 dB
 PL1W 35.18820572 W
 SFO1 100.6228298 MHz

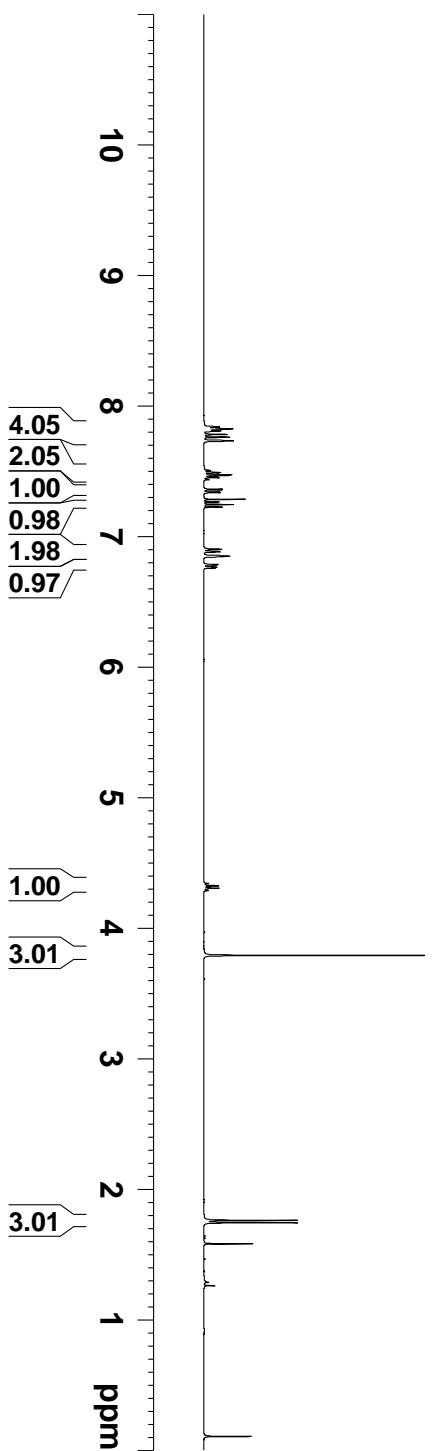
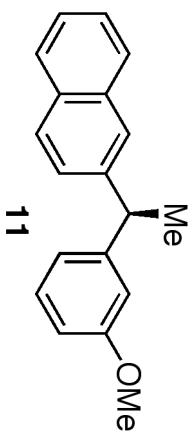
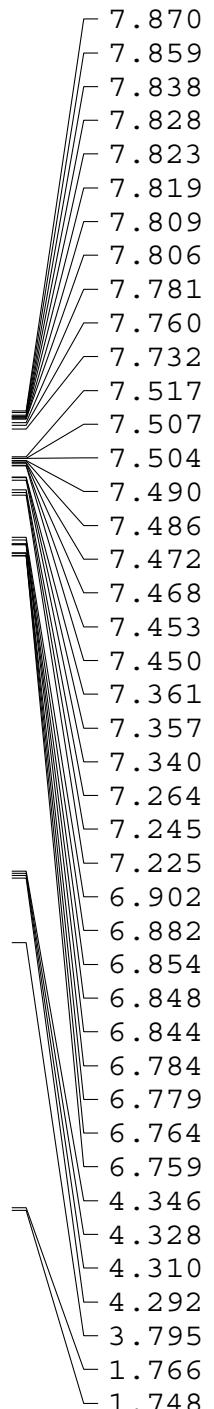
===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 4.90 dB
 PL12 20.46 dB
 PL13 21.00 dB
 PL2W 3.30822015 W
 PL12W 0.09195905 W
 PL13W 0.08120718 W
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127690 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Compound 11 1H NMR



S59



===== CHANNEL f1 =====
 NUC1 1H
 P1 15.00 usec
 PL1 4.90 dB
 PL1W 3.30822015 W
 SFO1 40.01324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 PC 1.00

Compound 11 13CNMR



S60

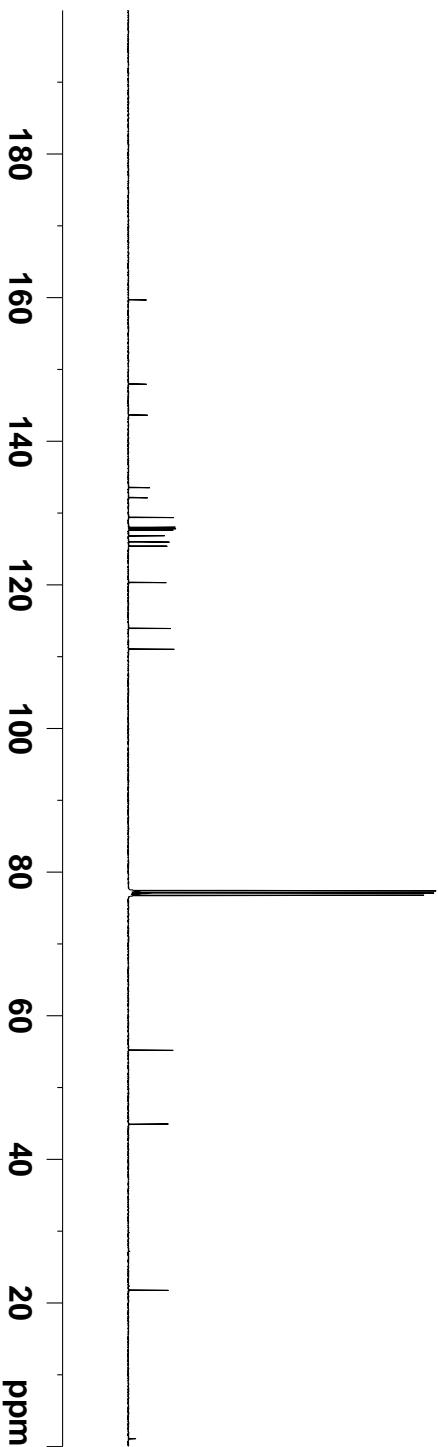
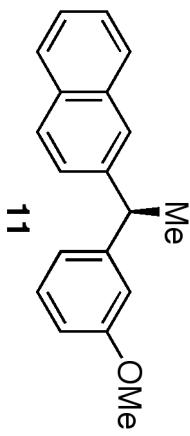
Current Data Parameters
 NAME HH-4-050A
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date 20121115
 Time 23.39
 INSTRUM spect
 PROBHD 5 mm CPQNP 1H/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 256
 DS 4
 SWH 2.3980-814 Hz
 FIDRES 0.365918 Hz
 AQ 1.3664756 sec
 RG 512
 DW 20.850 usec
 DE 18.00 usec
 TE 298.2 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TDO 1

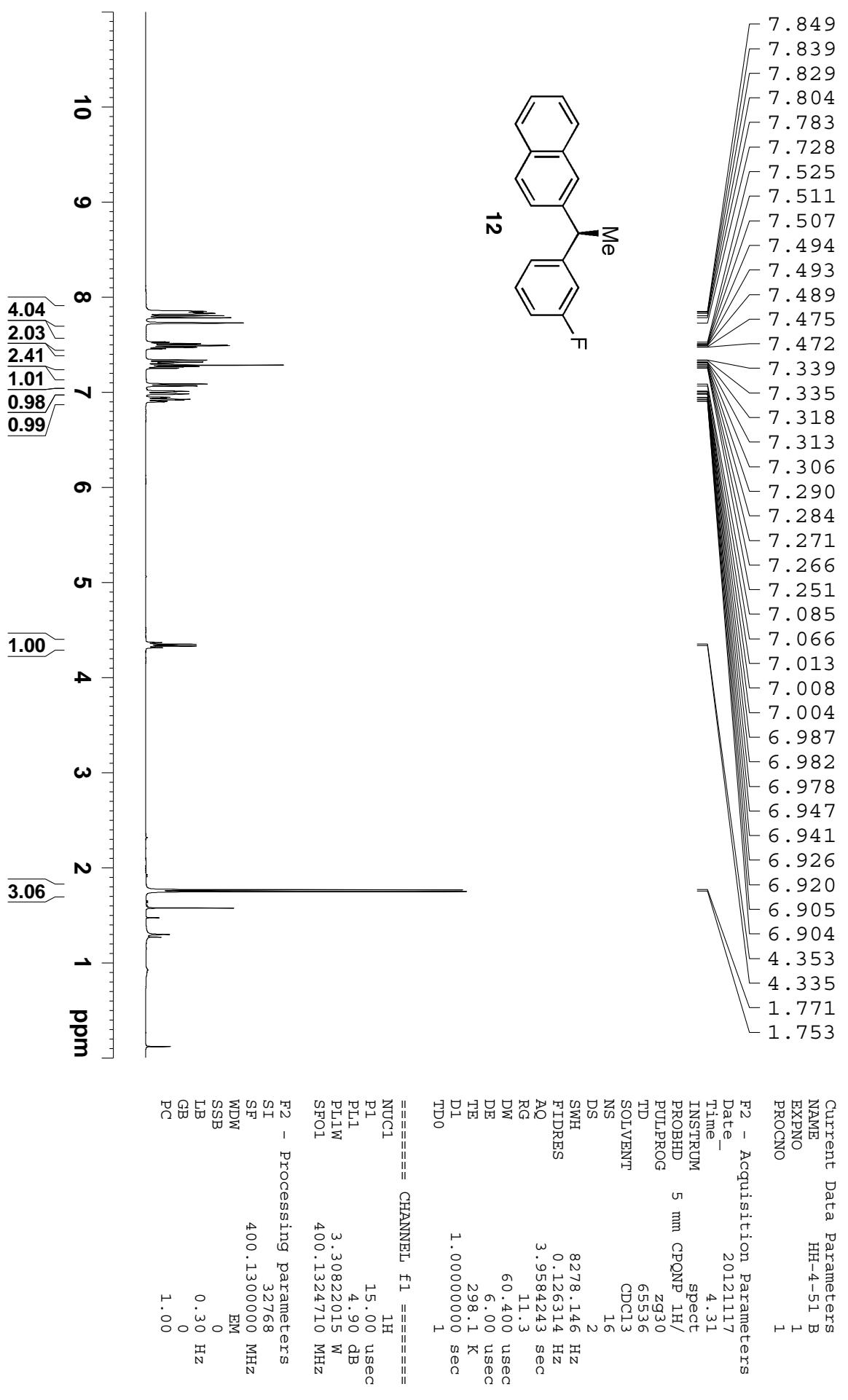
===== CHANNEL f1 =====
 NUC1 13C
 P1 9.25 usec
 PL1 0.55 dB
 PL1W 35.18820572 W
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 4.90 dB
 PL12 20.46 dB
 PL13 21.00 dB
 PL2W 3.30822015 W
 PL12W 0.09195905 W
 PL13W 0.08120718 W
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SP 100.6127690 MHz
 WDM EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



Compound 12 1H NMR



BRUKER

Compound 12 13CNMR



S62

F2 - Acquisition Parameters
 NAME HH-4-51 B
 EXPNO 2
 PROCNO 1

Time 4.47

INSTRUM spect

PROBHD 5 mm CPQNP 1H/
 PULPROG zgpg30

TD 65536

SOLVENT CDCl₃

NS 256

DS 4

SWH 23.980-814 Hz

FIDRES 0.365918 Hz

AQ 1.3664756 sec

RG 512

DW 20.850 usec

DE 18.000 usec

TE 298.2 K

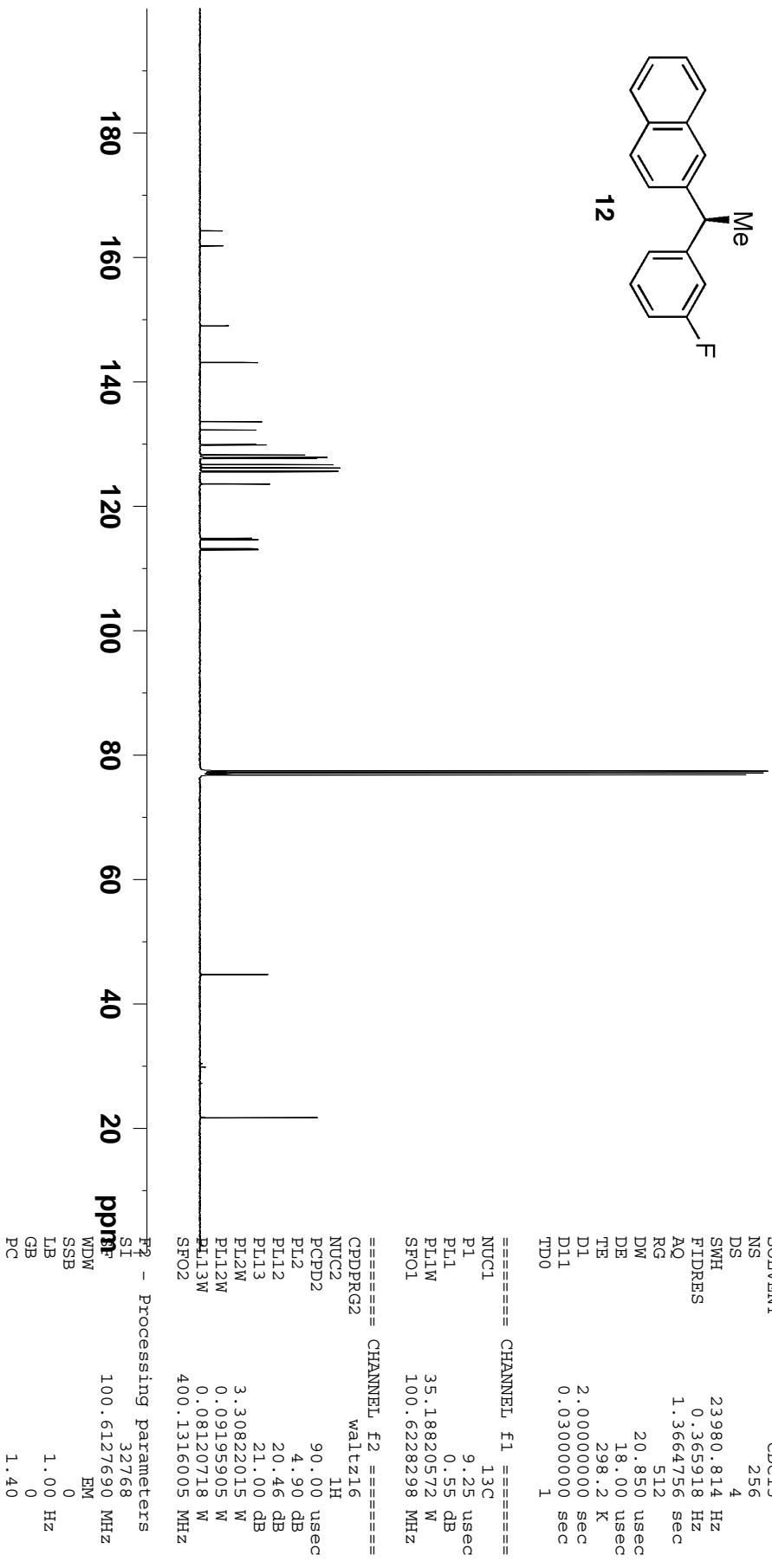
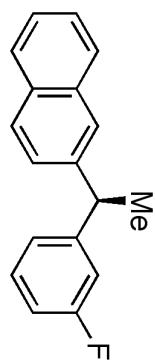
D1 2.0000000 sec

D11 0.03000000 sec

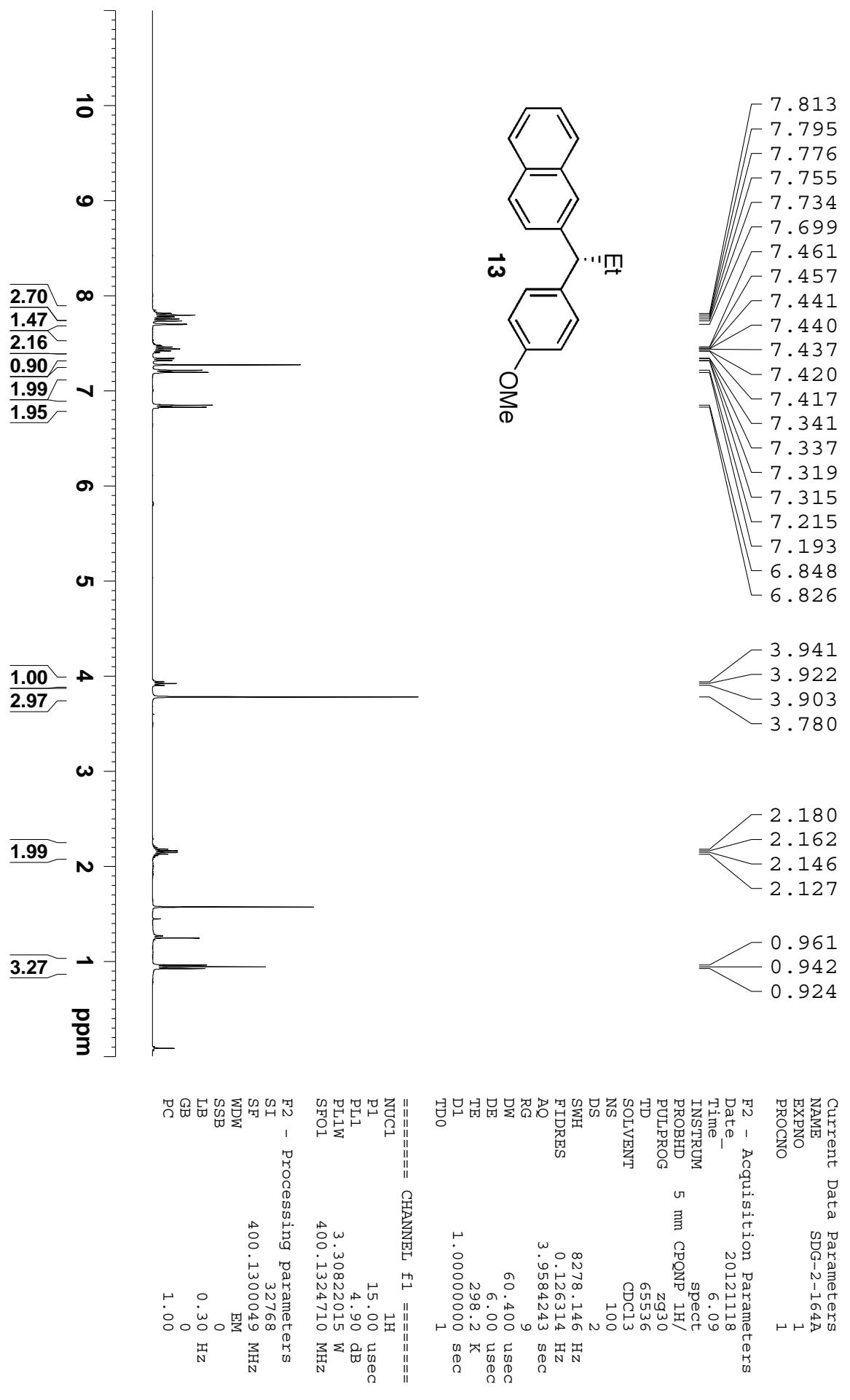
TD0 1

44.63

21.64



Compound 13 1H NMR



Compound 13 13CNMR



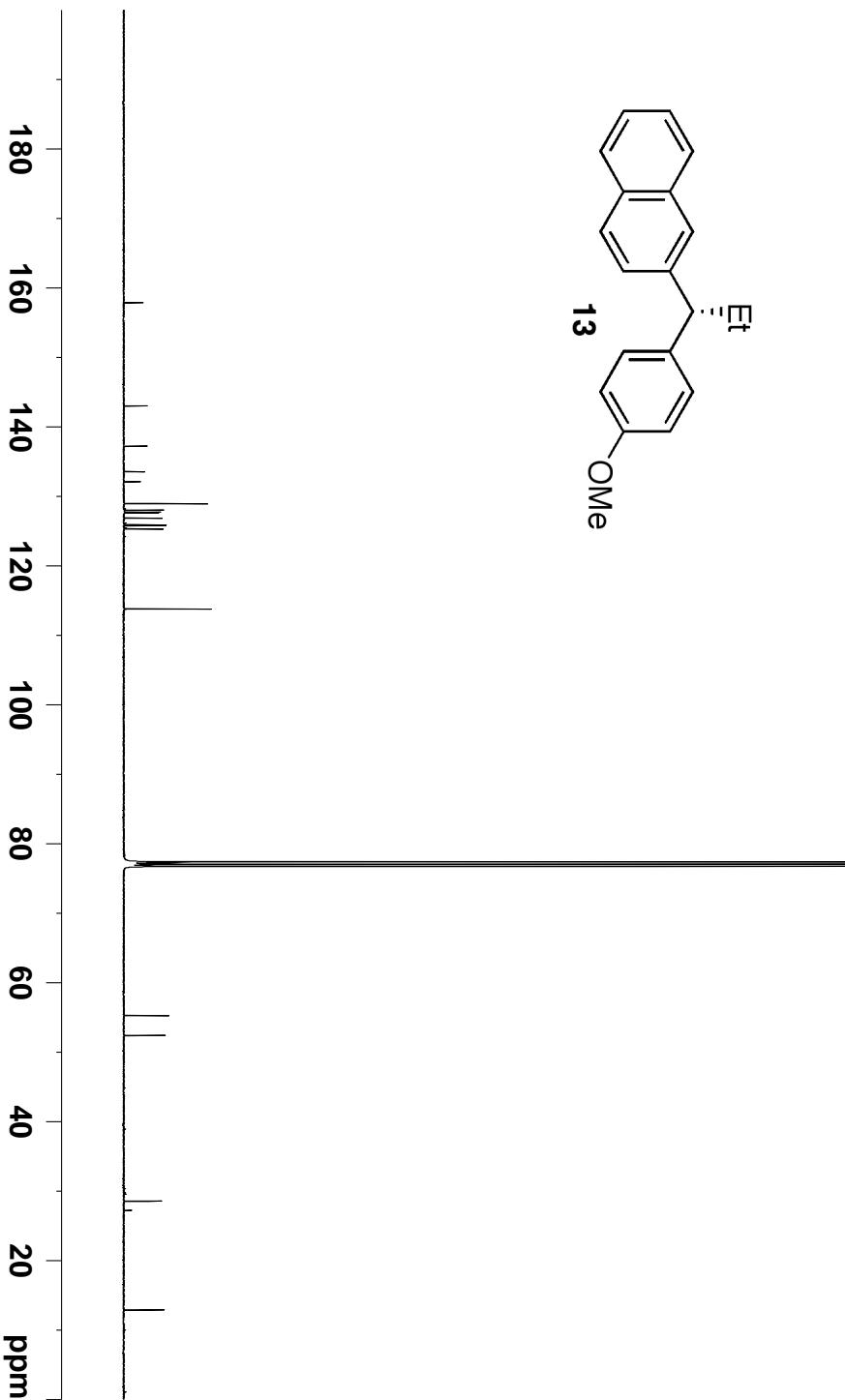
S64

F2 - Acquisition Parameters
 NAME SDG-2-164A
 EXPNO 2
 PROCNO 1

 Date 20121118
 Time 7.09
 INSTRUM spect
 PROBHD 5 mm CPQNP 1H/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1024
 DS 4
 SWH 23.980.814 Hz
 FIDRES 0.365918 Hz
 AQ 1.3664756 sec
 RG 512
 DW 20.850 usec
 DE 18.00 usec
 TE 298.1 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TDO 1

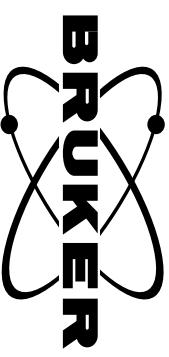
===== CHANNEL f1 =====
 NUC1 13C
 P1 9.25 usec
 PL1 0.55 dB
 PL1W 35.18820572 W
 SFO1 100.6228298 MHz

 ===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 4.90 dB
 PL12 20.46 dB
 PL13 21.00 dB
 PL2W 3.30822015 W
 PL12W 0.09195905 W
 PL13W 0.08120718 W
 SFO2 400.1316005 MHz

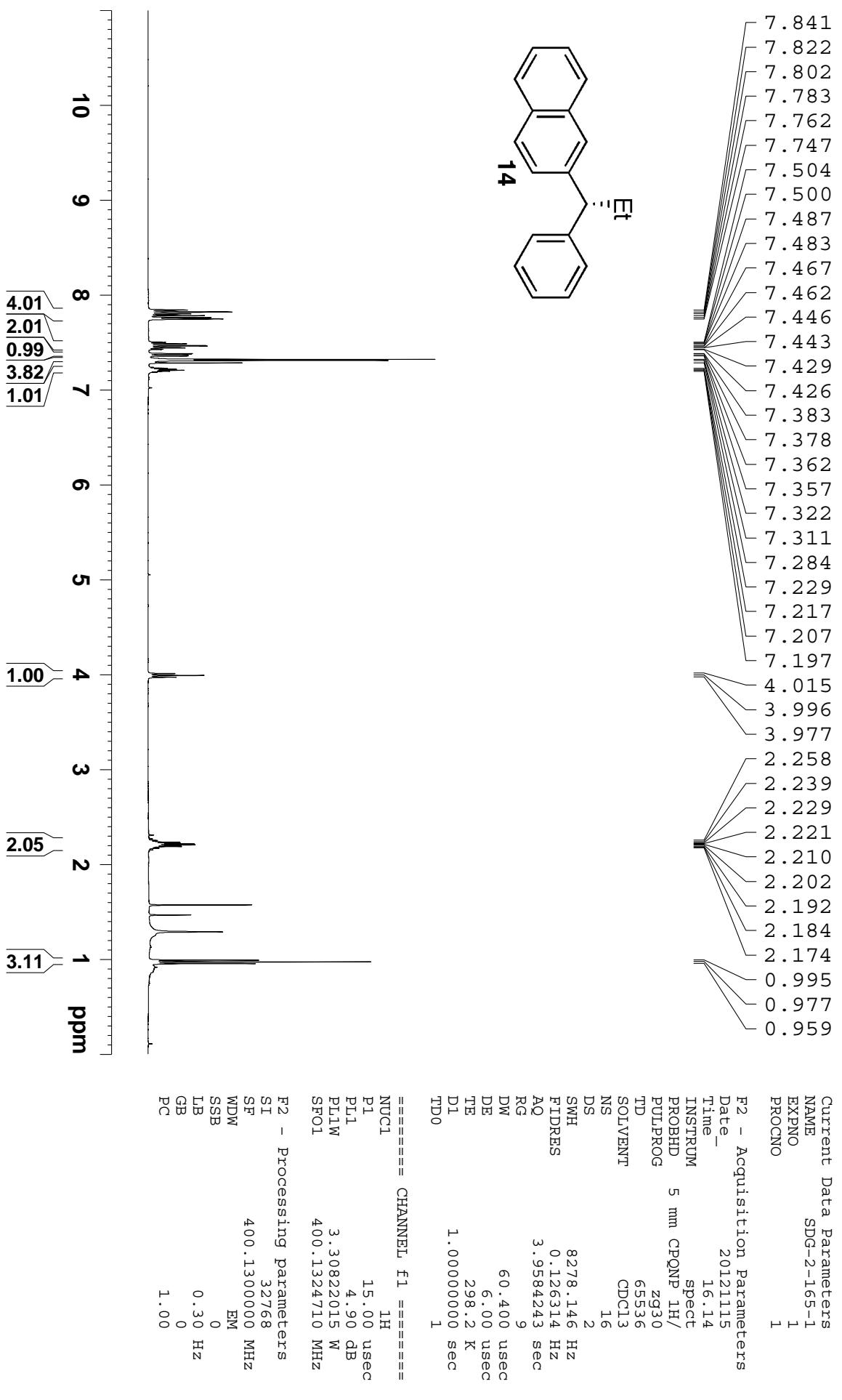


F2 - Processing parameters
 SI 32768
 SP 100.6127690 MHz
 WDM EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

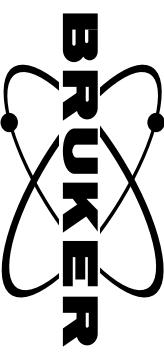
Compound 14 1H NMR



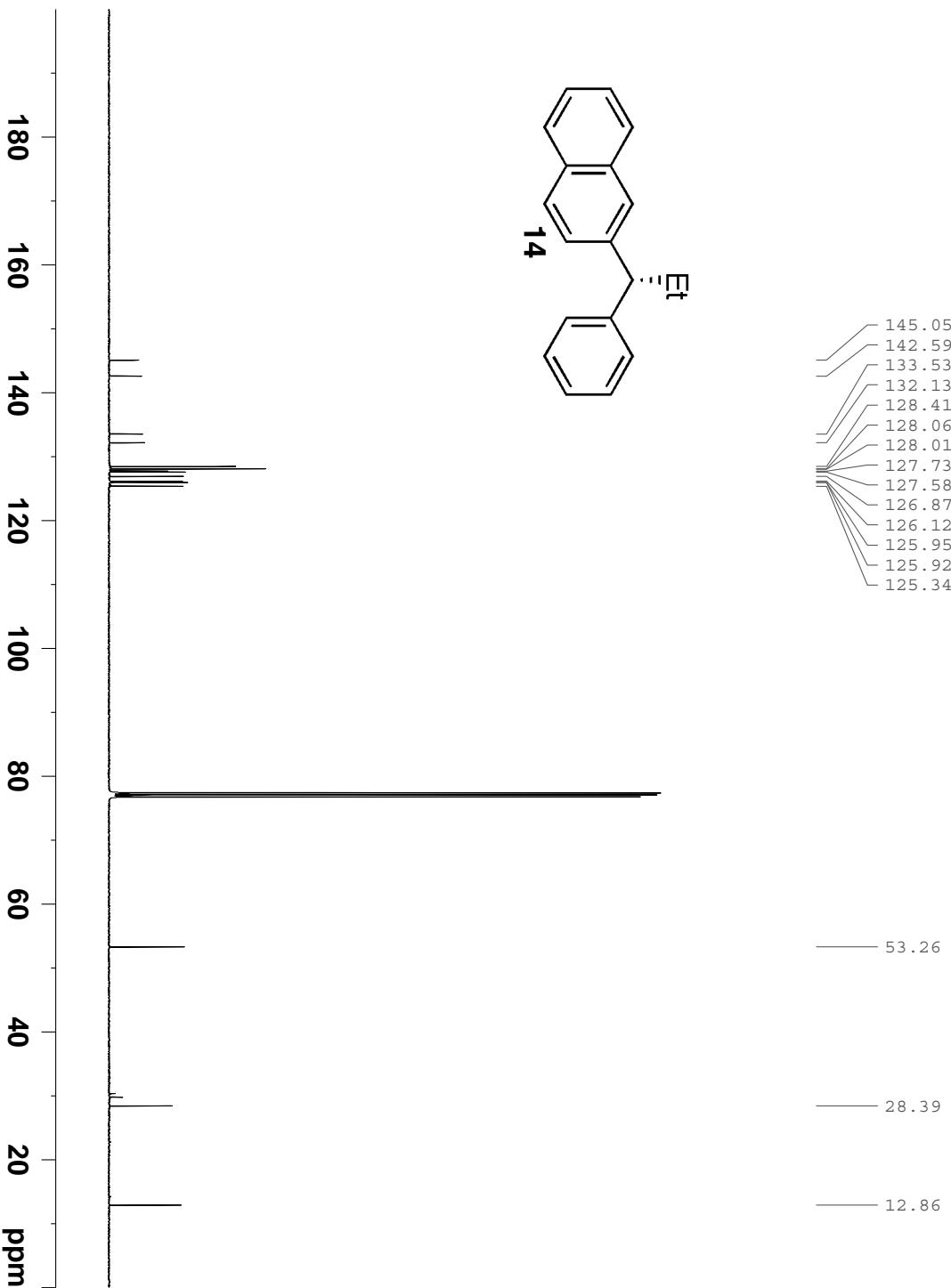
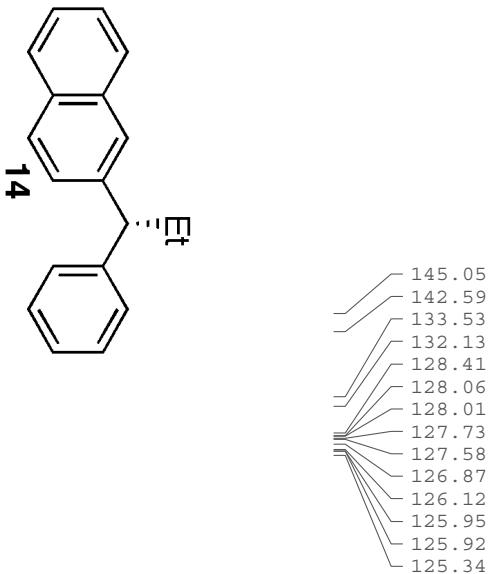
S65



Compound 14 13CNMR



S66



```

Current Data Parameters
NAME SDG-2-165-1
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121115
Time 22.41
INSTRUM spect
PROBHD 5 mm CPQNP 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 23.980-814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 512
DW 20.850 usec
DE 18.00 usec
TE 298.1 K
D1 2.0000000 sec
D11 0.03000000 sec
TD0 1

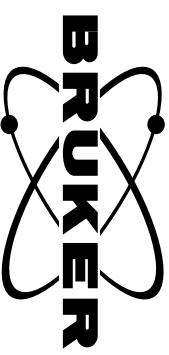
===== CHANNEL f1 =====
NUC1 13C
P1 9.25 usec
PL1 0.55 dB
PL1W 35.18820572 W
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 4.90 dB
PL12 20.46 dB
PL13 21.00 dB
PL2W 3.30822015 W
PL12W 0.09195905 W
PL13W 0.08120718 W
SFO2 400.1316005 MHz

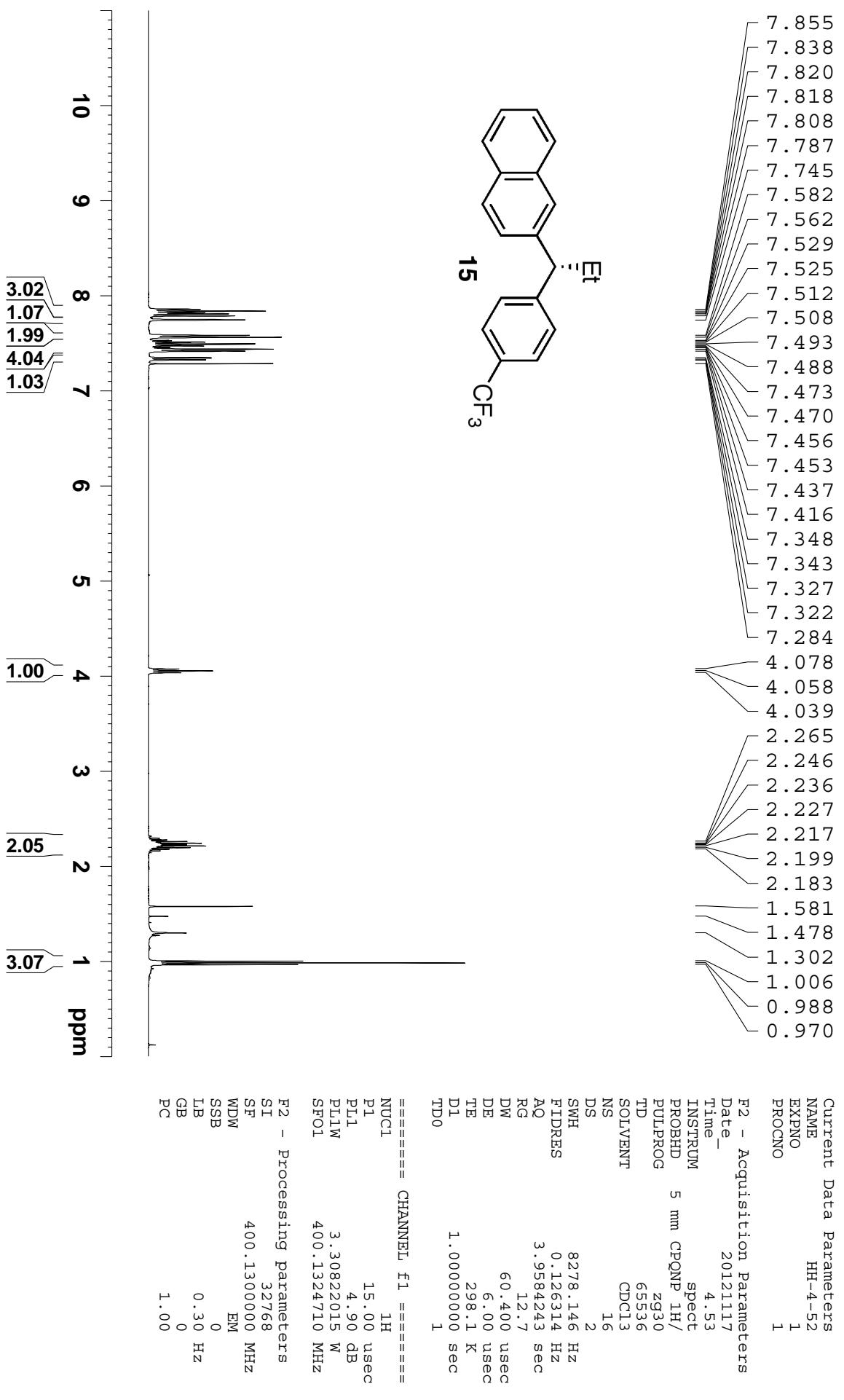
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

```

Compound 15 1H NMR



S67



Compound 15 13CNMR



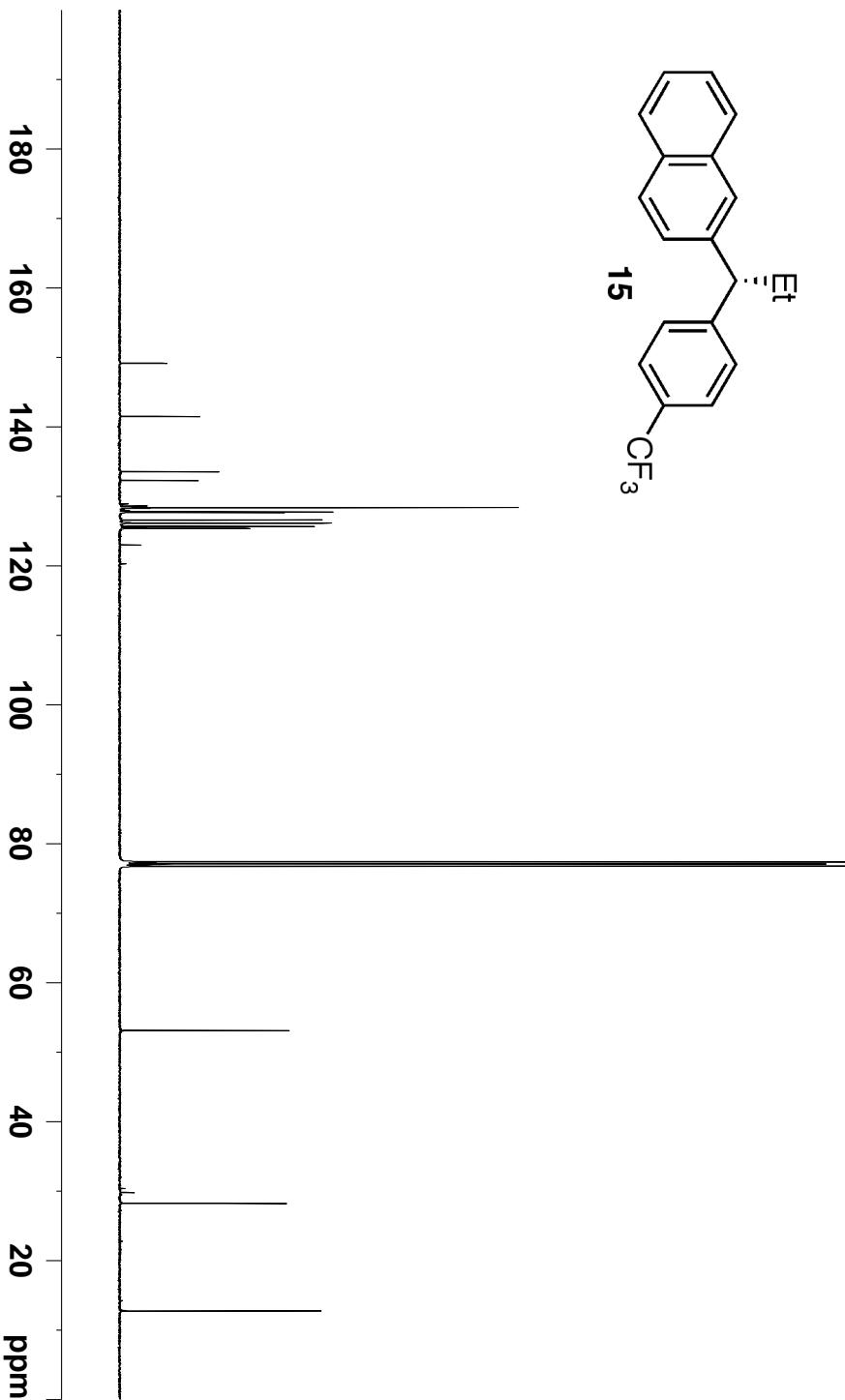
S68

Current Data Parameters
 NAME HH-4-52
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date 20121117
 Time 5.09
 INSTRUM spect
 PROBHD 5 mm CPQNP 1H/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 256
 DS 4
 SWH 23.980.814 Hz
 FIDRES 0.365918 Hz
 AQ 1.3664756 sec
 RG 512
 DW 20.850 usec
 DE 18.00 usec
 TE 298.2 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TDO 1

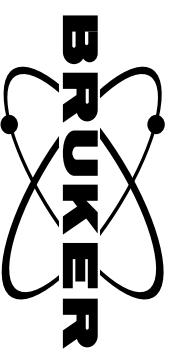
===== CHANNEL f1 =====
 NUC1 13C
 P1 9.25 usec
 PL1 0.55 dB
 PL1W 35.18820572 W
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 4.90 dB
 PL12 20.46 dB
 PL13 21.00 dB
 PL2W 3.30822015 W
 PL12W 0.09195905 W
 PL13W 0.08120718 W
 SFO2 400.1316005 MHz

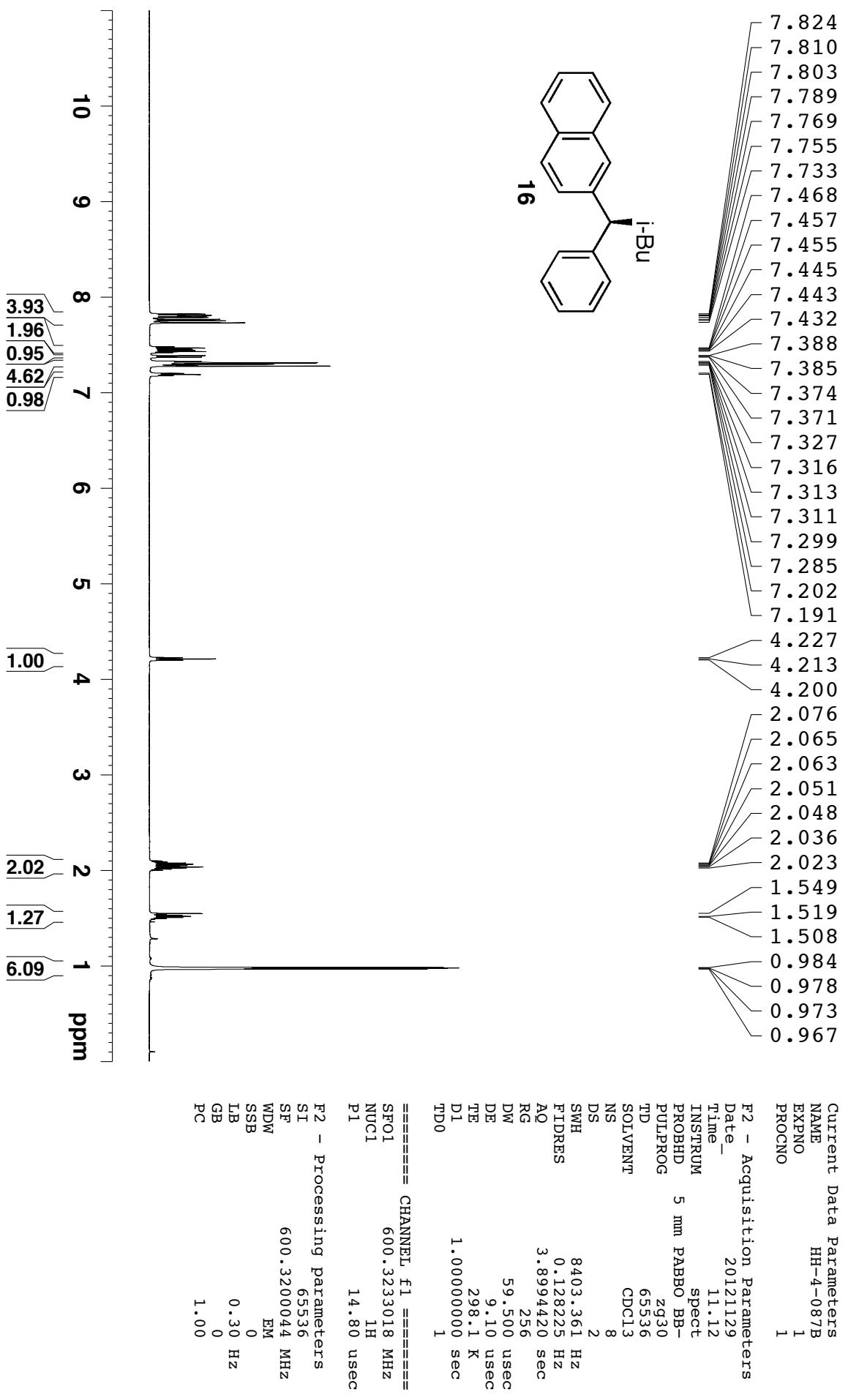


F2 - Processing parameters
 SI 32768
 SP 100.6127690 MHz
 WDM EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

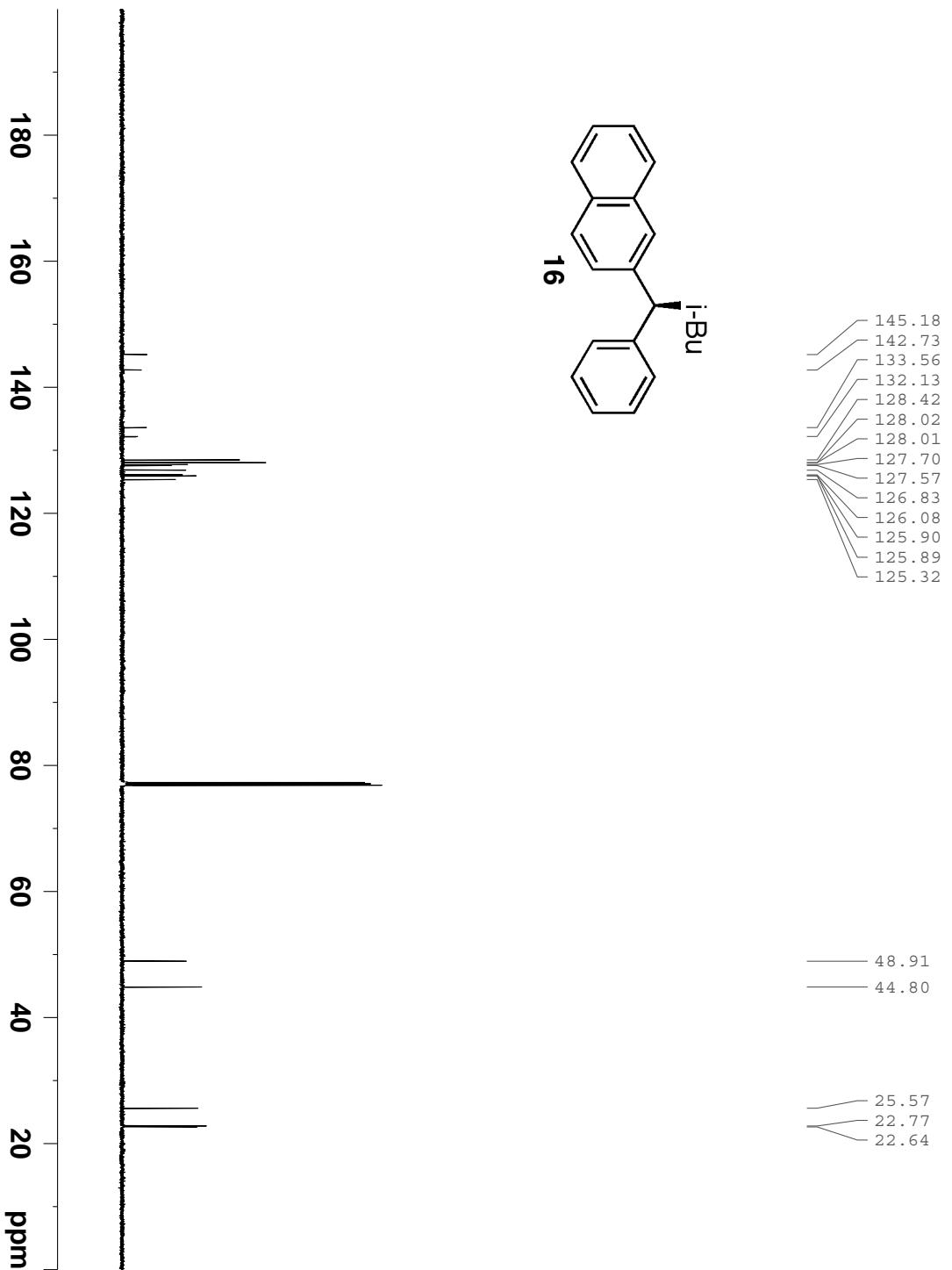
Compound 16 1H NMR



S69



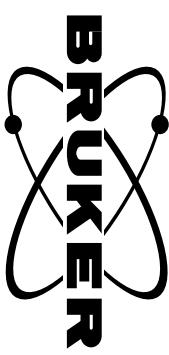
Compound 16 13CNMR



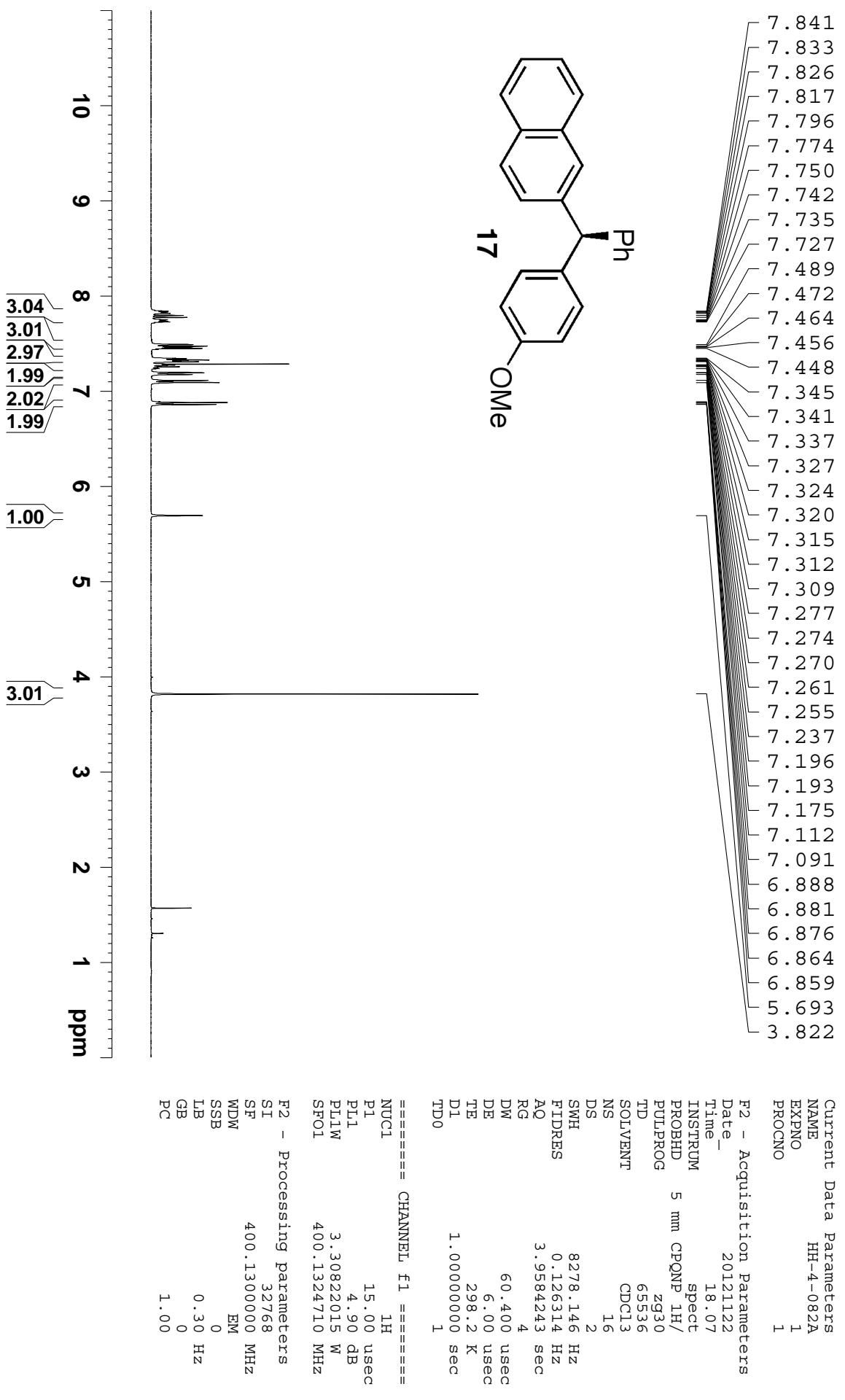
BRUKER

	Current	Data	Parameters
NAME	HH-4-087B		
EXPNO	2		
PROCNO	1		
F2 - Acquisition Parameters			
DATE	20121129		
TIME	11.22		
INSTRUM	spect		
PROBHD	5 mm	PABBO BB-	
PULPROG	zgppr55		
TD	65536		
SOLVENT	CDCl ₃		
NS	256		
DS	4		
SWH	34722.223 Hz		
FIDRES	0.529819 Hz		
AQ	0.9437684 sec		
RG	2050		
DW	14.400 usec		
DE	8.88 usec		
TE	298.1 K		
D1	1.1000002 sec		
D11	0.03000002 sec		
TDO	1 sec		
===== CHANNEL f1 =====			
SFO1	150.9656784 MHz		
NUC1	13C		
P1	9.00 usec		
F2 - Processing parameters			
SI	32768		
SF	150.9505840 MHz		
WDW	EM		
SSB	0		
LB	1.00 Hz		
GB	1.00 Hz		
PC	1.40		

Compound 17 1H NMR



S71



Compound 17 13CNMR



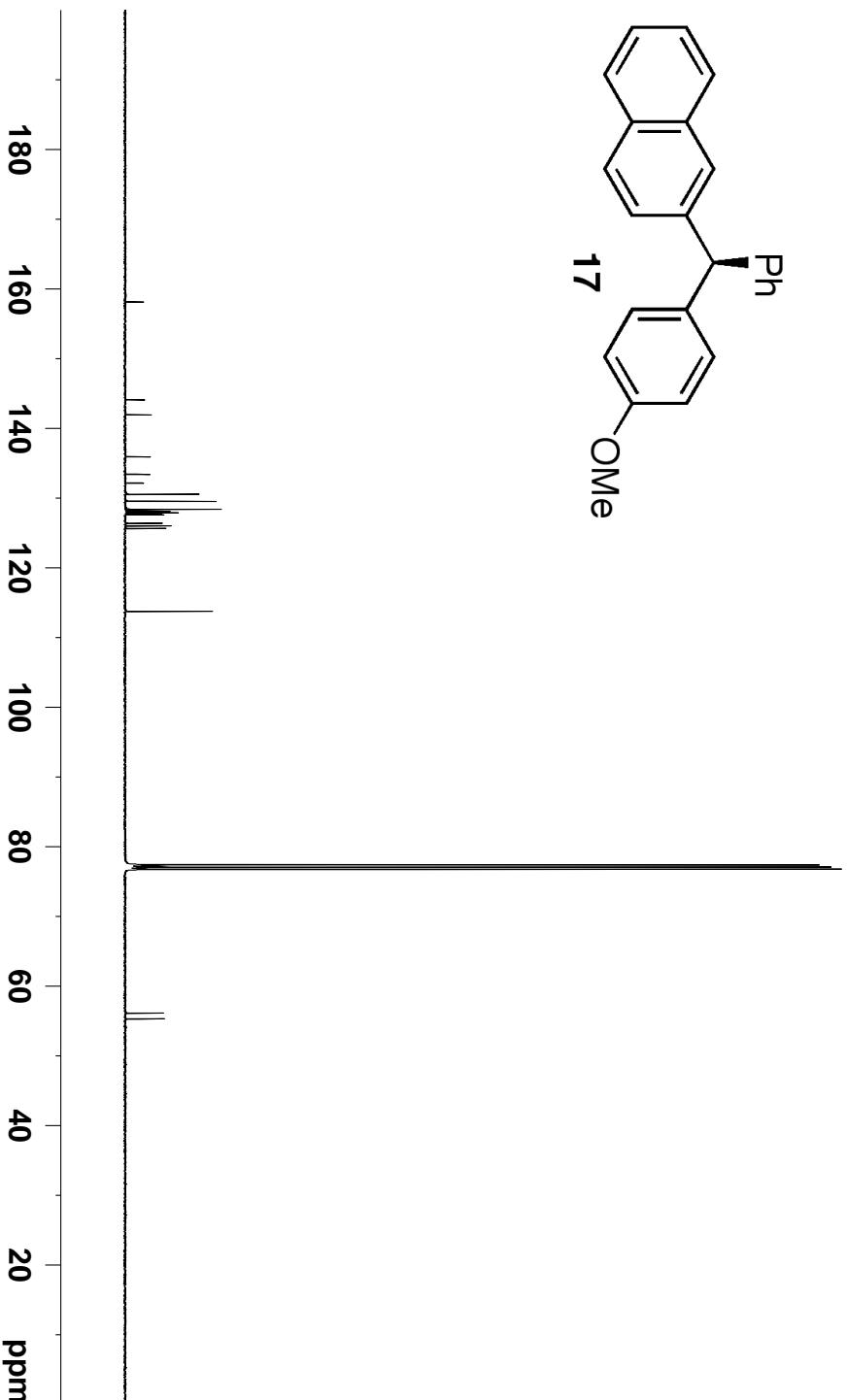
S72

Current Data Parameters
NAME HH-4-082A
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date 20121122
Time 18.28
INSTRUM spect
PROBHD 5 mm CPQNP 1H/
PULPROG zpgp30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 23.980-814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 512
DW 20.850 usec
DE 18.00 usec
TE 298.1 K
D1 2.0000000 sec
D11 0.03000000 sec
TD0 1

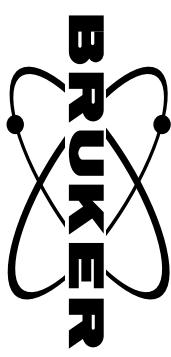
==== CHANNEL f1 =====
NUC1 13C
P1 9.25 usec
PL1 0.55 dB
PL1W 35.18820572 W
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 4.90 dB
PL12 20.46 dB
PL13 21.00 dB
PL2W 3.30822015 W
PL12W 0.09195905 W
PL13W 0.08120718 W
SFO2 400.1316005 MHz

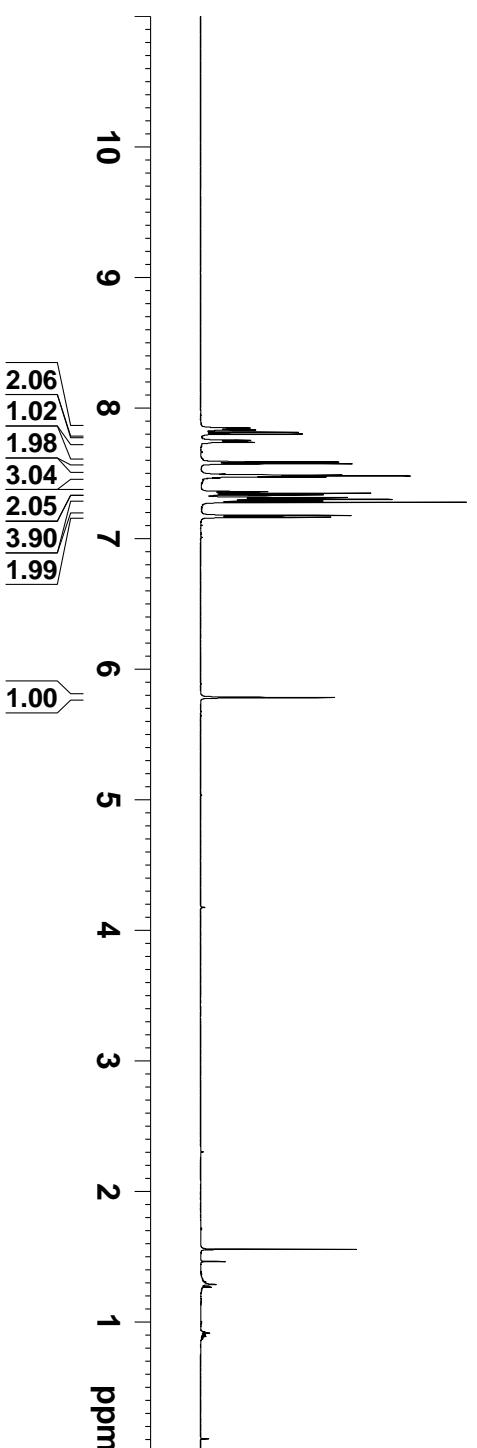
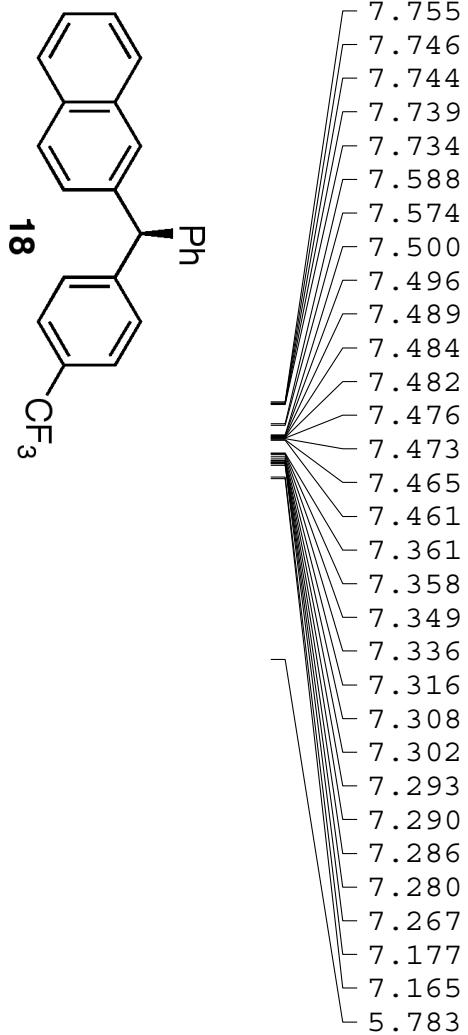


F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Compound 18 1H NMR



S73



Current Data Parameters
NAME QZ-3-139rs
EXPNO 1
PROCNO 1

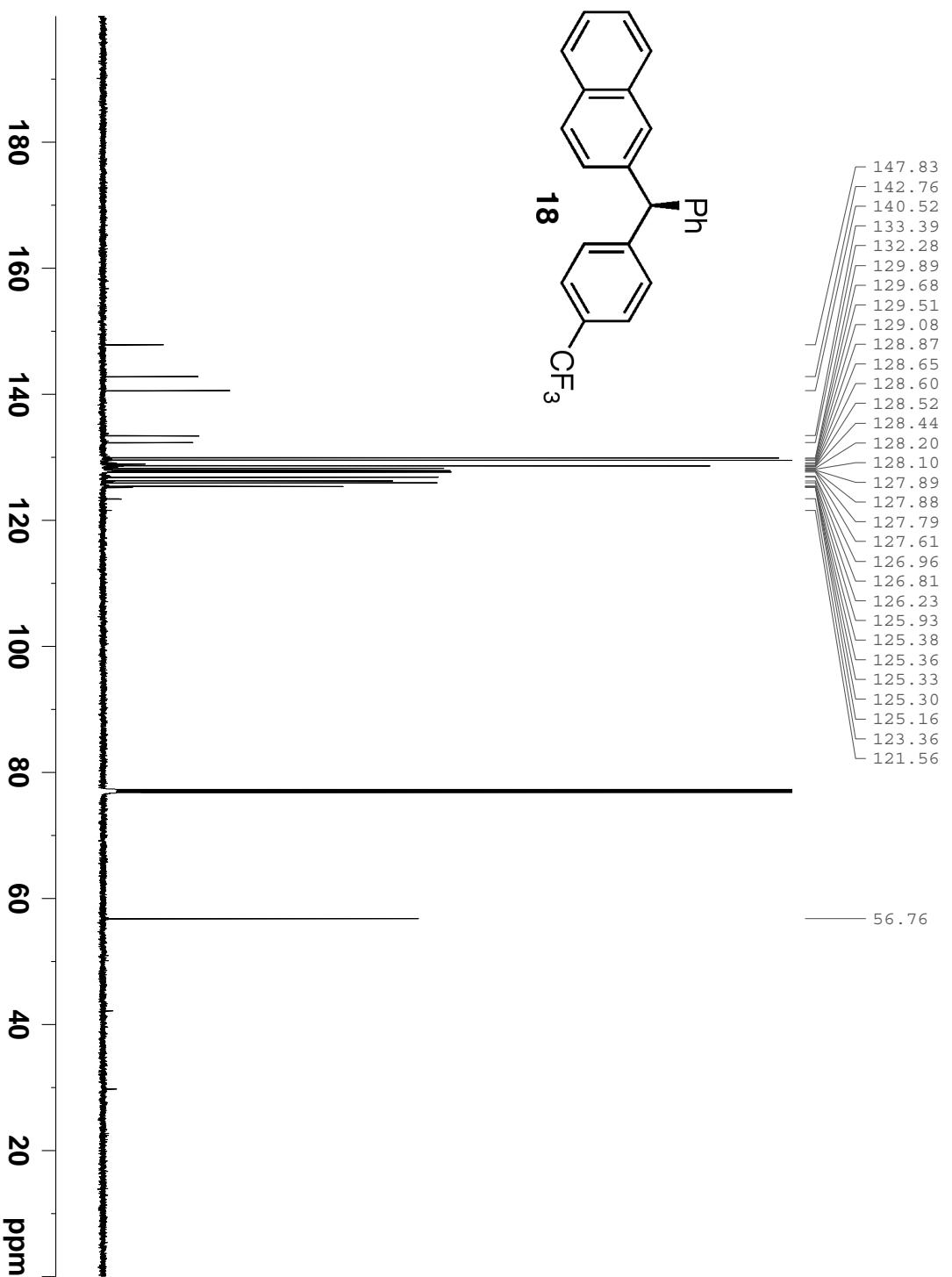
F2 - Acquisition Parameters
Date 20121130
Time 11.35
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl₃
NS 8
DS 2
SWH 8403.361 Hz
FIDRES 0.128225 Hz
AQ 3.8994420 sec
RG 256
DW 59.500 usec
DE 9.10 usec
TE 298.1 K
D1 1.0000000 sec
TD0

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

SFO1 600.3233018 MHz
NUC1 ¹H
P1 14.80 usec

F2 - Processing parameters
SI 65536
SF 600.3200039 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 1.00

Compound 18 13CNMR



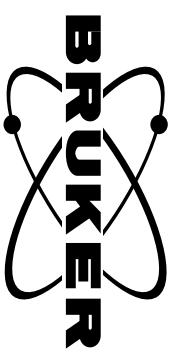
Current Data Parameters	
NAME	QZ-3-139 3rd tim
EXPNO	2
PROCNO	1
F2 - Acquisition Parameters	
Date	20121210
Time	18.42
INSTRUM	spec
PROBHD	5 mm PABBO BB/
PULPROG	zgpp95
TD	65536
SOLVENT	CDCl ₃
NS	1024
DS	4
SWH	34722.223 Hz
FIDRES	0.529819 Hz
AQ	0.9437684 sec
RG	2050
DW	14.400 usec
DE	19.34 usec
TE	298.1 K
D1	2.5000000 sec
D11	0.03000000 sec
TDO	1 sec

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

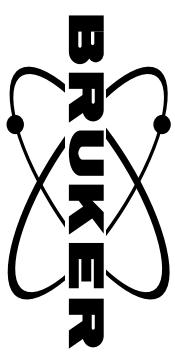
SFO1	150.9656784 MHz
NUC1	¹³ C
P1	10.63 usec

F2 - Processing parameters

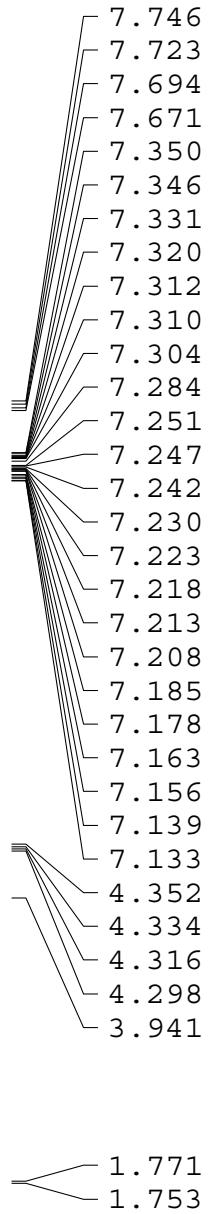
SI	32768
SF	150.9505840 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



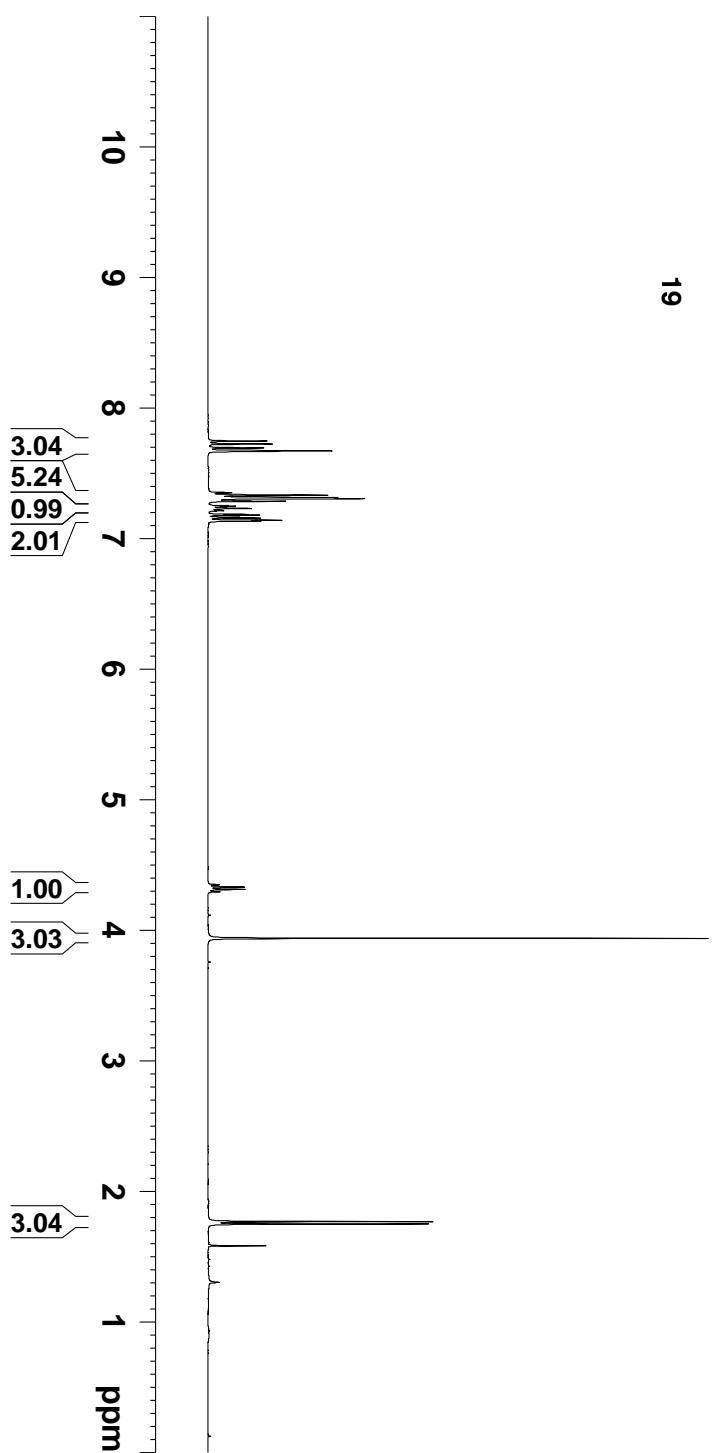
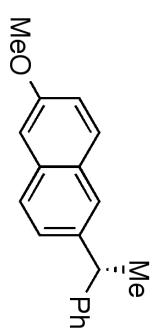
Compound 19 1H NMR



S75



1.771
1.753



Current Data Parameters	SDG-2-167-1
NAME	
EXPNO	1
PROCNO	1
F2 - Acquisition Parameters	
Date	20121115
Time	16.02
INSTRUM	spec
PROBHD	5 mm CPQNP 1H/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	8
DW	60.400 usec
DE	6.00 usec
TE	298.2 K
D1	1.0000000 sec
TD0	

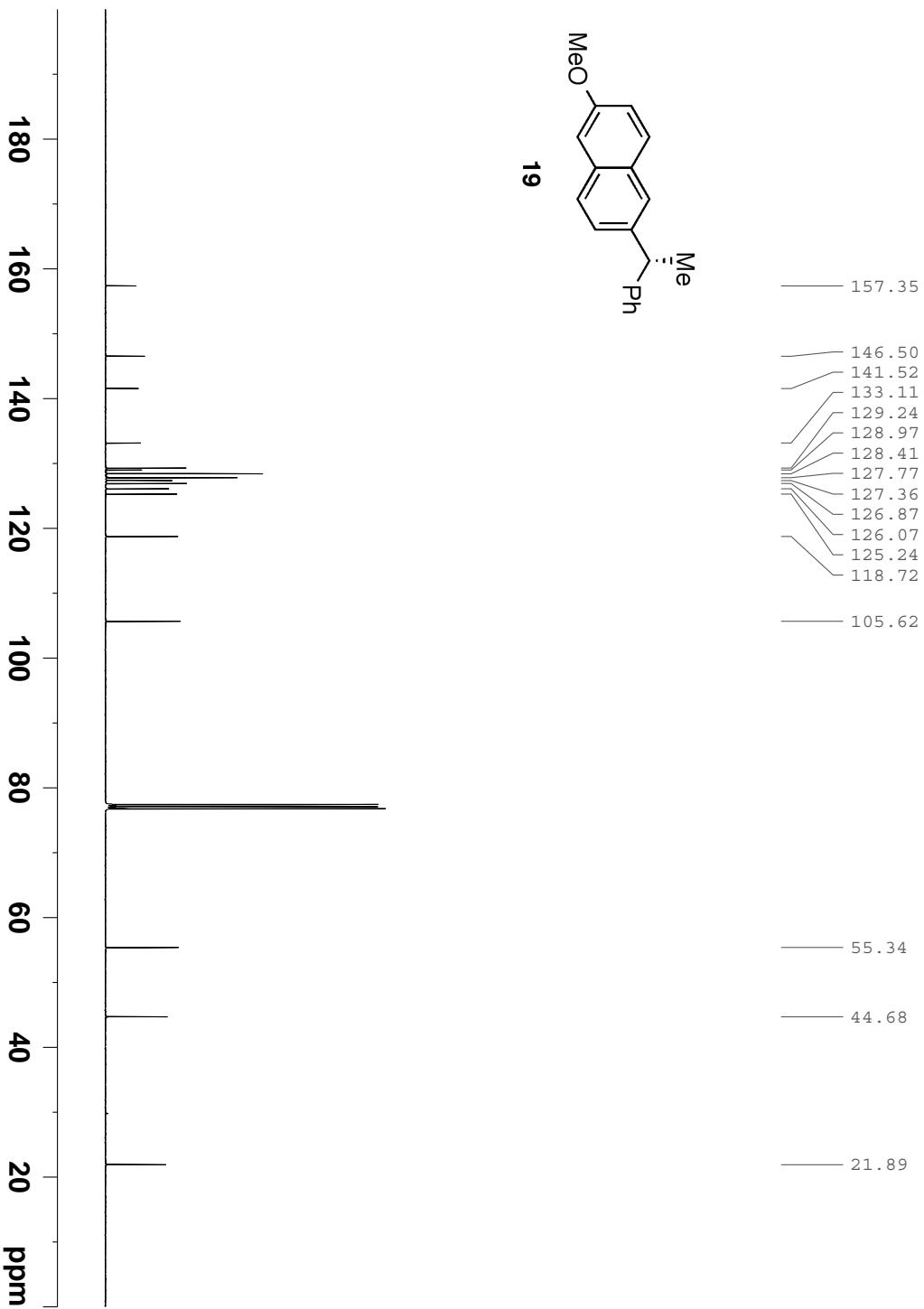
===== CHANNEL f1 =====	
NUC1	1H
P1	15.00 usec
PL1	4.90 dB
PL1W	3.30822015 W
SFO1	40.0.1324710 MHz

F2 - Processing parameters	
SI	32768
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	1.00
PC	

Compound 19 13CNMR

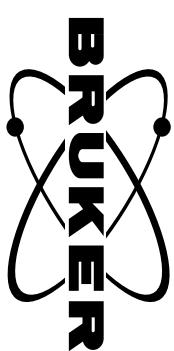


S76

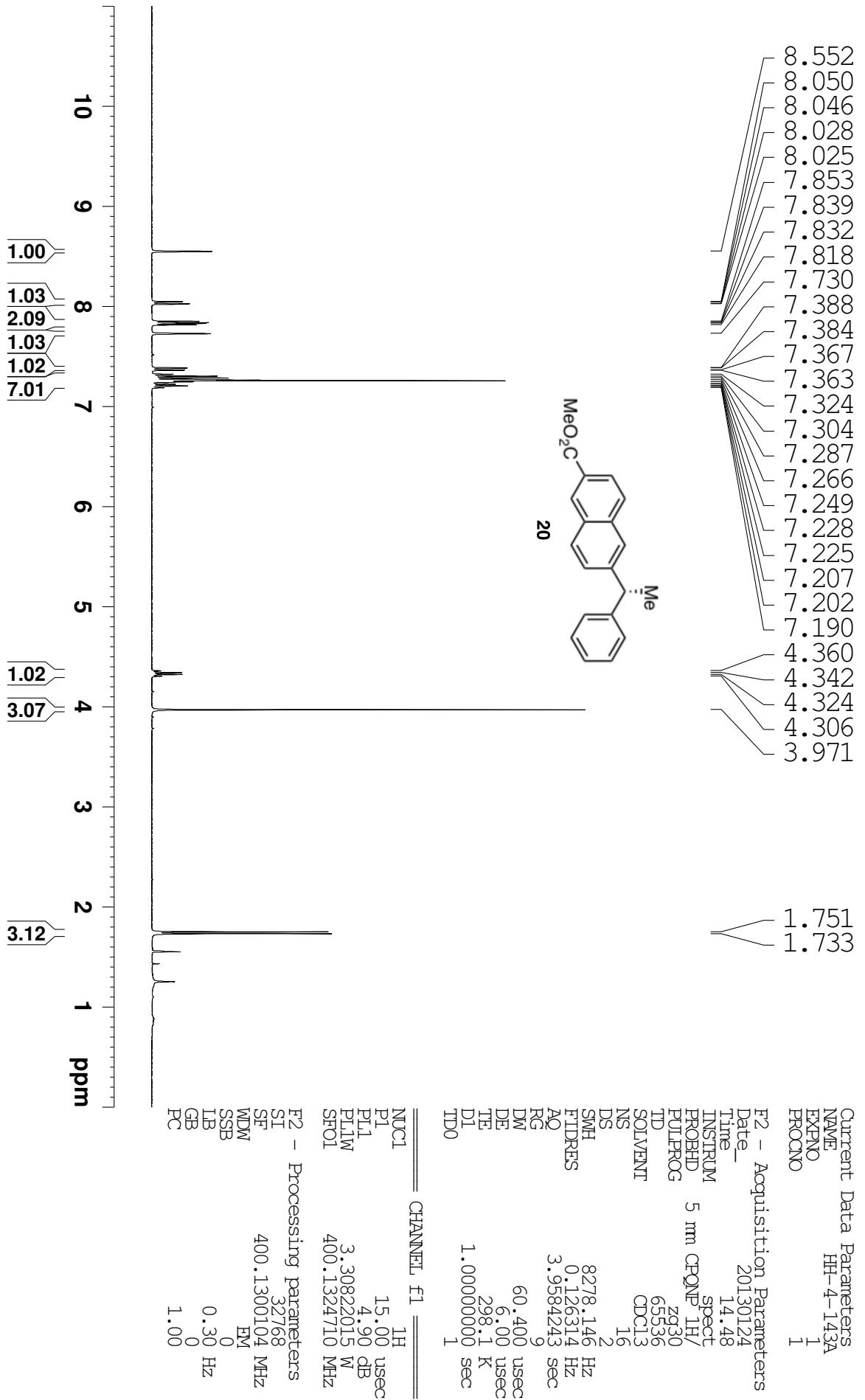


	Current	Data	Parameters
NAME	SDG-2-167-1		
EXPNO	2		
PROCNO	1		
F2 - Acquisition Parameters			
DATE		20121115	
TIME		22.03	
INSTRUM		spect	
PROBHD	5 mm	CPQNP 1H/	
PULPROG		ZGRG30	
TD		65536	
SOLVENT		CDCl3	
NS		256	
DS		4	
SWH	2.3980	8.14 Hz	
FIDRES	0.365918	Hz	
AQ	1.3664756	sec	
RG	512		
DW	20.850	usec	
DE	18.00	usec	
TE	298.2	K	
D1	2.00000000	sec	
D11	0.03000000	sec	
TD0	1		
===== CHANNEL f1 =====			
NUC1	13C		
P1	9.25	usec	
PL1	0.55	dB	
PL1W	35.18820572	W	
SFO1	100.6228298	MHz	
===== CHANNEL f2 =====			
CPDPRG2	waltz16		
NUC2	1H		
PCPD2	90.00	usec	
PL2	4.90	dB	
PL12	20.46	dB	
PL13	21.00	dB	
PL2W	3.30822015	W	
PL12W	0.09195905	W	
PL13W	0.08120718	W	
SFO2	400.1316005	MHz	
F2 - Processing parameters			
SI	32768		
SP	100.6127690	MHz	
WDW	EM		
SSB	0		
LB	1.00	Hz	
GB	0		
PC	1.40		

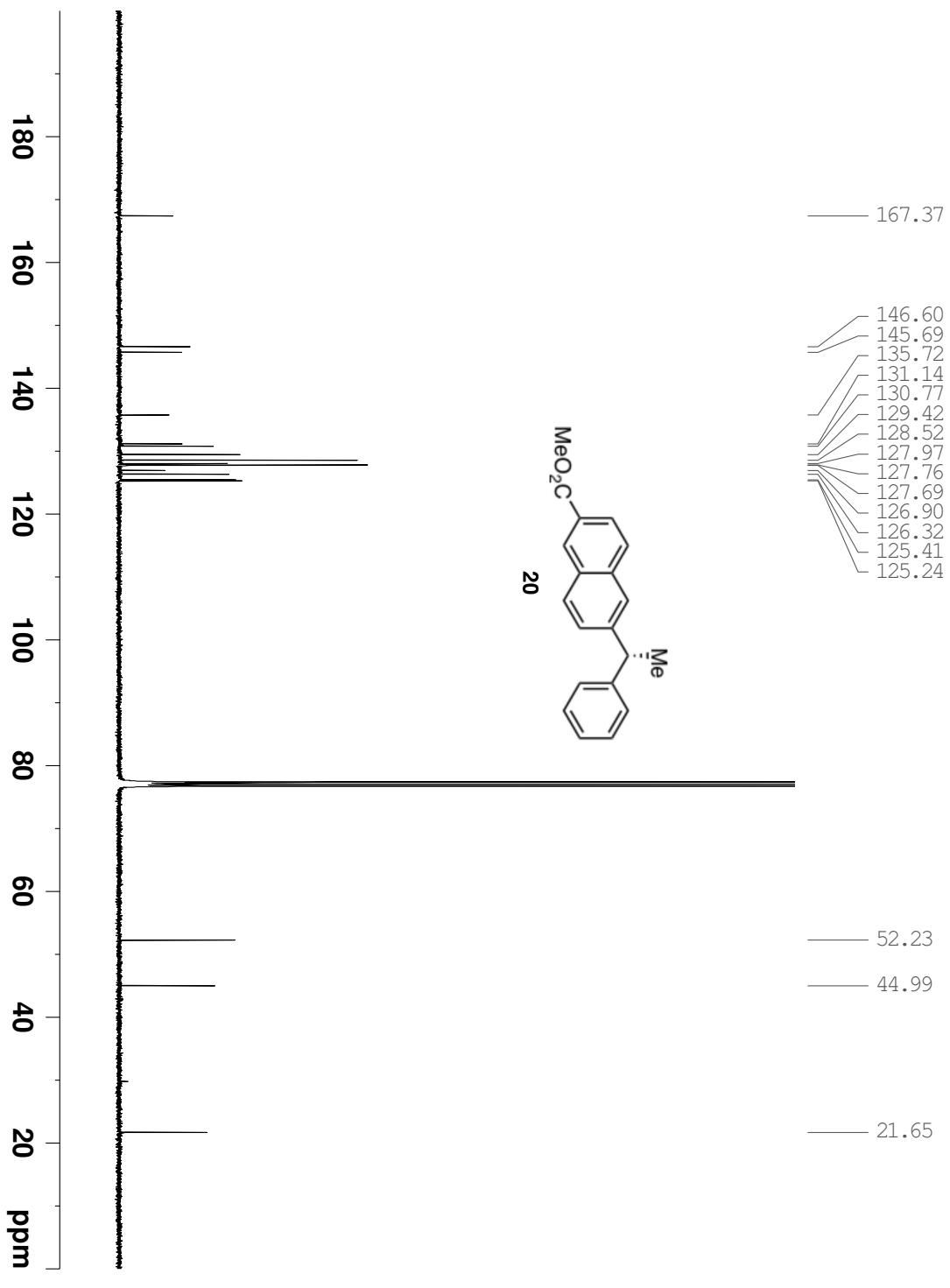
compound 20 1H NMR



S77



Compound 20 ^{13}C NMR



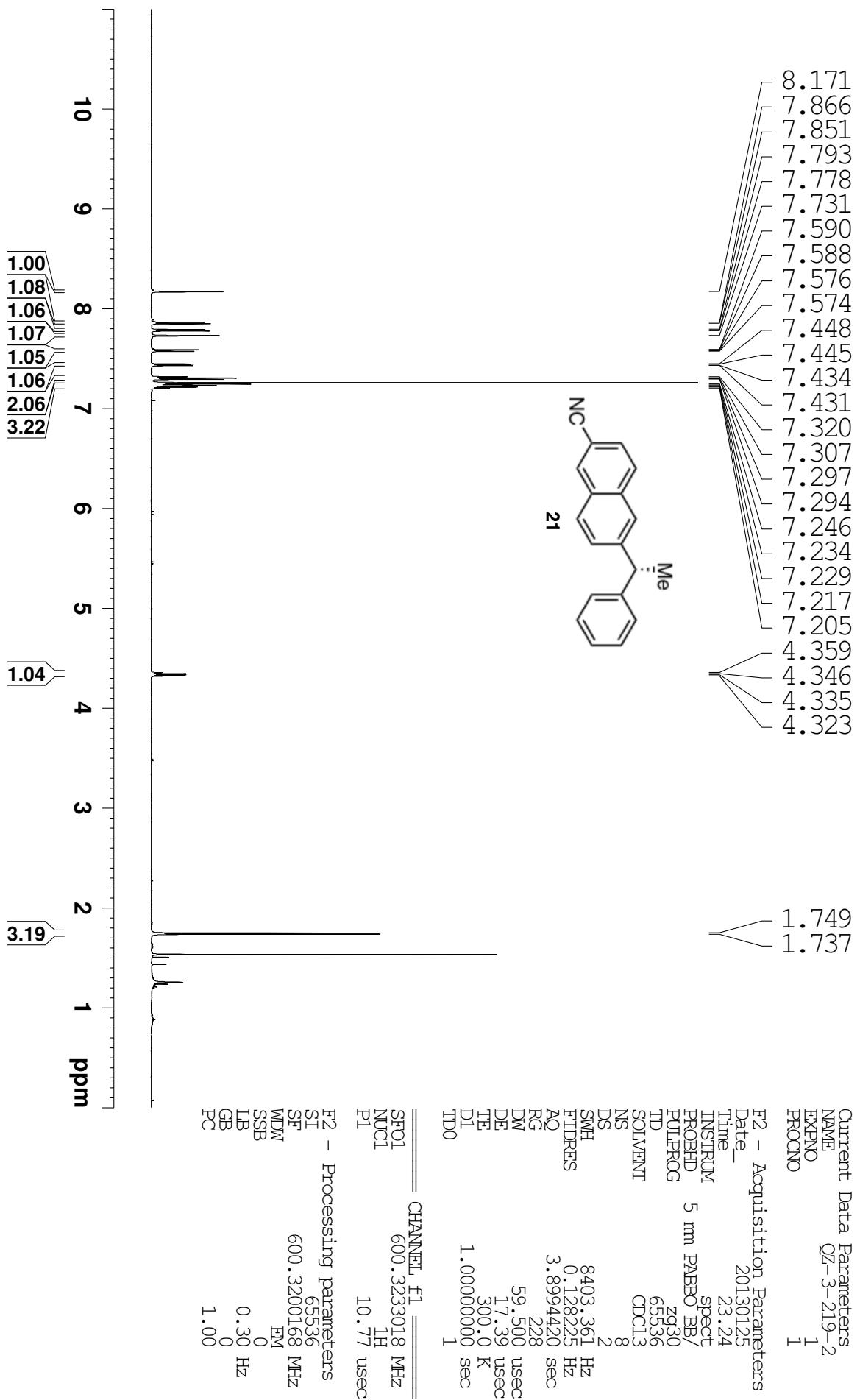
	Current Data	Parameters
NAME	HH-4-143A	2
EXPNO		1
PROCNO		
F2 - Acquisition Parameters		
INSTRM	5 mm CP/MR 1H/	spect
PROBHD	zgrg30	
PULFRQ	65536	
TD	CDCl ₃	
SOLVENT	1024	
NS	4	
DS	23980.814	Hz
SWH	0.365918	Hz
FIDRES	1.3664756	sec
AQ	512	
RG	20.850	usec
DW	18.00	usec
DE	298.2	K
TE		
DL	2.00000000	sec
D1	0.03000000	sec
D11	1	
TDO		
<hr/>		
CHANNEL f1		
NUC1	¹³ C	
PL1	9.25	usec
PLL1	0.55	dB
PL1W	35.18820572	W
SHF1	100.6228298	MHz
<hr/>		
CHANNEL f2		
NUC2	¹ H	
PCPD2	90.00	usec
PL2	4.90	dB
PLL2	20.46	dB
PLL3	21.00	dB
PL12W	3.30822015	W
PL12W	0.09195905	W
PL13W	0.08120718	W
SHF2	400.1316005	MHz

F2 - Processing parameters

SI	32768
ST	100.6127690
MDW	0
SSB	0
LB	1.00
GB	0
PC	1.40



compound 21 1H NMR



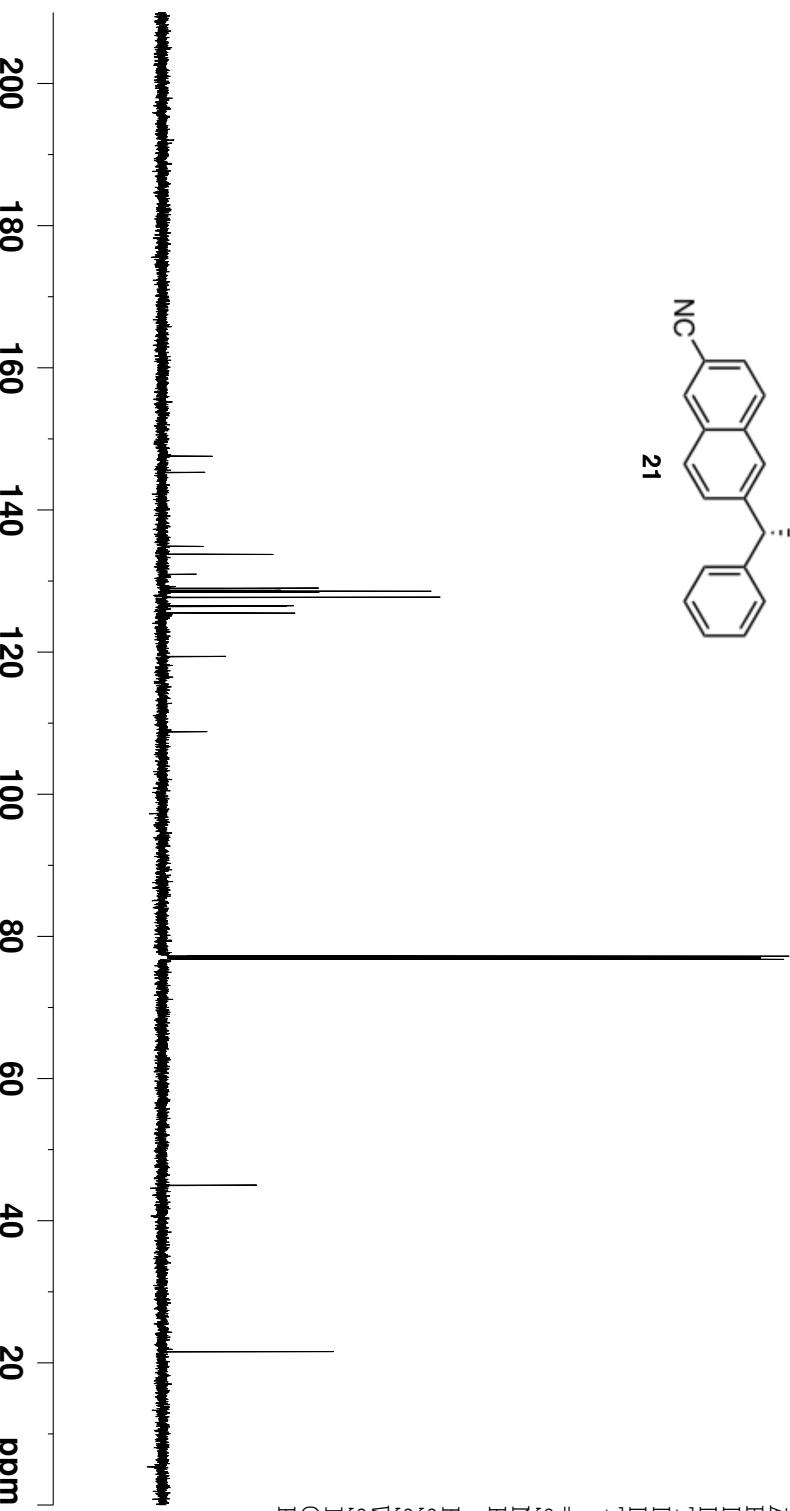
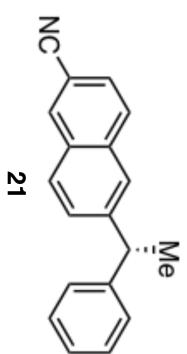
compound 21 13NMR



147.59
145.32
134.91
133.79
130.97
128.99
128.70
128.59
128.46
127.72
126.55
126.47
125.51
119.39
108.81

45.00

21.57



Current Data Parameters
NAME QZ-3-219-B-1
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters

Date 20130126
Time 1.08

INSTRUM spect
PROBID 5 mm PABBO BB/
PULPROG ztop55

TD 65536
SOLVENT CDCl₃
NS 256
DS 4

SWH 34722.223 Hz
FIDRES 0.9437684 sec

AQ 2050

RG 14.400 usec

DW 19.34 usec

DE 300.0 K

TE 1.10000002 sec

D1 0.03000000 sec

D11 1 sec

TDO 1 sec

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

SFO1 150.9656784 MHz
NUC1 ¹³C

P1 10.63 usec

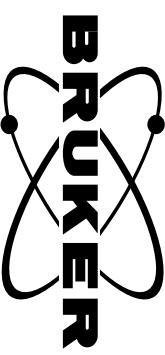
F2 - Processing parameters

SI 32768
SF 150.9505840 MHz
MDW 0

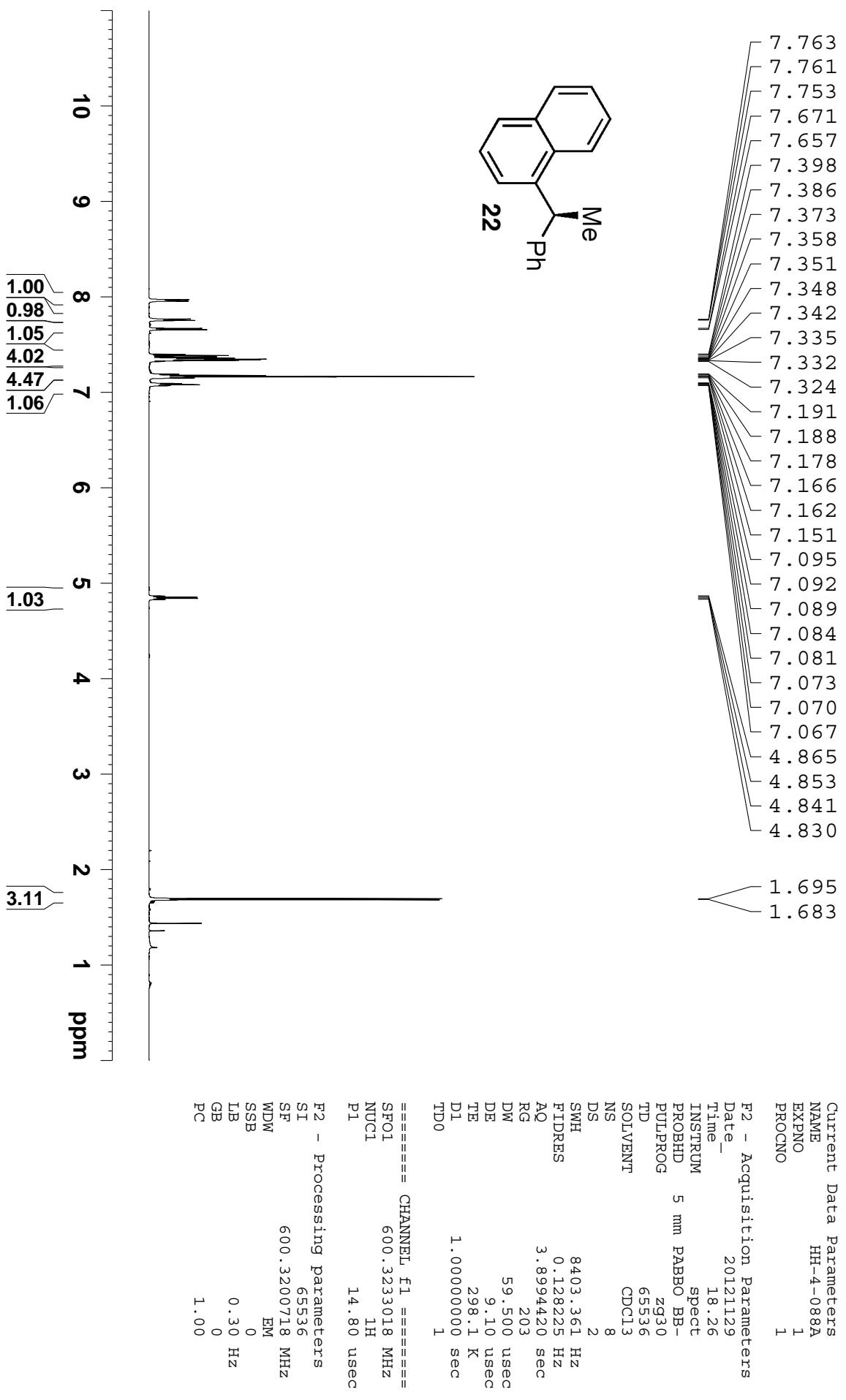
EM 0

SSB 1.00 Hz
LB 0
GB 1.40
PC

Compound 22 1H NMR



S81



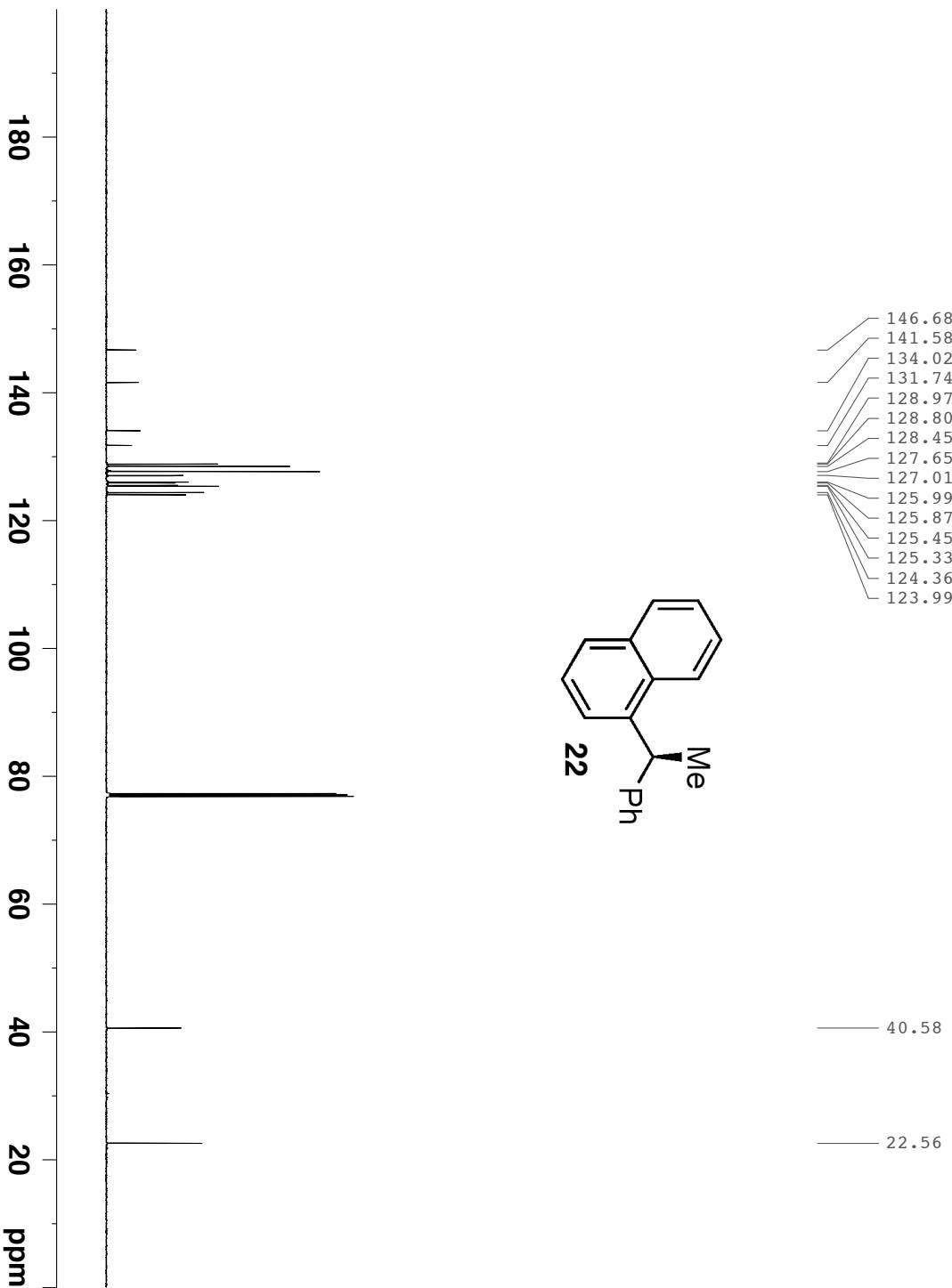
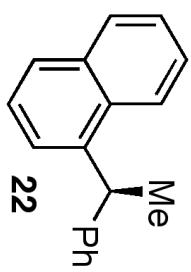
Compound 22 13CNMR



S82

146.68
141.58
134.02
131.74
128.97
128.80
128.45
127.65
127.01
125.99
125.87
125.45
125.33
124.36
123.99

40.58
22.56



Current	Data	Parameters
NAME	HH-4-088A	
EXPNO	2	
PROCNO	1	

F2 - Acquisition Parameters

Date	20121129
Time	22.39
INSTRUM	spct
PROBHD	5 mm PABBO BB-
PULPROG	zgpg55
TD	65536
SOLVENT	CDCl ₃
NS	3000
DS	4
SWH	34722.223 Hz
FIDRES	0.529819 Hz
AQ	0.9437684 sec
RG	2050
DW	14.400 usec
DE	8.88 usec
TE	298.1 K
D1	1.1000002 sec
D11	0.03000002 sec
TDO	1 sec

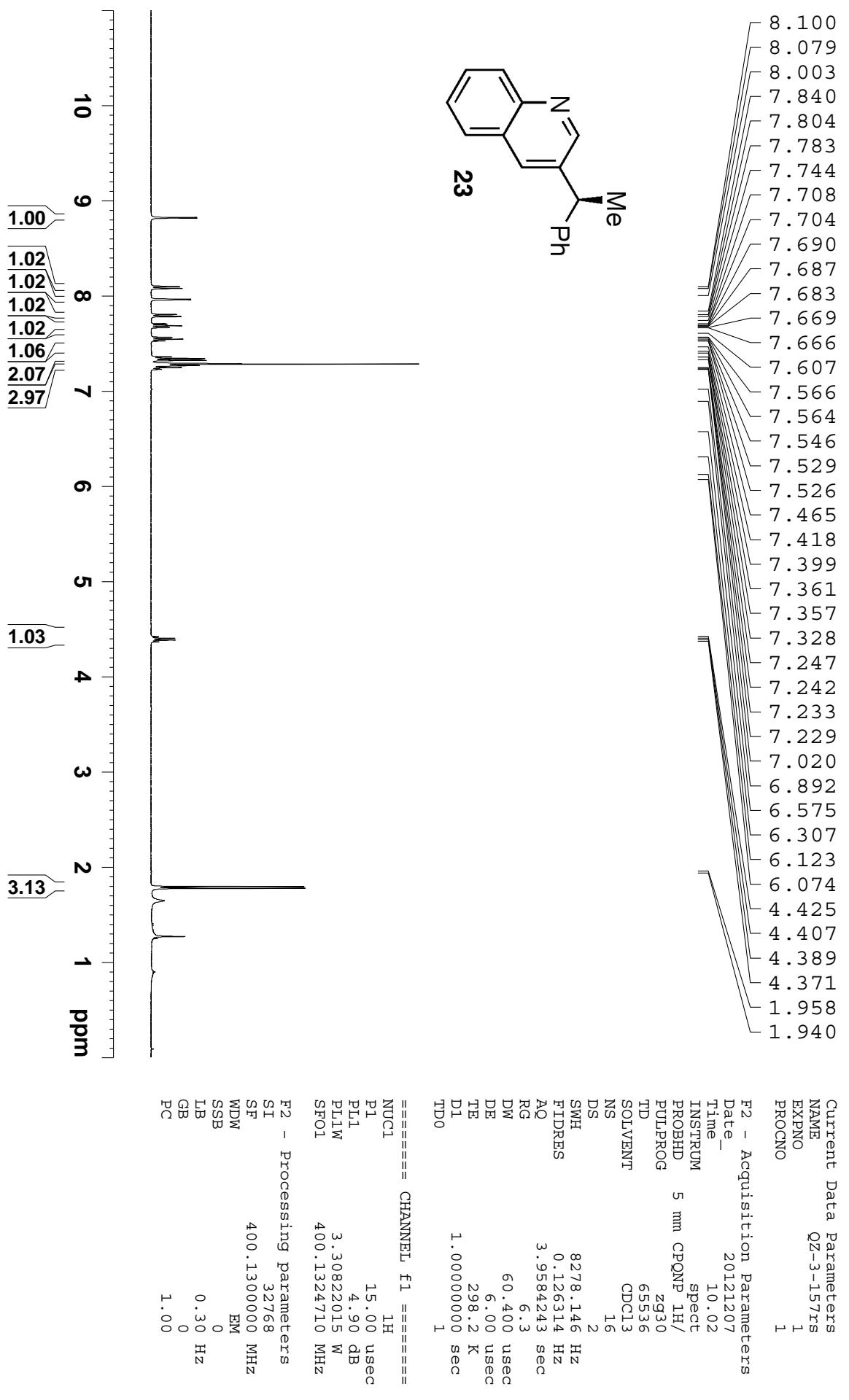
===== CHANNEL f1 =====

SFO1	f1	NUC1	spinlock
150.9656784 MHz	13C		
		9.00	usec

F2 - Processing parameters

SI	32768
SF	150.9505840 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

Compound 23 1H NMR



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Compound 23 13C NMR

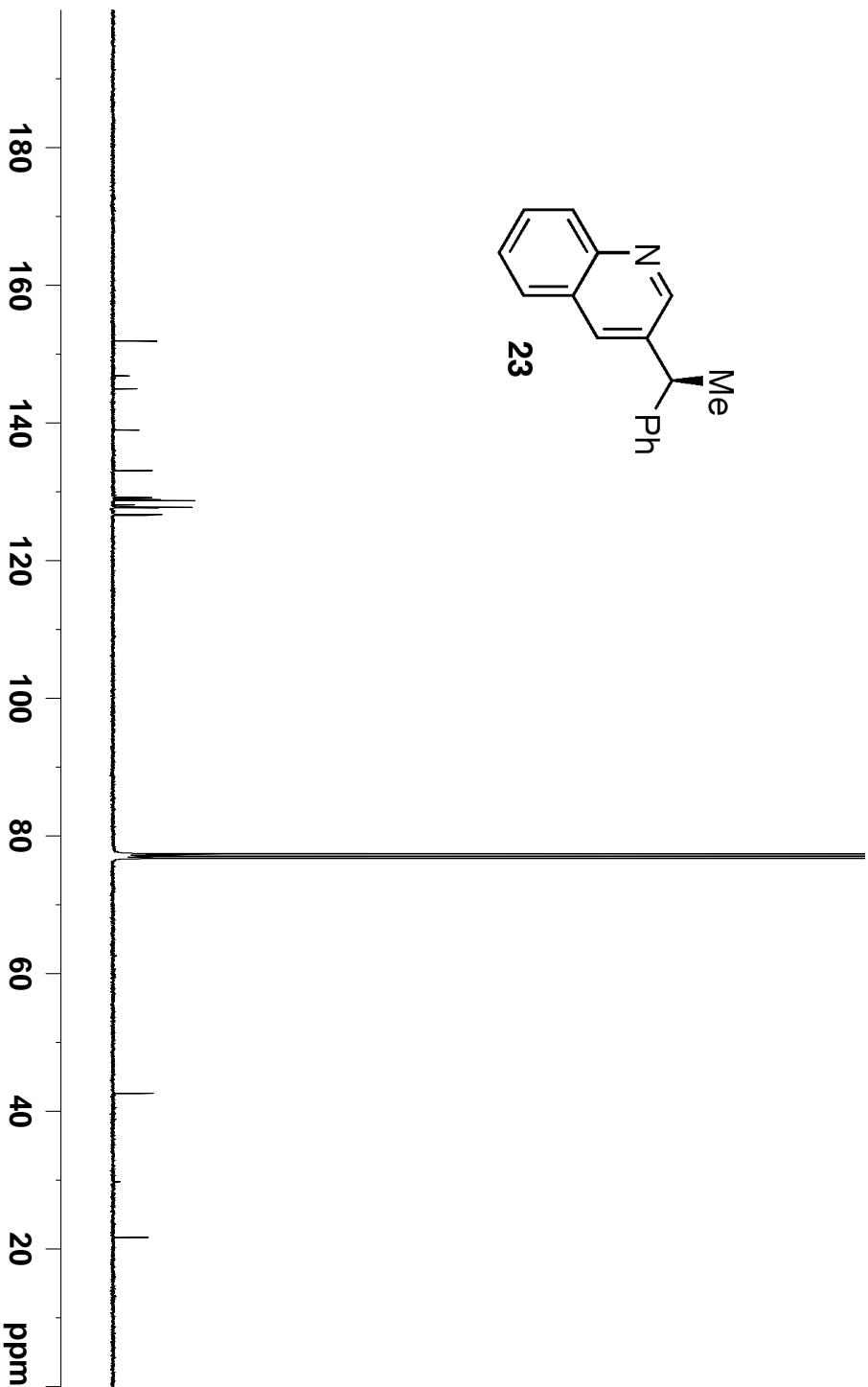
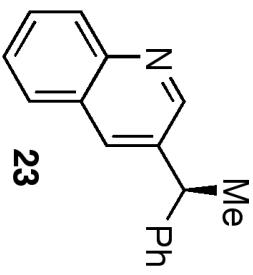


S84

151.88
146.82
144.93
138.91
133.03
129.13
128.85
128.68
128.06
127.70
127.62
126.66
126.56

42.56

21.65



==== CHANNEL f1 =====
 NUC1 13C
 P1 9.25 usec
 PLL 0.55 dB
 PL1W 35.18820572 W
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 4.90 dB
 PL12 20.46 dB
 PL13 21.00 dB
 PL2W 3.30822015 W
 PL12W 0.09195905 W
 PL13W 0.08120718 W
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127690 MHz
 WDM EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Current Data Parameters
 NAME QZ-3-157rs
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date 20121207
 Time 23.46
 INSTRUM spect
 PROBHD 5 mm CPQNP 1H/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 256
 DS 4
 SWH 23980.814 Hz
 FIDRES 0.365918 Hz
 AQ 1.3664756 sec
 RG 512
 DW 20.850 usec
 DE 18.000 usec
 TE 298.2 K
 D1 2.0000000 sec
 D11 0.03000000 sec
 TD0 1

==== CHANNEL f1 =====
 NUC1 13C
 P1 9.25 usec
 PLL 0.55 dB
 PL1W 35.18820572 W
 SFO1 100.6228298 MHz

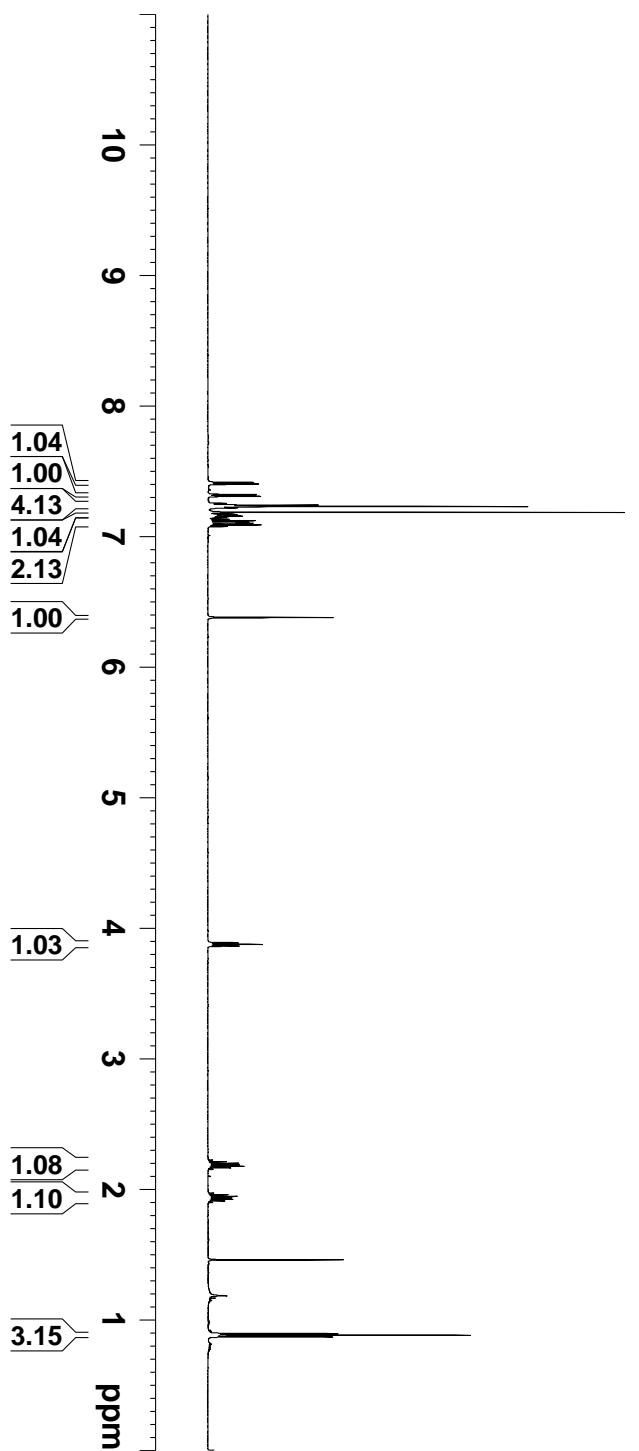
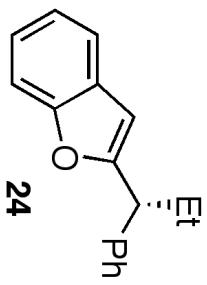
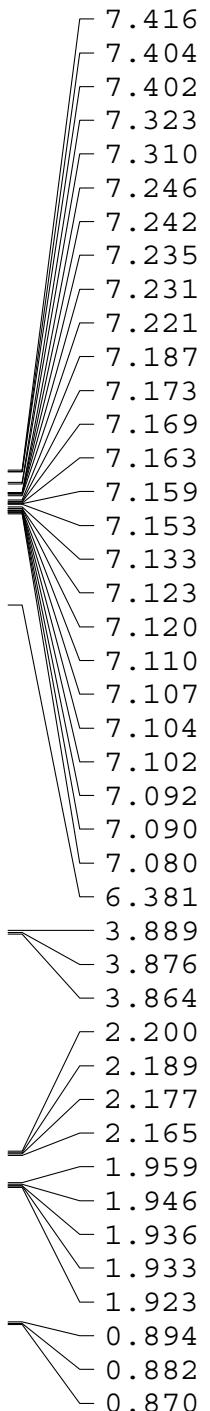
===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 4.90 dB
 PL12 20.46 dB
 PL13 21.00 dB
 PL2W 3.30822015 W
 PL12W 0.09195905 W
 PL13W 0.08120718 W
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127690 MHz
 WDM EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Compound 24 1H NMR



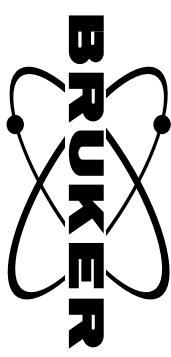
S85



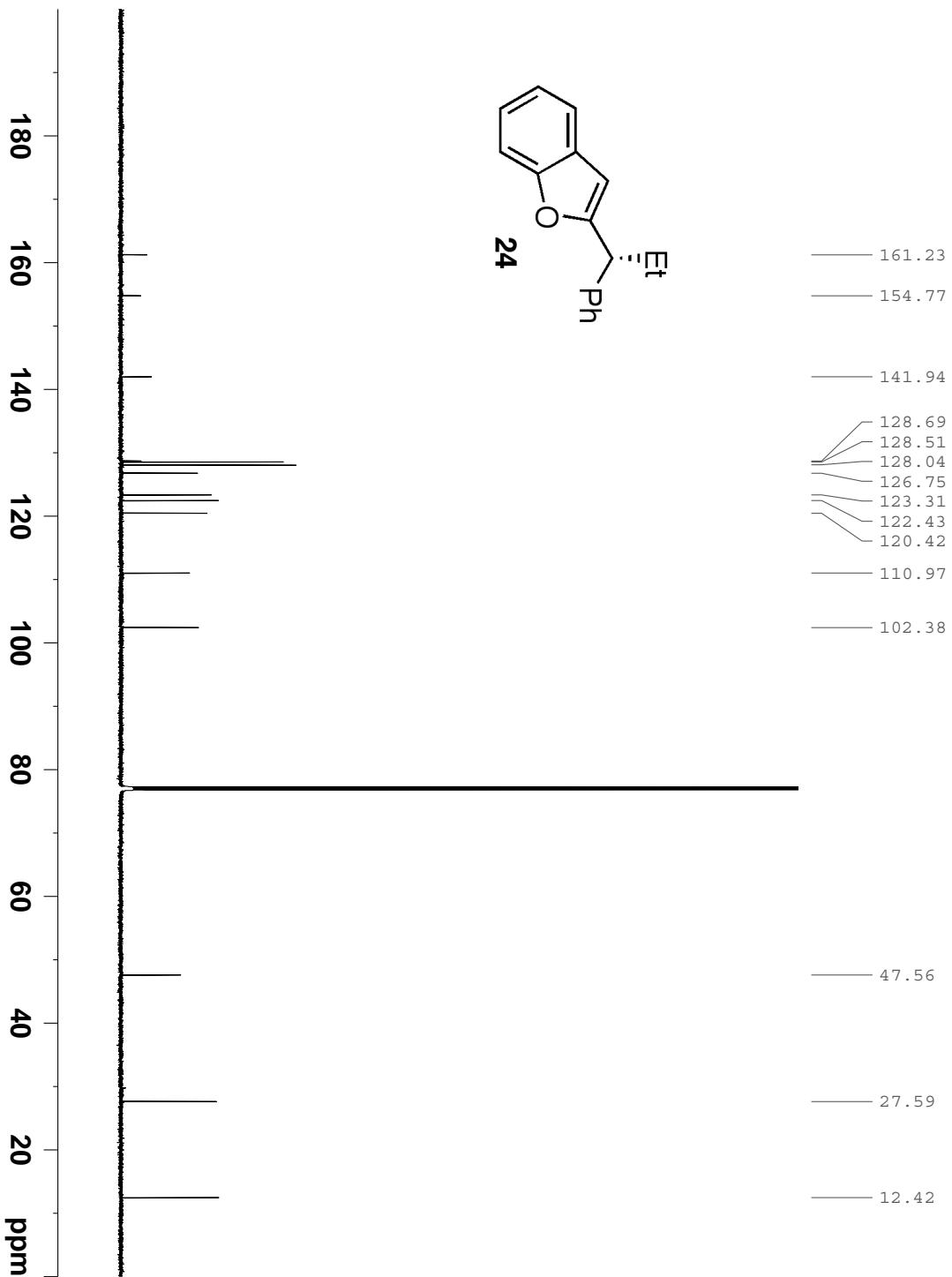
===== CHANNEL f1 ======
 SF01 600.3233018 MHz
 NUC1 1H
 P1 14.80 usec

F2 - Processing parameters
 SI 65536
 SF 600.3200598 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 1.00
 PC

Compound 24 13CNMR



S86



Current	Data	Parameters
NAME	HH-4-083	3rd time
EXPNO	2	
PROCNO	1	

F2 - Acquisition Parameters

Date	20121126
Time	3.32
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg55
TD	65536
SOLVENT	CDCl3
NS	3000
DS	4
SWH	34722.223 Hz
FIDRES	0.529819 Hz
AQ	0.9437684 sec
RG	2050
DW	14.400 usec
DE	8.88 usec
TE	298.1 K
D1	1.1000002 sec
D11	0.03000002 sec
TDO	1 sec

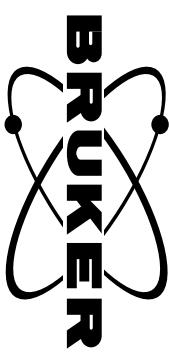
===== CHANNEL f1 =====

SFO1	150.9656784 MHz
NUC1	13C
P1	9.00 usec

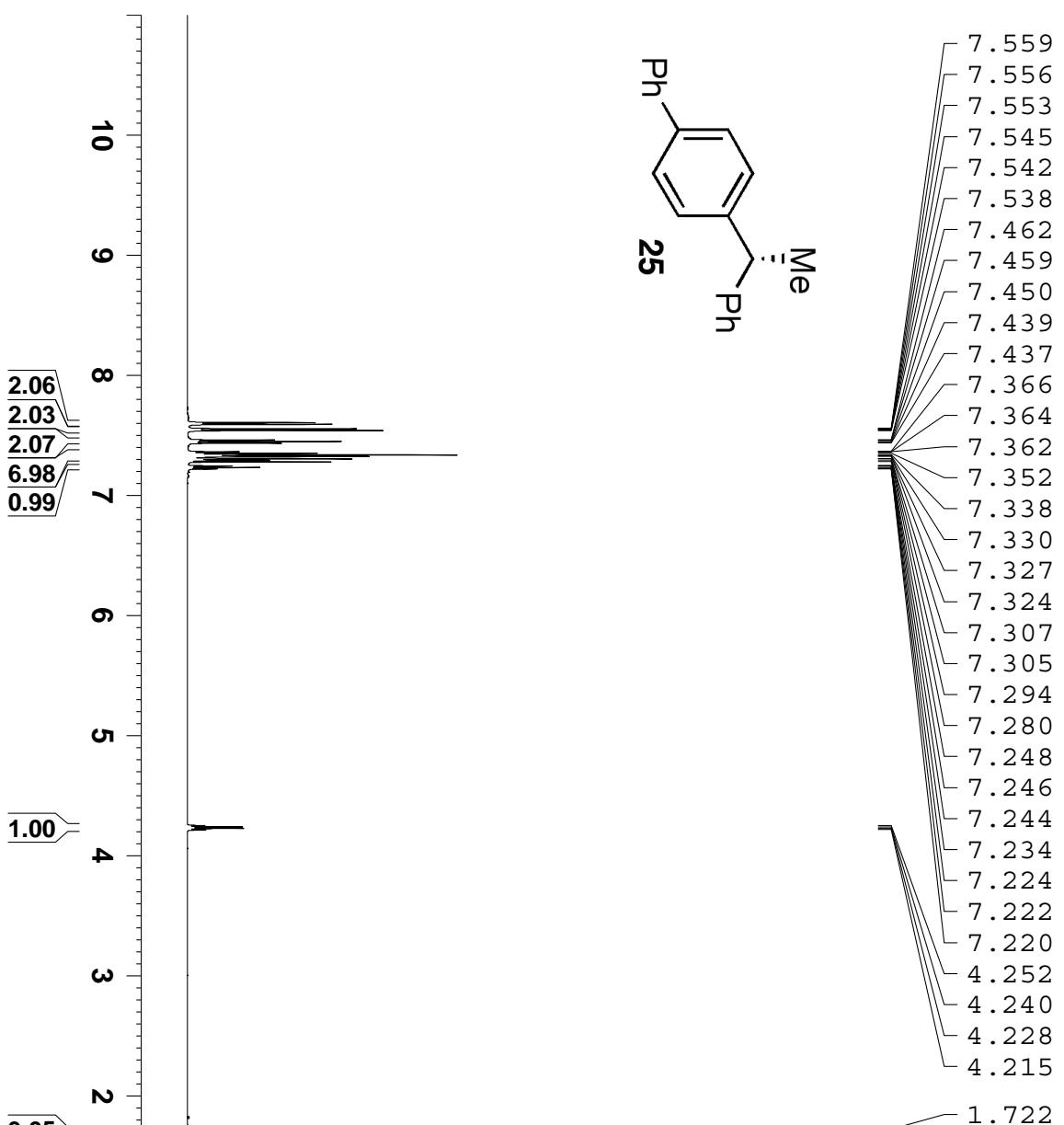
F2 - Processing parameters

SI	32768
SF	150.9505840 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	1.40
PC	

Compound 25 1H NMR

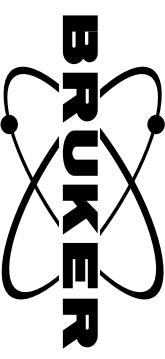


S87

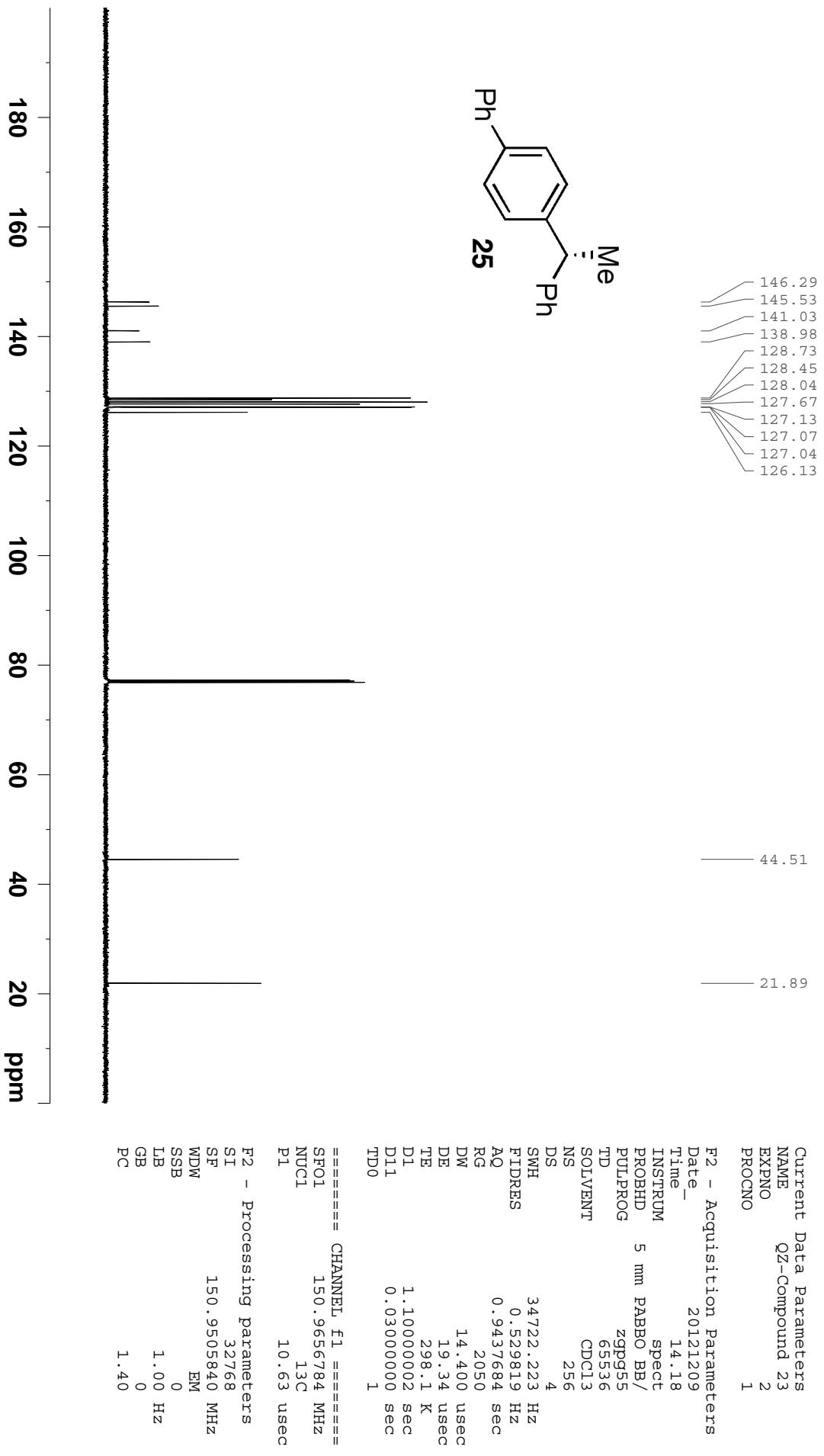


	Current Data Parameters
NAME	QZ-Compound 23
EXPNO	1
PROCNO	1
F2 - Acquisition Parameters	
DATE	20121209
TIME	14.07
INSTRUM	spec
PROBHD	5 mm PABBO BB/
PULPROG	zg30
TD	65536
SOLVENT	CDCl ₃
NS	8
DS	2
SWH	8403.361 Hz
FIDRES	0.128225 Hz
AQ	3.8994420 sec
RG	114
DW	59.500 usec
DE	17.39 usec
TE	298.1 K
D1	1.0000000 sec
TD0	1
===== CHANNEL f1 =====	
SFO1	600.3233018 MHz
NUC1	¹ H
P1	10.77 usec
F2 - Processing parameters	
SI	65536
SF	600.3200047 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	
PC	1.00

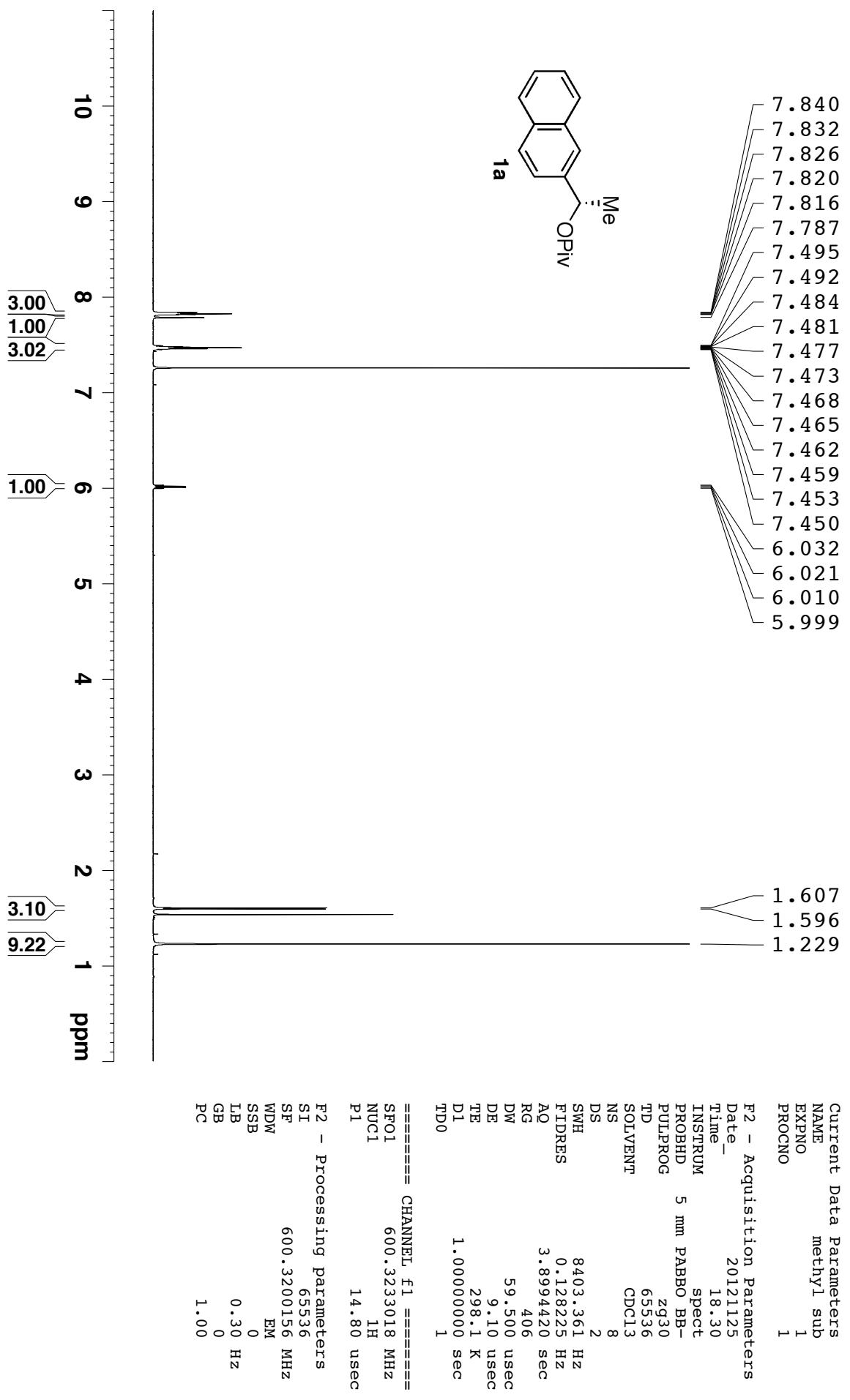
Compound 25 13CNMR



88

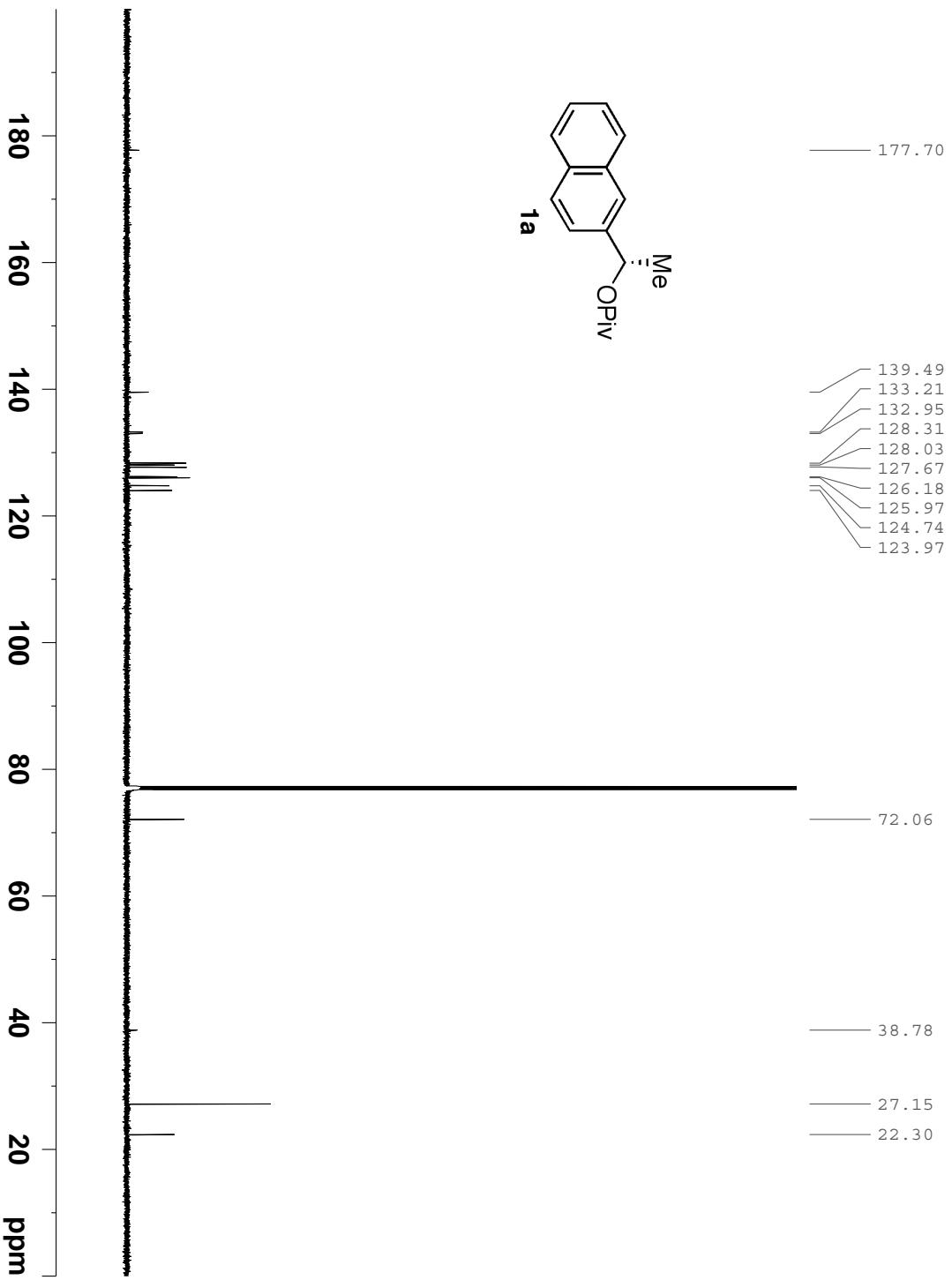


Compound 1a 1H NMR

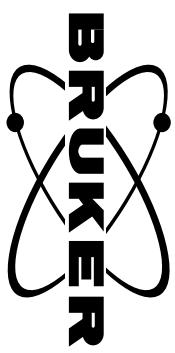


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Compound 1a 13C NMR



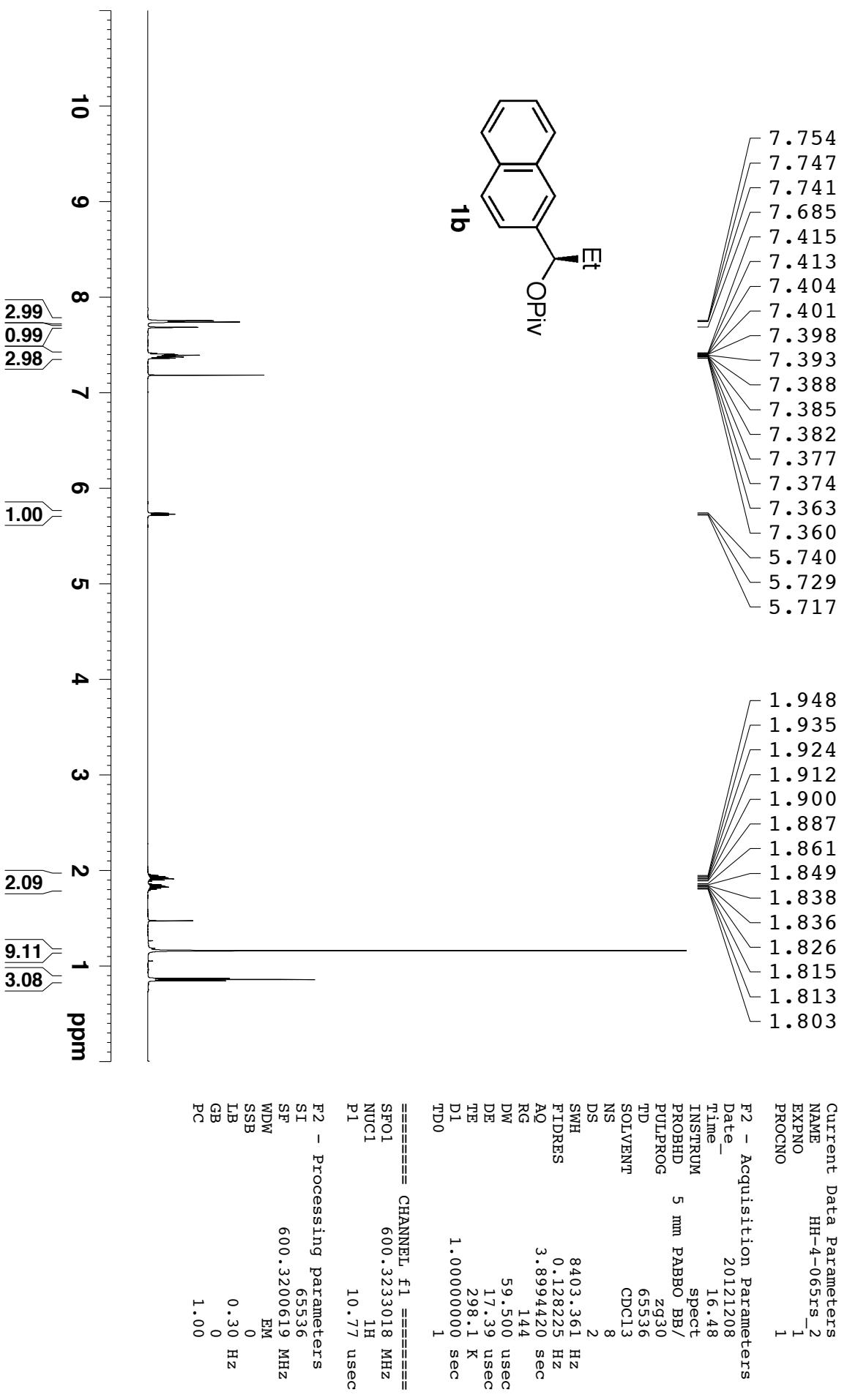
	Current	Data	Parameters
NAME		methyl sub	2
EXPNO			2
PROCNO			1
F2 - Acquisition Parameters			
Date	20121125		
Time	22.39		
INSTRUM	spect		
PROBHD	5 mm	PABBO BB-	
PULPROG	zgpp95		
TD	65536		
SOLVENT	CDCl ₃		
NS	2048		
DS	4		
SWH	34722.223 Hz		
FIDRES	0.529819 Hz		
AQ	0.9437684 sec		
RG	2050		
DW	14.400 usec		
DE	8.88 usec		
TE	298.1 K		
D1	1.1000002 sec		
D11	0.03000002 sec		
TD0	1		
===== CHANNEL f1 =====			
SFO1	150.9656784 MHz		
NUC1	13C		
P1	9.00 usec		
F2 - Processing parameters			
SI	32768		
SF	150.9505840 MHz		
WDW	EM		
SSB	0		
LB	1.00 Hz		
GB	0		
PC	1.40		



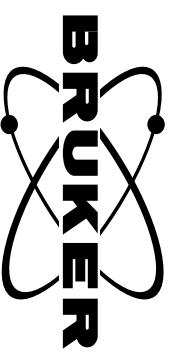
Compound 1b 1H NMR

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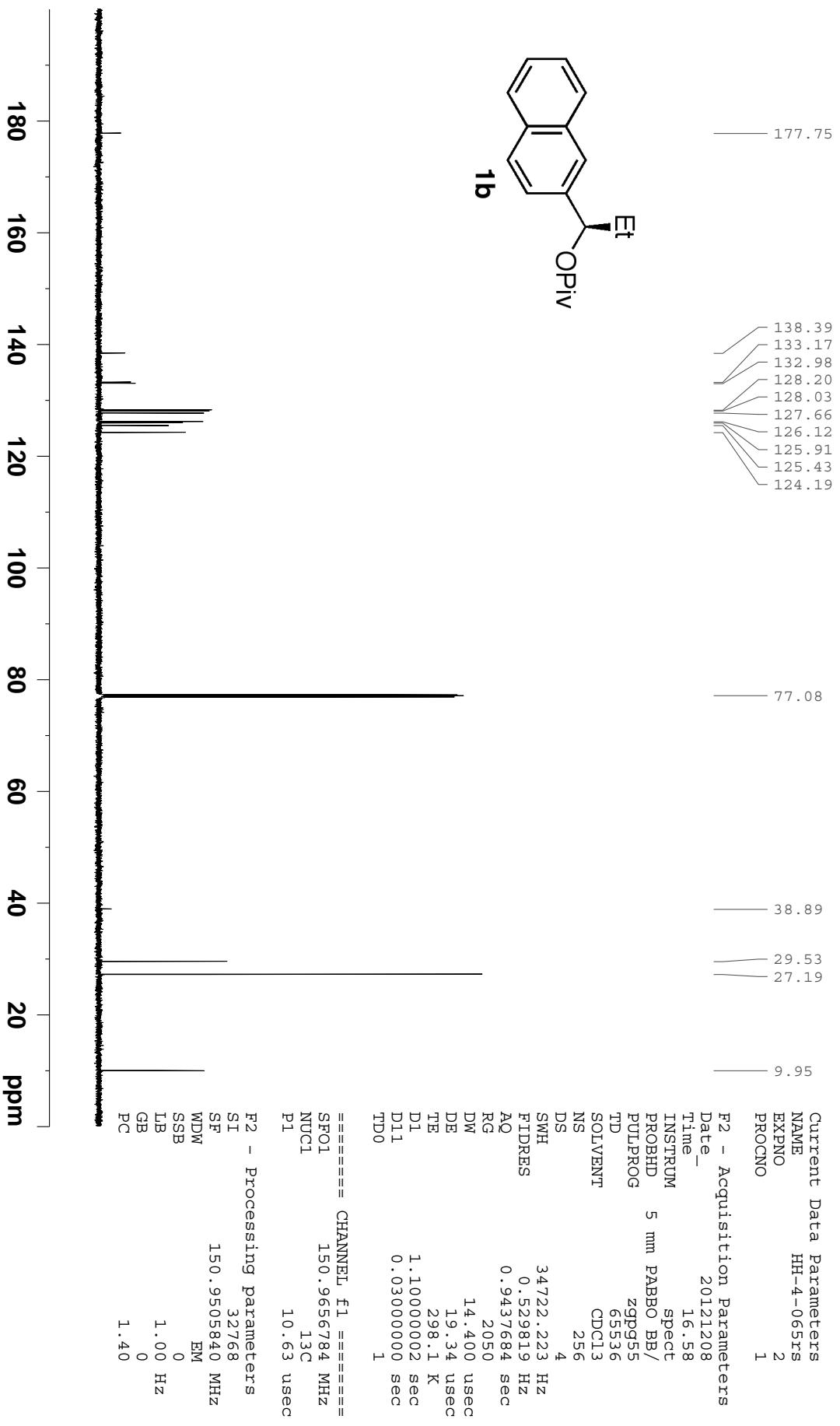
S91



Compound 1b 1H NMR



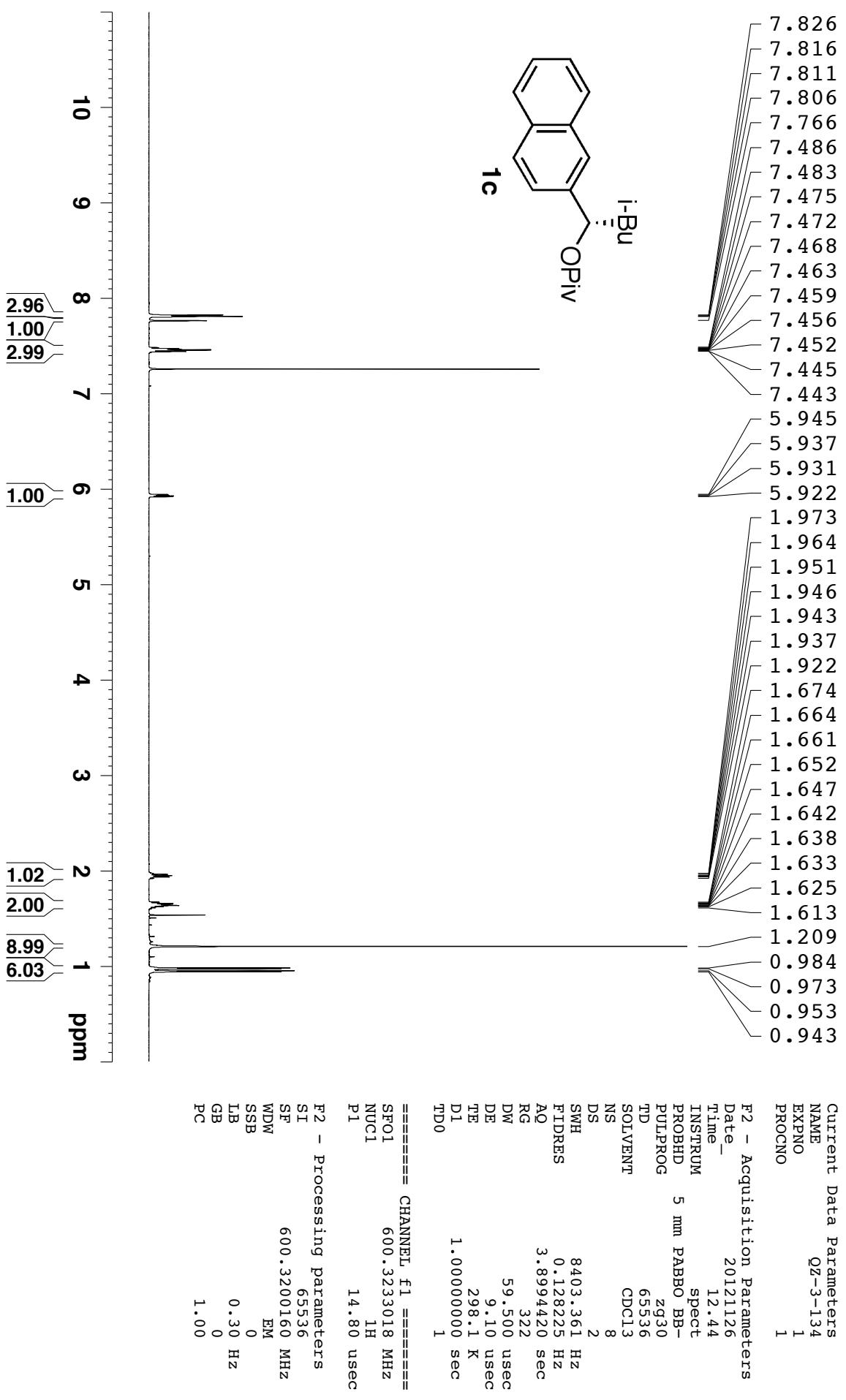
S92



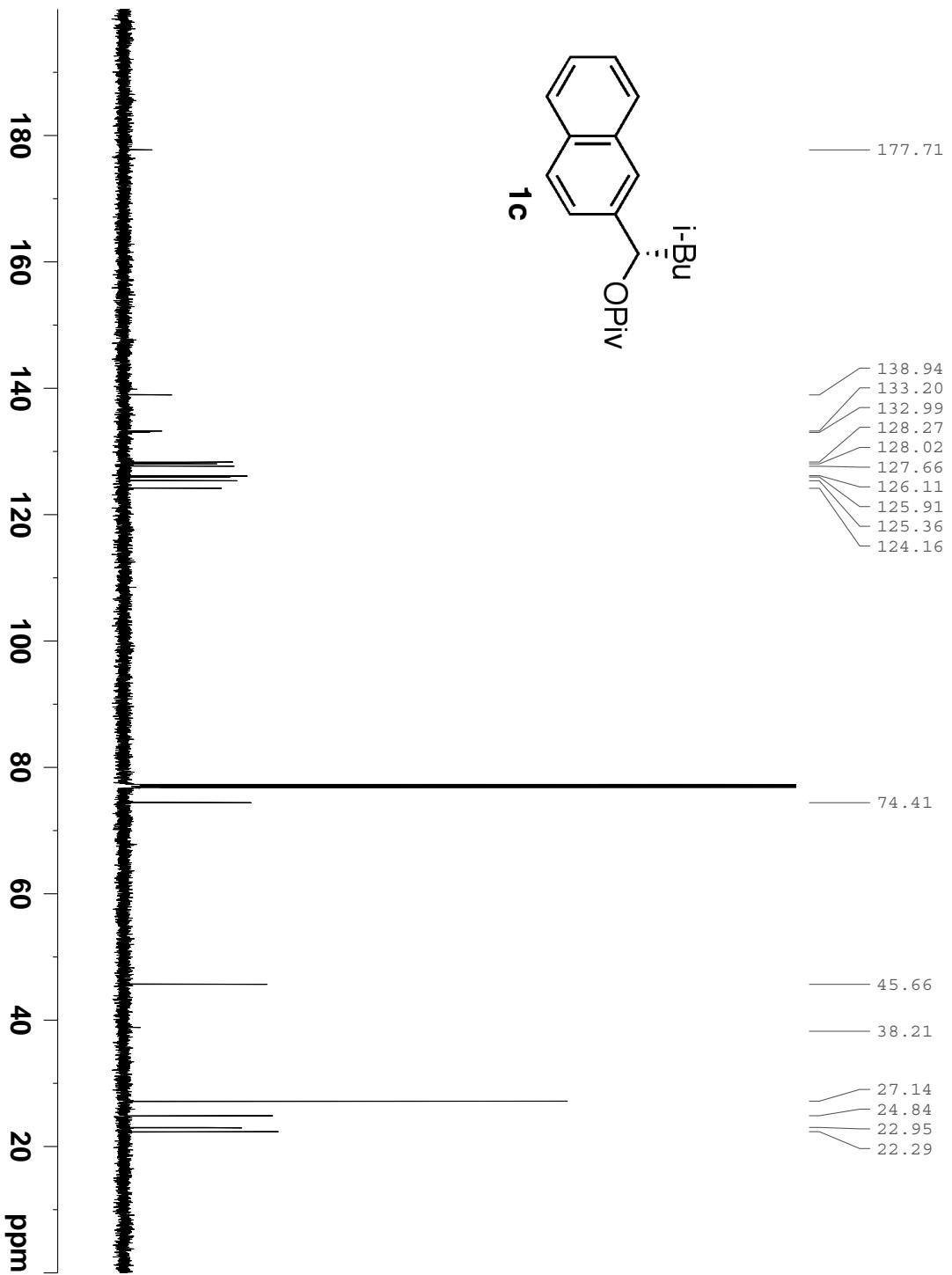
Compound 1c 1H NMR



S93



Compound 1c 13CNMR



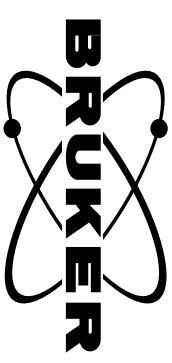
Current Data Parameters
 NAME QZ-3-134
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date 20121126
 Time 12.54
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg95
 TD 65536
 SOLVENT CDCl3
 NS 256
 DS 4
 SWH 34722.223 Hz
 FIDRES 0.529819 Hz
 AQ 0.9437684 sec
 RG 2050
 DW 14.400 usec
 DE 8.88 usec
 TE 298.1 K
 D1 1.1000002 sec
 D11 0.03000002 sec
 TDO 1 sec

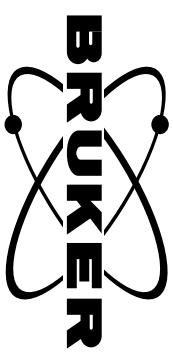
===== CHANNEL f1 =====

SFO1 150.9656784 MHz
 NUC1 13C
 P1 9.00 usec

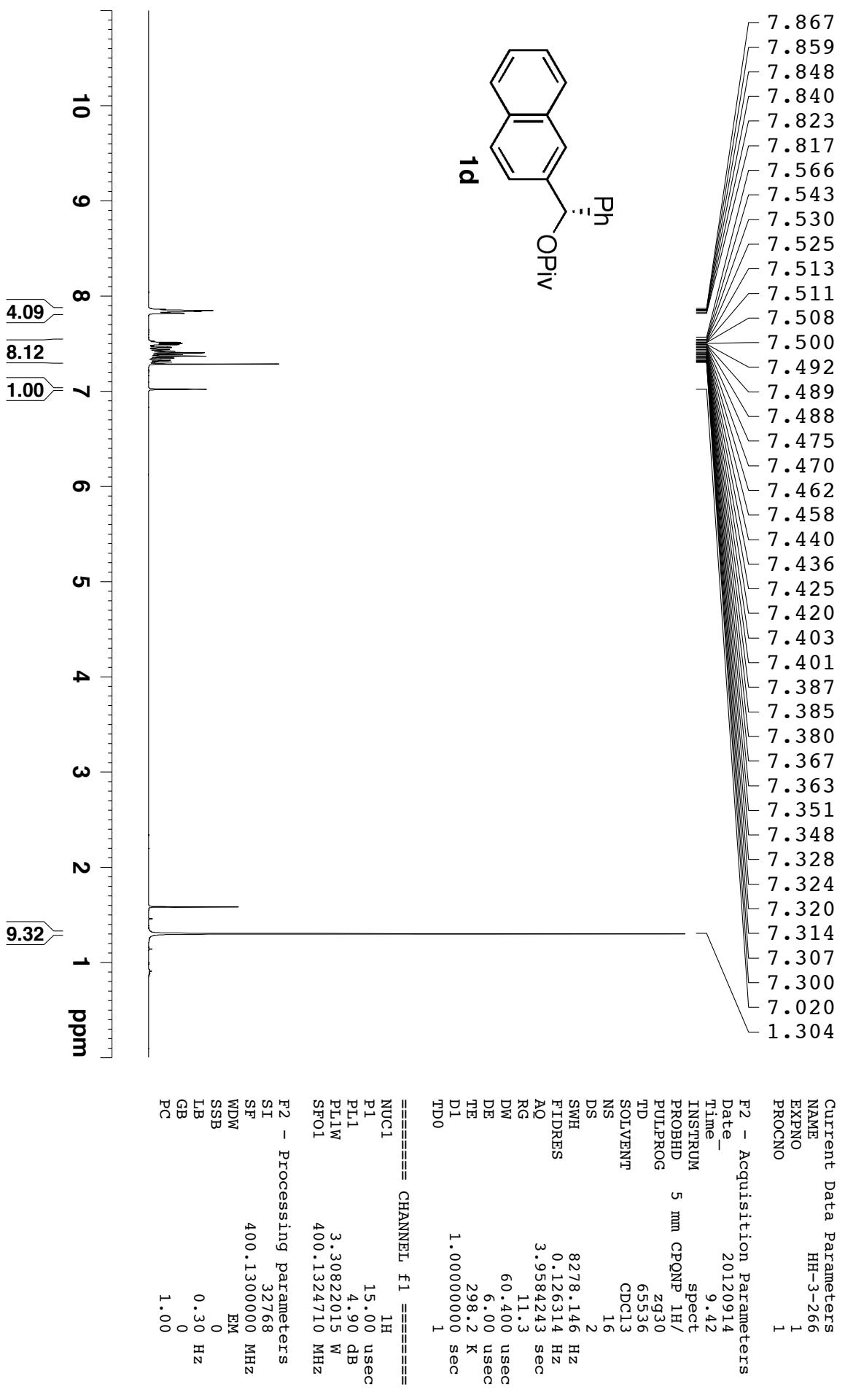
F2 - Processing parameters
 SI 32768
 SF 150.9505840 MHz
 WDW EM
 SSB 0
 LB 0
 GB 1.00 Hz
 PC 1.40



Compound 1d, ^1H NMR



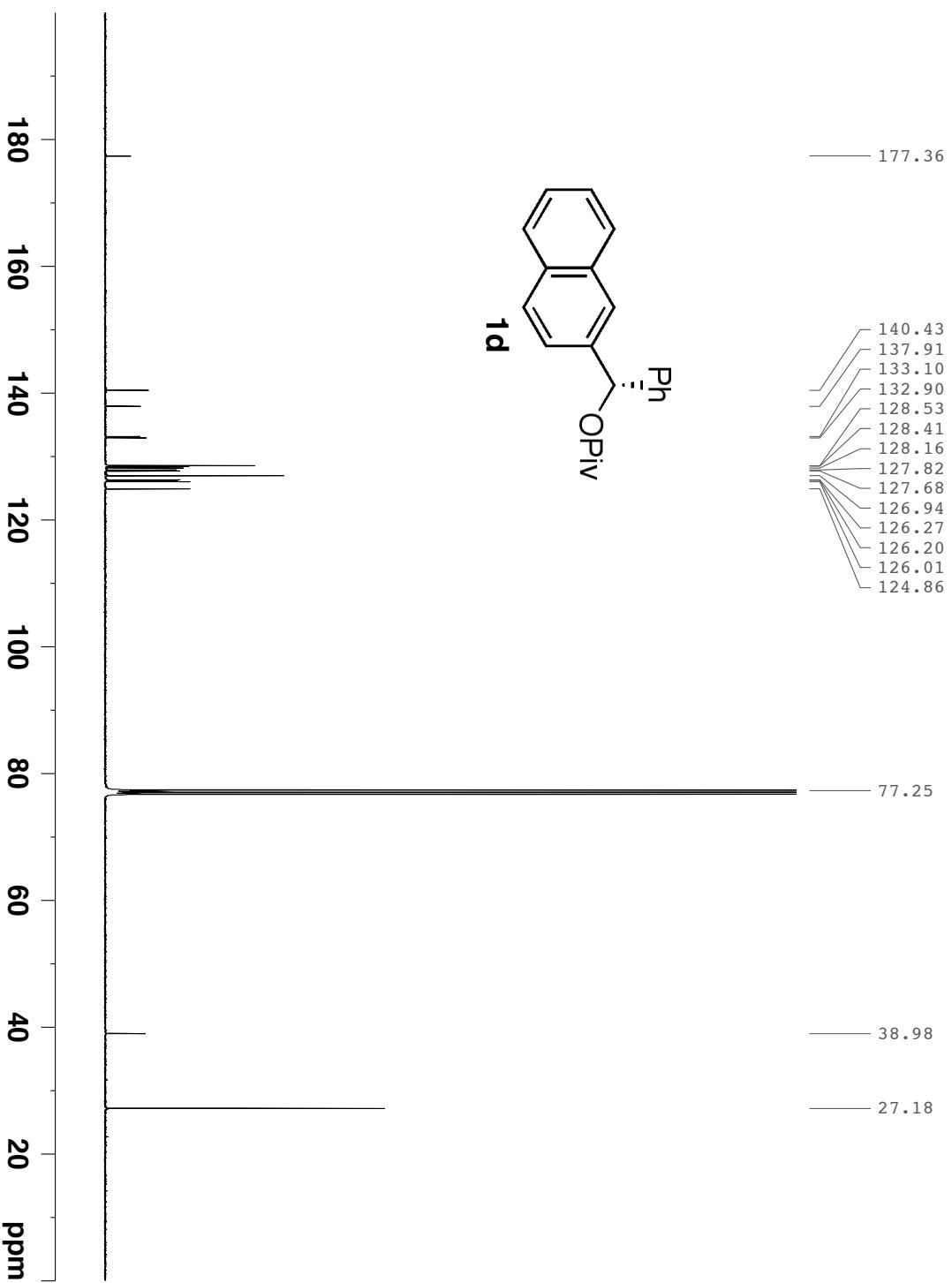
S95



Compound 1d 13C NMR



S96



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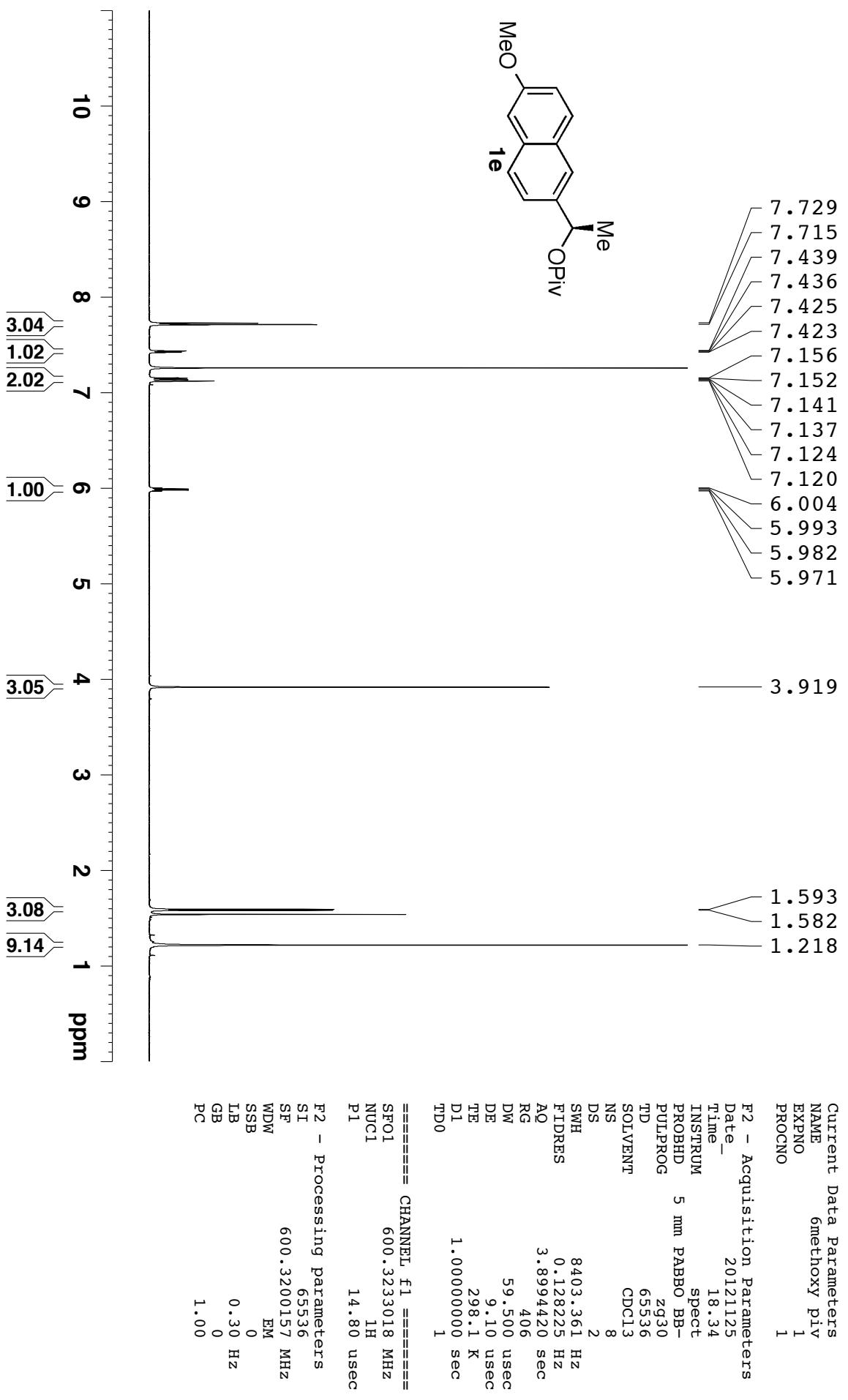
=====
Current Data Parameters
NAME      HH-3-266
EXPNO     2
PROCNO    1
F2 - Acquisition Parameters
Date_   20120916
Time_   14.48
INSTRUM spect
PROBHD  5 mm CPQNP 1H/
PULPROG zgpg30
TD      65536
SOLVENT  CDCl3
NS      1024
DS      4
SWH    2.3980-814 Hz
FIDRES 0.365918 Hz
AQ     1.3664756 sec
RG      512
DW      20.850 usec
DE      18.000 usec
TE      298.2 K
D1      2.0000000 sec
D11     0.03000000 sec
TD0      1
===== CHANNEL f1 =====
NUC1    13C
P1      9.25 usec
PL1     0.55 dB
PL1W    35.18820572 W
SFO1    100.6228298 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2    1H
PCPD2   90.00 usec
PL2     4.90 dB
PL12    20.46 dB
PL13    21.00 dB
PL2W    3.30822015 W
PL12W   0.09195905 W
PL13W   0.08120718 W
SFO2    400.1316005 MHz
=====
F2 - Processing parameters
SI      32768
SF      100.6127690 MHz
WDW    EM
SSB    0
LB     1.00 Hz
GB     0
PC     1.40

```

Compound 1e 1H NMR

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S97



Compound 1e 13CNMR



S98

Current	Data	Parameters
NAME	6methoxy piv	
EXPNO	2	
PROCNO	1	

F2 - Acquisition Parameters

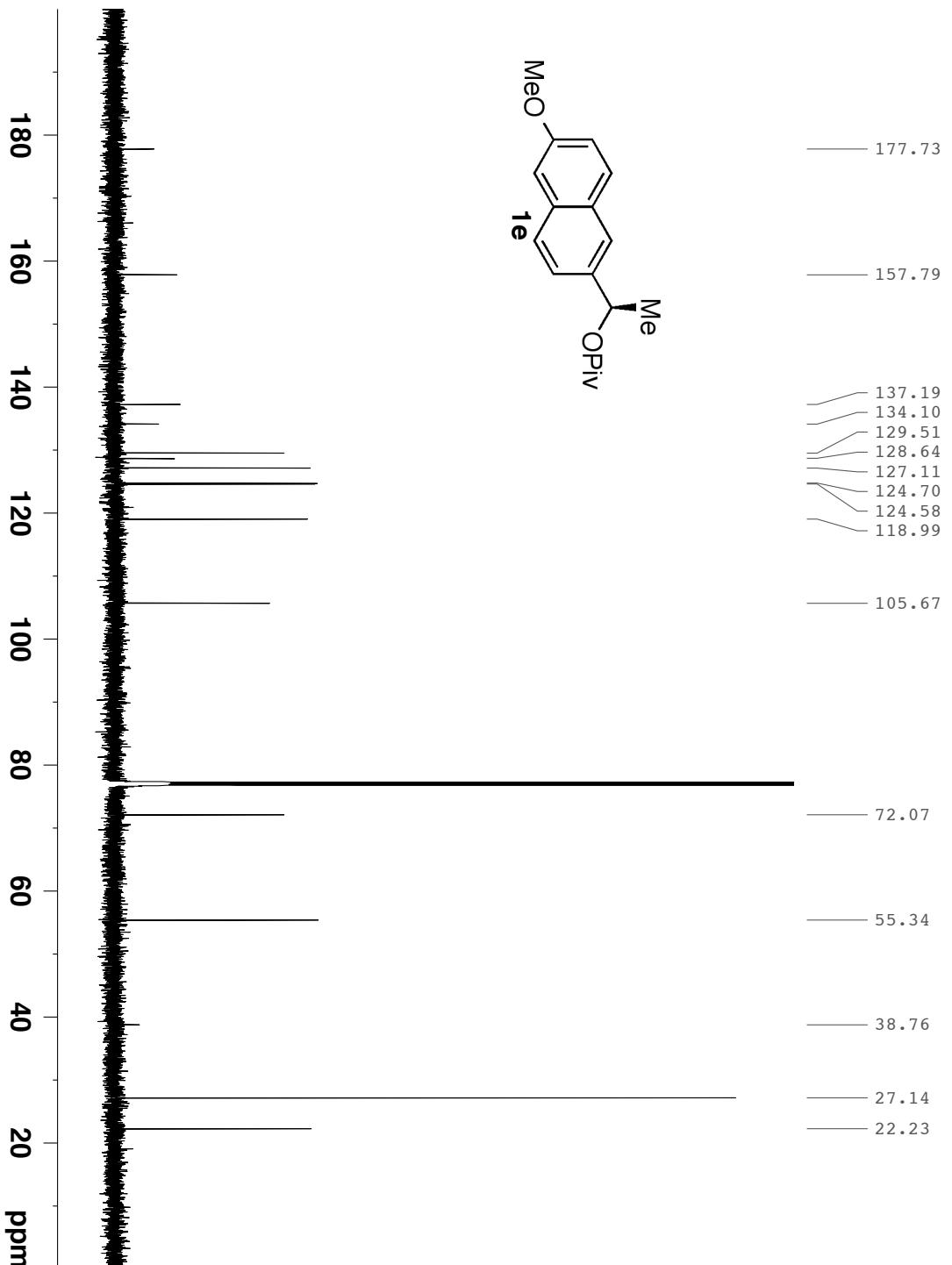
Date	20121125
Time	23.58
INSTRUM	spec
PROBHD	5 mm PABBO BB-
PULPROG	zgpg55
TD	65536
SOLVENT	CDCl ₃
NS	2048
DS	4
SWH	34722.223 Hz
FIDRES	0.529819 Hz
AQ	0.9437684 sec
RG	2050
DW	14.400 usec
DE	8.88 usec
TE	298.1 K
D1	1.1000002 sec
D11	0.03000002 sec
TDO	1 sec

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

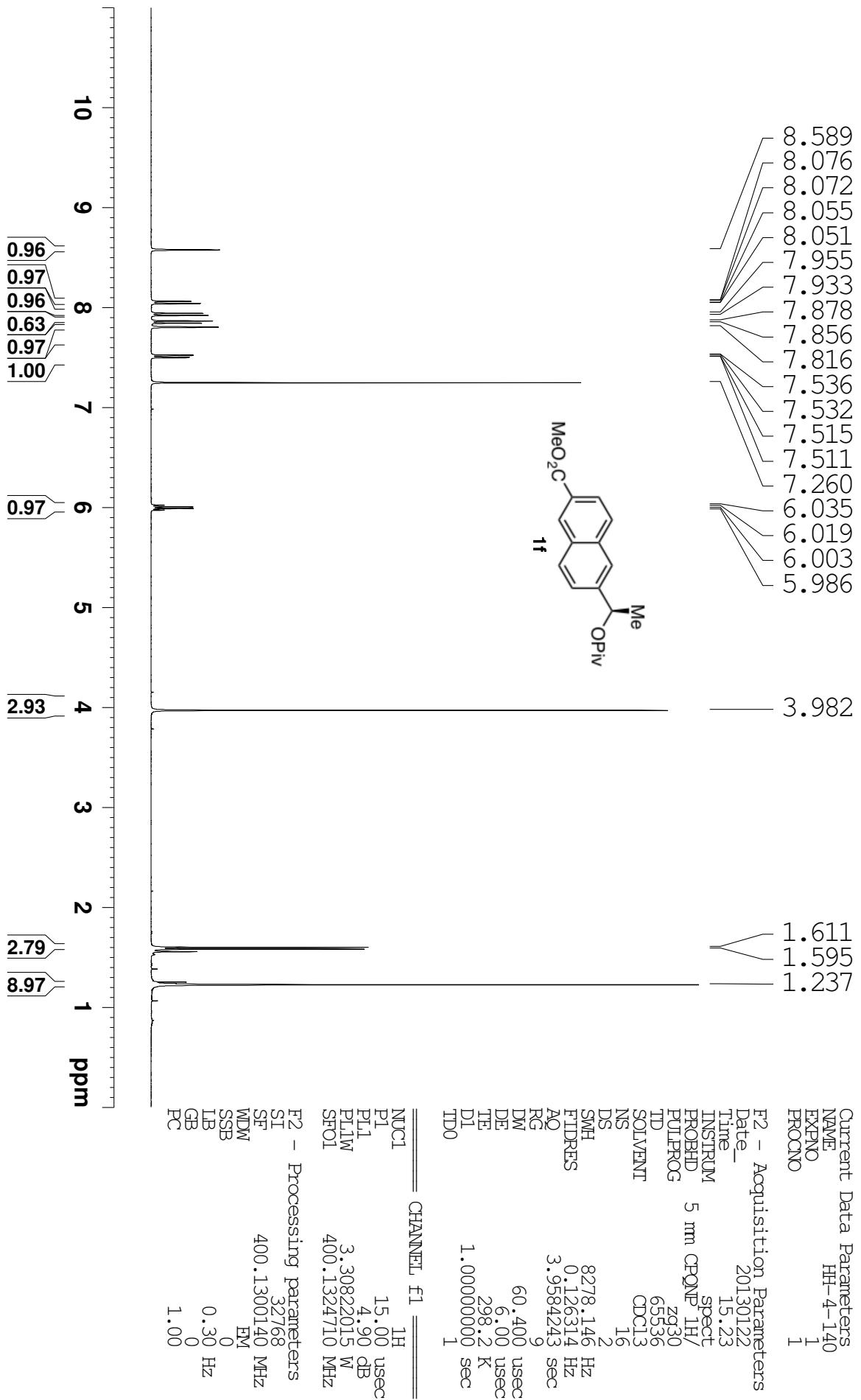
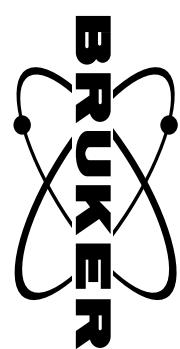
SFO1	150.9656784 MHz
NUC1	¹³ C
P1	9.00 usec

F2 - Processing parameters

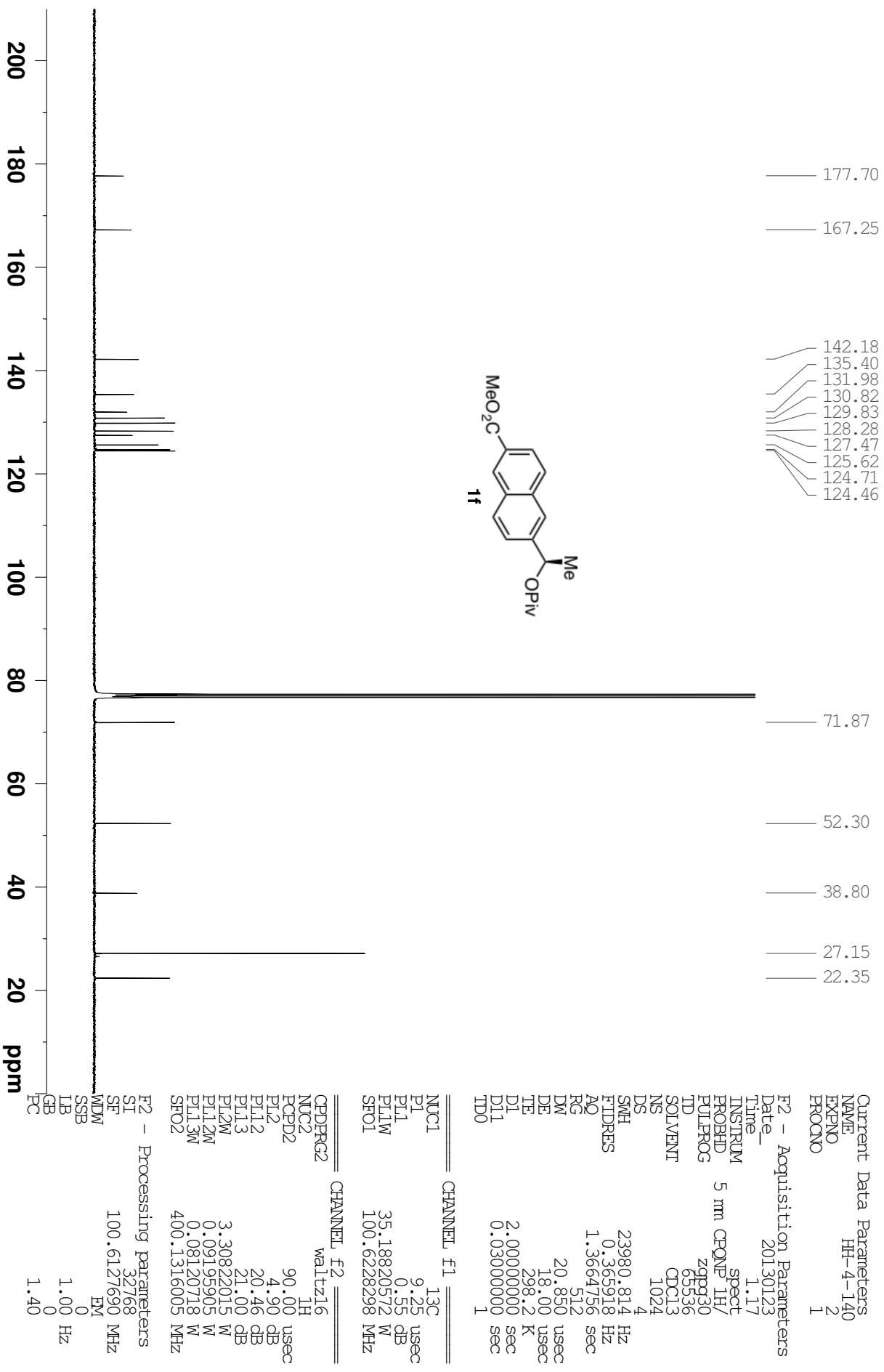
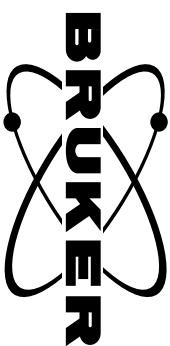
SI	32768
SF	150.9505840 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



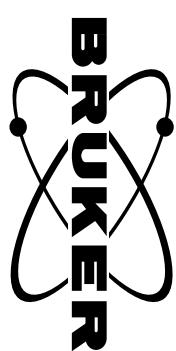
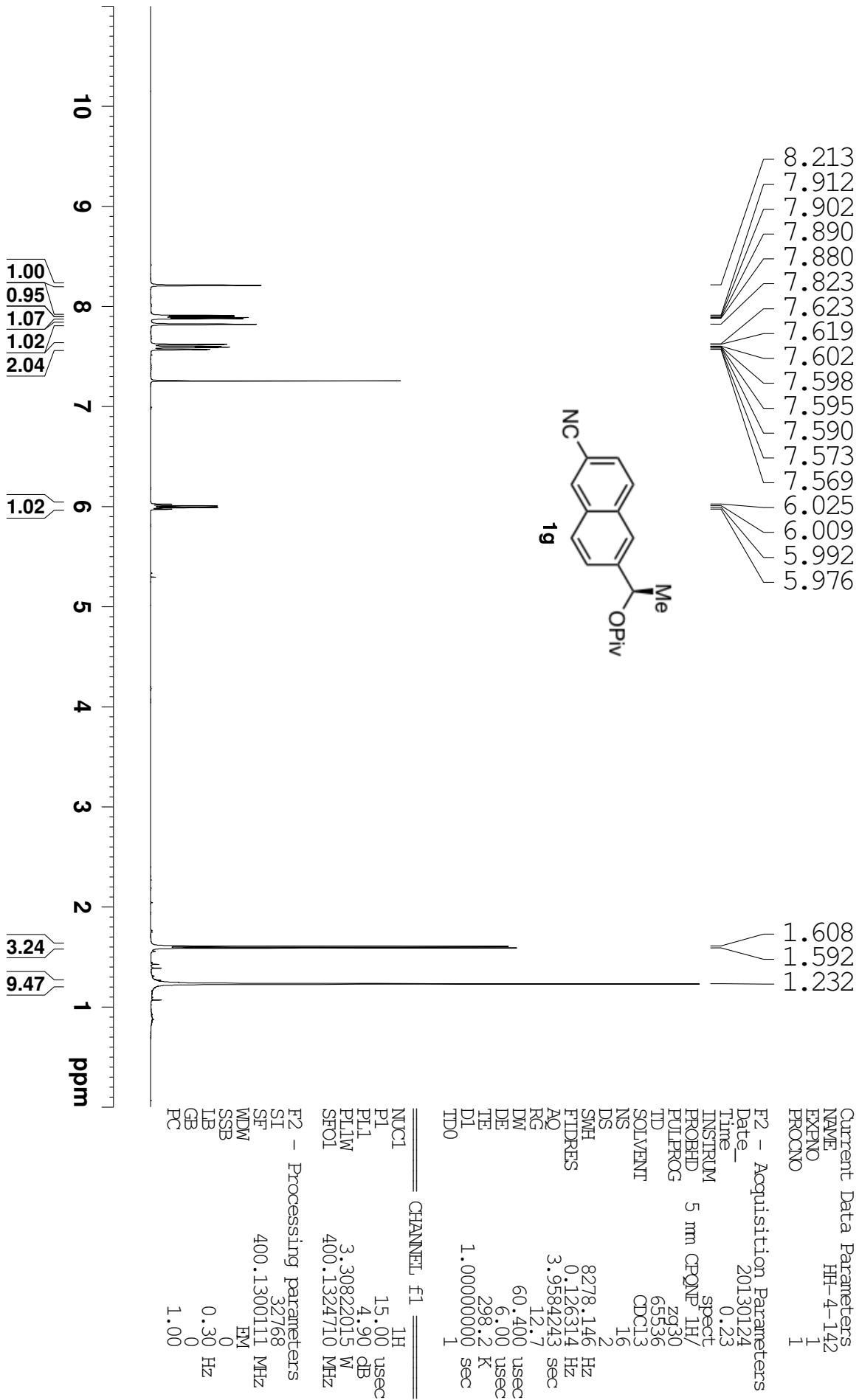
compound 1f 1H NMR



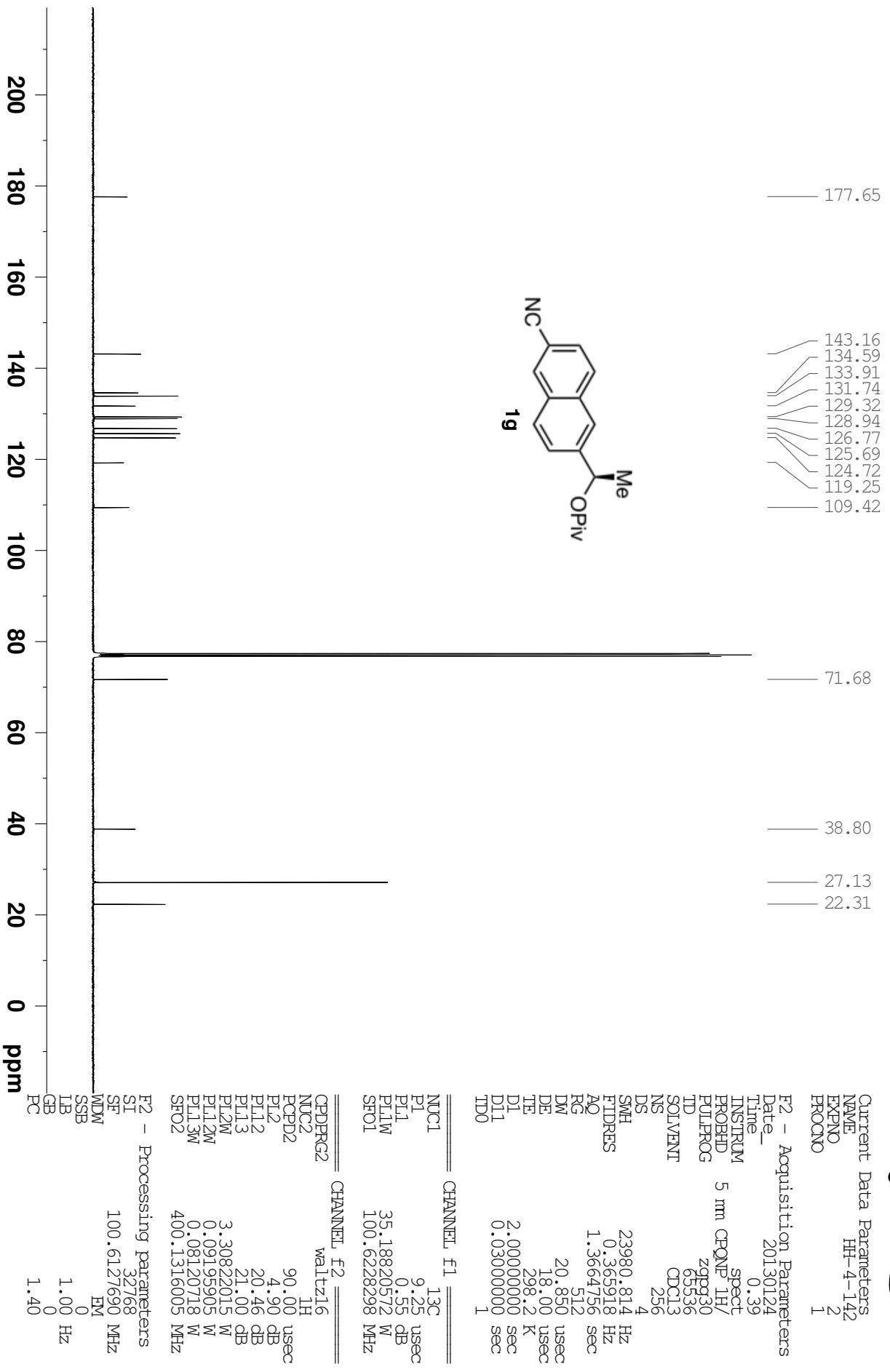
compound 1f 13CNMR



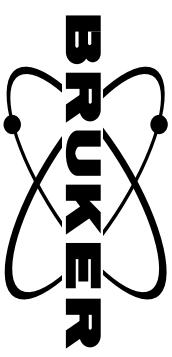
compound 1g 1H NMR



compound 1g 13CNMR



Compound 1h 1H NMR



S103

Current Data Parameters
NAME Q2-Compound 1f 1H NMR
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20121210
Time 9.04

INSTRUM spect

PROBHD 5 mm PABBO BB/

PULPROG zg30

TD 65536

SOLVENT CDCl3

NS 8

DS 2

SWH 8403.361 Hz

FIDRES 0.128225 Hz

AQ 3.8994420 sec

RG 90.5

DW 59.500 usec

DE 17.39 usec

TE 298.1 K

D1 1.0000000 sec

TDO 1

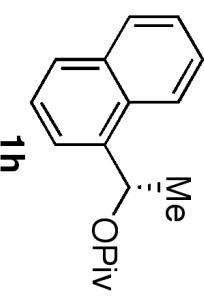
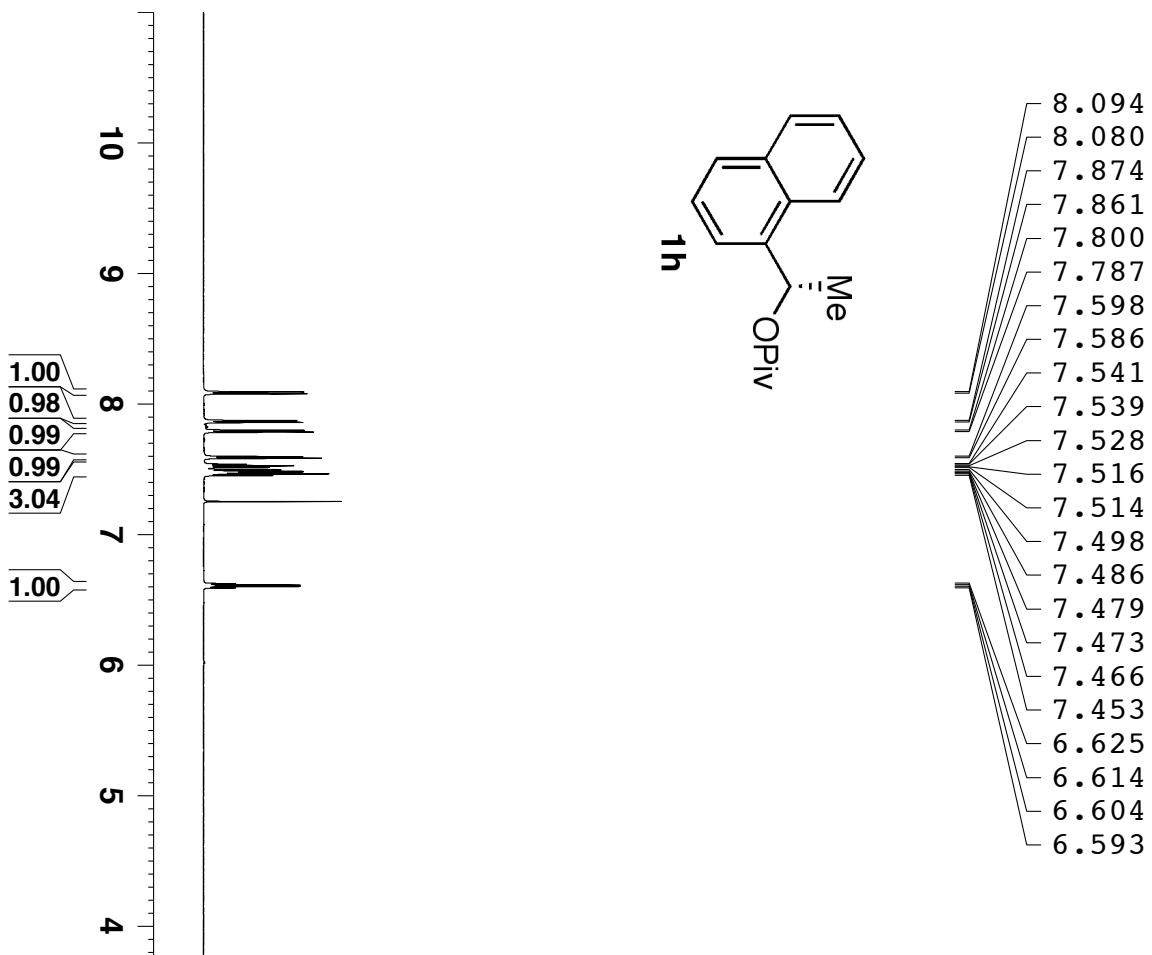
===== CHANNEL f1 =====

SFO1 600.3233018 MHz
NUC1 1H
P1 10.77 usec

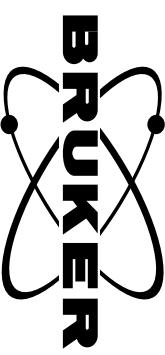
F2 - Processing parameters

SI 65536
SF 600.3200200 MHz
WDW EM

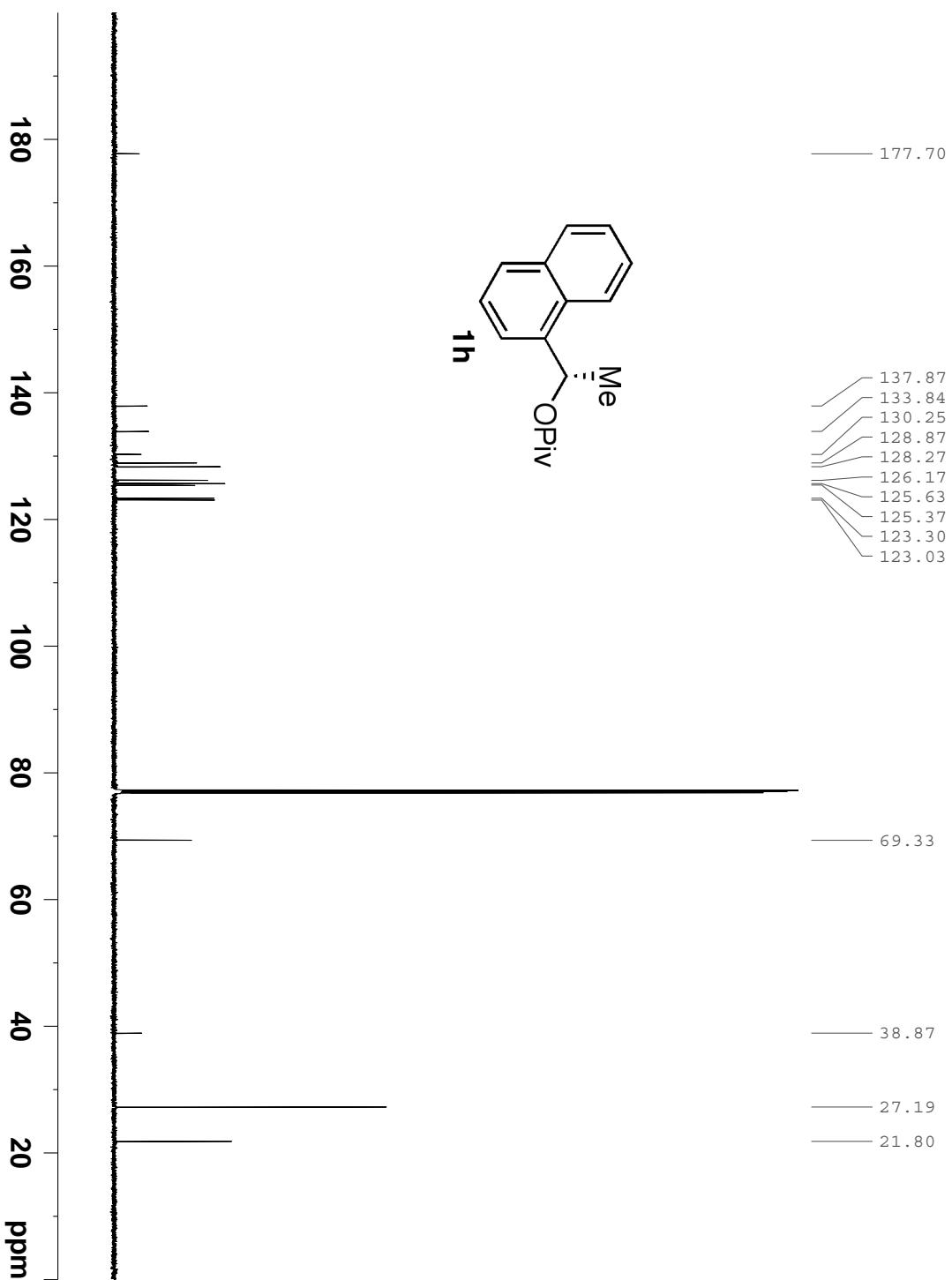
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



Compound 1h 13CNMR

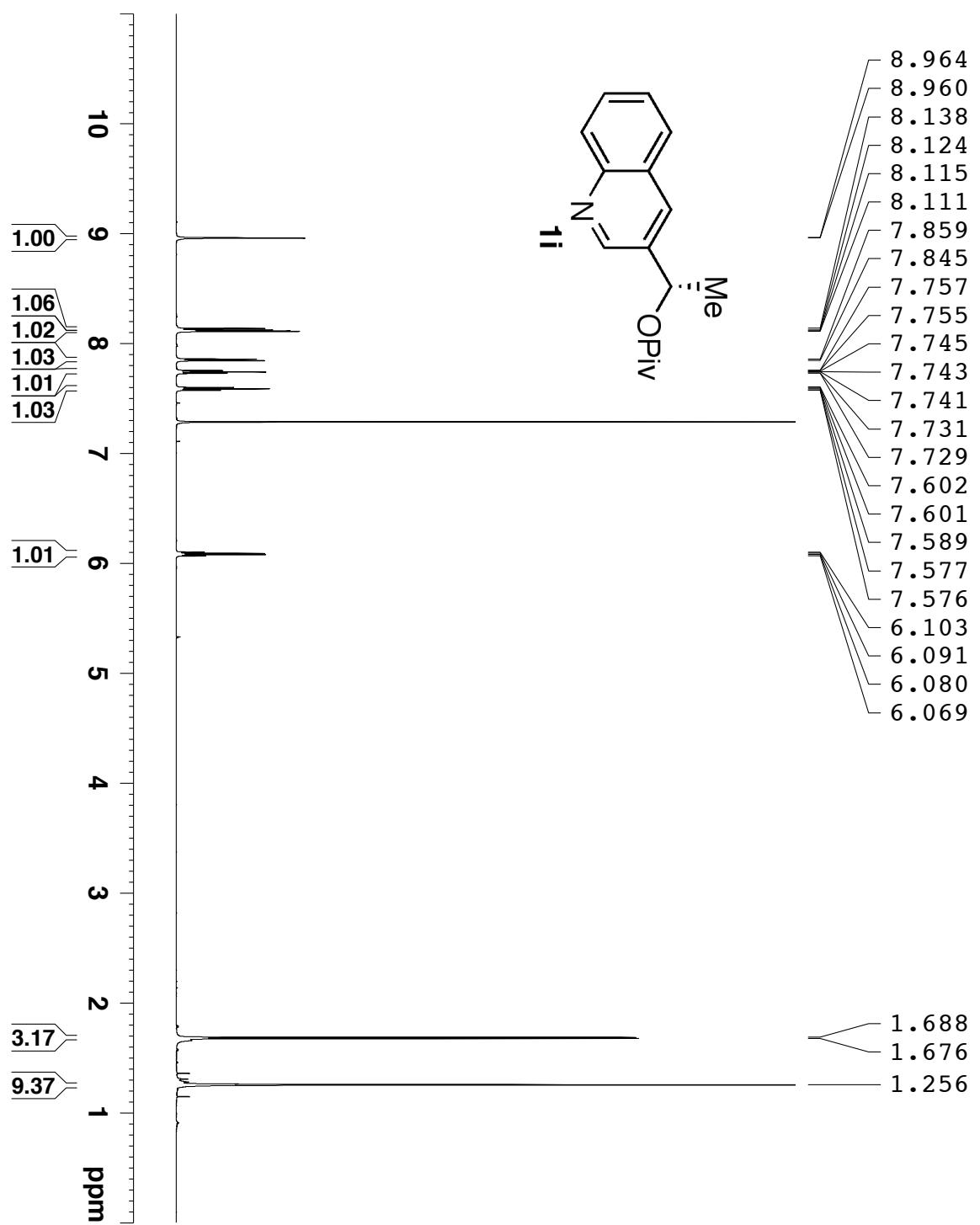


S104



Current	Data	Parameters
NAME	HH-4-077	
EXPNO	3	
PROCNO	1	
F2 - Acquisition Parameters		
Date	20121129	
Time	20.50	
INSTRUM	spect	
PROBHD	5 mm	PABBO BB-
PULPROG	zgppr55	
TD	65536	
SOLVENT	CDCl ₃	
NS	2048	
DS	4	
SWH	34722.223 Hz	
FIDRES	0.529819 Hz	
AQ	0.9437684 sec	
RG	1030	
DW	14.400 usec	
DE	8.88 usec	
TE	298.1 K	
D1	1.1000002 sec	
D11	0.030000002 sec	
TDO	1 sec	
===== CHANNEL f1 =====		
SFO1	150.9656784 MHz	
NUC1	13C	
P1	9.00 usec	
F2 - Processing parameters		
SI	32768	
SF	150.9505840 MHz	
WDW	EM	
SSB	0	
LB	1.00 Hz	
GB	0	
PC	1.40	

Compound 1i 1H NMR

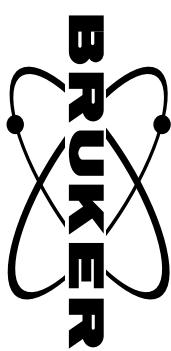


Current Data Parameters
NAME QZ-2-290-2
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date 20121203
Time 22.51
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8403.361 Hz
FIDRES 0.128225 Hz
AQ 3.8994420 sec
RG 161
DW 59.500 usec
DE 17.39 usec
TE 298.1 K
D1 1.0000000 sec
TD0 1

===== CHANNEL f1 =====
SF01 600.3233018 MHz
NUC1 1H
P1 10.77 usec

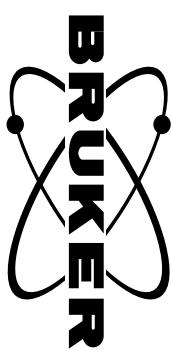
F2 - Processing parameters
SI 65536
SF 600.3200000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 1.00
PC 1.00



Compound 1i 13CNMR



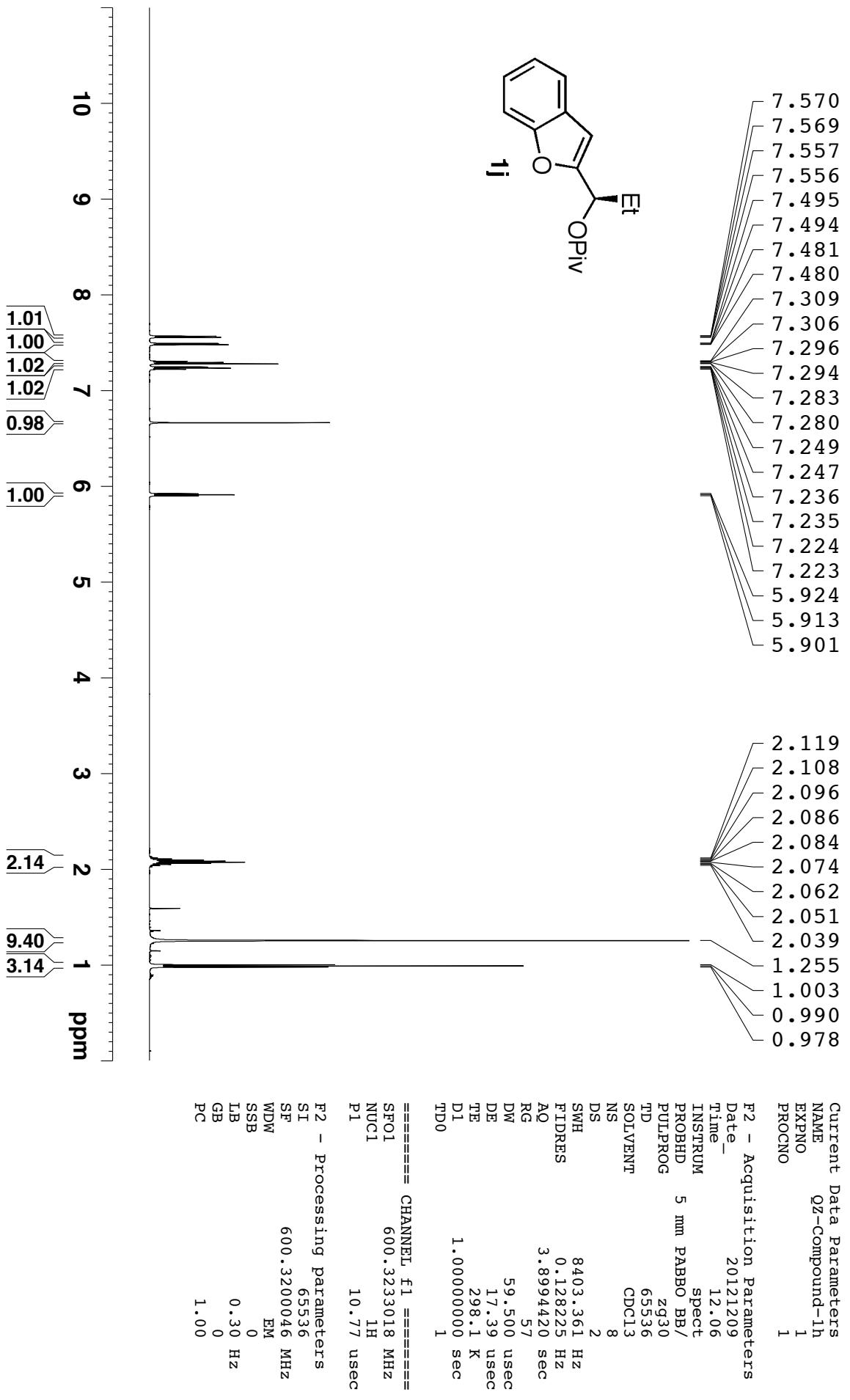
	Current	Data	Parameters
NAME		QZ-2-290-2	
EXPNO		2	
PROCNO		1	
F2 - Acquisition Parameters			
DATE	20121203		
TIME	23.01		
INSTRUM	spect		
PROBHD	5 mm PABBO BB/		
PULPROG	zgpg55		
TD	65536		
SOLVENT	CDCl ₃		
NS	256		
DS	4		
SWH	34722.223 Hz		
FIDRES	0.529819 Hz		
AQ	0.9437684 sec		
RG	2050		
DW	14.400 usec		
DE	19.34 usec		
TE	298.1 K		
D1	1.1000002 sec		
D11	0.030000002 sec		
TDO	1 sec		
===== CHANNEL f1 =====			
SFO1	150.9656784 MHz		
NUC1	¹³ C		
P1	10.63 usec		
F2 - Processing parameters			
SI	32768		
SF	150.9505840 MHz		
WDW	EM		
SSB	0		
LB	1.00 Hz		
GB	1.40		
PC			



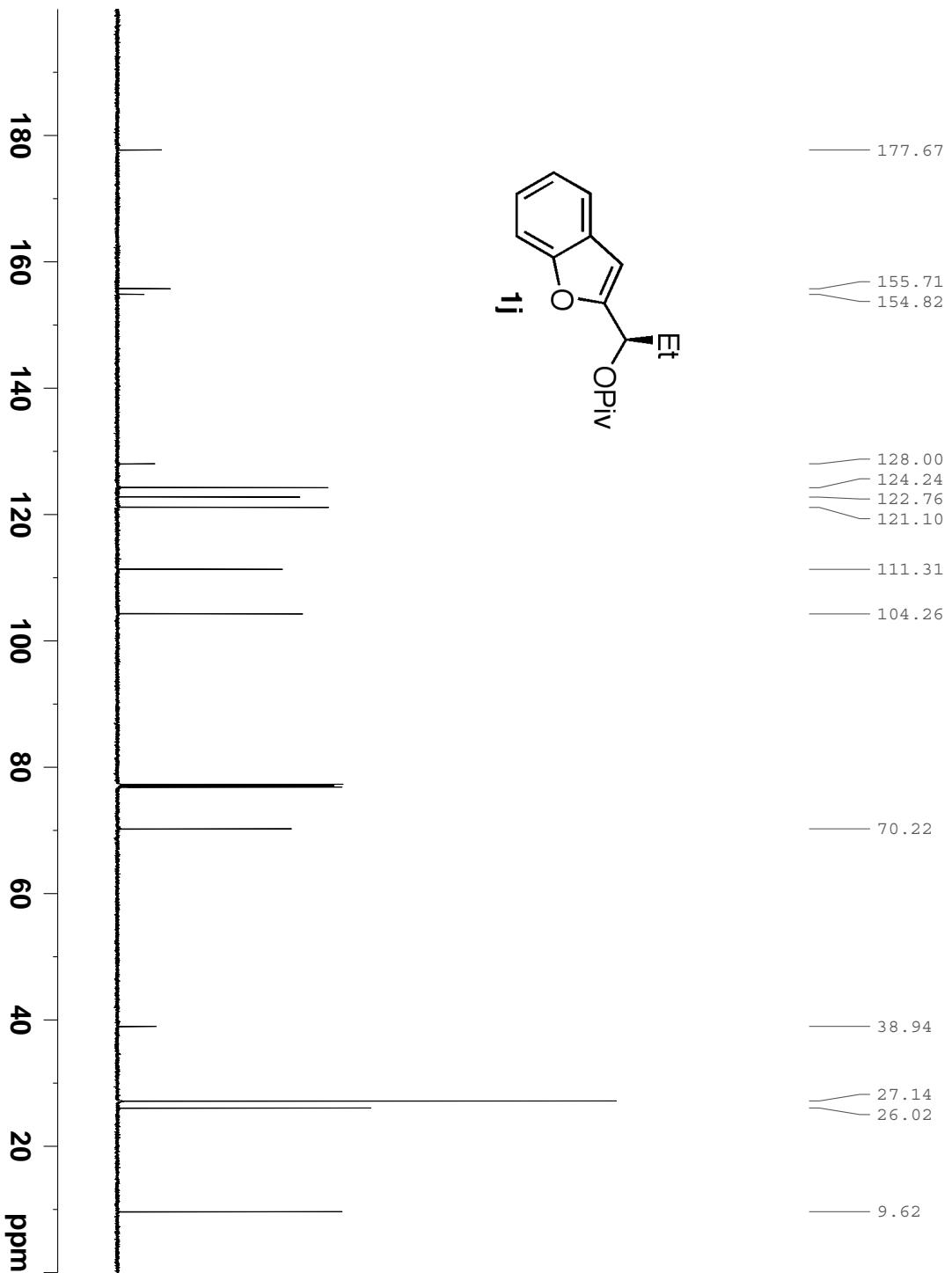
Compound 1j 1H NMR

BRUKER

S107



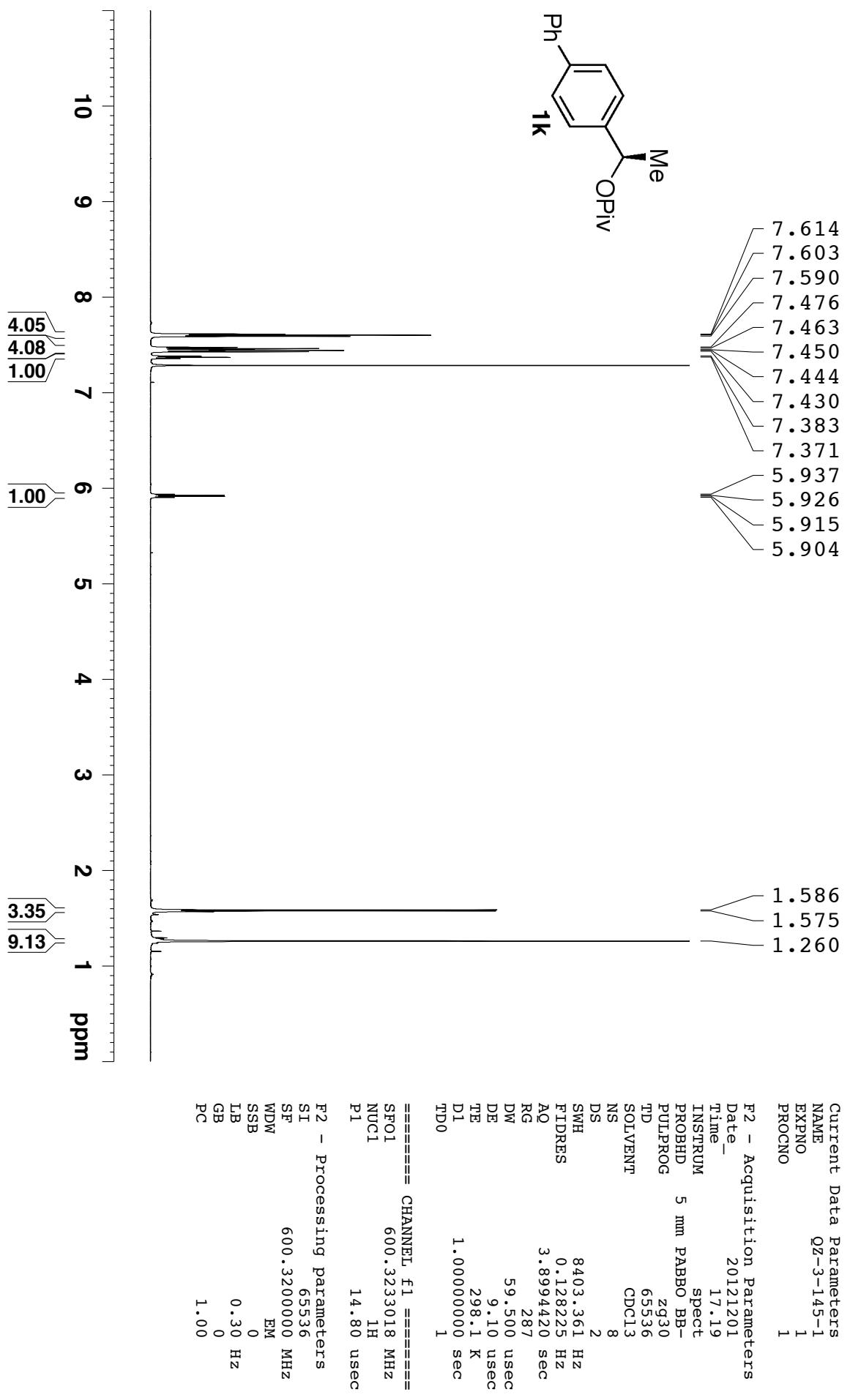
Compound 1j 13CNMR



BRUKER

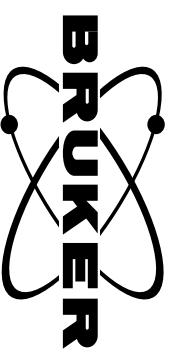
	Current Data Parameters
NAME	QZ-Compound-1h
EXPNO	2
PROCNO	1
 F2 - Acquisition Parameters	
Time	12.16
INSTRUM	spec
PROBHD	5 mm PABBO BB/
PULPROG	zgpg95
TD	65536
SOLVENT	CDCl3
NS	256
DS	4
SWH	34722.223 Hz
FIDRES	0.529819 Hz
AQ	0.9437684 sec
RG	2050
DW	14.400 usec
DE	19.34 usec
TE	298.1 K
D1	1.1000002 sec
D11	0.03000002 sec
TDO	1 sec
 ===== CHANNEL f1 =====	
SFO1	150.9656784 MHz
NUC1	13C
P1	10.63 usec
 F2 - Processing parameters	
SI	32768
SF	150.9505840 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	1.40
PC	

Compound 1k 1H NMR

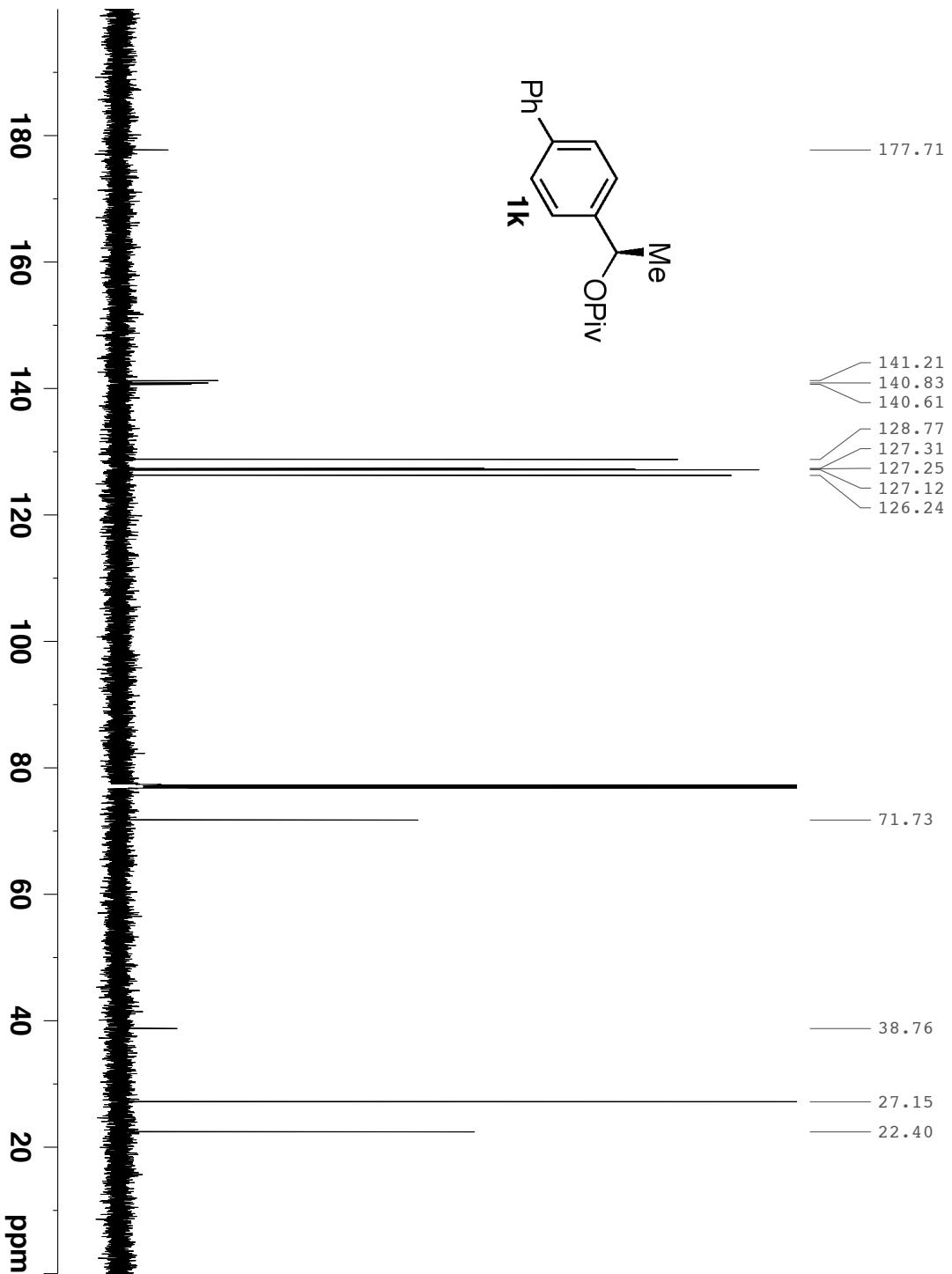


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Compound 1k 13CNMR



S110



Current Data Parameters
NAME QZ-3-145-1
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date 20121201
Time 17.29
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg55
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 34722.223 Hz
FIDRES 0.529819 Hz
AQ 0.9437684 sec
RG 2050
DW 14.400 usec
DE 8.88 usec
TE 298.1 K
D1 1.1000002 sec
D11 0.03000002 sec
TDO 1 sec

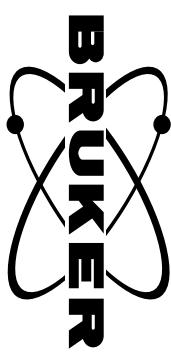
===== CHANNEL f1 =====

SFO1 150.9656784 MHz
NUC1 13C
P1 9.00 usec

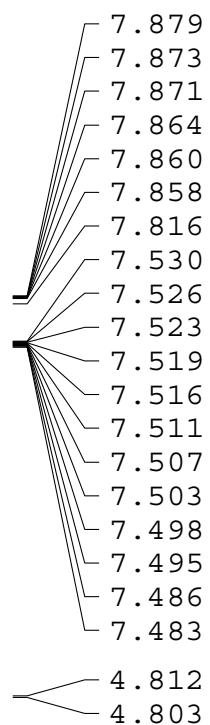
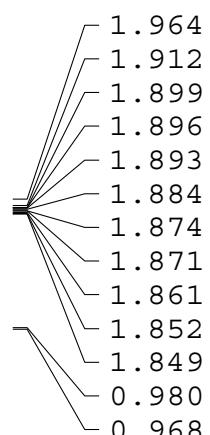
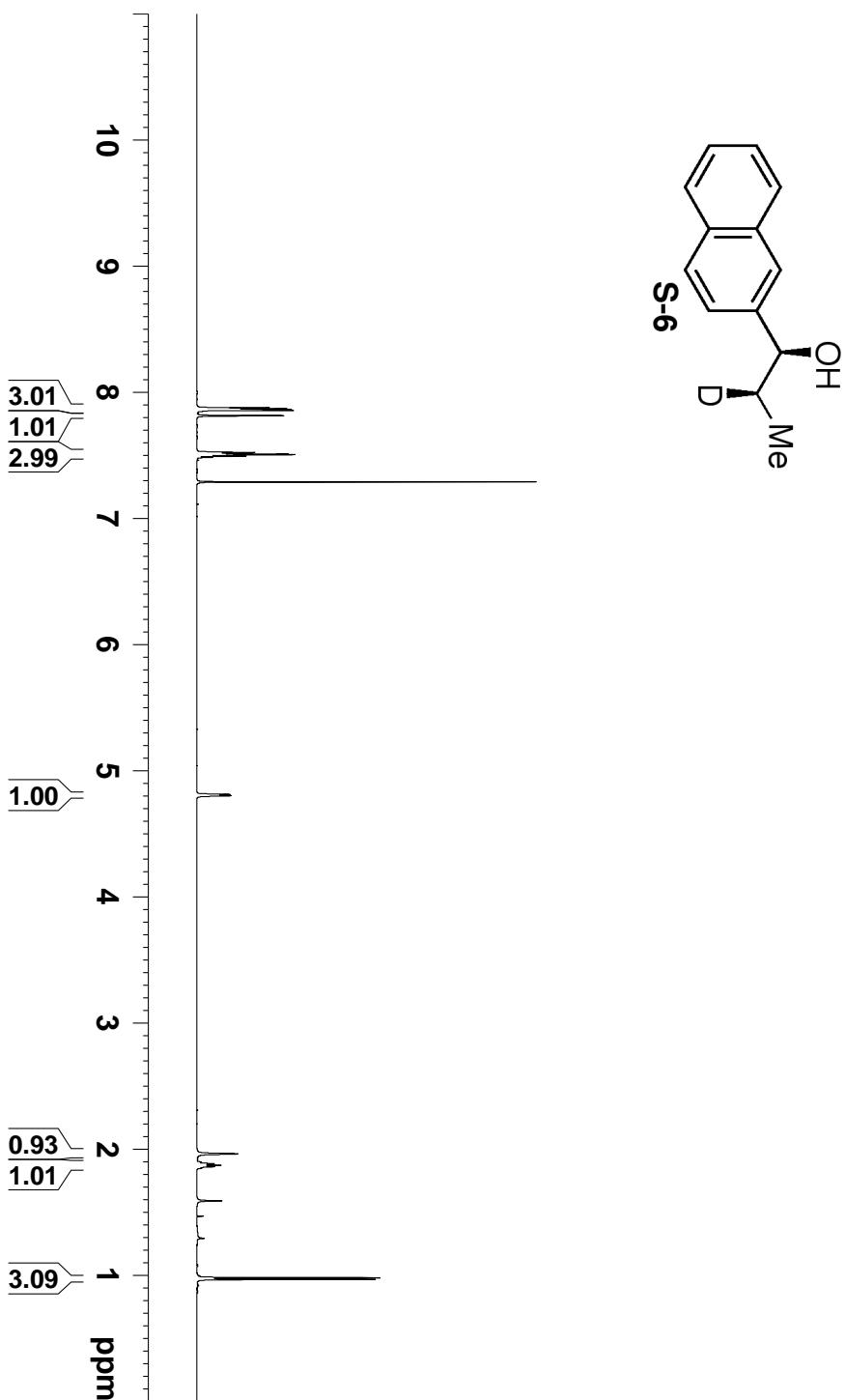
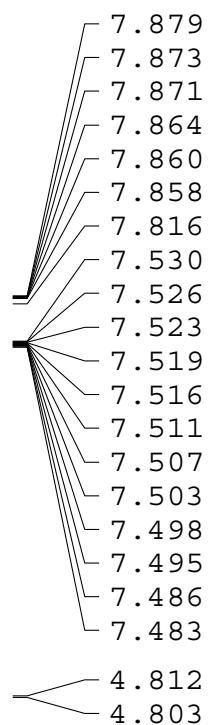
F2 - Processing parameters

SI	32768
SF	150.9505840 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

Compound S-6 1H NMR

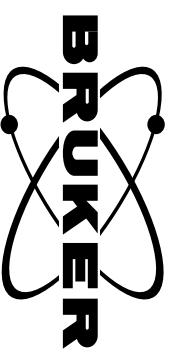


S111



S111

Compound S-6 13C NMR



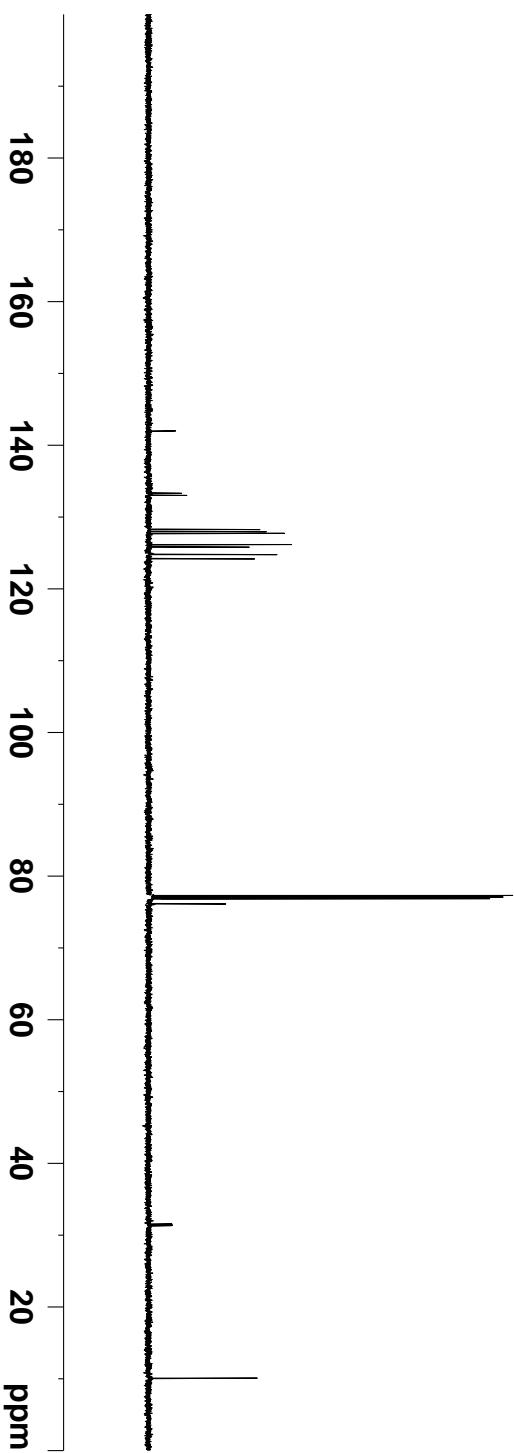
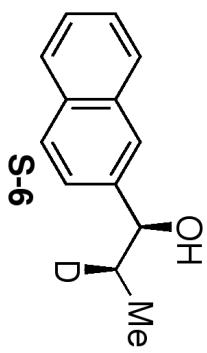
S112

141.94
133.29
133.01
128.26
127.94
127.70
126.13
125.80
124.74
124.16

76.11

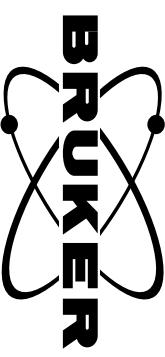
31.54
31.41
31.28

10.04

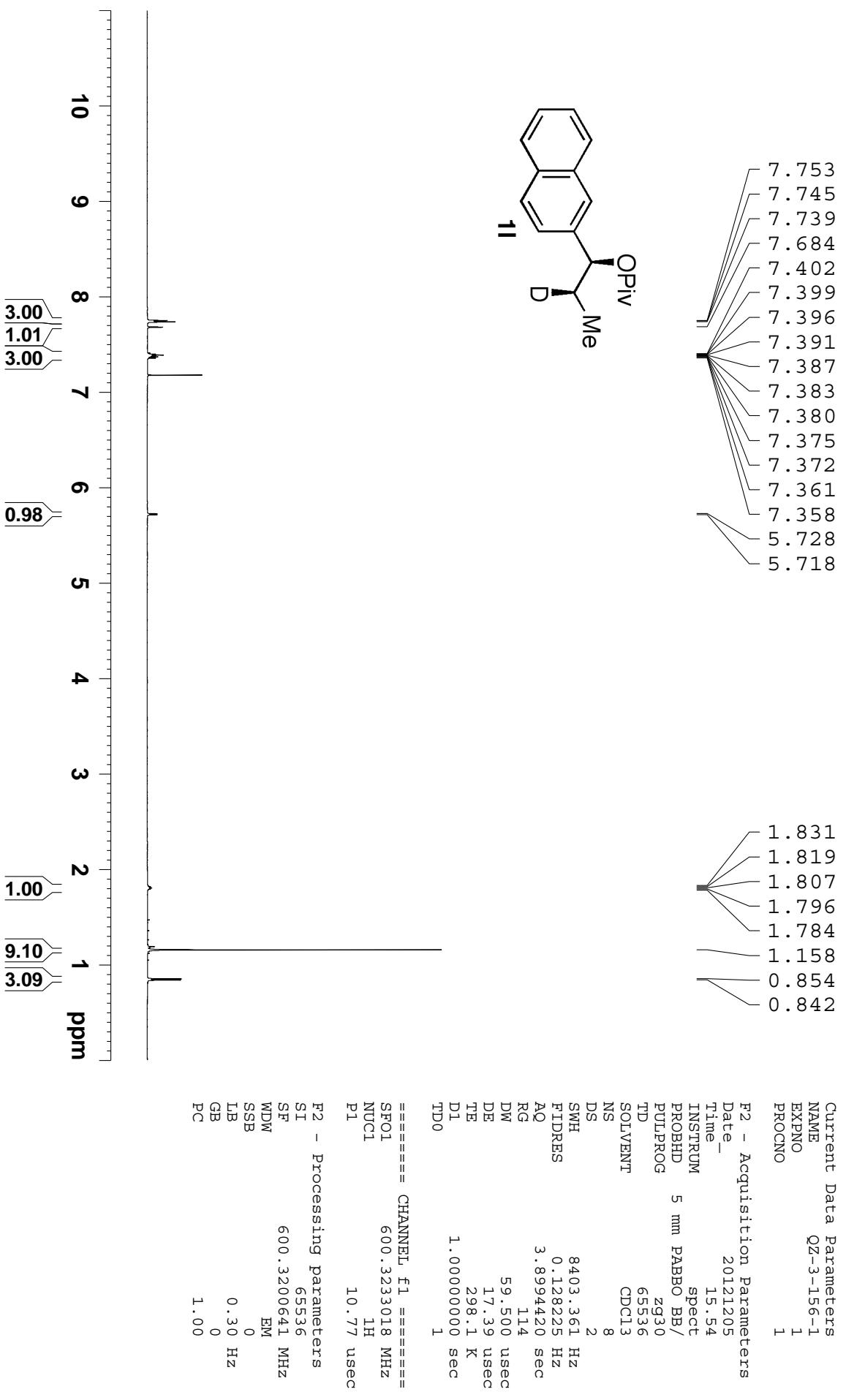


Current Data	Parameters
NAME	QZ-3-132-1
EXPNO	4
PROCNO	1
F2 - Acquisition Parameters	
DATE	20121203
TIME	22.48
INSTRUM	spec
PROBHD	5 mm PABBO BB/
PULPROG	zgpg95
TD	65536
SOLVENT	CDCl3
NS	256
DS	4
SWH	34722.223 Hz
FIDRES	0.529819 Hz
AQ	0.9437684 sec
RG	2050
DW	14.400 usec
DE	19.34 usec
TE	298.1 K
D1	1.1000002 sec
D11	0.03000002 sec
TDO	1 sec
===== CHANNEL f1 =====	
SFO1	150.9656784 MHz
NUC1	13C
P1	10.63 usec
F2 - Processing parameters	
SI	32768
SF	150.9505840 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	1.40
PC	

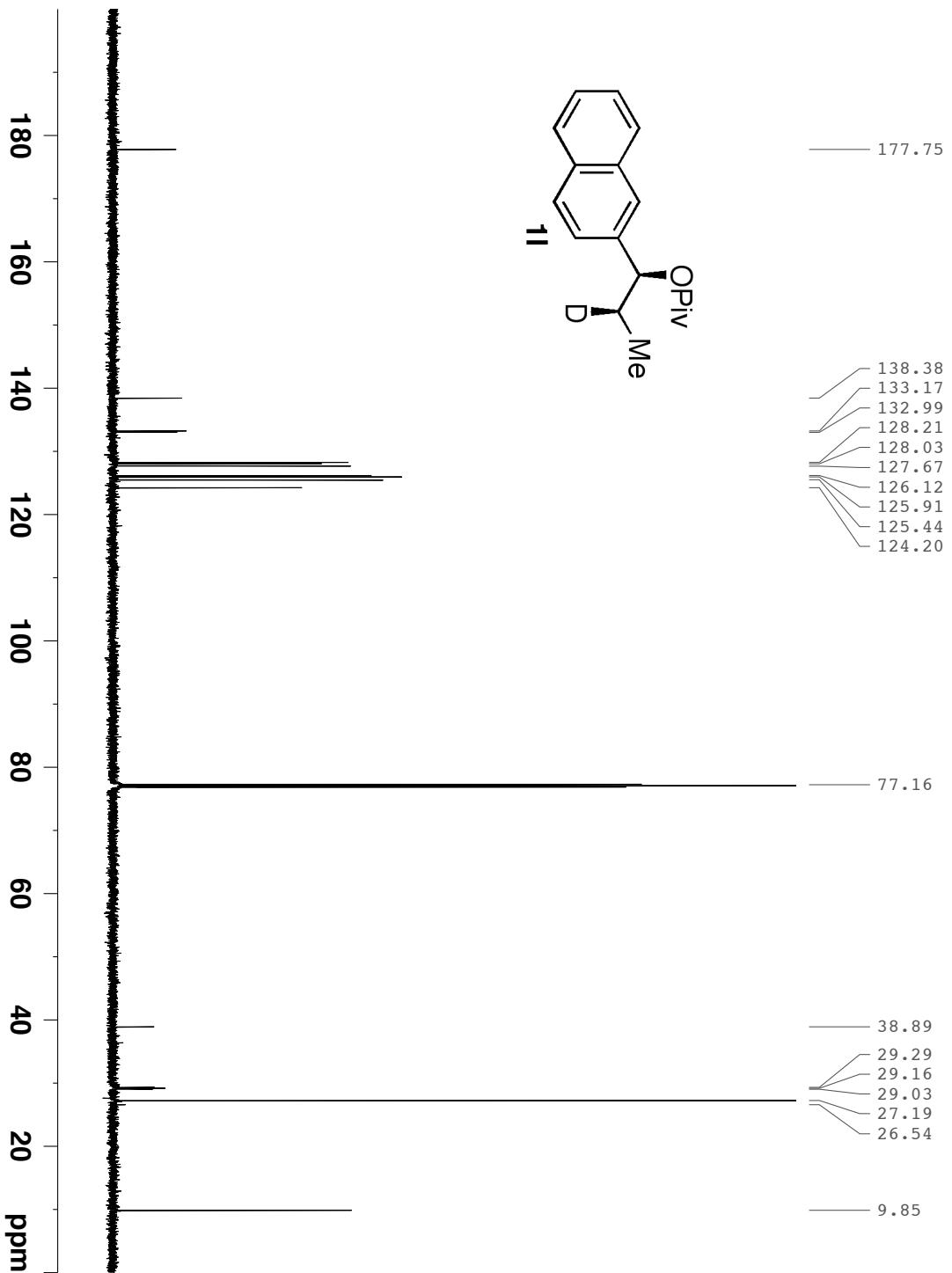
Compound 11 1H NMR



S113

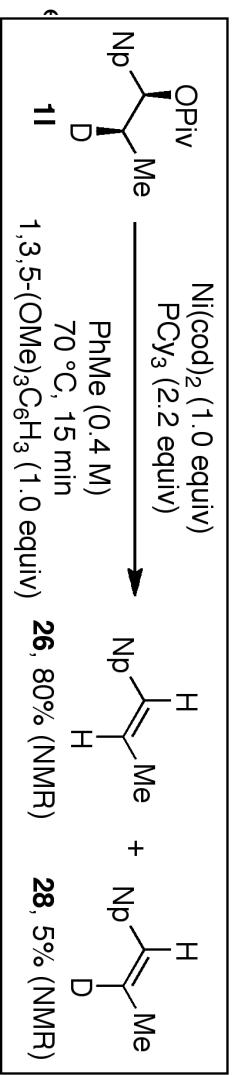


Compound 11 13CNMR

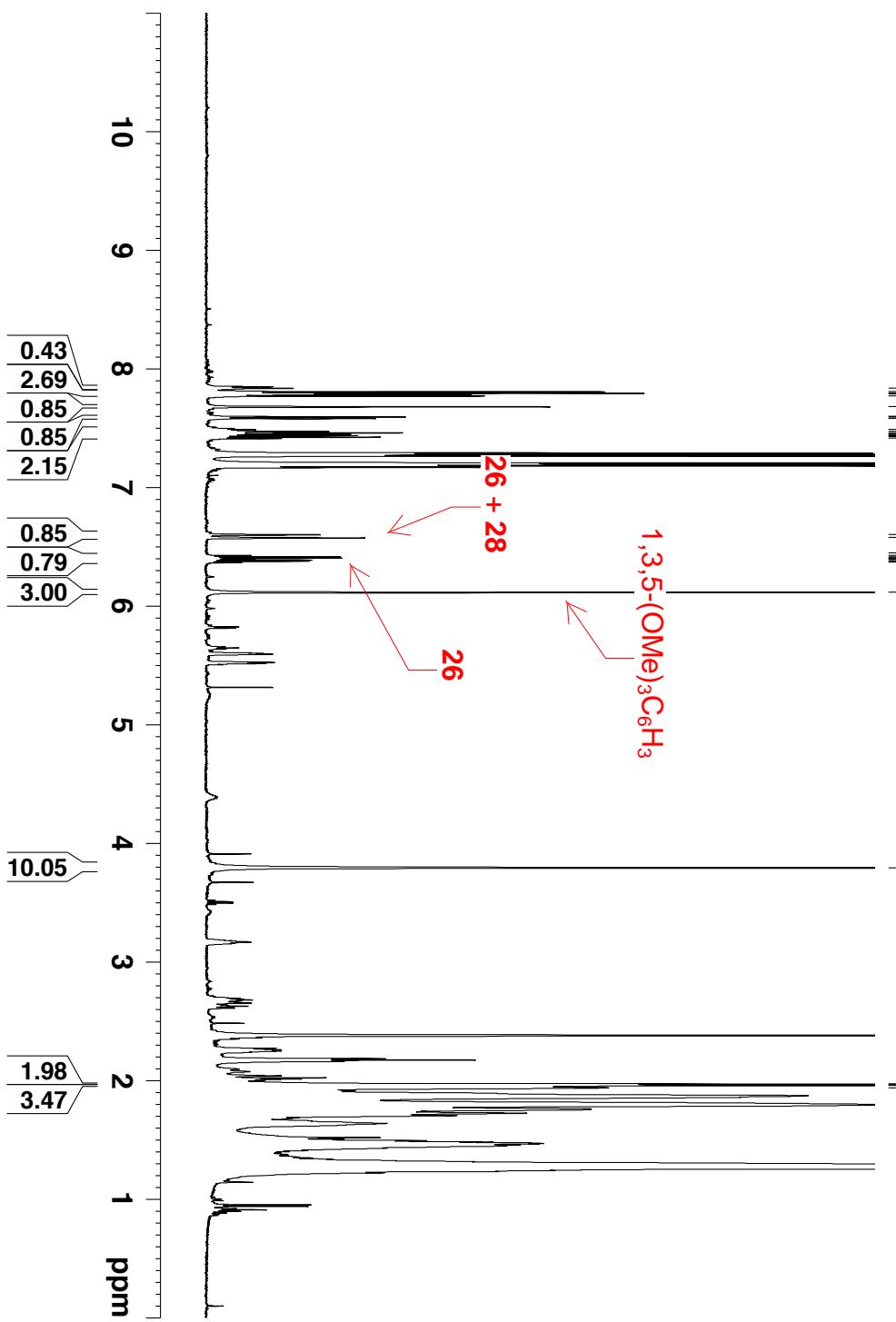


BRUKER

Crude ¹H NMR for beta-hydride elimination



S115



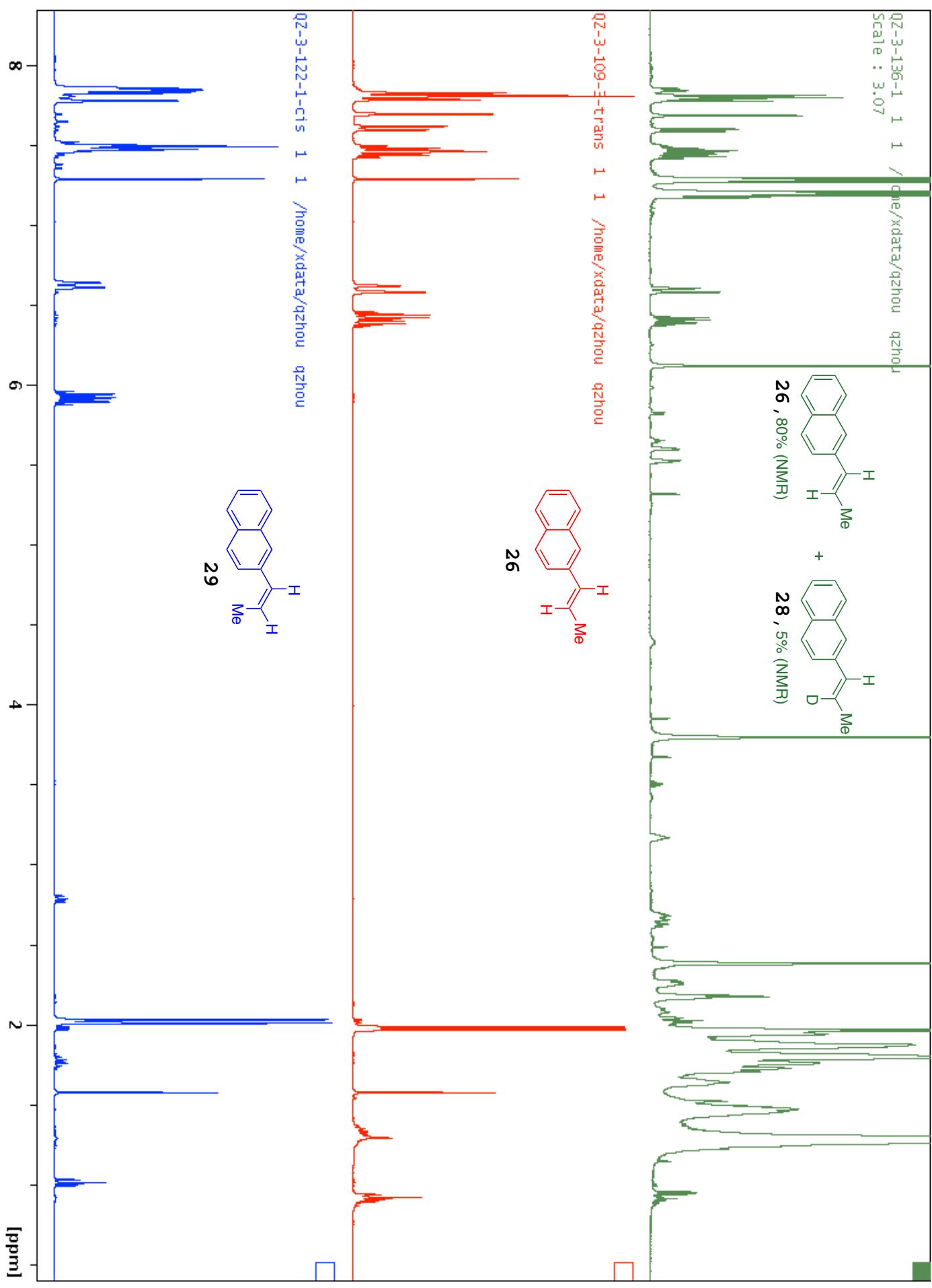
Current Data Parameters
 NAME QZ-3-136-1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date 20121125
 Time 22.43
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl₃
 NS 8
 DS 2
 SWH 8403.361 Hz
 FIDRES 0.128225 Hz
 AQ 3.8994420 sec
 RG 144
 DW 59.500 usec
 DE 9.10 usec
 TE 298.1 K
 D1 1.0000000 sec
 TD0 1

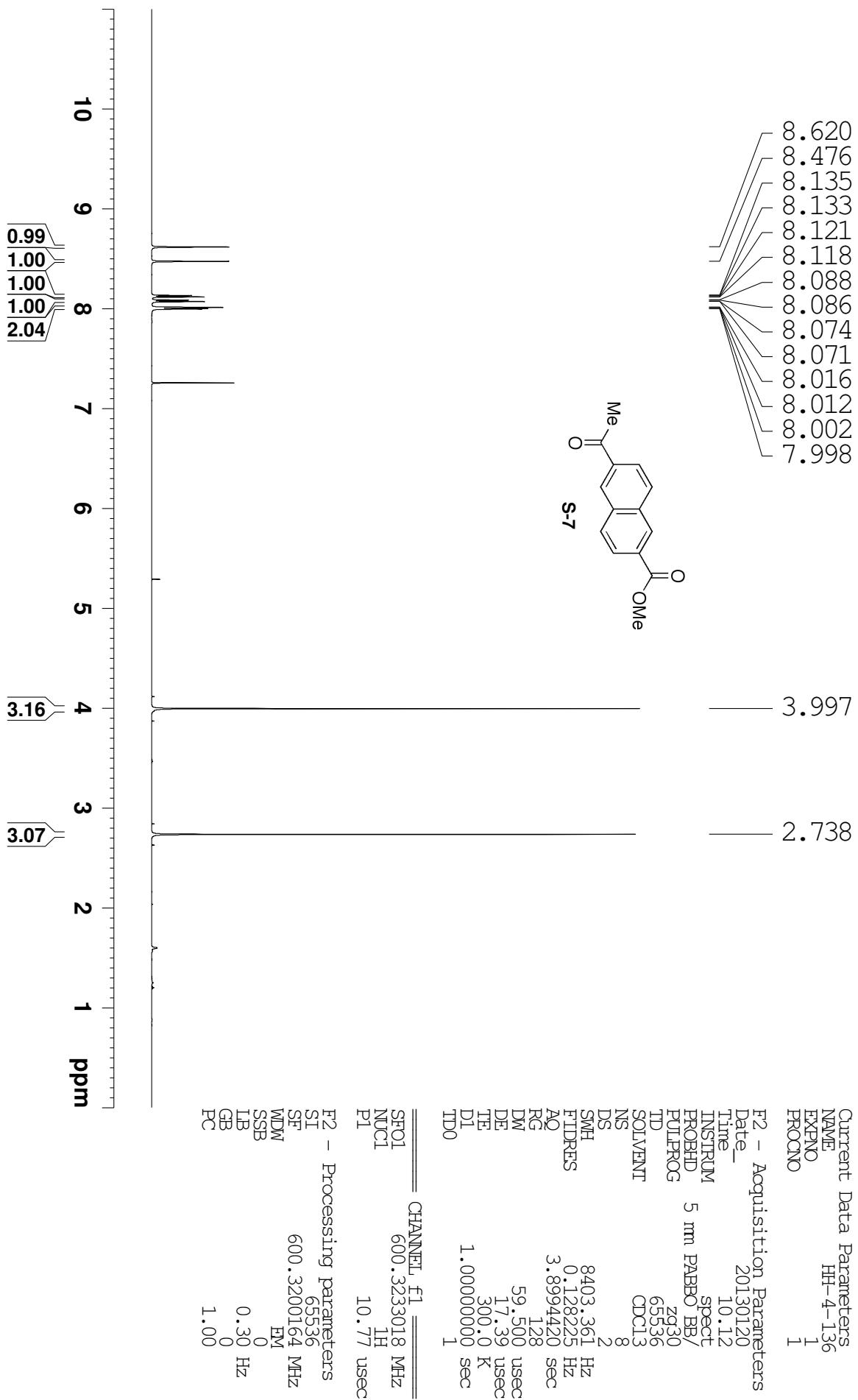
===== CHANNEL f1 =====

SFO1 600.3233018 MHz
 NUC1 ¹H
 P1 14.80 usec

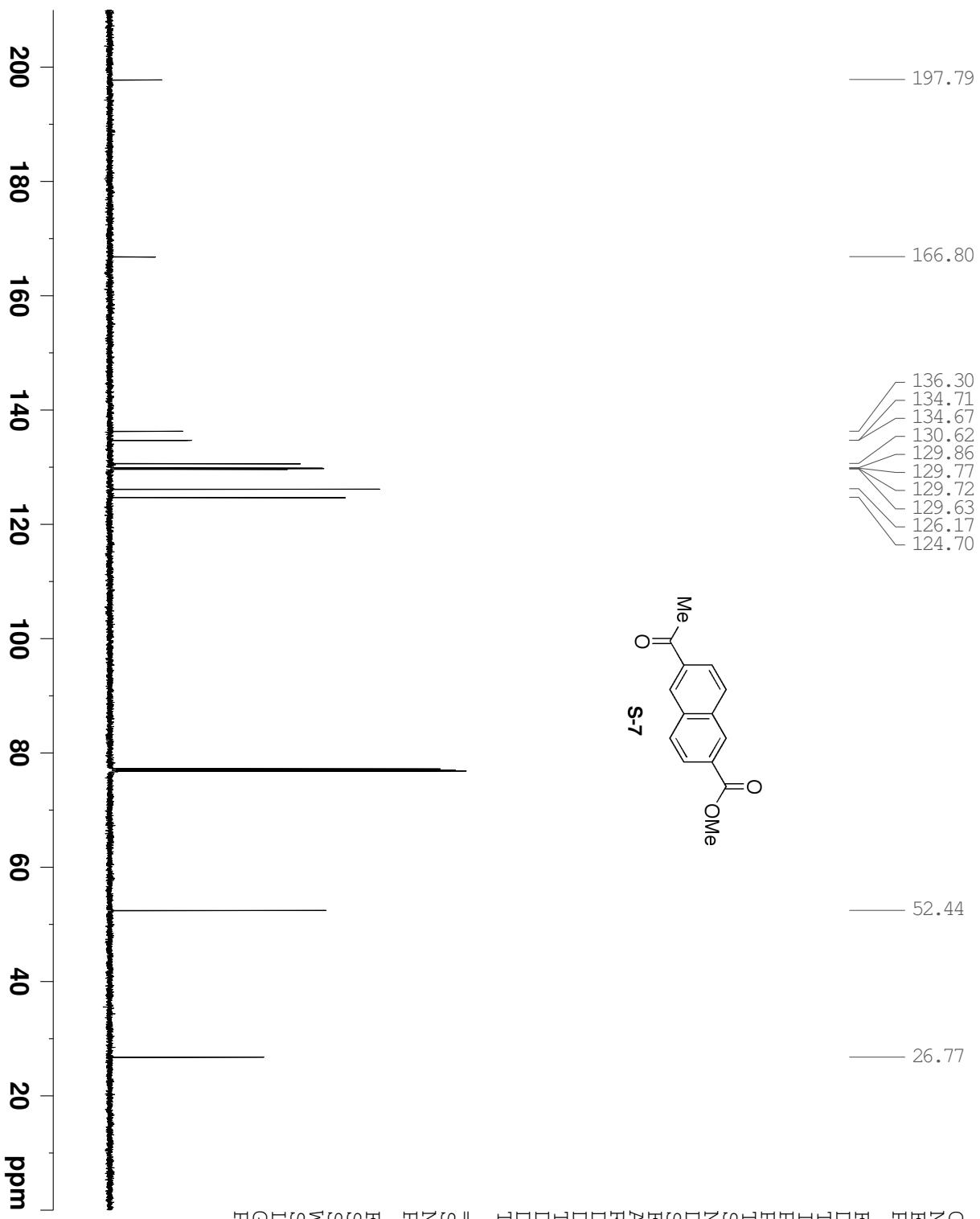
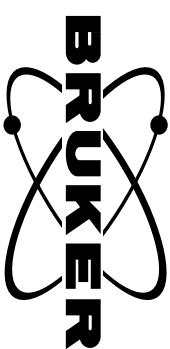
F2 - Processing parameters
 SI 65536
 SF 600.3200037 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 1.00
 PC



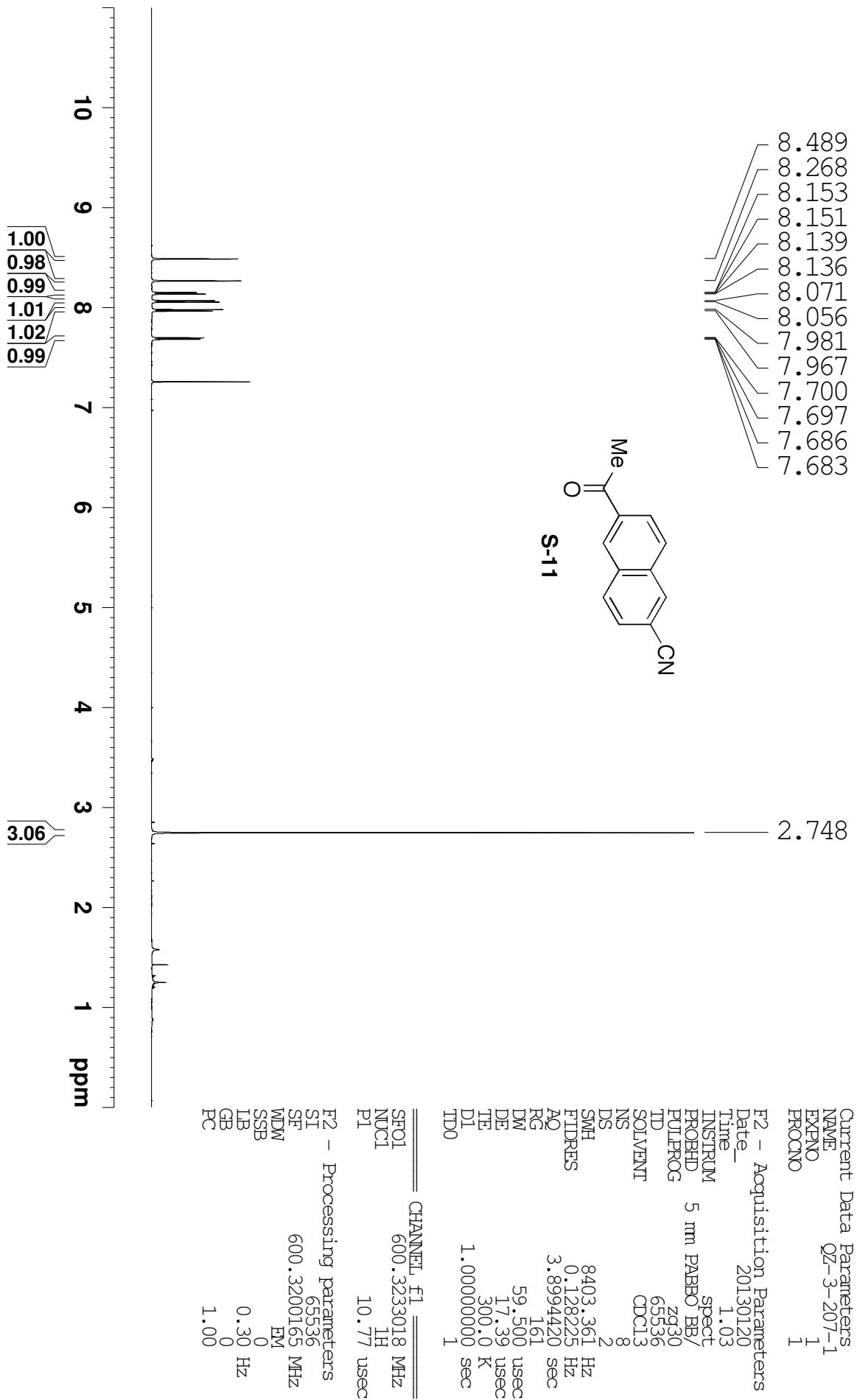
compound S-7 1HNMR



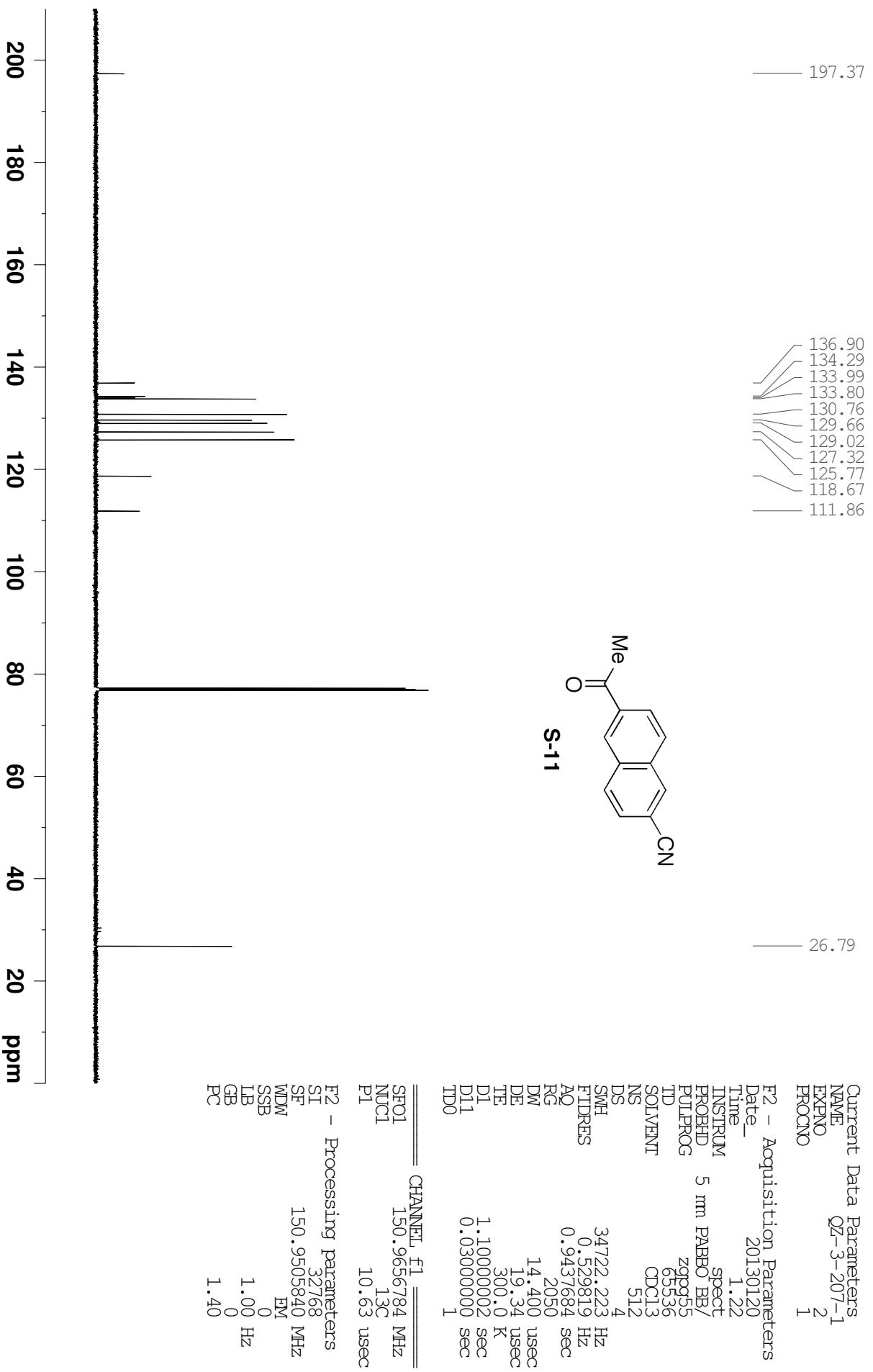
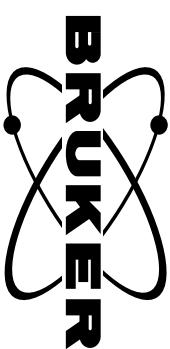
compound S-7 13CNMR



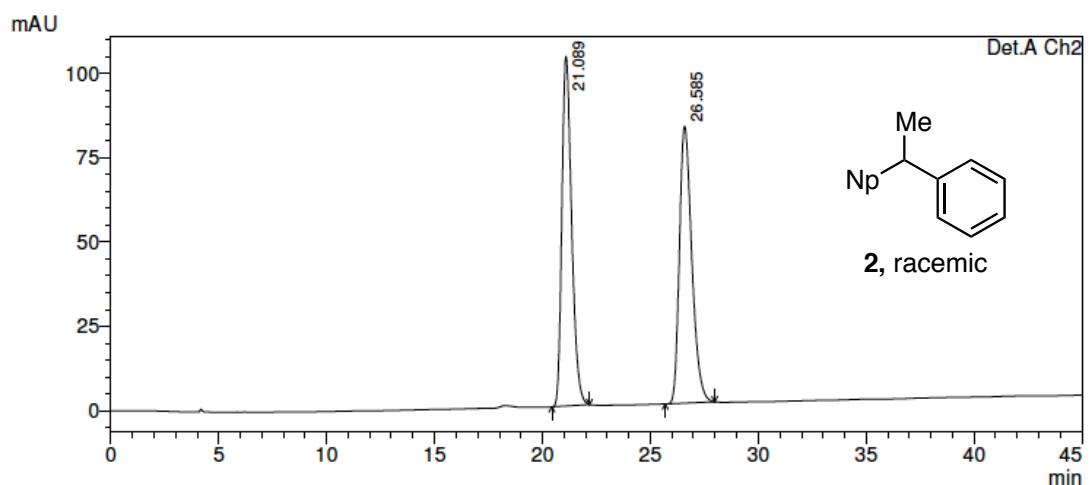
compound S-11 1H NMR



compound S-11 13C NMR



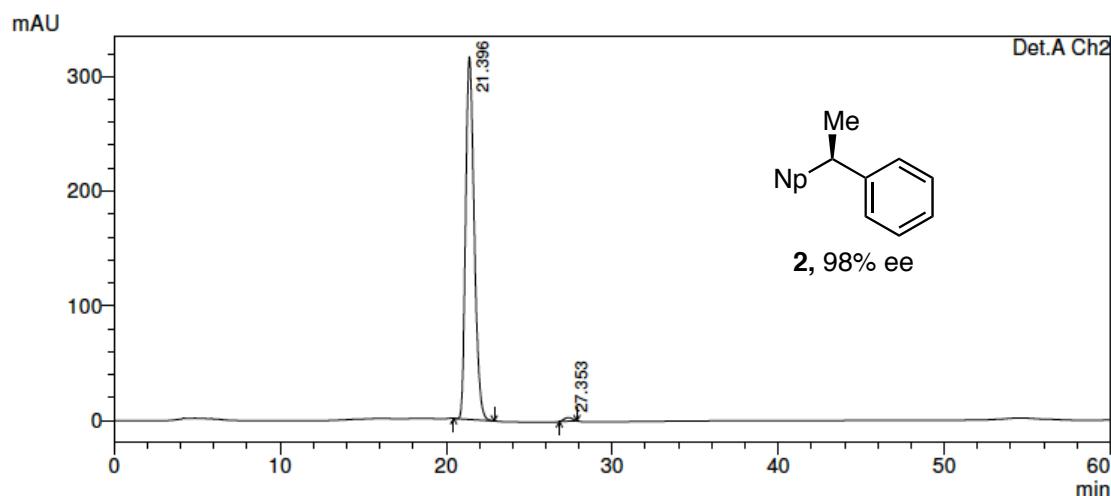
Compound 2, racemic



Detector A Ch2 220nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	21.089	3262561	103718	49.915	55.809
2	26.585	3273635	82125	50.085	44.191
Total		6536196	185843	100.000	100.000

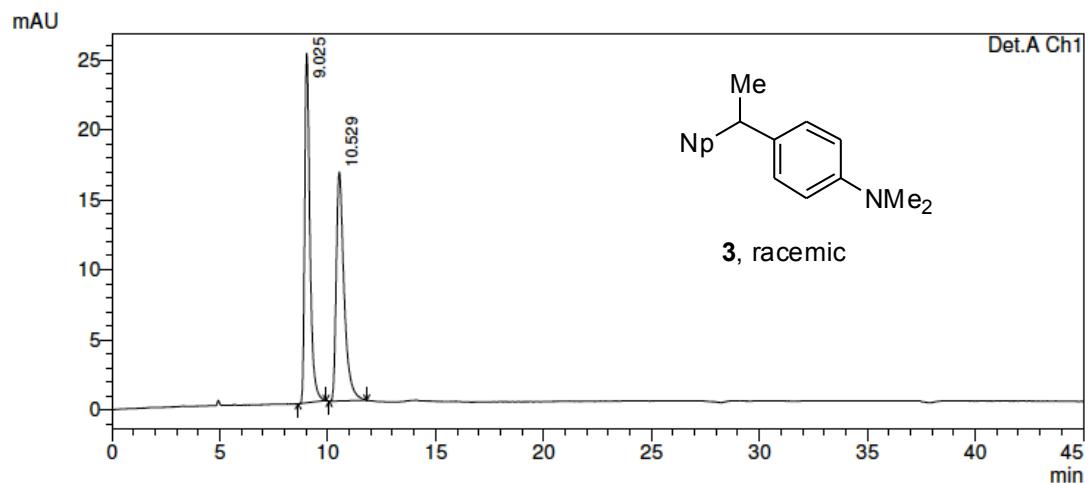
Compound 2, 98% ee



Detector A Ch2 220nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	21.396	11355848	316283	99.047	99.024
2	27.353	109219	3118	0.953	0.976
Total		11465067	319400	100.000	100.000

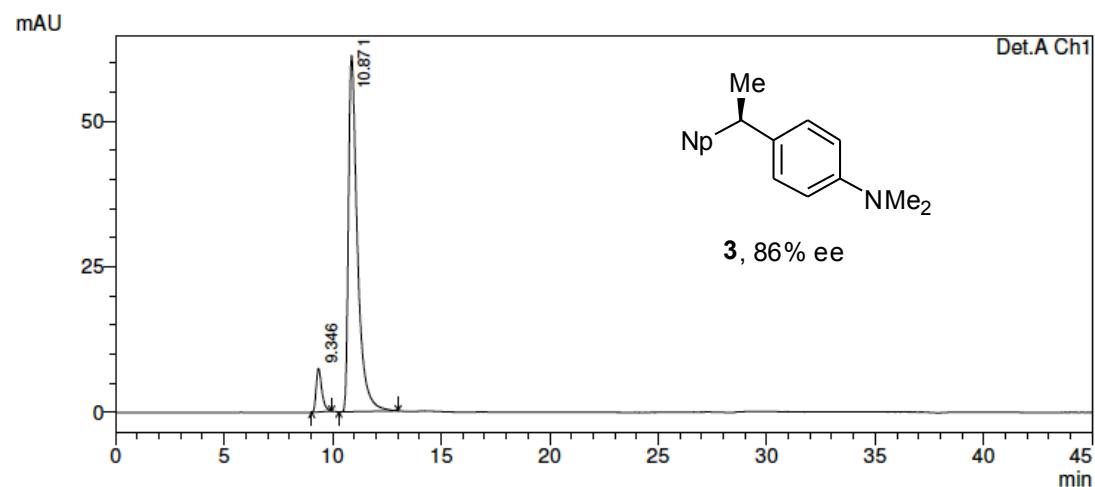
Compound 3, racemic



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.025	420349	24972	50.369	60.385
2	10.529	414192	16383	49.631	39.615
Total		834541	41355	100.000	100.000

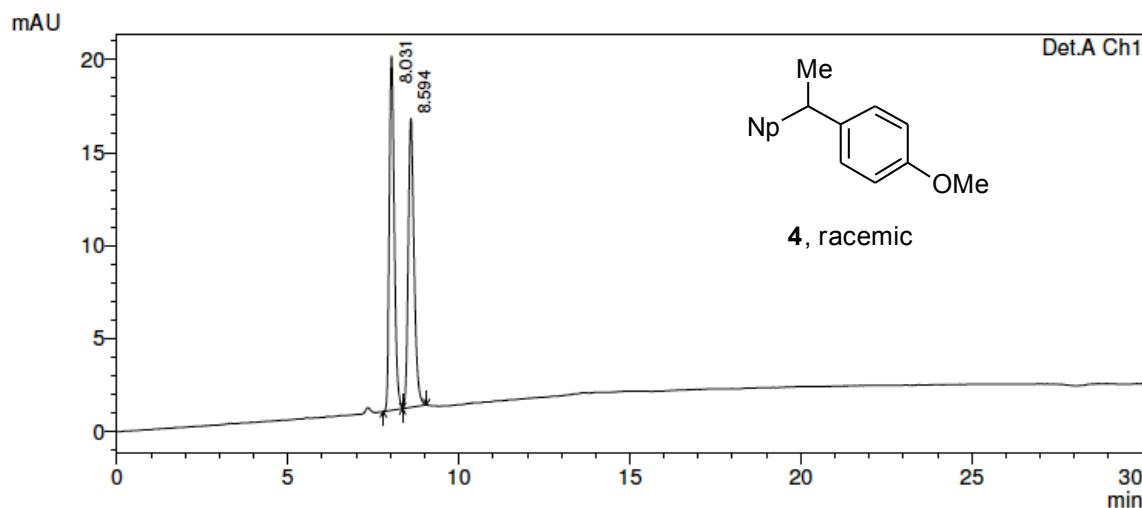
Compound 3, 86% ee



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.346	136637	7475	7.169	10.895
2	10.871	1769349	61137	92.831	89.105
Total		1905986	68612	100.000	100.000

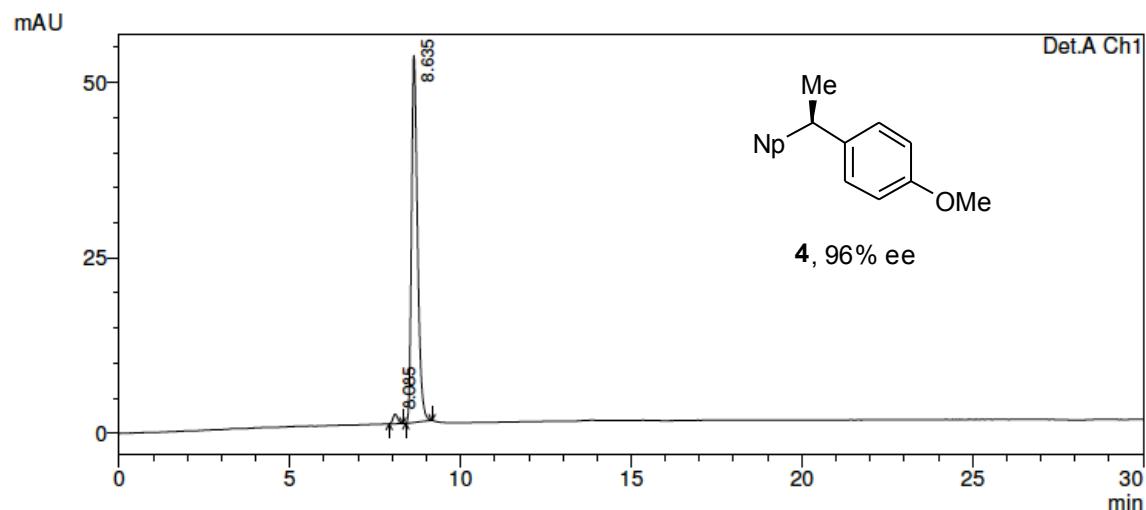
Compound 4, racemic



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.031	191673	19028	50.578	55.069
2	8.594	187296	15524	49.422	44.931
Total		378969	34552	100.000	100.000

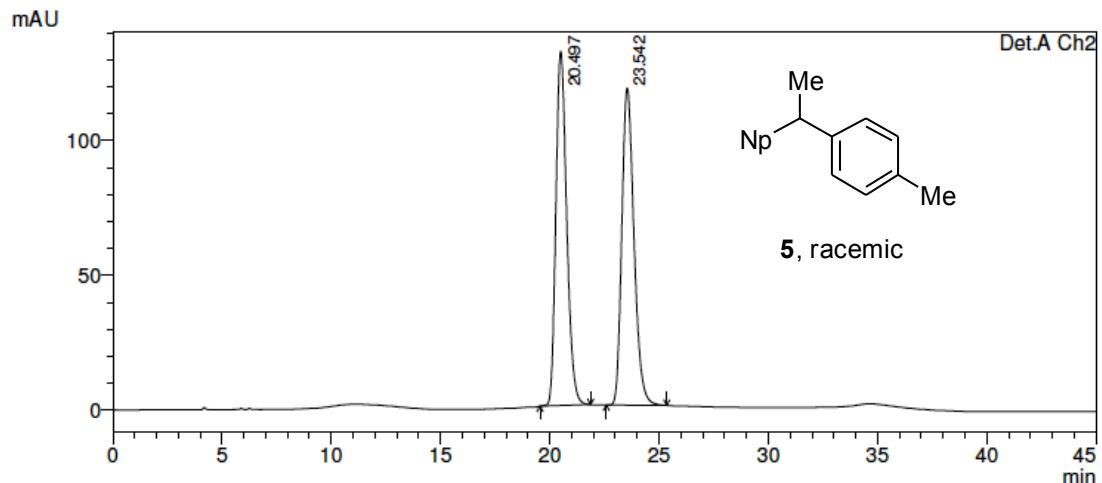
Compound 4, 96% ee



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.085	13175	1356	1.992	2.529
2	8.635	648308	52277	98.008	97.471
Total		661483	53633	100.000	100.000

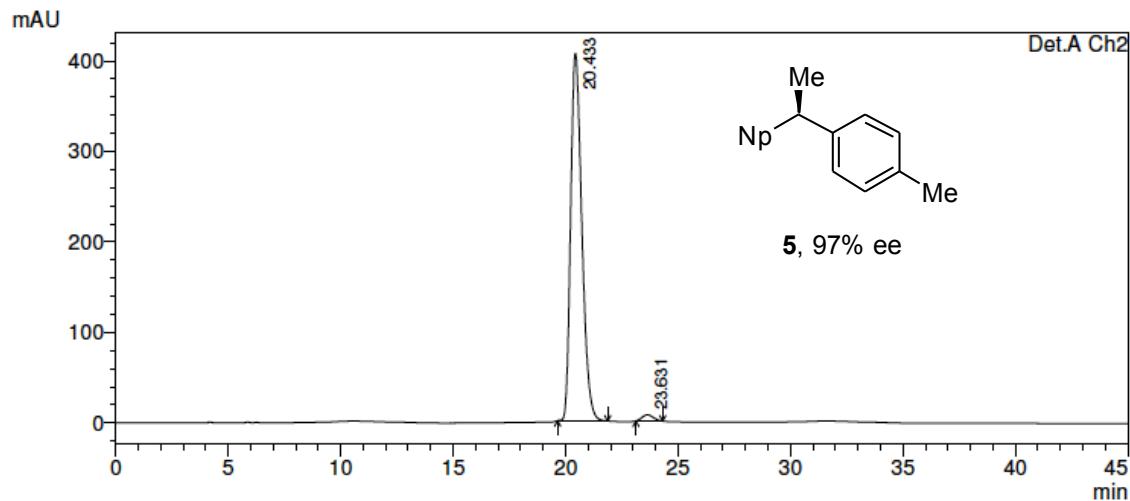
Compound 5, racemic



Detector A Ch2 220nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	20.497	4523871	131568	49.803	52.771
2	23.542	4559688	117752	50.197	47.229
Total		9083559	249320	100.000	100.000

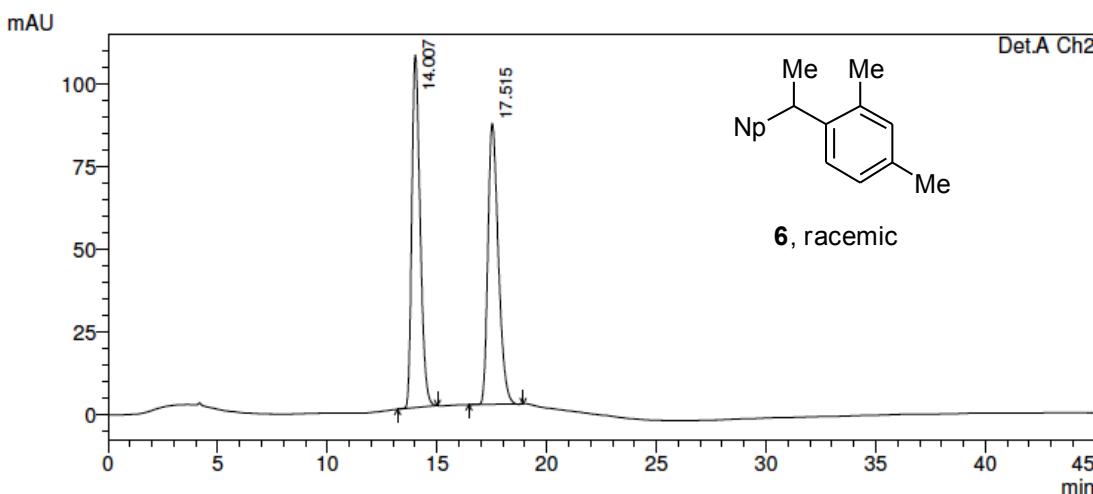
Compound 5, 97% ee



Detector A Ch2 220nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	20.433	14024628	406137	98.341	98.352
2	23.631	236613	6803	1.659	1.648
Total		14261242	412940	100.000	100.000

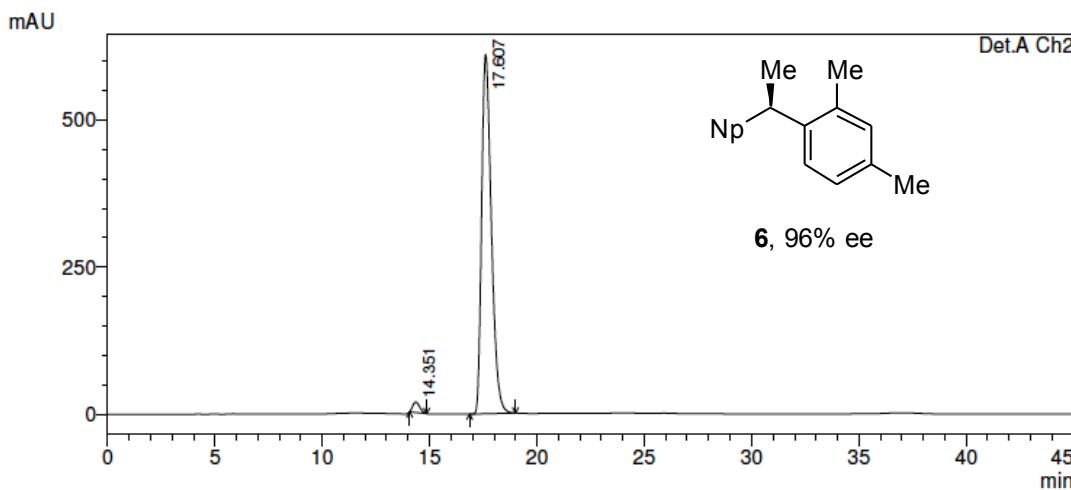
Compound 6, racemic



Detector A Ch2 220nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	14.007	2808009	106586	50.097	55.666
2	17.515	2797140	84887	49.903	44.334
Total		5605149	191474	100.000	100.000

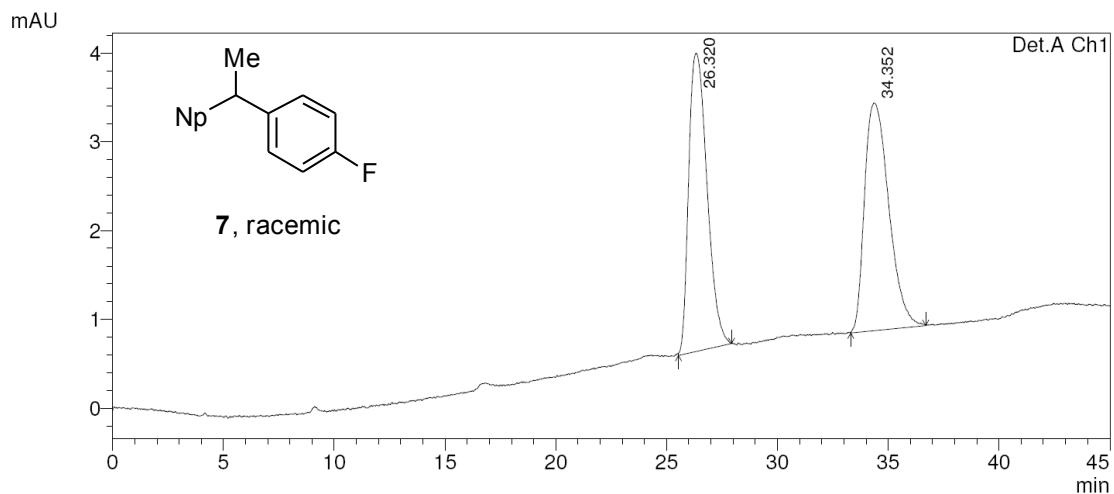
Compound 6, 96% ee



Detector A Ch2 220nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	14.351	393869	17724	2.008	2.823
2	17.607	19217322	610132	97.992	97.177
Total		19611191	627856	100.000	100.000

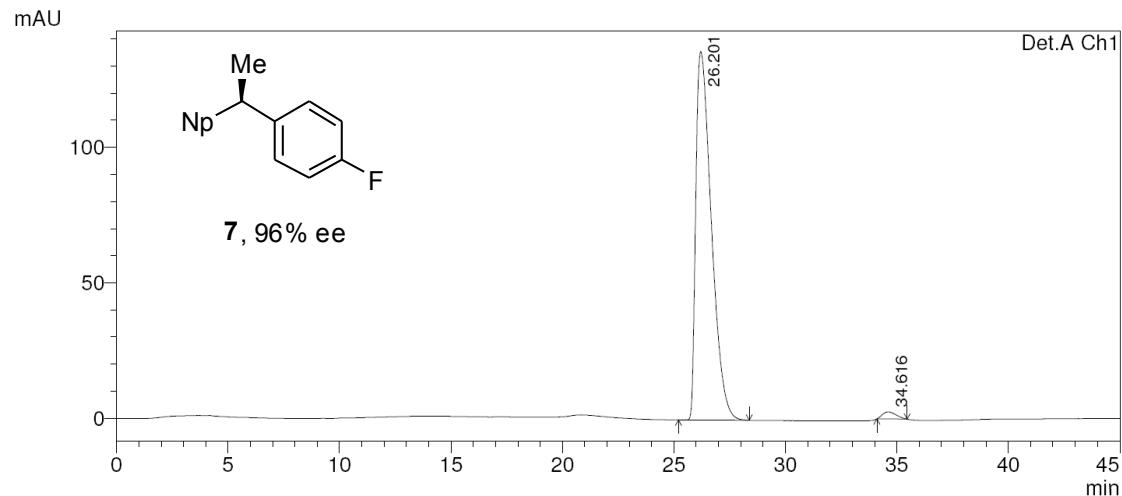
Compound 7, racemic



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	26.320	193738	3360	49.747	56.717
2	34.352	195710	2564	50.253	43.283
Total		389449	5925	100.000	100.000

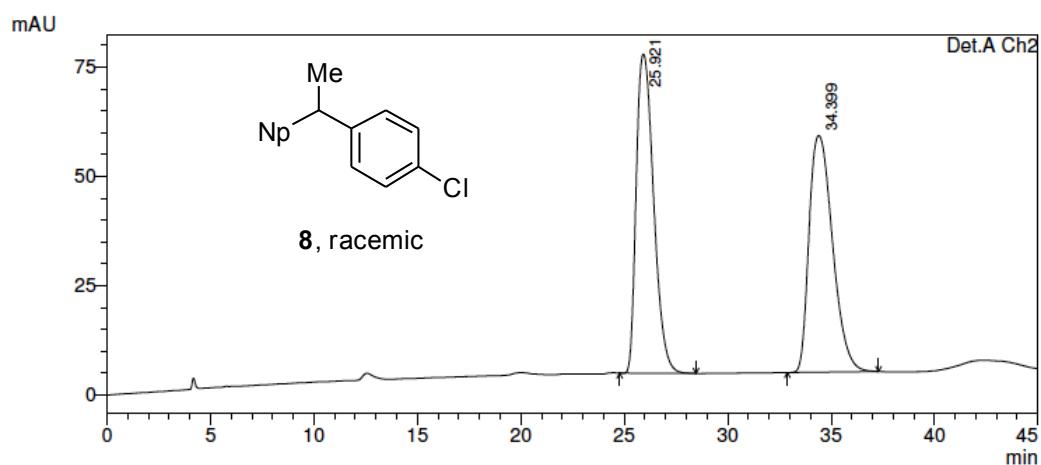
Compound 7, 96% ee



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	26.201	6602080	135918	98.328	98.110
2	34.616	112284	2618	1.672	1.890
Total		6714364	138536	100.000	100.000

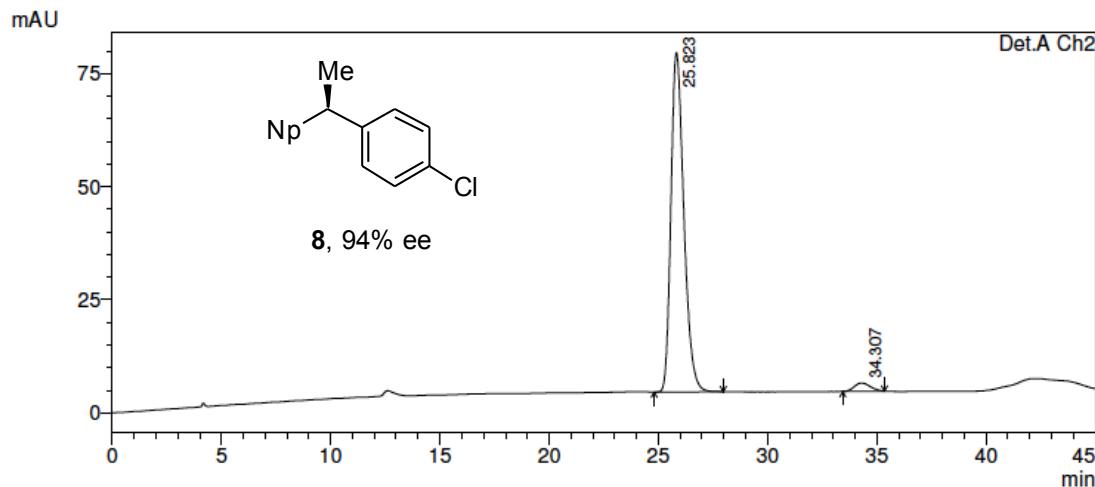
Compound 8, racemic



Detector A Ch2 220nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	25.921	4270293	73019	49.968	57.422
2	34.399	4275844	54144	50.032	42.578
Total		8546138	127164	100.000	100.000

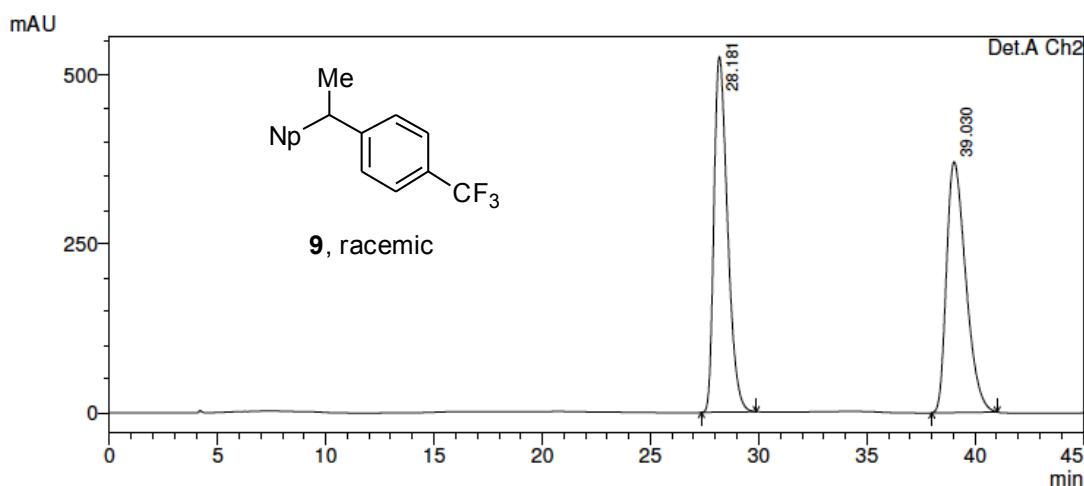
Compound 8, 94% ee



Detector A Ch2 220nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	25.823	3087241	75048	97.037	97.589
2	34.307	94277	1854	2.963	2.411
Total		3181518	76902	100.000	100.000

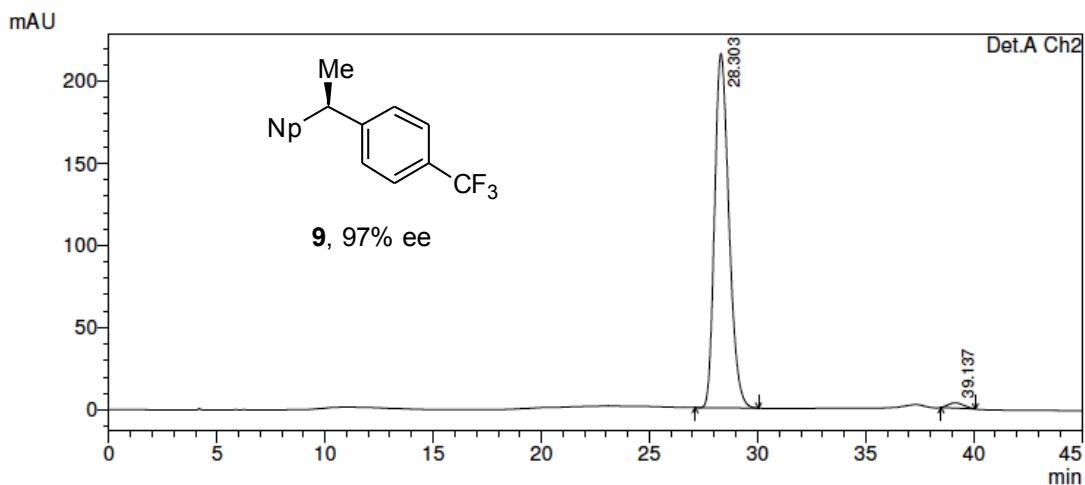
Compound 9, racemic



Detector A Ch2 220nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	28.181	23034505	527033	50.014	58.657
2	39.030	23021610	371471	49.986	41.343
Total		46056115	898503	100.000	100.000

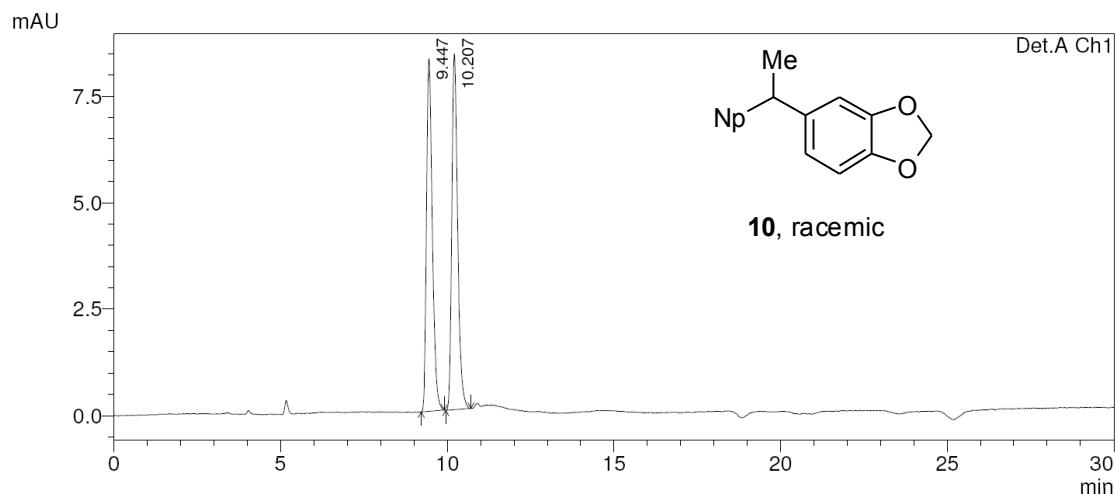
Compound 9, 97% ee



Detector A Ch2 220nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	28.303	9967337	215692	98.419	98.556
2	39.137	160085	3159	1.581	1.444
Total		10127422	218851	100.000	100.000

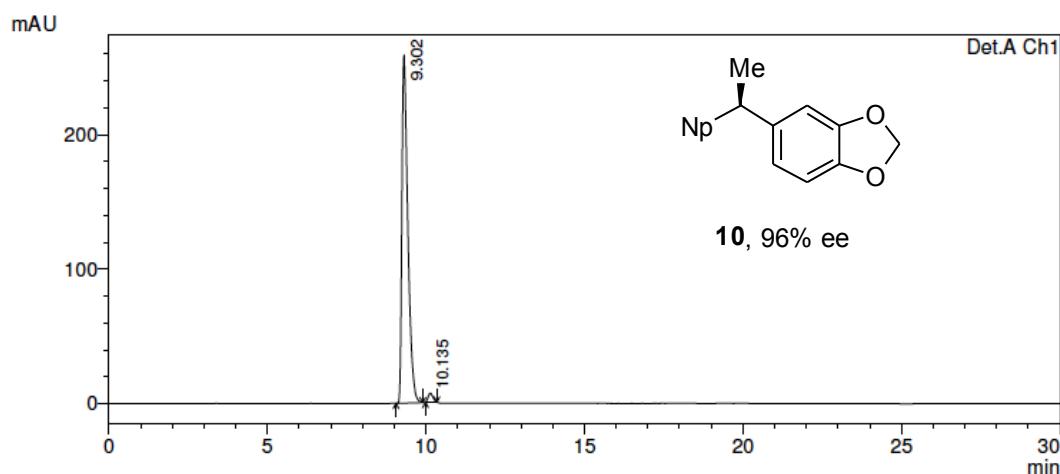
Compound 10, racemic



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.447	99369	8286	49.582	49.778
2	10.207	101046	8360	50.418	50.222
Total		200415	16646	100.000	100.000

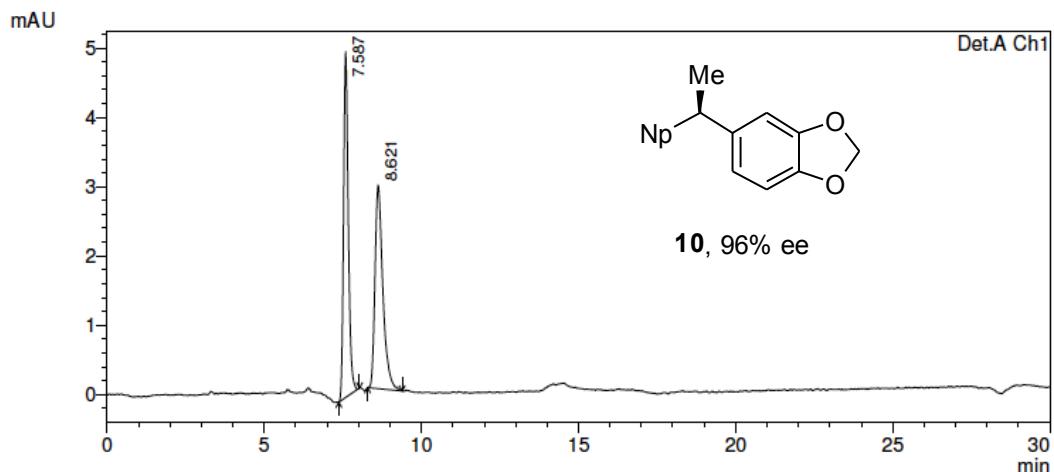
Compound 10, 96% ee



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.302	3412628	258849	98.030	97.482
2	10.135	68590	6685	1.970	2.518
Total		3481218	265533	100.000	100.000

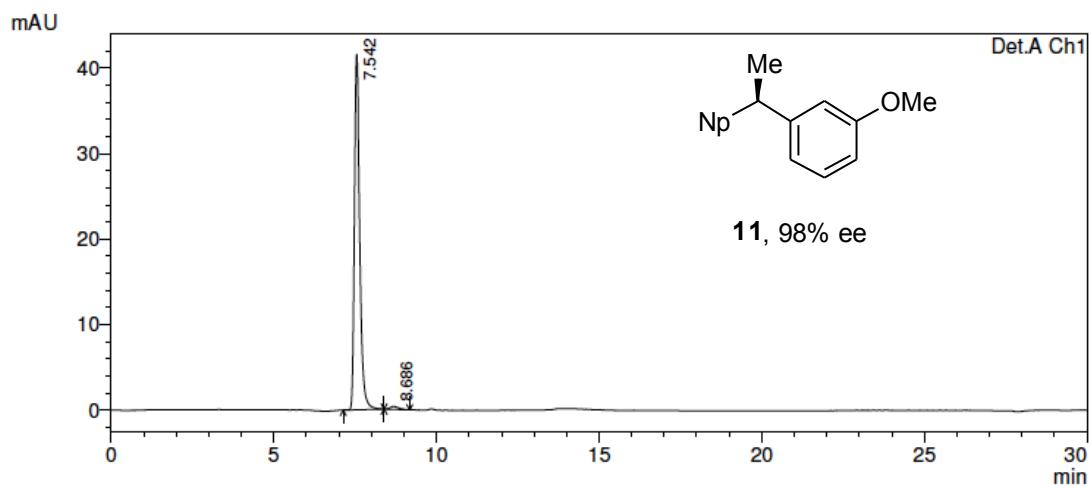
Compound 11, racemic



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.587	53165	4992	50.782	62.930
2	8.621	51528	2941	49.218	37.070
Total		104693	7933	100.000	100.000

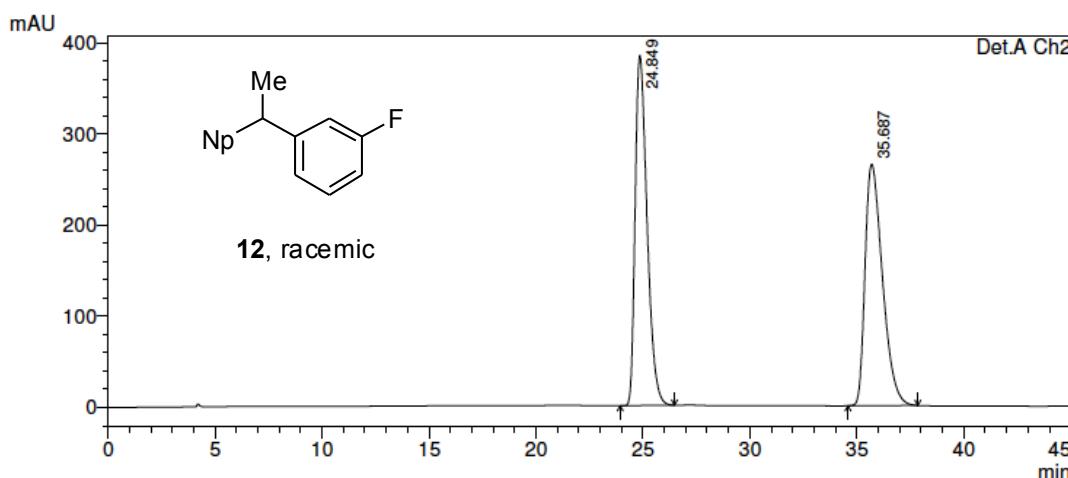
Compound 11, 98% ee



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.542	475460	41545	98.690	99.195
2	8.686	6310	337	1.310	0.805
Total		481770	41882	100.000	100.000

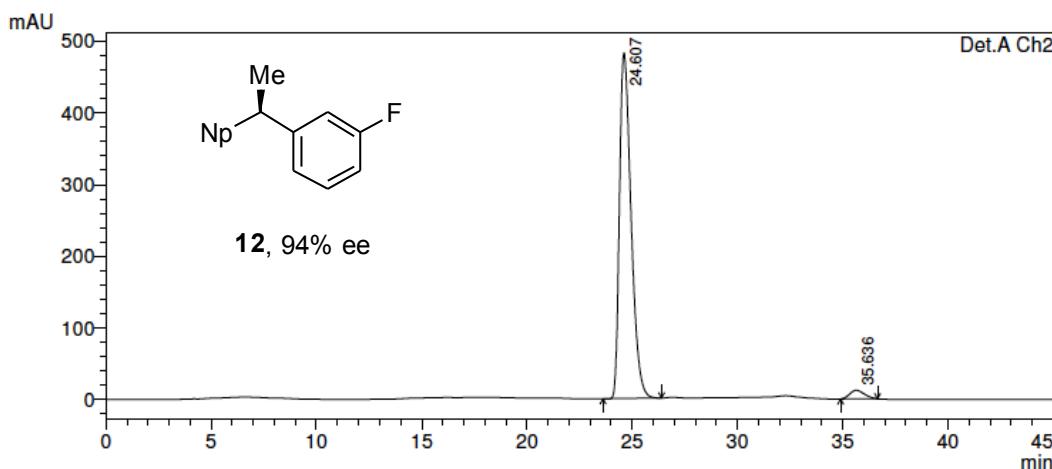
Compound 12, racemic



Detector A Ch2 220nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	24.849	14888201	384482	49.841	59.188
2	35.687	14983179	265111	50.159	40.812
Total		29871380	649593	100.000	100.000

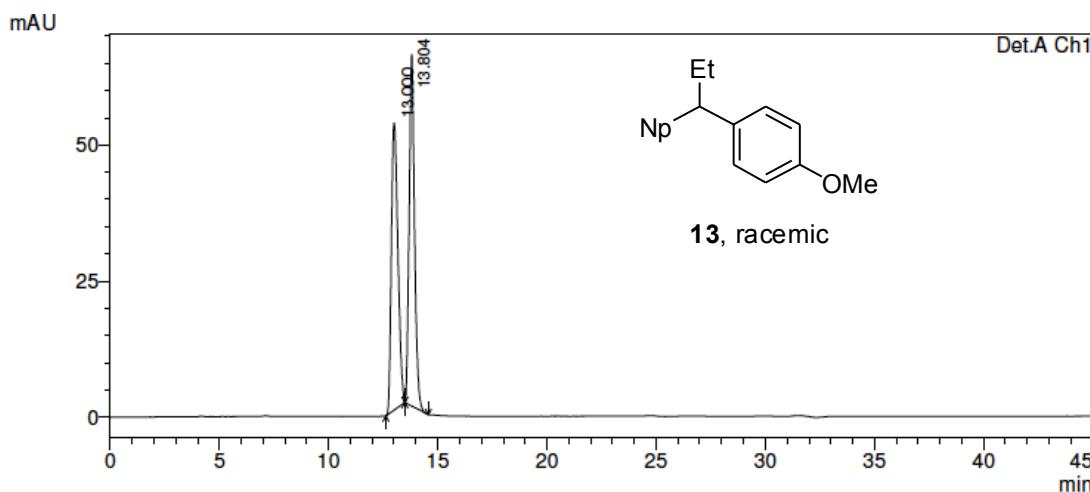
Compound 12, 94% ee



Detector A Ch2 220nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	24.607	18437656	482435	96.983	97.593
2	35.636	573579	11899	3.017	2.407
Total		19011234	494333	100.000	100.000

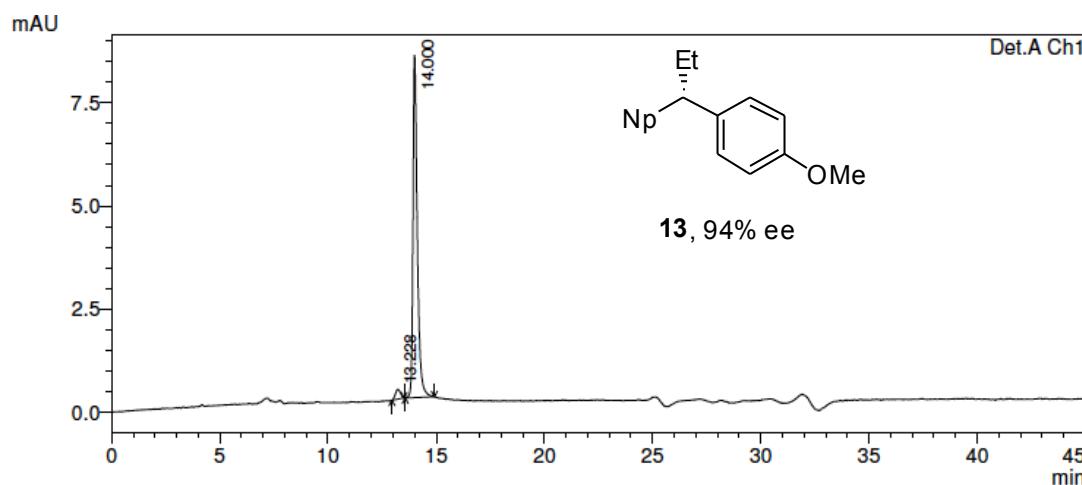
Compound 13, racemic



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	13.000	1095755	52759	49.972	44.969
2	13.804	1096990	64565	50.028	55.031
Total		2192745	117324	100.000	100.000

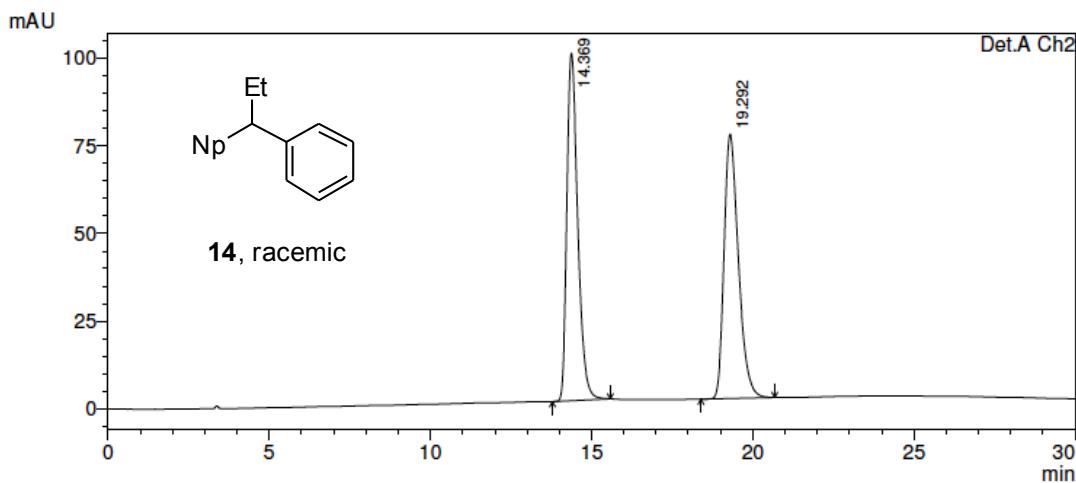
Compound 13, 94% ee



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	13.228	3845	237	3.216	2.779
2	14.000	115716	8281	96.784	97.221
Total		119561	8518	100.000	100.000

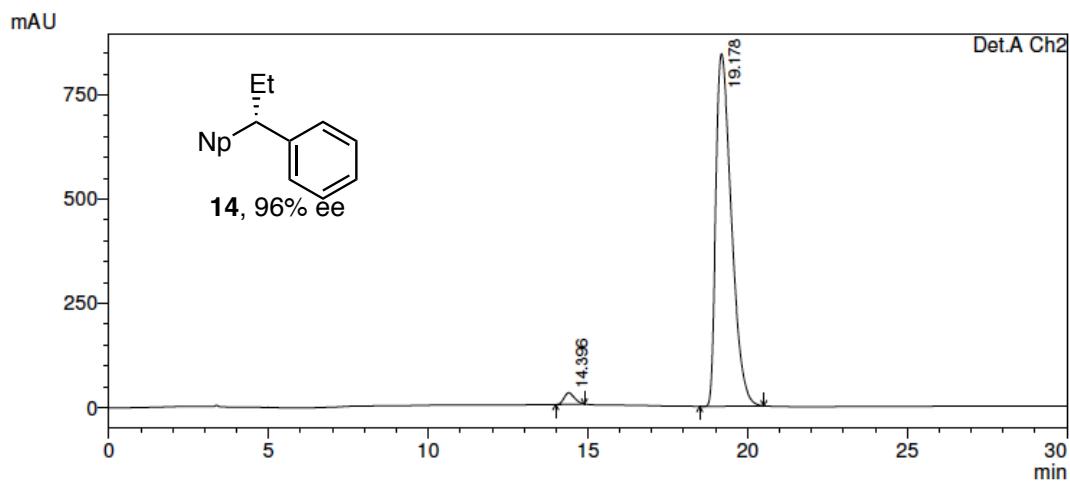
Compound 14, racemic



Detector A Ch2 220nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	14.369	2317727	98945	49.927	56.791
2	19.292	2324539	75281	50.073	43.209
Total		4642266	174226	100.000	100.000

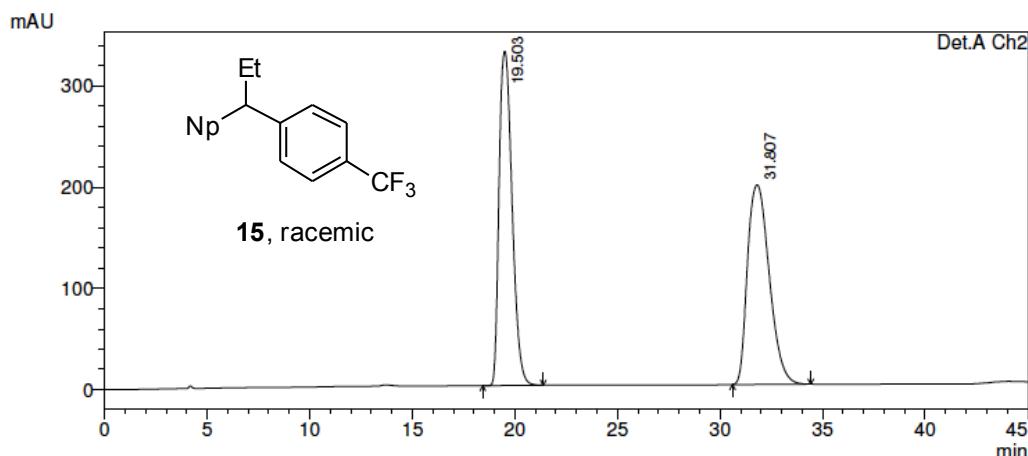
Compound 14, 96% ee



Detector A Ch2 220nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	14.396	619653	27486	2.118	3.152
2	19.178	28642737	844555	97.882	96.848
Total		29262391	872041	100.000	100.000

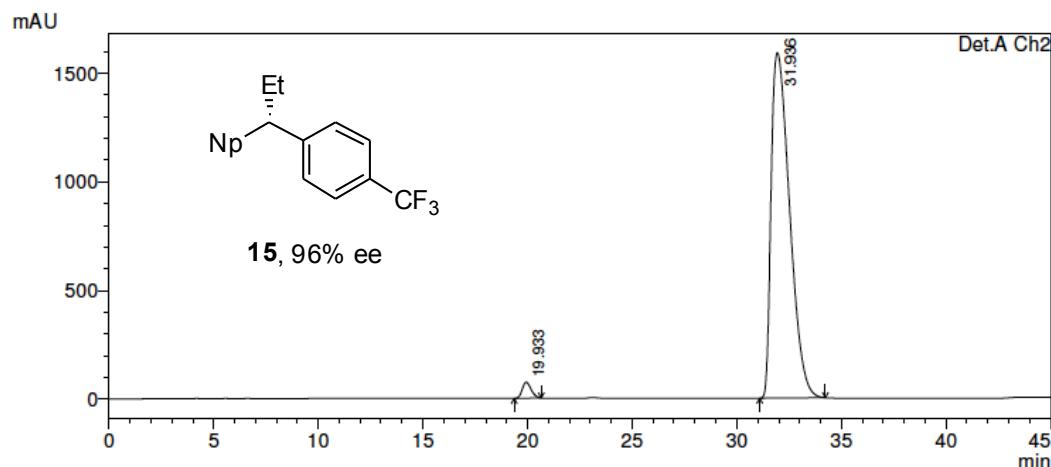
Compound 15, racemic



Detector A Ch2 220nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	19.503	14527364	330016	49.946	62.606
2	31.807	14558791	197114	50.054	37.394
Total		29086155	527130	100.000	100.000

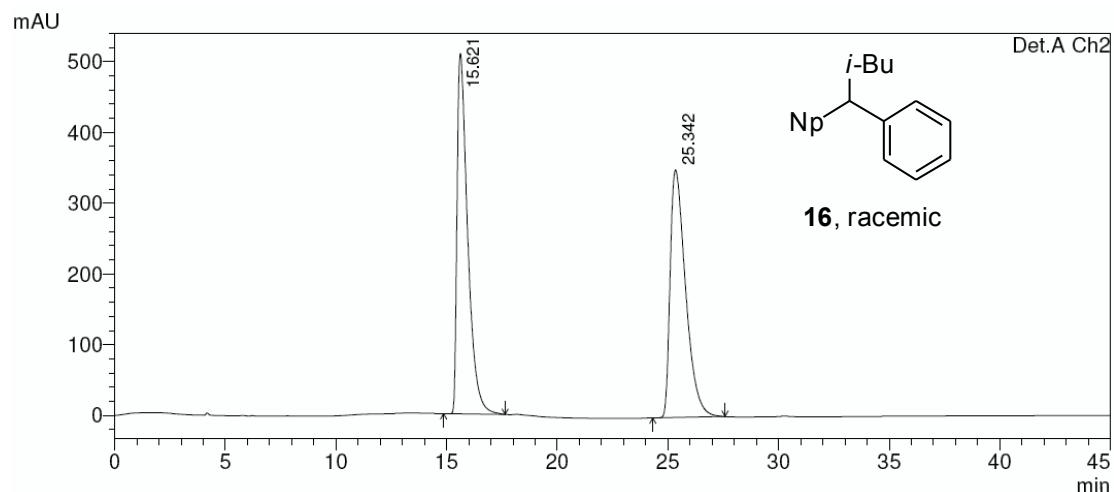
Compound 15, 96% ee



Detector A Ch2 220nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	19.933	2186019	73861	2.187	4.435
2	31.936	97776569	1591639	97.813	95.565
Total		99962589	1665500	100.000	100.000

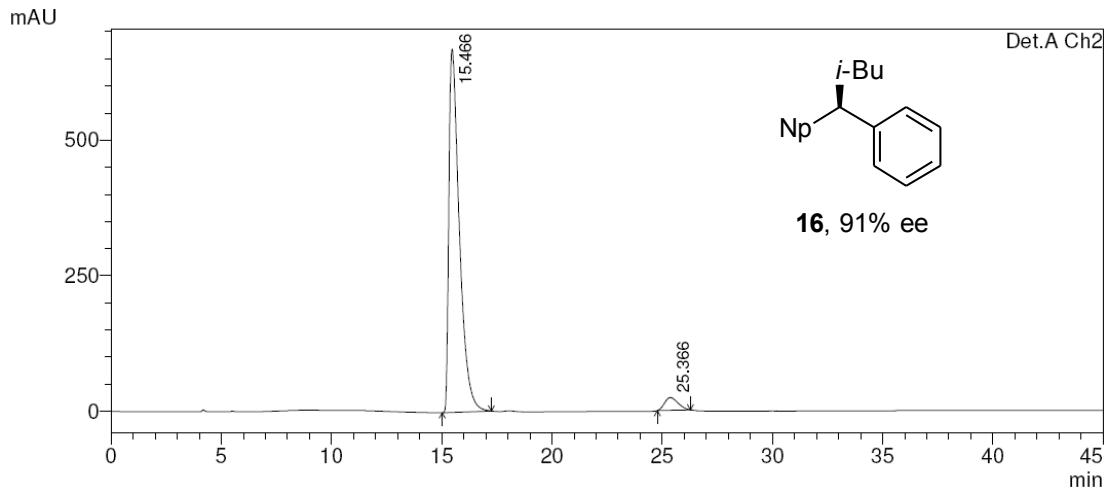
Compound 16, racemic



Detector A Ch2 220nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	15.621	16984566	509077	50.009	59.278
2	25.342	16978126	349718	49.991	40.722
Total		33962693	858795	100.000	100.000

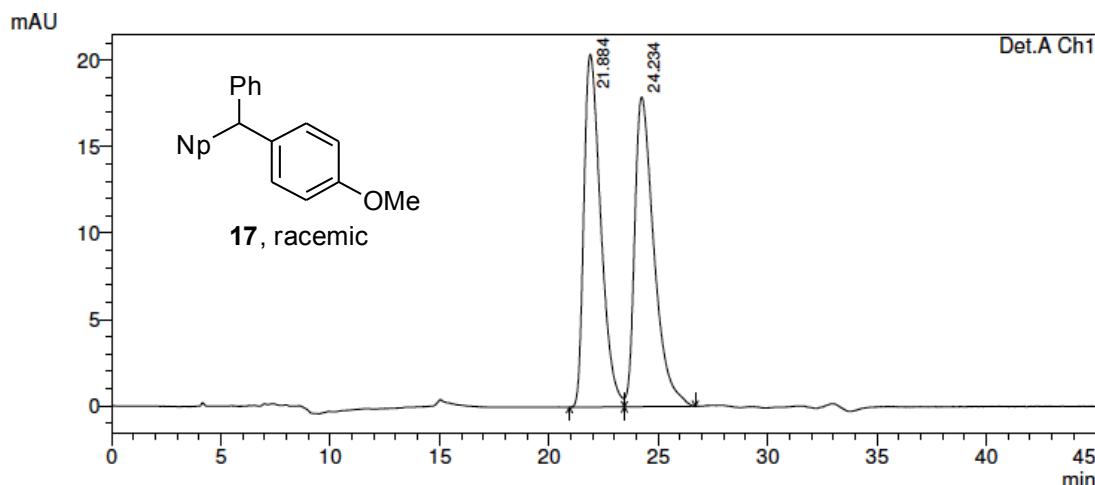
Compound 16, 91% ee



Detector A Ch2 220nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	15.466	22211081	670111	95.721	96.614
2	25.366	992790	23482	4.279	3.386
Total		23203871	693593	100.000	100.000

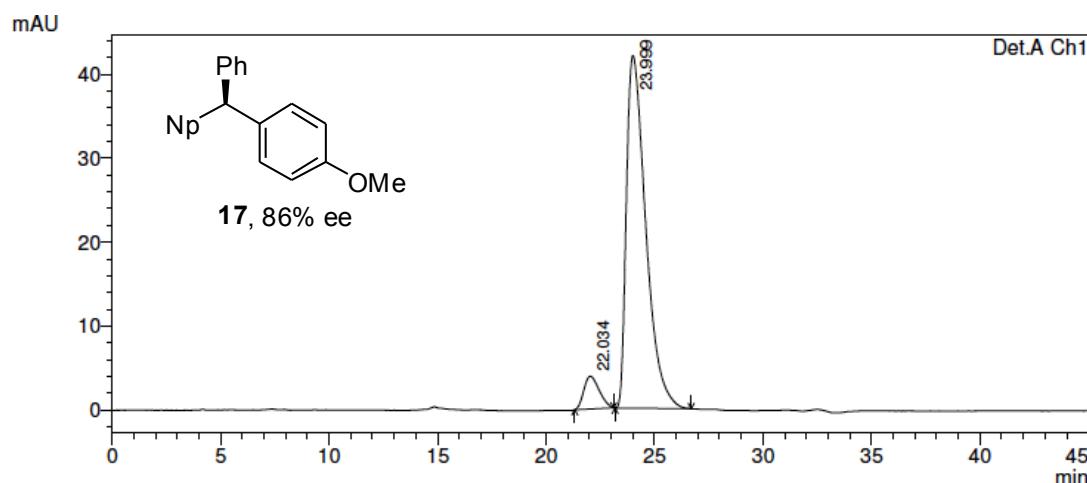
Compound 17, racemic



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	21.884	1094878	20418	49.887	53.263
2	24.234	1099828	17916	50.113	46.737
Total		2194707	38334	100.000	100.000

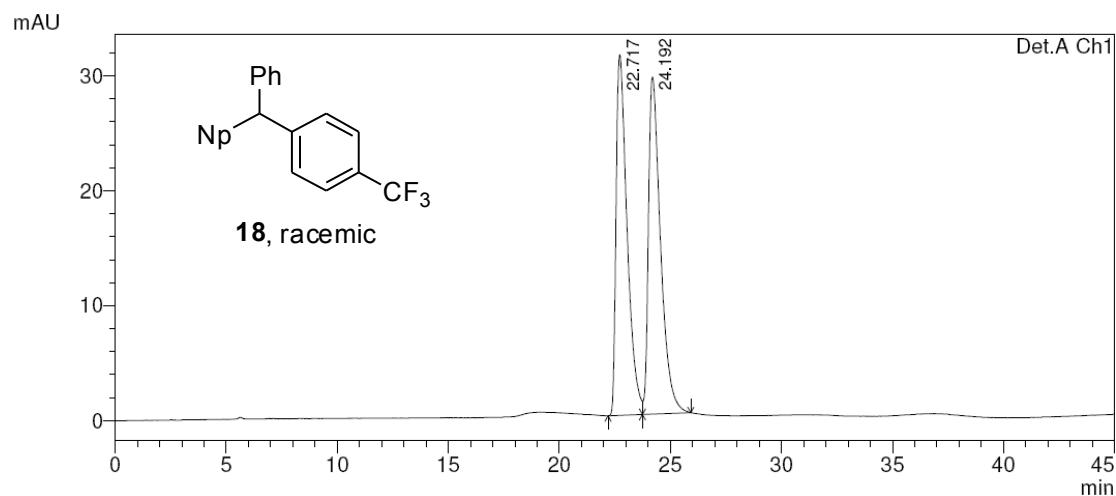
Compound 17, 86% ee



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	22.034	191831	3896	6.858	8.490
2	23.999	2605492	41994	93.142	91.510
Total		2797324	45890	100.000	100.000

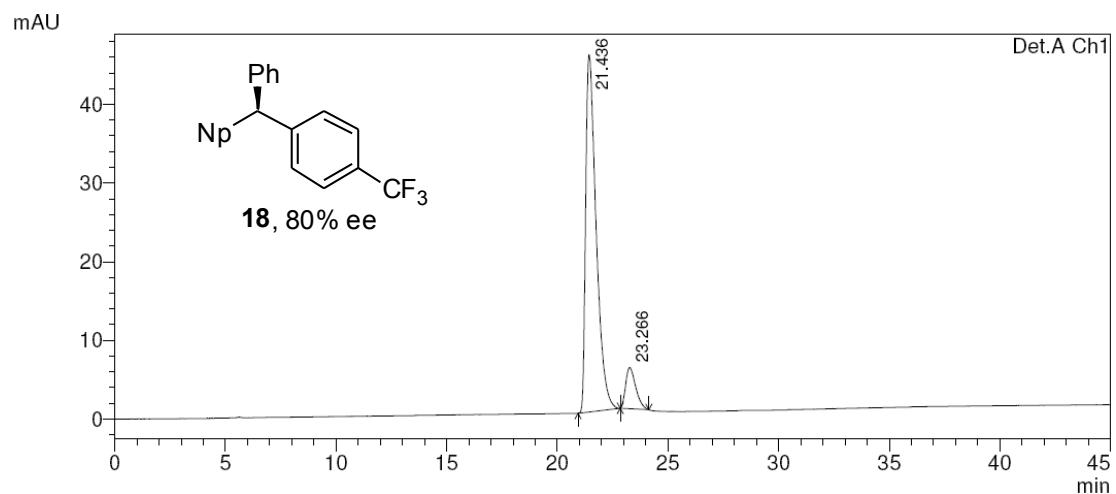
Compound 18, racemic



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	22.717	1082361	31364	49.305	51.676
2	24.192	1112865	29329	50.695	48.324
Total		2195226	60693	100.000	100.000

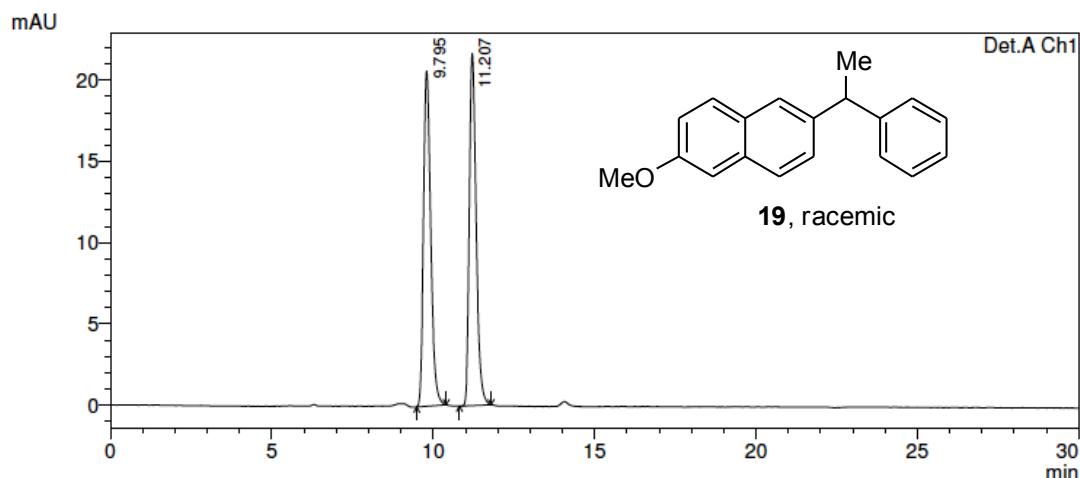
Compound 18, 80% ee



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	21.436	1487789	45441	89.984	89.669
2	23.266	165603	5235	10.016	10.331
Total		1653392	50676	100.000	100.000

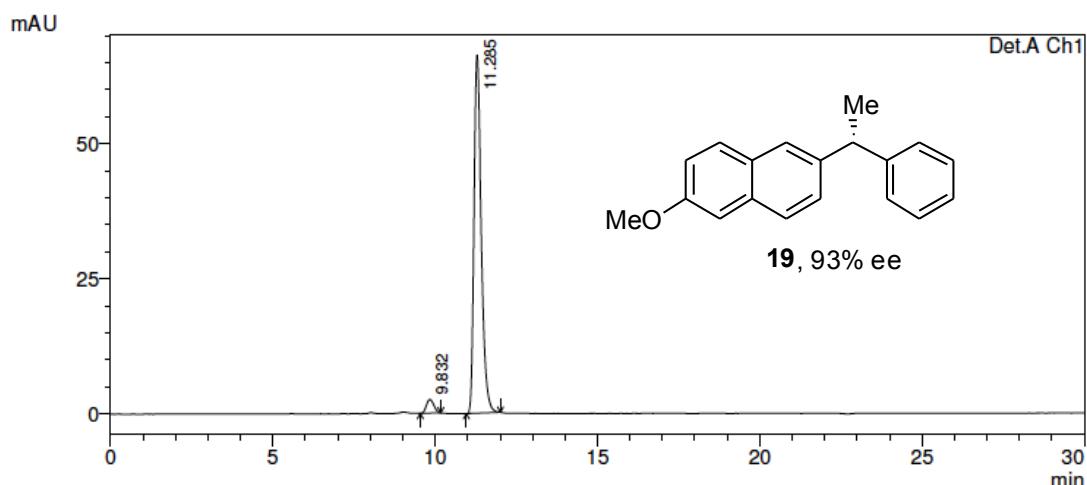
Compound 19, racemic



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.795	319664	20638	50.251	48.794
2	11.207	316471	21658	49.749	51.206
Total		636135	42296	100.000	100.000

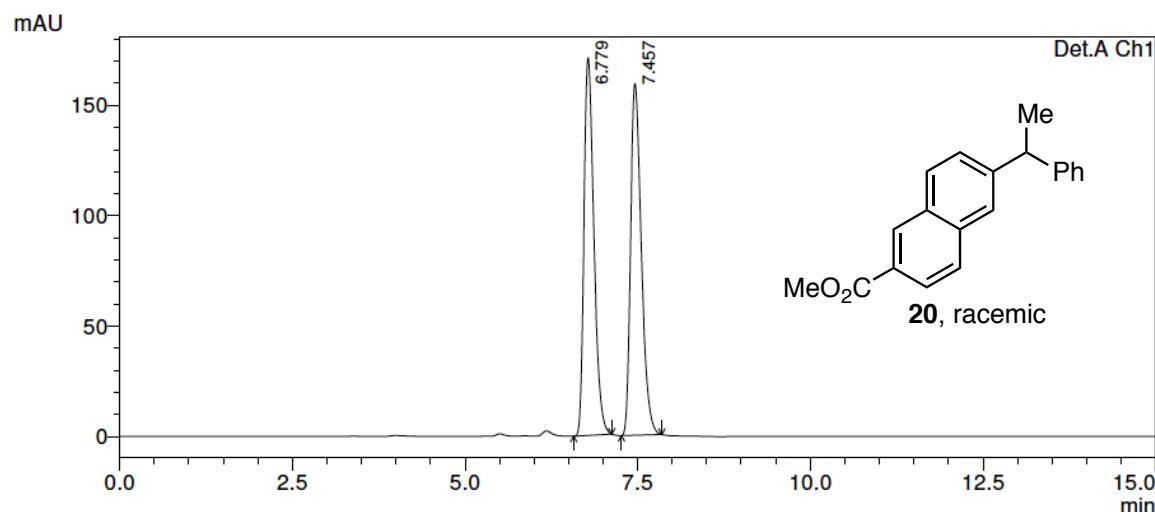
Compound 19, 93% ee



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.832	40139	2543	3.773	3.694
2	11.285	1023789	66290	96.227	96.306
Total		1063928	68833	100.000	100.000

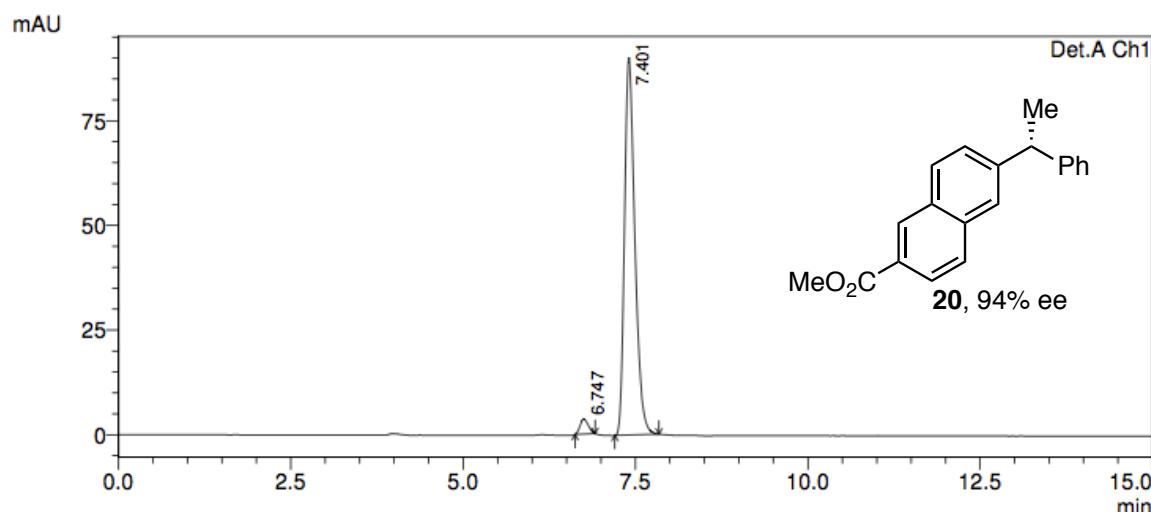
Compound 20, racemic



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.779	1705134	171182	49.744	51.809
2	7.457	1722698	159230	50.256	48.191
Total		3427831	330412	100.000	100.000

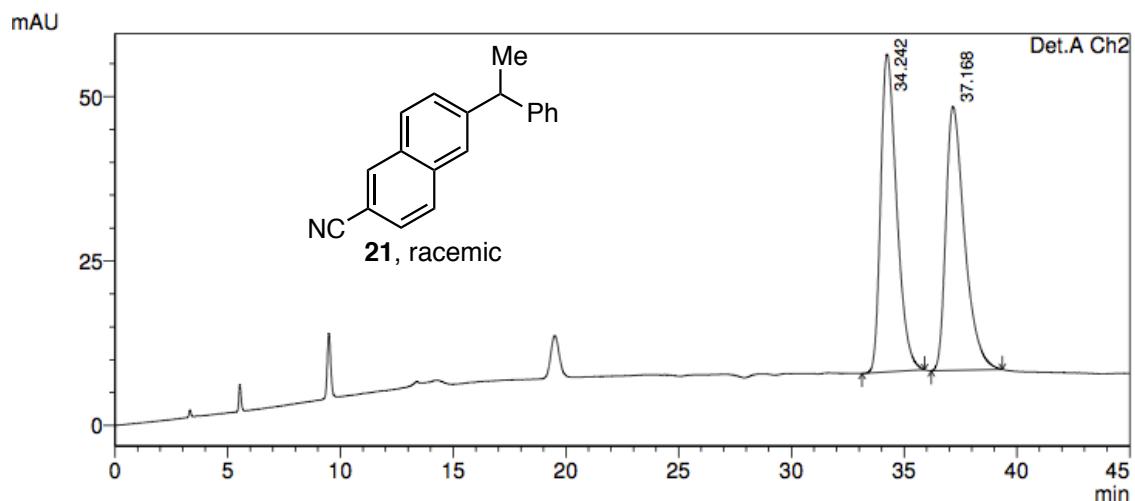
Compound 20, 94% ee



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.747	30236	3609	3.099	3.847
2	7.401	945469	90198	96.901	96.153
Total		975705	93807	100.000	100.000

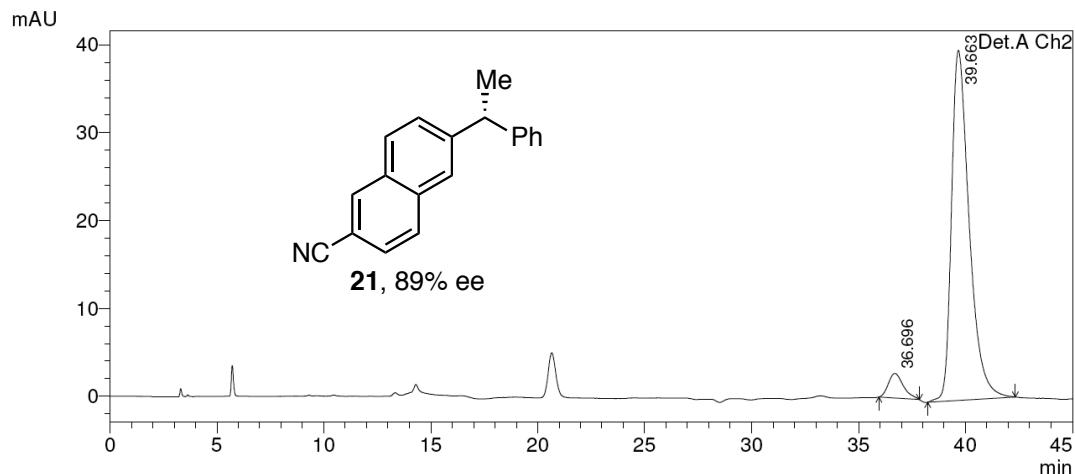
Compound 21, racemic



Detector A Ch2 230nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	34.242	2385648	48276	50.718	54.631
2	37.168	2318059	40092	49.282	45.369
Total		4703707	88368	100.000	100.000

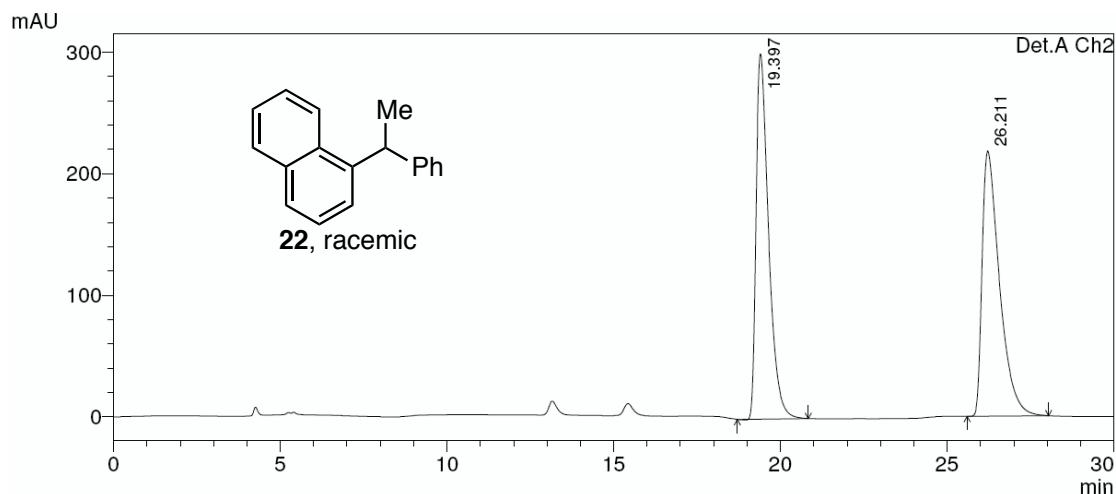
Compound 21, 89% ee



Detector A Ch2 230nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	32.051	57812	1412	5.285	6.564
2	34.791	1036032	20095	94.715	93.436
Total		1093843	21507	100.000	100.000

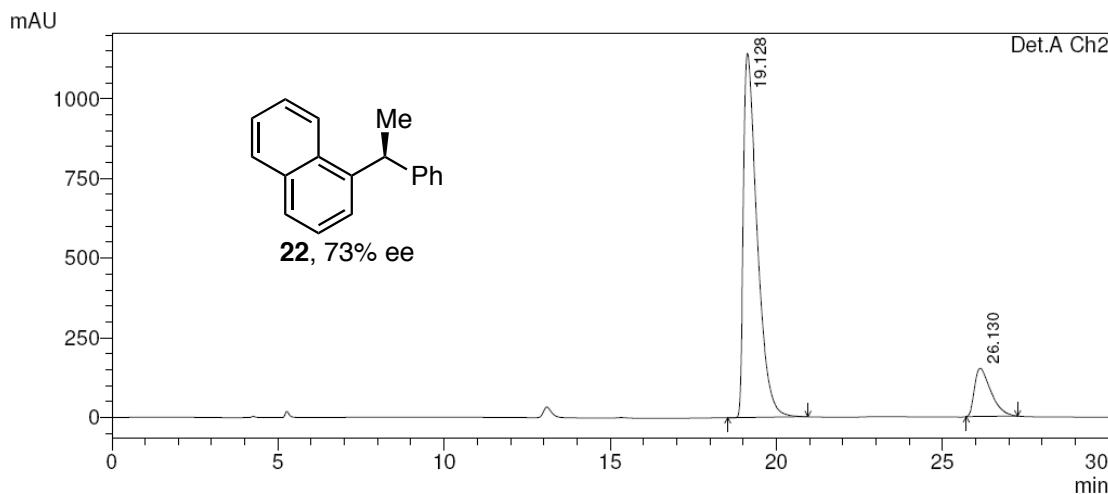
Compound 22, racemic



Detector A Ch2 220nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	19.397	7810522	300253	49.720	57.922
2	26.211	7898490	218125	50.280	42.078
Total		15709011	518378	100.000	100.000

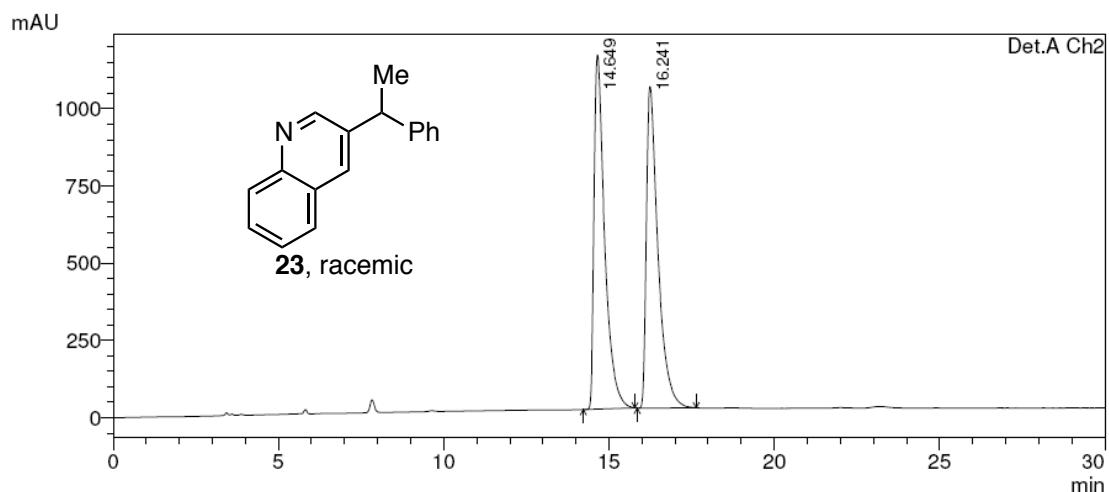
Compound 22, 73% ee



Detector A Ch2 220nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	19.128	32816995	1143059	86.387	88.309
2	26.130	5171503	151332	13.613	11.691
Total		37988498	1294391	100.000	100.000

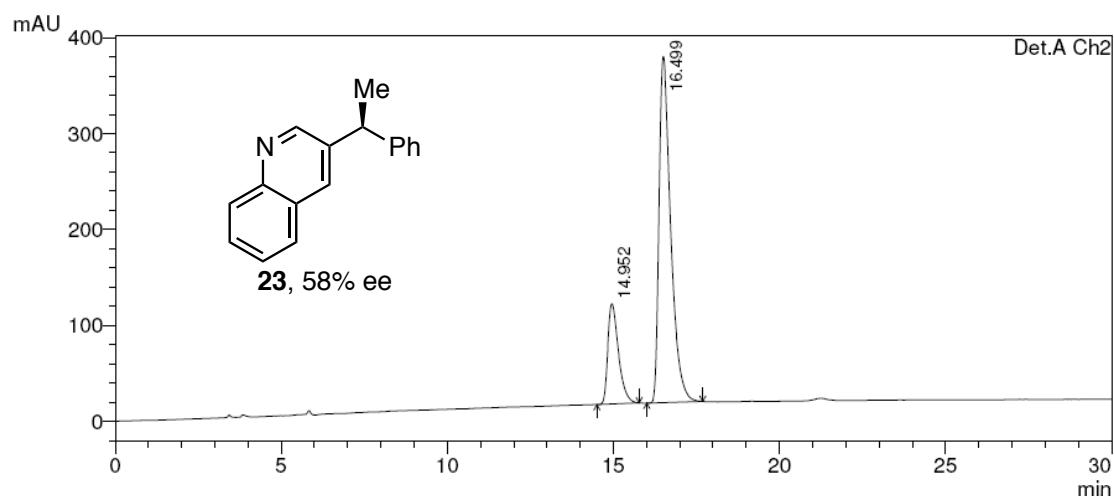
Compound 23, racemic



Detector A Ch2 210nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	14.649	25499869	1145829	50.011	52.413
2	16.241	25488158	1040344	49.989	47.587
Total		50988027	2186173	100.000	100.000

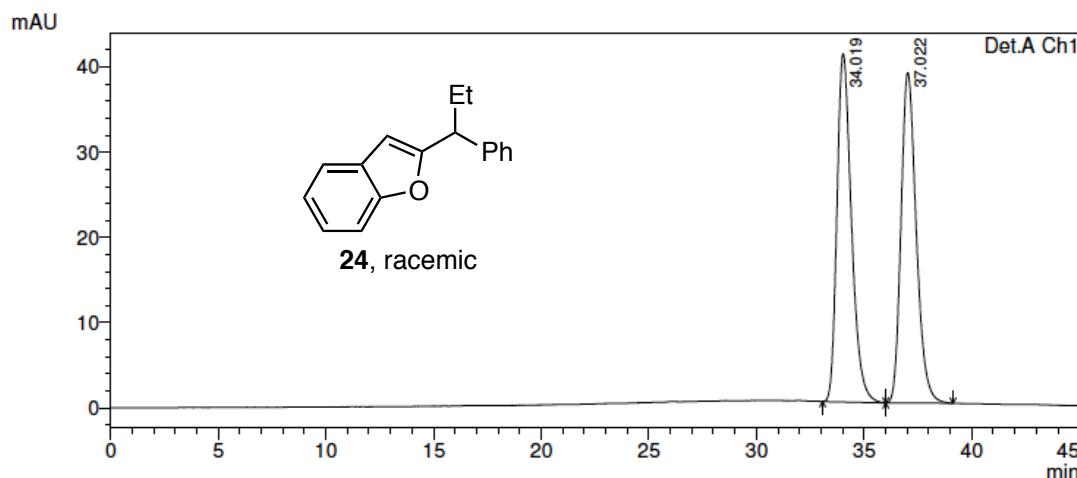
Compound 23, 58% ee



Detector A Ch2 210nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	14.952	2329178	104604	20.956	22.464
2	16.499	8785565	361045	79.044	77.536
Total		11114743	465649	100.000	100.000

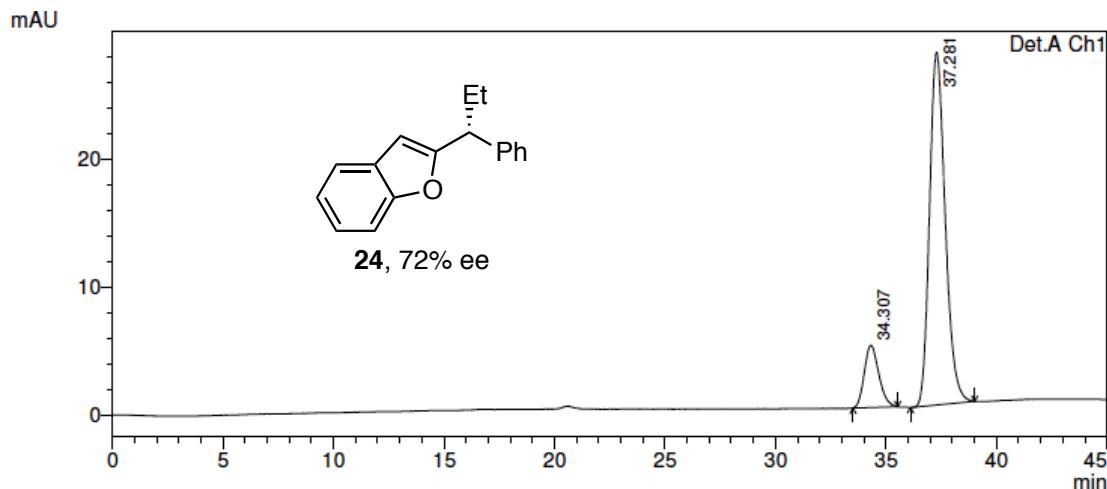
Compound 24, racemic



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	34.019	1909990	40855	50.048	51.323
2	37.022	1906321	38748	49.952	48.677
Total		3816311	79603	100.000	100.000

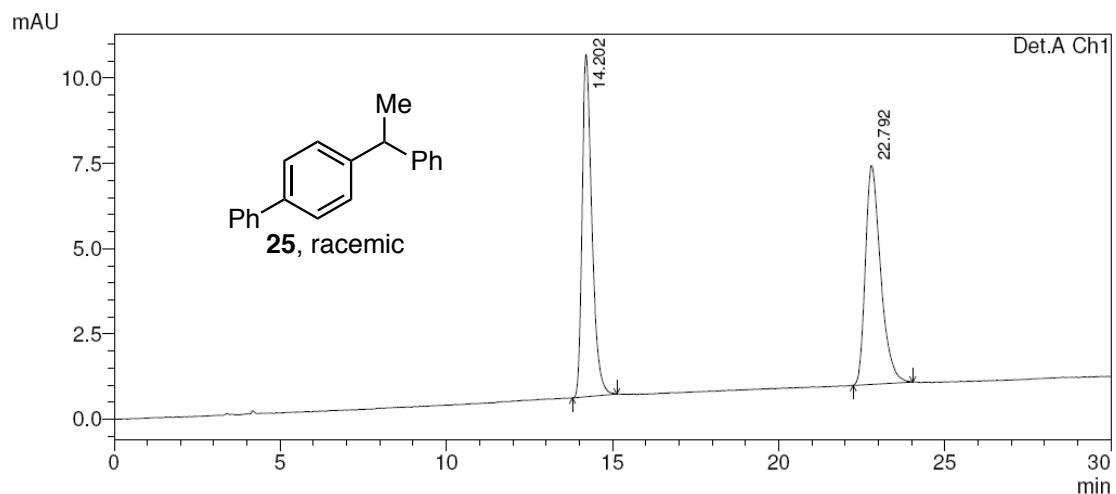
Compound 24, 72% ee



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	34.307	221554	4844	14.011	14.963
2	37.281	1359765	27528	85.989	85.037
Total		1581318	32372	100.000	100.000

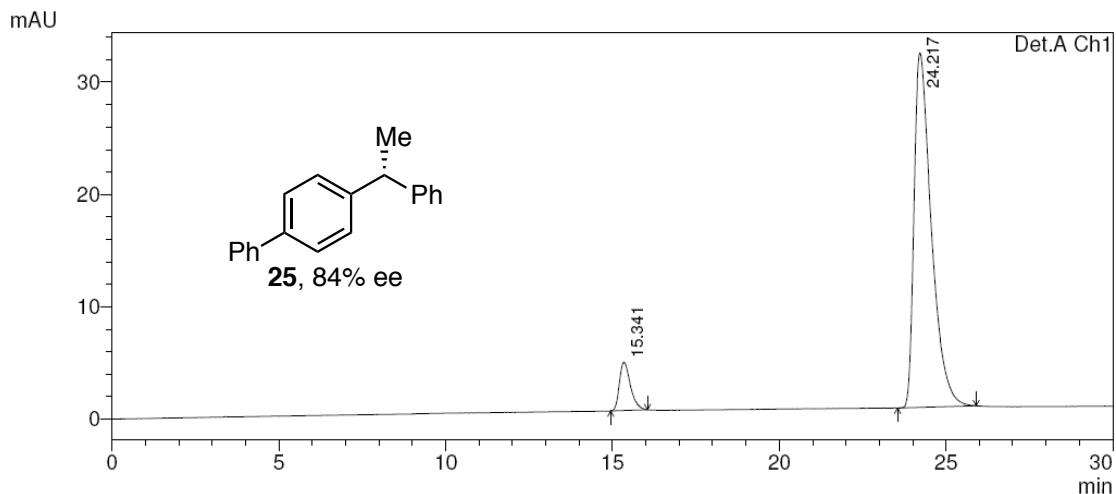
Compound 25, racemic



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	14.202	205998	10036	50.049	60.984
2	22.792	205596	6420	49.951	39.016
Total		411595	16456	100.000	100.000

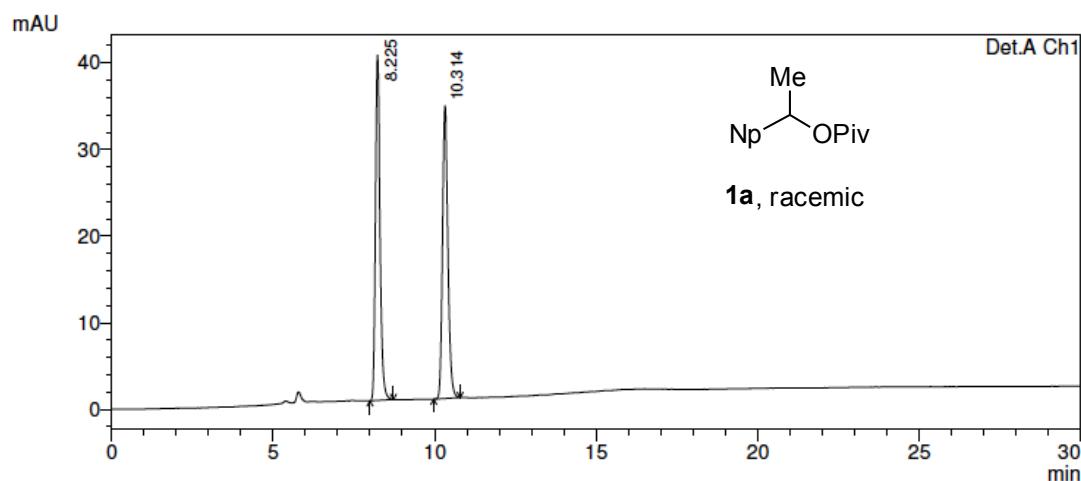
Compound 25, 84% ee



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	15.341	98693	4298	7.974	11.991
2	24.217	1138964	31549	92.026	88.009
Total		1237657	35848	100.000	100.000

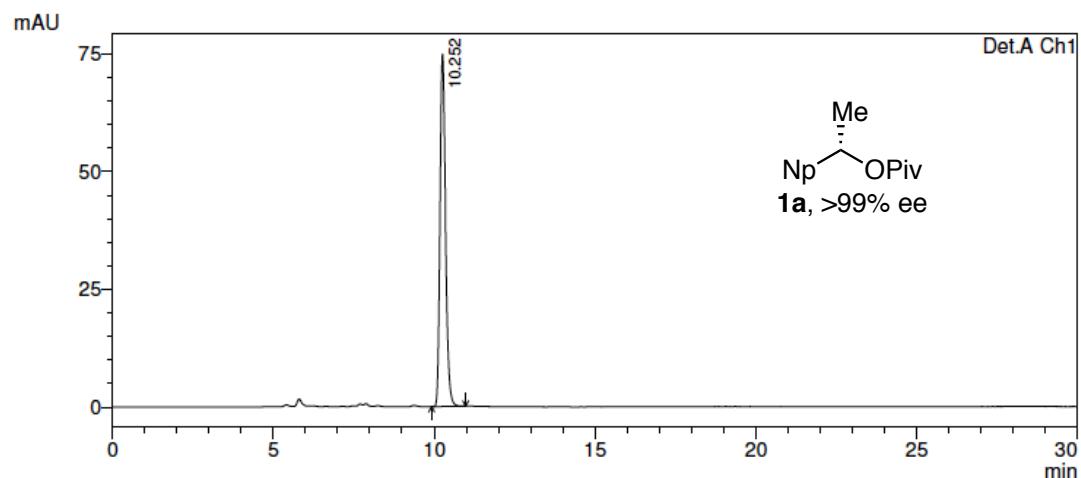
Compound 1a, racemic



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.225	396397	39831	49.995	54.121
2	10.314	396480	33765	50.005	45.879
Total		792877	73597	100.000	100.000

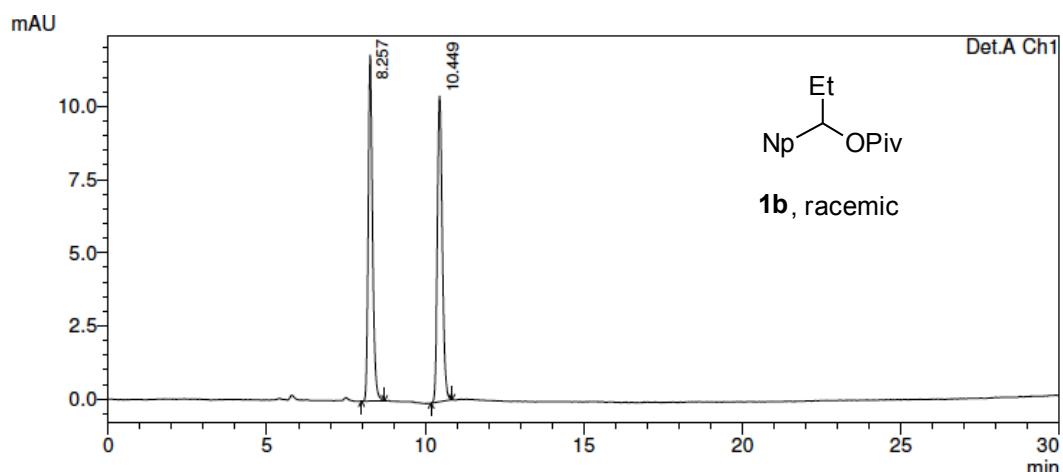
Compound 1a, >99% ee



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	10.252	897607	74803	100.000	100.000
Total		897607	74803	100.000	100.000

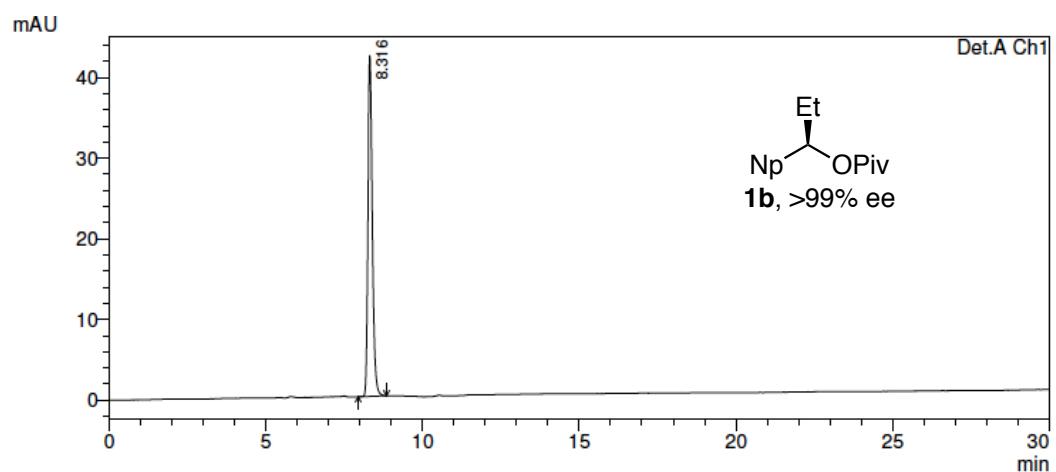
Compound 1b, racemic



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.257	114388	11828	50.272	53.100
2	10.449	113152	10447	49.728	46.900
Total		227540	22276	100.000	100.000

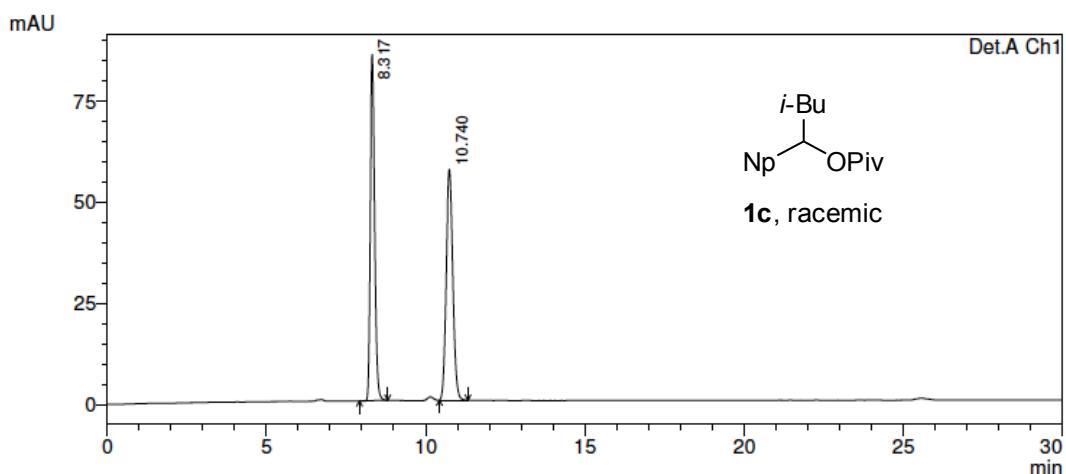
Compound 1b, >99% ee



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.316	415101	42242	100.000	100.000
Total		415101	42242	100.000	100.000

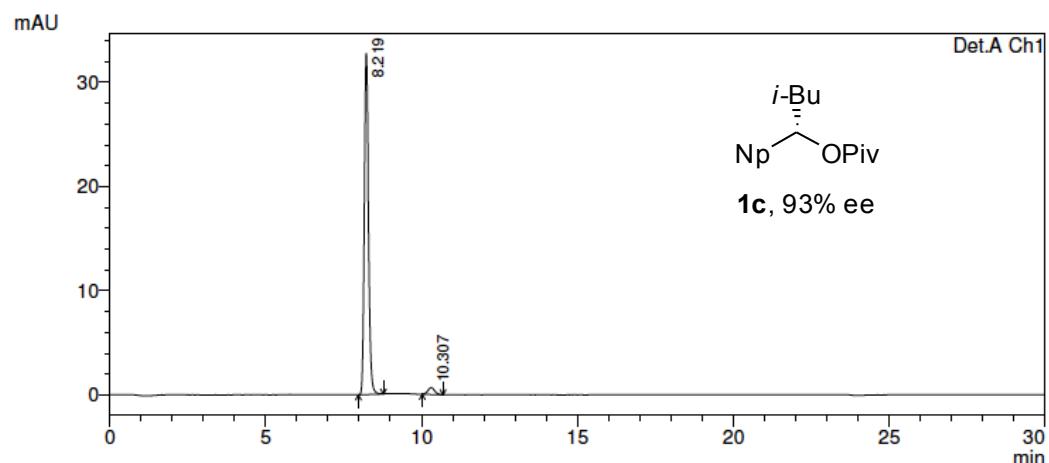
Compound 1c, racemic



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.317	820067	85600	49.944	59.987
2	10.740	821921	57098	50.056	40.013
Total		1641989	142698	100.000	100.000

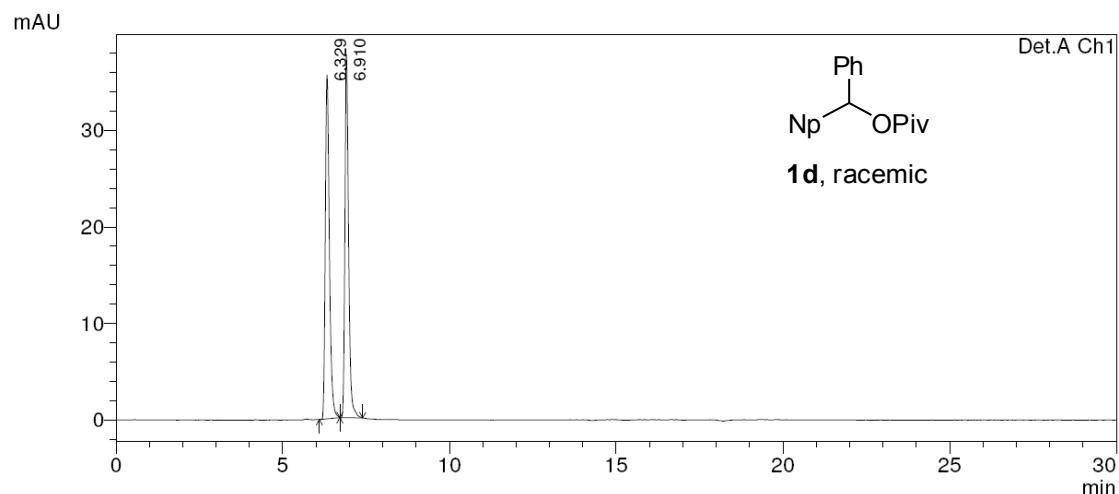
Compound 1c, 93% ee



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.219	308202	32752	96.654	97.974
2	10.307	10668	677	3.346	2.026
Total		318870	33429	100.000	100.000

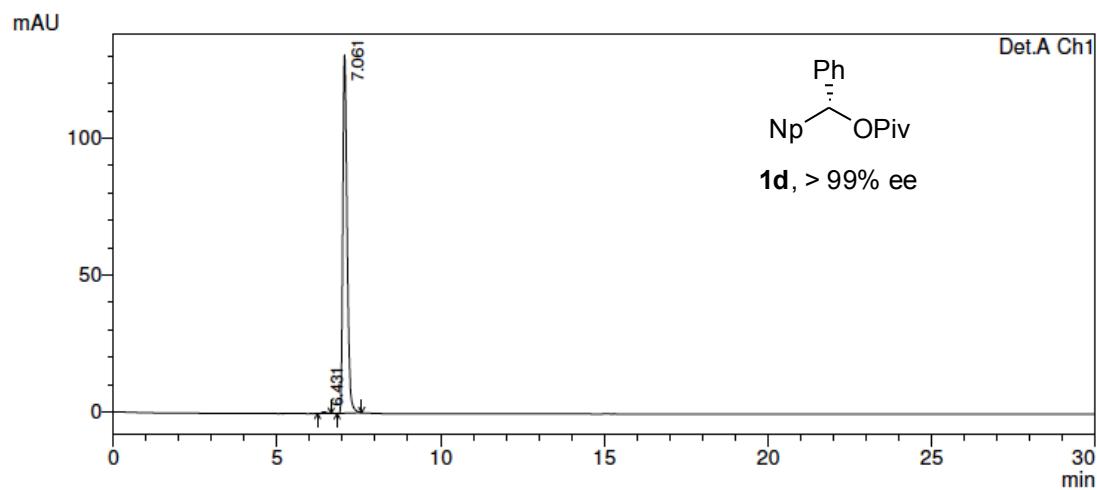
Compound 1d, racemic



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.329	297105	35629	49.891	48.644
2	6.910	298408	37615	50.109	51.356
Total		595513	73244	100.000	100.000

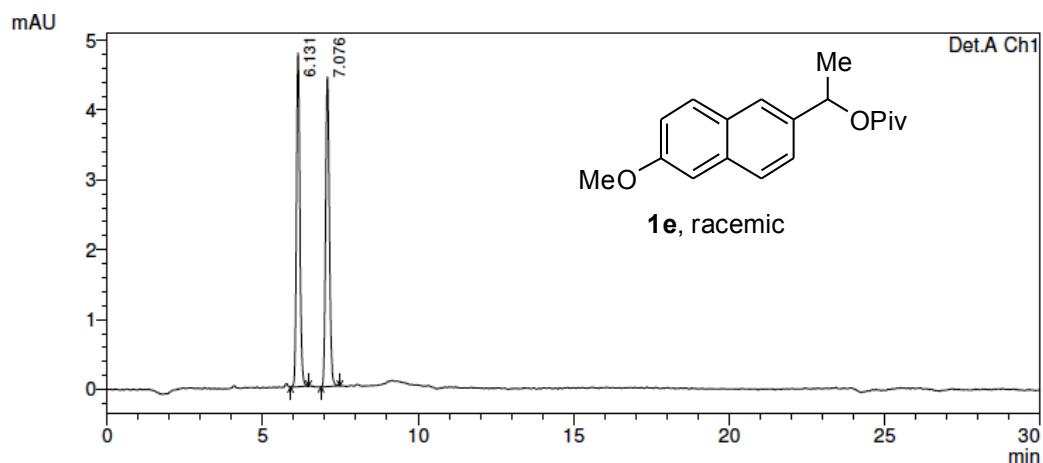
Compound 1d, >99% ee



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.431	5458	661	0.464	0.503
2	7.061	1171050	130897	99.536	99.497
Total		1176509	131558	100.000	100.000

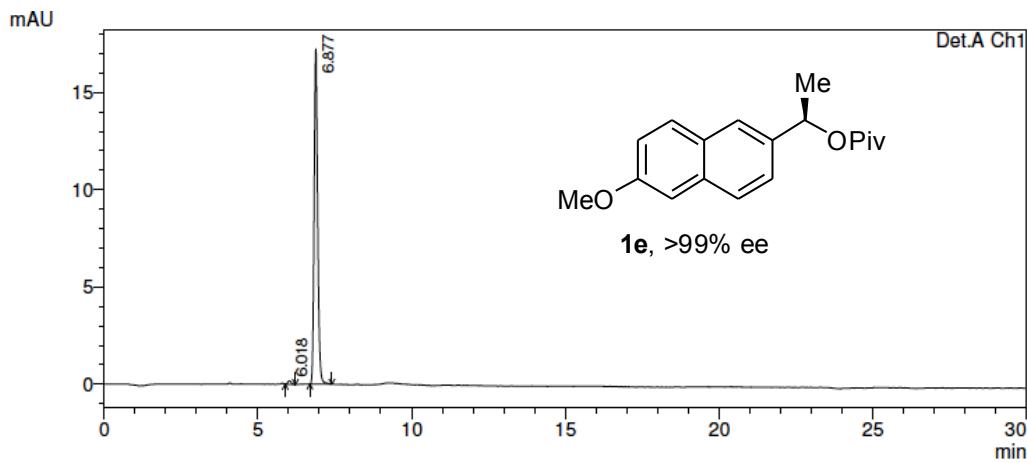
Compound 1e, racemic



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.131	37054	4787	50.237	51.891
2	7.076	36705	4438	49.763	48.109
Total		73759	9224	100.000	100.000

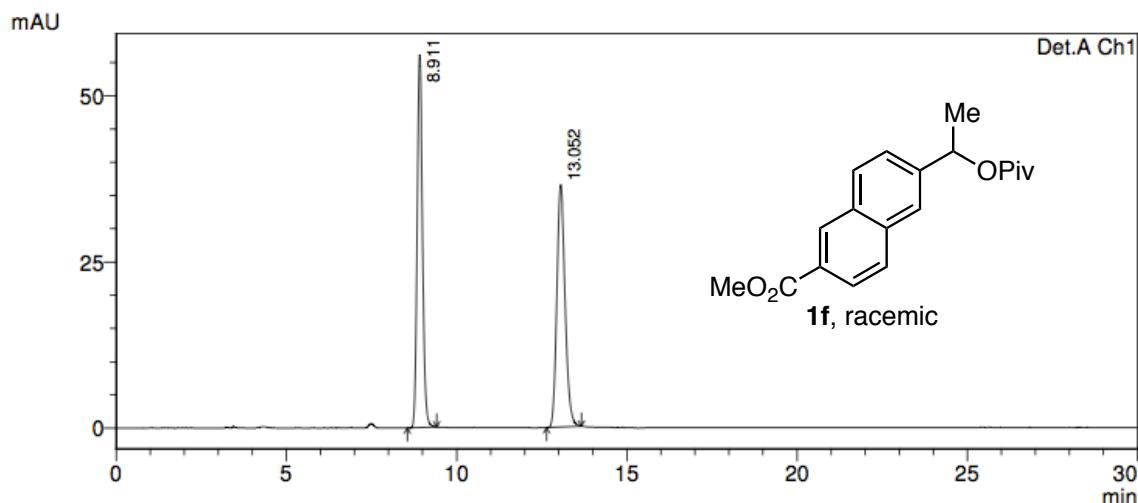
Compound 1e, >99% ee



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.018	1376	185	0.962	1.060
2	6.877	141653	17224	99.038	98.940
Total		143029	17409	100.000	100.000

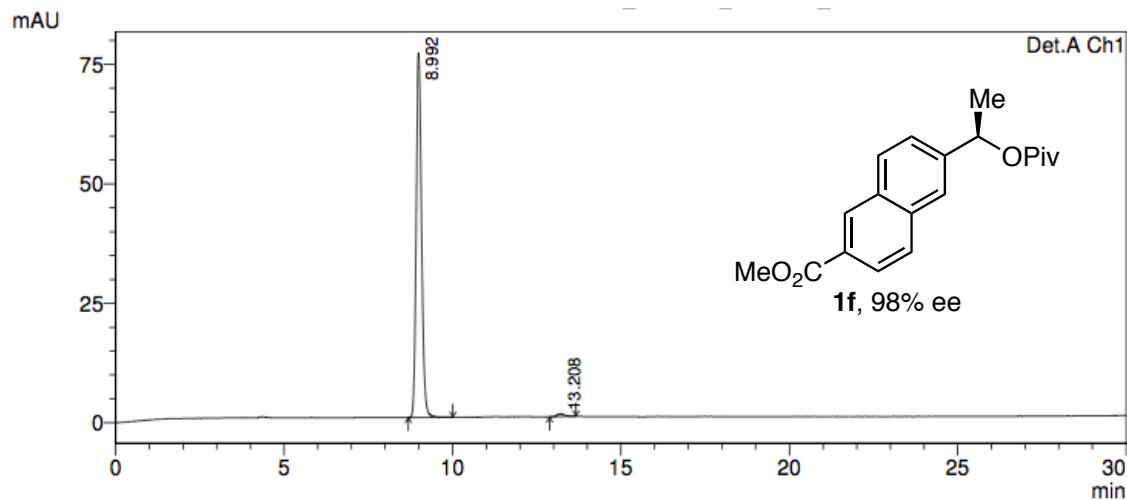
Compound 1f, racemic



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.911	613049	56085	50.015	60.609
2	13.052	612685	36451	49.985	39.391
Total		1225734	92536	100.000	100.000

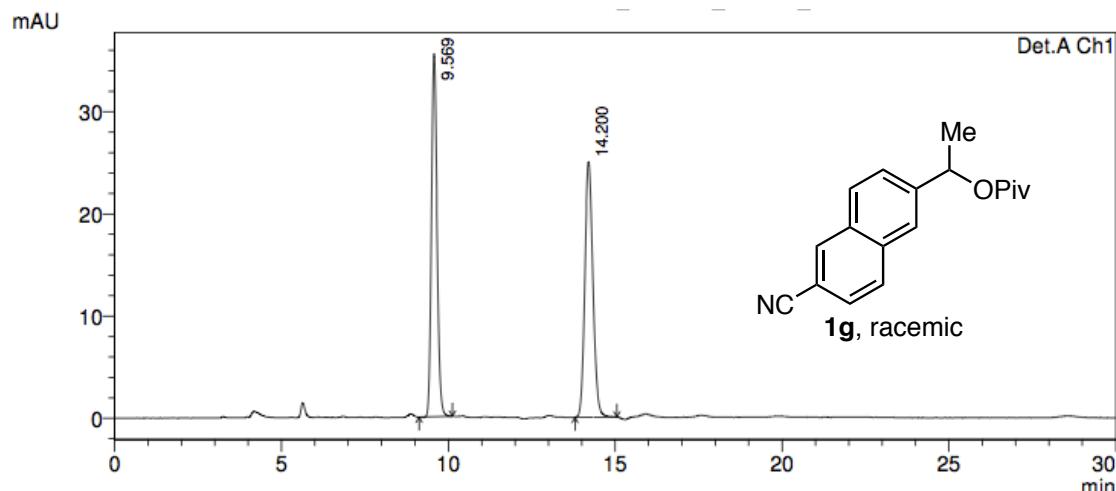
Compound 1f, 98% ee



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.992	851729	76272	99.004	99.274
2	13.208	8567	557	0.996	0.726
Total		860296	76830	100.000	100.000

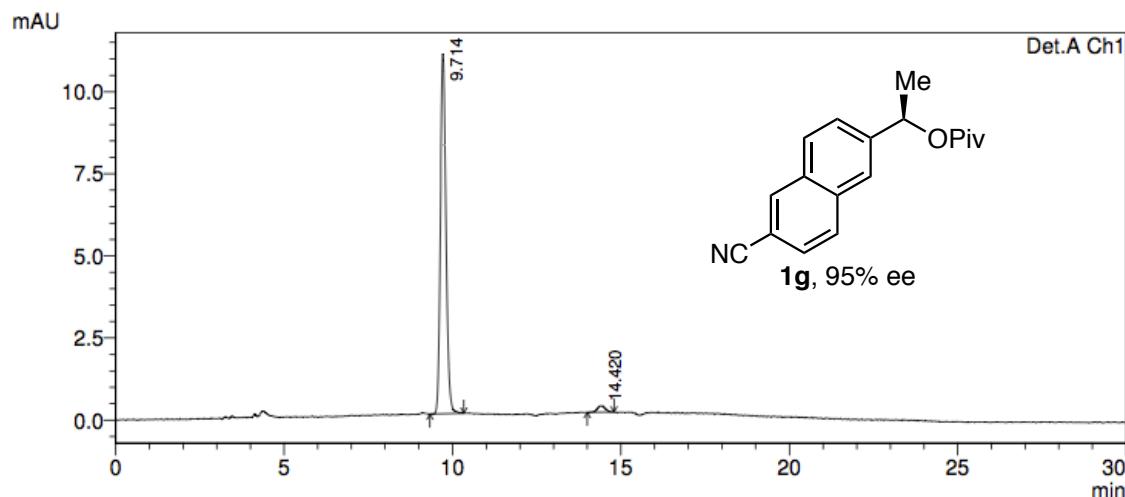
Compound 1g, racemic



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.569	415129	35588	49.625	58.719
2	14.200	421402	25019	50.375	41.281
Total		836531	60608	100.000	100.000

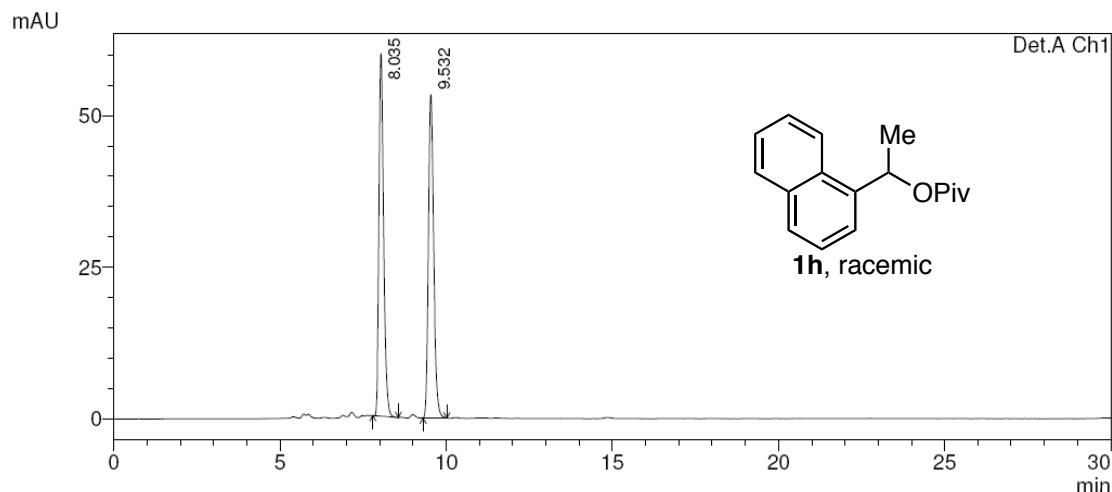
Compound 1g, 95% ee



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.714	126744	10973	97.412	98.176
2	14.420	3367	204	2.588	1.824
Total		130111	11177	100.000	100.000

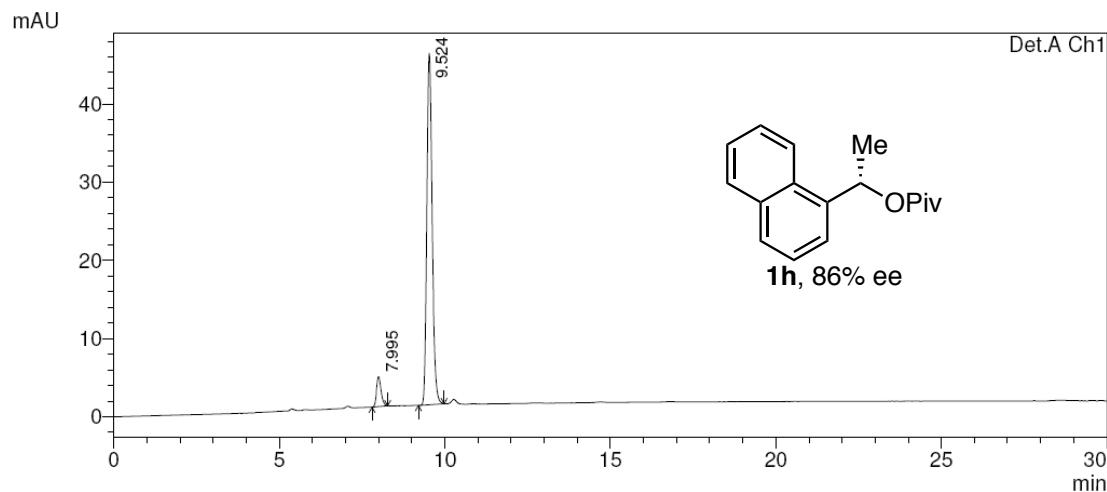
Compound 1h, racemic



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.035	579672	59706	49.676	52.828
2	9.532	587238	53313	50.324	47.172
Total		1166910	113019	100.000	100.000

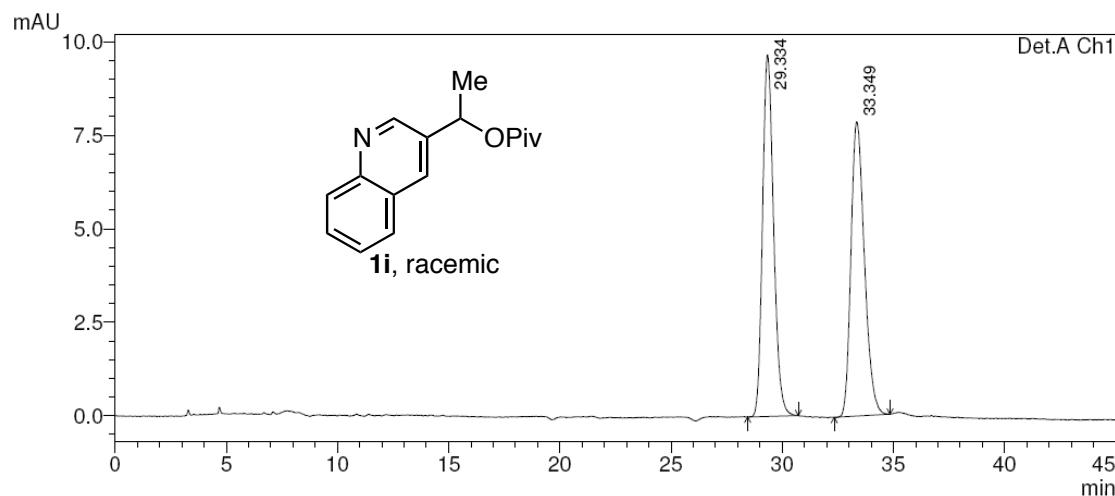
Compound 1h, 86% ee



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.995	36367	3814	6.791	7.825
2	9.524	499160	44924	93.209	92.175
Total		535527	48738	100.000	100.000

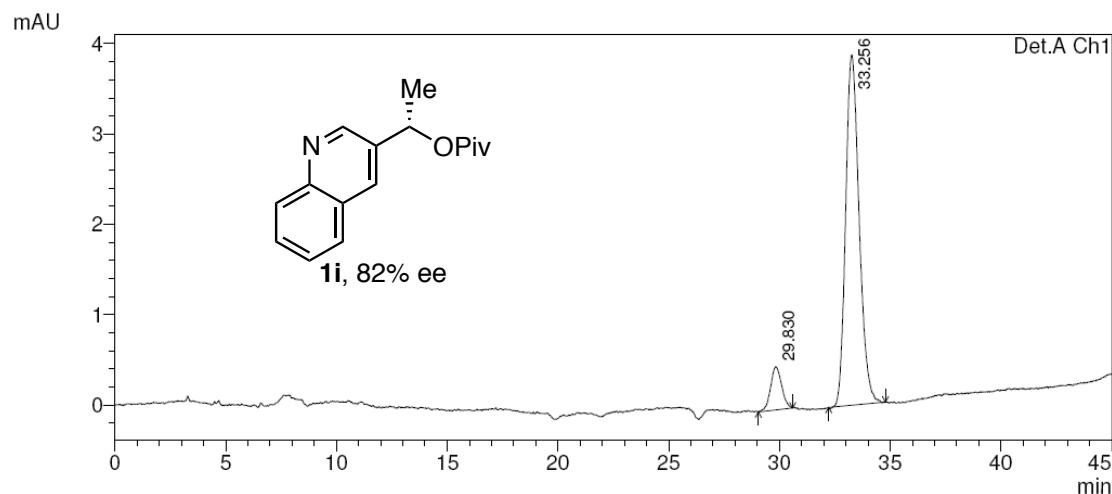
Compound 1i, racemic



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	29.334	333054	9664	50.134	55.117
2	33.349	331269	7870	49.866	44.883
Total		664323	17534	100.000	100.000

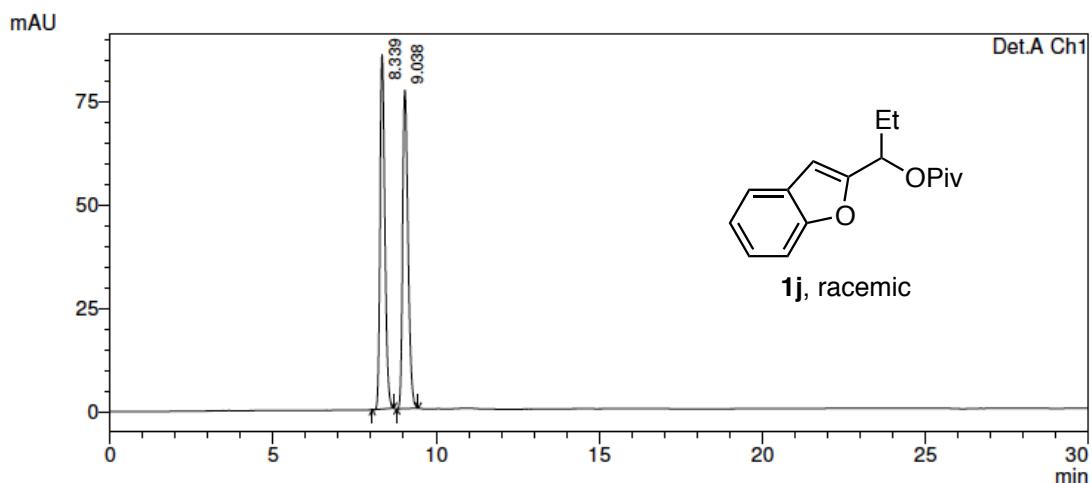
Compound 1i, 82% ee



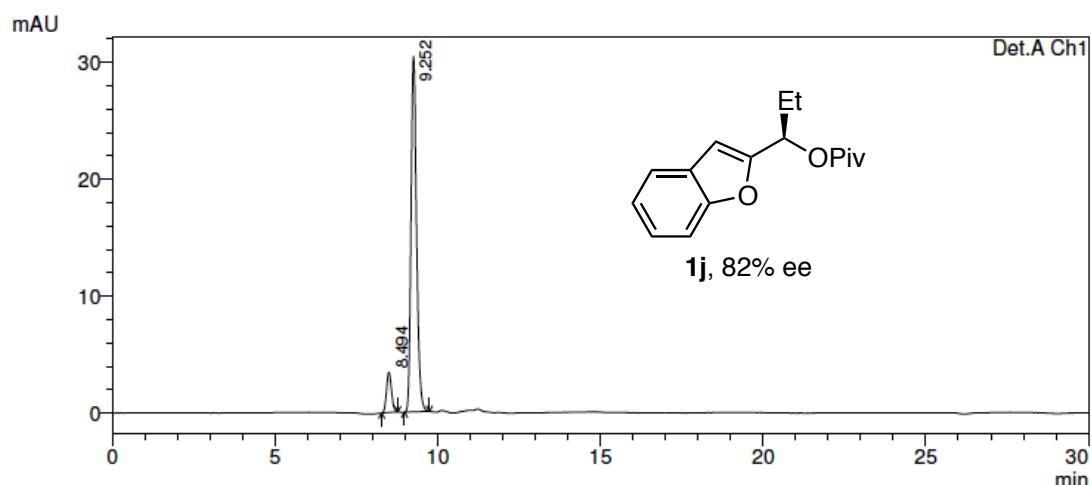
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	29.830	16673	481	9.218	11.024
2	33.256	164207	3881	90.782	88.976
Total		180880	4362	100.000	100.000

Compound 1j, racemic



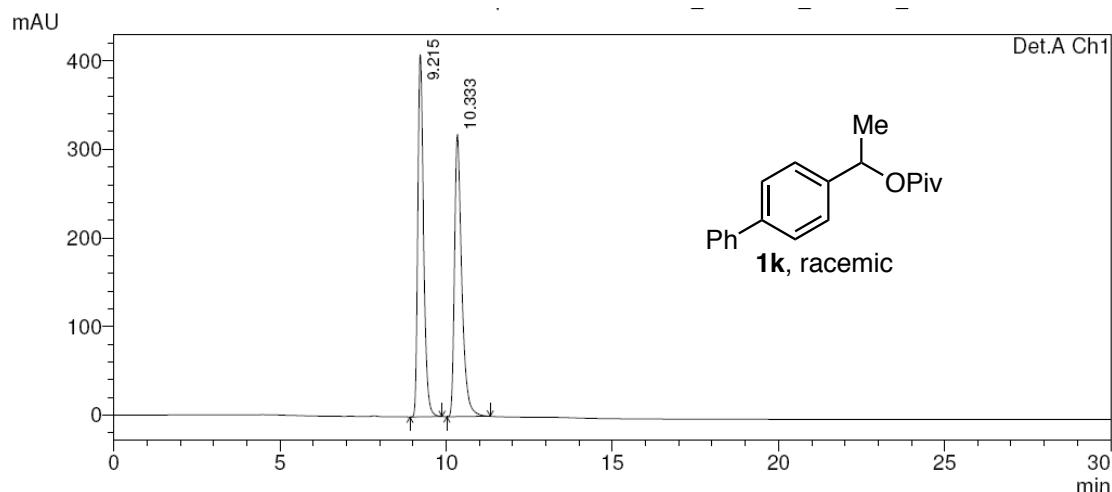
Compound 1j, 82% ee



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.494	36076	3440	9.012	10.169
2	9.252	364222	30391	90.988	89.831
Total		400298	33832	100.000	100.000

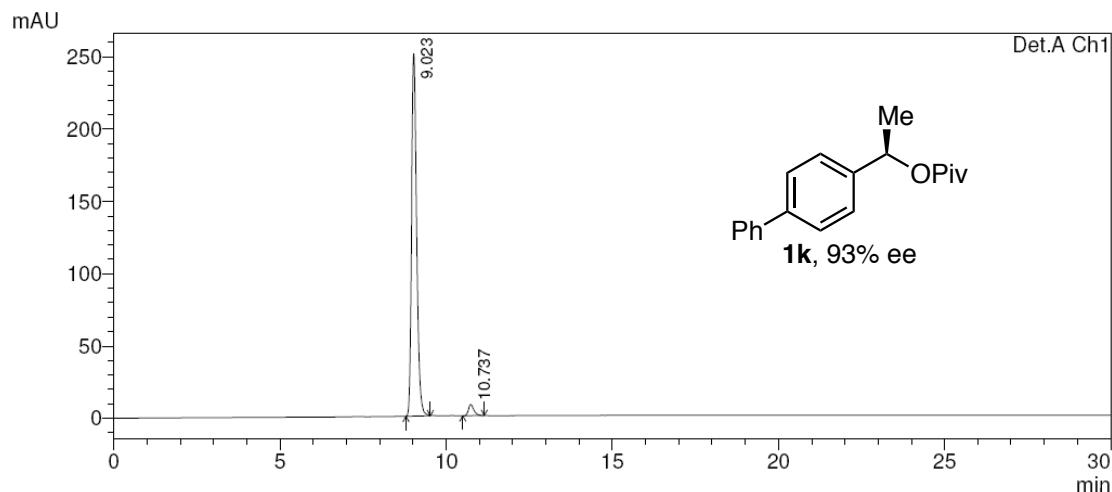
Compound 1k, racemic



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.215	4946210	408528	50.233	56.198
2	10.333	4900271	318419	49.767	43.802
Total		9846481	726947	100.000	100.000

Compound 1k, 93% ee



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.023	2670459	250803	96.736	96.996
2	10.737	90112	7769	3.264	3.004
Total		2760571	258572	100.000	100.000