Enantiospecific, Nickel-Catalyzed Cross-Couplings of Allylic Pivalates and Arylboroxines

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Supporting Information

General Information

Reactions were performed either in a N_2-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 N_2. 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) unless otherwise noted. 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, CH_2Cl_2, dioxane, MeCN and Et_2O were dried by passing through drying columns.[1] Toluene and MeCN were then degassed by sparging with N_2 and stored over activated 4Å MS in a N_2-atmosphere glovebox. Pivaloyl chloride was purchased from Sigma Aldrich and distilled before use. Boronic acids were purchased from Combi Blocks and were converted to the boroxines according to literature procedure.[2] Racemic products **2**–**24** were prepared from the corresponding racemic pivalates using the General Procedure for the cross coupling described below. $CDCl_3$ was stored over oven-dried potassium carbonate. Proton nuclear magnetic resonance (^{1}H NMR) spectra and carbon nuclear magnetic resonance (^{13}C 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 ($CHCl_3$ = δ 7.26; $(CD_3)_2CO$ = δ 2.07). Chemical shifts for carbon are reported in parts per million downfield from tetramethylsilane and are referenced to the carbon resonances of the solvent ($CDCl_3$ = δ 77.2; $(CD_3)_2CO$ = δ 28.94). 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

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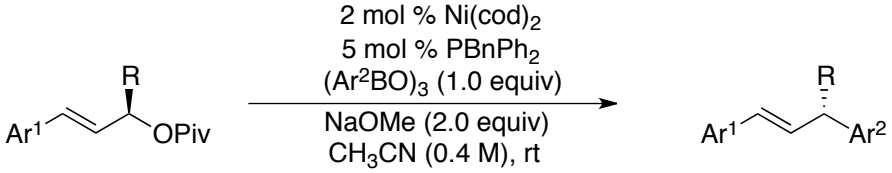
1 Pangborn, A. B.; Giardello, M. A.; Grubbs, R. H.; Rosen, R. K.; Timmers, F. J. *Organometallics* **1996**, *15*, 1518.

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

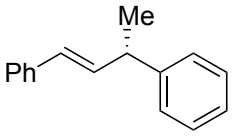
obtained at the University of Delaware mass spectrometry facility. Optical rotations were measured using a 2.5 mL cell with a 0.1 dm path length. Melting points were taken on a Stuart SMP10 instrument.

Stereospecific Cross Coupling of Allylic Pivalates with Boroxines

General Procedure for the Stereospecific, Nickel-Catalyzed Coupling of Allylic Pivalates with Boroxines

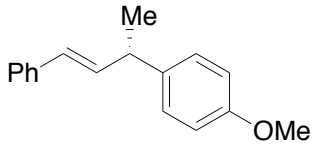


In a N_2-atmosphere glovebox, $Ni(cod)_2$ (1.7 mg, 0.06 mmol, 2 mol %), benzyl diphenyl phosphine (4.2 mg, 0.015 mmol, 5 mol %) and NaOMe (32.5 mg, 0.6 mmol, 2.0 equiv) were weighed into a 1-dram vial. Allylic pivalate (0.30 mmol, 1.0 equiv) and boroxine (0.30 mmol, 1.0 equiv) were added, followed by acetonitrile (0.75 mL, 0.4 M). The vial was capped with a Teflon-lined cap and removed from the glovebox. The mixture was stirred at room temperature for the time specified below. 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 then purified by silica gel chromatography to give the arylated product.

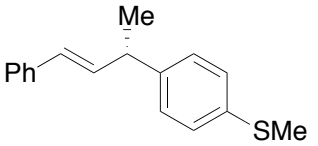


(*S*,*E*)-But-1-ene-1,3-diyldibenzene (2). Product **2** was prepared via the General Procedure using pivalate **1a** (prepared in 96% ee). The reaction was stirred at room temperature for 4 h. The crude material was purified by silica gel chromatography (100% hexanes) to give compound **2** (run 1: 55.6 mg, 89%; run 2: 54.3 mg, 87%) as colorless oil. The enantiomeric excess was determined to be 95% (run 1: 95% ee; run 2: 95% ee) by chiral HPLC analysis (CHIRALCEL OD-H, 1.0 mL/min, 0.1% *i*-PrOH/hexane λ=254 nm); t_R(major)=6.07 min, t_R(minor)=6.48 min. $[\alpha]_D^{24}$ = –21.1° (c 1.42, $CHCl_3$): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 – 7.36 (m, 2H), 7.35 – 7.27 (m, 6H), 7.25 – 7.19 (m, 2H), 6.46 – 6.37 (m, 2H), 3.69 – 3.63(m, 1H), 1.49 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, $(CD_3)_2CO$) δ 145.8, 137.6, 135.2, 128.5, 128.43, 128.40, 127.1,

127.0, 126.11, 126.07, 42.6, 20.9. The spectral data for this compound matches that reported in the literature.[3]



(*S*)-2-(1-Phenylethyl)naphthalene (3). Compound **3** was prepared via the General Procedure using pivalate **1a** (prepared in 96% ee). The crude material was purified by silica gel chromatography (2% Et_2O/hexanes) to give compound **3** (run 1: 65.9 mg, 92%; run 2: 63.0 mg, 88%) as colorless oil. The enantiomeric excess was determined to be 88% (run 1: 87% ee; run 2: 89% ee) by chiral HPLC analysis (CHIRALCEL OD-H, 0.8 mL/min, 0.1% *i*-PrOH/hexane, λ=254 nm); t_R(major)=14.47 min, t_R(minor)=13.13 min. $[\alpha]_D^{24}$ = –2.8° (c 3.56, $CHCl_3$): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 – 7.35 (m, 2H), 7. 34 – 7.26 (m, 2H), 7.25 – 7.16 (m, 3H), 6.93 – 6.84 (m, 2H), 6.45 – 6.34 (m, 2H), 3.81 (s, 3H), 3.66 – 3.55 (m, 1H), 1.46 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 158.1, 137.8, 137.7, 135.7, 128.6, 128.39, 128.36, 127.2, 126.3, 114.0, 55.4, 41.9, 21.5. The spectral data for this compound matches that reported in the literature.[4]



((*S*,*E*)-Methyl(4-(4-phenylbut-3-en-2-yl)phenyl)sulfane (4). Compound **4** was prepared via the General Procedure using pivalate **1a** (prepared in 96% ee). The crude material was purified by silica gel chromatography (100% hexanes) to give compound **4** (run 1: 73.2 mg, 96%; run 2: 70.6 mg, 93%) as a white amorphous solid (mp 49–51 °C). The enantiomeric excess was determined to be 93% (run 1: 93% ee; run 2: 92% ee) by chiral HPLC analysis (CHIRALPAK IA, 1.0 mL/min, 100% hexanes, λ=254 nm); t_R(major)=13.90 min, t_R(minor)=17.71 min. $[\alpha]_D^{24}$ = –8.3° (c 1.44, $CHCl_3$): ^{1}H NMR (600 MHz, $(CD_3)_2CO$) δ 7.43 – 7.38 (m, 2H), 7.32 – 7.16 (m, 7H), 6.48 – 6.44 (m, 2H), 3.68 – 3.60 (m, 1H), 2.46 (s, 3H), 1.43 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (151 MHz,

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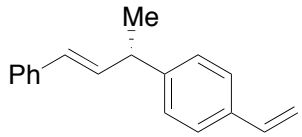
[3] Sanz, R.; Miguel, D.; Martínez, A.; Álvarez-Gutiérrez, J. M.; Rodríguez, F. *Org. Lett*. **2007**, 9, 727.

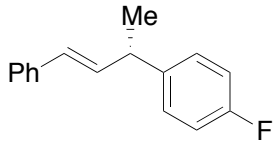
[4] Malkov, A. V.; Davis, S. L.; Baxendale, I. R.; Mitchell, W. L.; Kocovský , P. *J. Org. Chem*. **1999**, 64, 2751.

$CDCl_3$) δ 142.8, 137.5, 135.9, 135.0, 128.6, 128.5, 127.9, 127.2, 127.1, 126.2, 42.1, 21.2, 16.3; FTIR (NaCl/thin film) 3023, 2963, 2920, 2869, 1891, 1799, 1598, 1493, 1446, 1010, 964, 746; HRMS (EI+) $[M]^+$ calculated for $C_{17}H_{18}S$: 254.1129, found: 254.1134.



(*S*,*E*)-1-(4-Phenylbut-3-en-2-yl)-4-vinylbenzene (5).

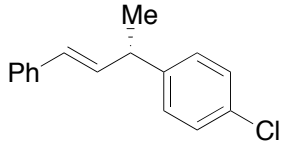
Compound **5** was prepared via the General Procedure using pivalate **1a** (prepared in 96% ee). The crude material was purified by silica gel chromatography (100% hexanes) to give compound **5** (run 1: 65.4 mg, 93%; run 2: 64.8 mg, 92%) as yellow oil. The enantiomeric excess was determined to be 91% (run 1: 91% ee; run 2: 91% ee) by chiral HPLC analysis (CHIRALPAK IA, 1.0 mL/min, 100% hexanes, λ=254 nm); t_R(major)=7.82 min, t_R(minor)=9.24 min. $[\alpha]_D^{24}$ = –22.6 ° (c 2.21, $CHCl_3$): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 – 7.35 (m, 4H), 7.33 – 7.17 (m, 5H), 6.73 (dd, *J* = 17.6, 10.9 Hz, 1H), 6.49 – 6.34 (m, 2H), 5.75 (dd, *J* = 17.6, 1.0 Hz, 1H), 5.23 (dd, *J* = 10.8, 1.0 Hz, 1H), 3.66 (qd, *J* = 6.9, 5.4 Hz, 1H), 1.48 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 145.5, 137.7, 136.7, 135.8, 135.2, 128.74, 128.67, 127.6, 127.3, 126.5, 126.3, 113.4, 42.5, 21.3; FTIR (NaCl/thin film) 3023, 2964, 2928, 1629, 1509, 1494, 965, 838, 746 cm^{-1}; HRMS (EI+) $[M]^+$ calculated for $C_{18}H_{18}$: 234.1409, found: 234.1412.



***(S,E)*-1-Fluoro-4-(4-phenylbut-3-en-2-yl)benzene (6).**

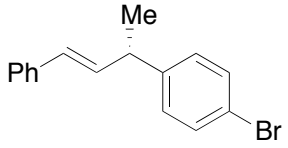
Compound **6** was prepared via the General Procedure using pivalate **1a** (prepared in 95% ee). The crude material was purified by silica gel chromatography (100% hexanes) to give compound **6** (run 1: 57.6 mg, 87%; run 2: 58.9 mg, 85%) as a yellow oil. The enantiomeric excess was determined to be 93% (run 1: 93% ee; run 2: 93% ee) by chiral HPLC analysis (CHIRALPAK IB, 1.0 mL/min, 100% hexanes, λ=254 nm); t_R(major)=7.86 min, t_R(minor)=7.41 min. $[\alpha]_D^{24}$ = –15.4° (c 2.34, $CHCl_3$): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 – 7.18 (m, 7H), 7.07 – 6.97 (m, 2H), 6.46 – 6.31 (m, 2H), 3.69 – 3.60 (m, 1H), 1.47 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.5 (d, $J_{C–F}$ = 243.8 Hz), 141.4 (d, $J_{C–F}$ = 3.2 Hz), 137.5, 135.1, 128.9, 128.8 (d, $J_{C–F}$ = 1.4 Hz), 128.7,

127.3, 126.3, 115.4 (d, J_{C-F} = 21.0 Hz), 42.0, 21.5. The spectral data for this compound matches that reported in the literature.[5]



(*S*,*E*)-1-Chloro-4-(4-phenylbut-3-en-2-yl)benzene (7).

Compound **7** was prepared via the General Procedure using pivalate **1a** (prepared in 96% ee). The crude material was purified by silica gel chromatography (100% hexanes) to give compound **7** (run 1: 53.7 mg, 74%; run 2: 54.6 mg, 75%) as a yellow oil. The enantiomeric excess was determined to be 94% (run 1: 94% ee; run 2: 94% ee) by chiral HPLC analysis (CHIRALPAK IA, 1.0 mL/min, 100% hexanes, λ =254 nm); t_R(major)=7.38 min, t_R(minor)=8.11 min. $[\alpha]_D^{24}$ = –13.9 ° (c 3.36 $CHCl_3$): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 – 7.28 (m, 6H), 7.25 – 7.18 (m, 3H), 6.46 – 6.29 (m, 2H), 3.68 – 3.59 (m, 1H), 1.46 (d, J = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 144.2, 137.5, 134.7, 132.0, 129.0, 128.9, 128.74, 128.71, 127.4, 126.3, 42.1, 21.3. The spectral data for this compound matches that reported in the literature.[6]



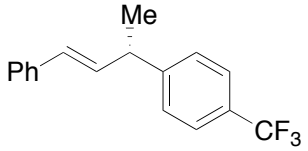
(*S*,*E*)-1-Bromo-4-(4-phenylbut-3-en-2-yl)benzene (8).

Compound **8** was prepared via the General Procedure using pivalate **1a** (prepared in 96% ee). The crude material was purified by silica gel chromatography (100% hexanes) to give compound **7** (run 1: 53.4 mg, 62%; run 2: 54.2 mg, 63%) as a yellow oil. The enantiomeric excess was determined to be 96% (run 1: 96% ee; run 2: 95% ee) by chiral HPLC analysis (CHIRALPAK IA, 1.0 mL/min, 100% hexanes, λ=254 nm); t_R(major)=8.22 min, t_R(minor)=9.44 min. $[\alpha]_D^{24}$ = –5.8° (c 1.38, $CHCl_3$): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 – 7.42 (m, 2H), 7.38 – 7.34 (m, 2H), 7.30 (dd, J = 8.5, 6.7 Hz, 2H), 7.24 – 7.19 (m, 1H), 7.18 – 7.13 (m, 2H), 6.47 – 6.29 (m, 2H), 3.66 – 3.57 (m, 1H), 1.45 (d, J = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 144.7, 137.4, 134.6, 131.7, 129.3,

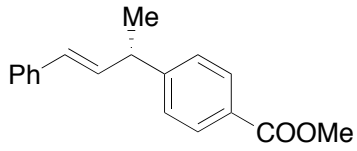
5 Mauleón, P.; Carretero, J. C. *Org. Lett.* **2004**, 6, 3195.

6 Li, M.-B.; Wang, Y.; Tian, S.-K. *Angew. Chem. Int. Ed.* **2012** ,51, 2968.

129.1, 128.7, 127.4, 126.3, 120.1, 42.2, 21.3. The spectral data for this compound matches that reported in the literature.[7]



(*S*,*E*)-1-(4-Phenylbut-3-en-2-yl)-4-(trifluoromethyl)benzene (9). Compound **9** was prepared via the General Procedure using pivalate **1a** (prepared in 96% ee). The crude material was purified by silica gel chromatography (100% hexanes) to give compound **9** (run 1: 64.6 mg, 78%; run 2: 63.8 mg, 77%) as a colorless oil. The enantiomeric excess was determined to be 91% (run 1: 90% ee; run 2: 91% ee) by chiral HPLC analysis (CHIRALCEL OD-H, 0.6 mL/min, 0.1% *i*-PrOH/hexane, λ=254 nm); t_R(major)=11.14 min, t_R(minor)=12.14 min. $[\alpha]_D^{24}$ = –9.4° (c 2.32, $CHCl_3$): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 (d, J = 8.1 Hz, 2H), 7.43 – 7.35 (m, 4H), 7.31 (dd, J = 8.5, 6.7 Hz, 2H), 7.25 – 7.19 (m, 1H), 6.47 – 6.32 (m, 2H), 3.76 – 3.68 (m, 1H), 1.49 (d, J = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 149.8 (q, $J_{C–F}$ = 1.3 Hz), 137.3, 134.2, 129.5, 128.72, 128.69 (q, $J_{C–F}$ = 32.2 Hz), 127.8, 127.5, 125.6 (q, $J_{C–F}$ = 3.8 Hz), 124.5 (q, $J_{C–F}$ = 271.6 Hz), 120.4, 42.6, 21.2. The spectral data for this compound matches that reported in the literature.[8]



(*S*,*E*)-Methyl 4-(4-phenylbut-3-en-2-yl)benzoate (10). Compound **10** was prepared via the General Procedure using pivalate **1a** (prepared in 96% ee). The crude material was purified by silica gel chromatography (5% Et_2O/hexanes) to give compound **10** (run 1: 75.1 mg, 94%; run 2: 73.4mg, 92%) as a colorless oil. The enantiomeric excess was determined to be 89% (run 1: 89% ee; run 2: 89% ee) by chiral HPLC analysis (CHIRALPAK IA, 0.8 mL/min, 1% *i*-PrOH/hexane, λ=254 nm); t_R(major)=21.99 min, t_R(minor)=19.68 min. $[\alpha]_D^{24}$ = –30.2° (c 2.91, $CHCl_3$):

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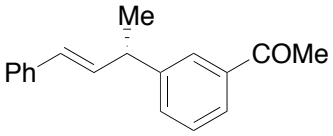
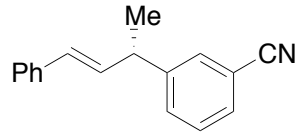
7 Zhou, T.; Peters, B.; Maldonado, M. F.; Govender, T.; Andersson, P. G. *J. Am. Chem. Soc.* **2012**, 134, 13592**.**

8 Zhao, J.; Ye, J.; Zhang, Y. J. *Adv. Synth. Catal.* **2013**, 355, 491.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 – 7.97 (m, 2H), 7.43 – 7.28 (m, 6H), 7.25 – 7.18 (m, 1H), 6.50 – 6.30 (m, 2H), 3.92 (s, 3H), 3.75 – 3.67 (m, 1H), 1.49 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.2, 151.1, 137.4, 134.3, 130.0, 129.3, 128.7, 128.3, 127.5, 127.4, 126.3, 52.2, 42.8, 21.2. The spectral data for this compound matches that reported in the literature.[9]



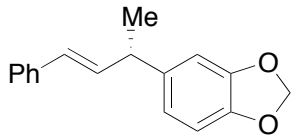
(*S*,*E*)-1-(3-(4-Phenylbut-3-en-2-yl)phenyl)ethanone (11). Compound **11** was prepared via the General Procedure using pivalate **1a** (prepared in 95% ee). The crude material was purified by silica gel chromatography (6% Et_2O/hexanes) to give compound **11** (run 1: 65.4 mg, 87%; run 2: 64.2 mg, 85%) as a colorless oil. The enantiomeric excess was determined to be 91% ee (run 1: 91% ee; run 2: 91% ee) by chiral HPLC analysis (CHIRALPAK IB, 1.0 mL/min, 0.5% *i*-PrOH/hexane, λ=254 nm); t_R(major)=18.72 min, t_R(minor)=21.37 min. $[\alpha]_D^{24}$ = –26.1° (c 2.64, $CHCl_3$): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (t, *J* = 1.8 Hz, 1H), 7.81 (dt, *J* = 7.6, 1.5 Hz, 1H), 7.49 (dt, *J* = 7.7, 1.6 Hz, 1H), 7.42 (t, *J* = 7.6 Hz, 1H), 7.39 – 7.34 (m, 2H), 7.33 – 7.27 (m, 2H), 7.24 – 7.18 (m, 1H), 6.57 – 6.22 (m, 2H), 3.76 – 3.68 (m, 1H), 2.62 (s, 3H), 1.50 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.5, 146.4, 137.5, 137.4, 134.6, 132.4, 129.2, 128.9, 128.7, 127.4, 127.1, 126.7, 126.3, 42.7, 26.9, 21.3; FTIR (NaCl/thin film) 3025, 2965, 2927, 1681, 1598, 1448, 1356, 1264, 966, 750, 693 cm^{-1}; HRMS (EI+) $[M]^+$ calculated for $C_{18}H_{18}O$: 250.1358, found: 250.1376.



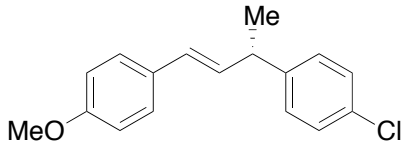
(*S*,*E*)-3-(4-Phenylbut-3-en-2-yl)benzonitrile (12). Compound **12** was prepared via the General Procedure using pivalate **1a** (prepared in 96% ee). The crude material was purified by silica gel chromatography (2% Et_2O/hexanes) to give compound **12** (run 1: 60.2 mg, 85%; run 2: 58.4 mg, 83%) as a colorless oil. The enantiomeric excess was determined to be 93% (run 1: 92% ee; run 2: 93% ee) by chiral HPLC analysis (CHIRALPAK IA, 1.0 mL/min, 2.0% *i*-PrOH/hexane, λ=254 nm);

9 Liao, L.; Sigman, M. S. *J. Am. Chem. Soc.* **2010**, 132, 10209.

t_R(major)=9.53 min, t_R(minor)=10.52 min. $[\alpha]_D^{24}$ = –19.5° (c 2.40, $CHCl_3$): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57 (t, *J* = 1.7 Hz, 1H), 7.52 (dd, *J* = 8.2, 1.4 Hz, 2H), 7.46 – 7.28 (m, 5H), 7.26 – 7.20 (m, 1H), 6.48 – 6.40 (m, 1H), 6.31 (dd, *J* = 15.9, 6.7 Hz, 1H), 3.73 – 3.64 (m, 1H), 1.48 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 147.1, 137.1, 133.6, 132.2, 131.1, 130.2, 129.8, 129.4, 128.7, 127.6, 126.3, 119.2, 112.6, 42.3, 21.1; FTIR (NaCl/thin film) 3025, 2967, 2930, 2228, 1590, 1481, 1446, 960, 798, 751 cm^{-1}; HRMS (EI+) $[M]^+$ calculated for $C_{17}H_{15}N$: 233.1204, found: 233.1221.

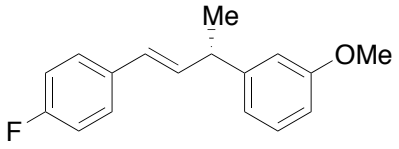


(*S*,*E*)-5-(4-Phenylbut-3-en-2-yl)benzo[*d*][1,3]dioxole (13). Compound **13** was prepared via the General Procedure using pivalate **1a** (prepared in 96% ee). The crude material was purified by silica gel chromatography (2% Et_2O/hexanes) to give compound **13** (run 1: 67.3 mg, 89%; run 2: 65.1 mg, 86%) as a colorless oil. The enantiomeric excess was determined to be 92% (run 1: 92% ee; run 2: 91% ee) by chiral HPLC analysis (CHIRALPAK IA, 0.6 mL/min, 0.1% *i*-PrOH/hexane, λ=254 nm); t_R(major)=13.70 min, t_R(minor)=14.68 min. $[\alpha]_D^{24}$ = –7.7° (c 2.98, $CHCl_3$): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 – 7.34 (m, 2H), 7.29 (dd, *J* = 8.5, 6.8 Hz, 2H), 7.24 – 7.16 (m, 1H), 6.84 – 6.70 (m, 3H), 6.44 – 6.30 (m, 2H), 5.93 (s, 2H), 3.61 – 3.52 (m, 1H), 1.43 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 147.8, 146.0, 139.8, 137.6, 135.4, 128.6, 128.5, 127.2, 126.3, 120.2, 108.3, 108.0, 101.0, 42.4, 21.5; FTIR (NaCl/thin film) 3024, 2964, 2873, 2228, 1486, 1437, 1245, 1040, 938, 693 cm^{-1}; HRMS (EI+) $[M]^+$ calculated for $C_{17}H_{16}O_2$: 252.1150, found: 252.1156.

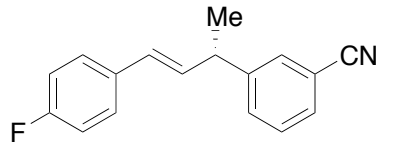


(*S*,*E*)-1-Chloro-4-(4-(4-methoxyphenyl)but-3-en-2-yl)benzene (14). Compound **14** was prepared via the General Procedure using pivalate **1b** (prepared in 82% ee). The crude material was purified by silica gel chromatography (4% Et_2O/hexanes) to give compound **14** (run 1: 68.2 mg, 83%; run 2: 62.5 mg, 82%) as a white solid (mp 58–61 °C). The enantiomeric excess was determined to be 76% ee (run 1: 76% ee; run 2: 75% ee) by chiral HPLC analysis (CHIRALPAK IA, 0.8 mL/min, 0.1%

i-PrOH/hexane, λ=254 nm); t_R(major)=11.45min, t_R(minor)=13.71 min. $[\alpha]_D^{24}$ = –8.3° (c 1.07, $CHCl_3$): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 – 7.27 (m, 4H), 7.23 – 7.18 (m, 2H), 6.88 – 6.81 (m, 2H), 6.40 – 6.30 (m, 1H), 6.19 (dd, *J* = 15.9, 6.7 Hz, 1H), 3.80 (s, 3H), 3.66 – 3.55 (m, 1H), 1.44 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 159.1, 144.5, 132.6, 131.9, 130.3, 128.9, 128.7, 128.4, 127.4, 114.1, 55.5, 42.1, 21.4; FTIR (NaCl/thin film) 3030, 2963, 2931, 2835, 1607, 1510, 1251, 1174, 1035, 816 cm^{-1}; HRMS (EI+) $[M]^+$ calculated for $C_{17}H_{17}ClO$: 272.0968, found: 272.0960.

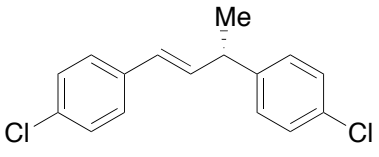


(*S*,*E*)-1-(4-(4-Fluorophenyl)but-3-en-2-yl)-3-methoxybenzene (15). Compound **15** was prepared via the General Procedure using pivalate **1c** (prepared in 98% ee). The crude material was purified by silica gel chromatography (2% Et_2O/hexanes) to give compound **15** (run 1: 69.4 mg, 90%; run 2: 68.2 mg, 89%) as a colorless oil. The enantiomeric excess was determined to be 91% (run 1: 91% ee; run 2: 91% ee) by chiral HPLC analysis (CHIRALPAK IB, 0.6 mL/min, 0.1% *i*-PrOH/hexane, λ=254 nm); t_R(major)=14.19 min, t_R(minor)=17.07 min. $[\alpha]_D^{24}$ = –18.9° (c 2.80, $CHCl_3$): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 – 7.29 (m, 2H), 7.29 – 7.23 (m, 1H), 7.02 – 6.95 (m, 2H), 6.90 – 6.75 (m, 3H), 6.39 (d, *J* = 15.9 Hz, 1H), 6.29 (dd, *J* = 15.9, 6.5 Hz, 1H), 3.82 (s, 3H), 3.65 – 3.56 (m, 1H), 1.47 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.1 (d, $J_{C–F}$ = 245.9 Hz), 159.9, 147.4, 134.93 (d, $J_{C–F}$ = 2.3 Hz), 133.80 (d, $J_{C–F}$ = 3.2 Hz), 129.64, 127.72 (d, $J_{C–F}$ = 7.8 Hz), 127.53, 119.83, 115.47 (d, $J_{C–F}$ = 21.5 Hz), 113.46, 111.34, 55.33, 42.73, 21.30; FTIR (NaCl/thin film) 2964, 2834, 1600, 1508, 1259, 1227, 1157, 779 cm^{-1}; HRMS (EI+) $[M]^+$ calculated for $C_{17}H_{17}FO$: 256.1263, found: 256.1252.



(*S*,*E*)-3-(4-(4-Fluorophenyl)but-3-en-2-yl)benzonitrile (16). Compound **16** was prepared via the General Procedure using pivalate **1c** (prepared in 98% ee). The crude material was purified by silica gel chromatography (5% Et_2O/hexanes) to give compound **16** (run 1: 56.4 mg, 75%; run 2: 55.2 mg, 73%) as a

colorless oil. The enantiomeric excess was determined to be 96% (run 1: 96% ee; run 2: 95% ee) by chiral HPLC analysis (CHIRALPAK IA, 1.0 mL/min, 0.1% *i*-PrOH/hexane, λ=254 nm); t_R(major)=11.64 min, t_R(minor)=12.68 min. $[\alpha]_D^{24}$ = –5.0° (c 2.80, $CHCl_3$): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61 – 7.47 (m, 3H), 7.42 (t, *J* = 7.7 Hz, 1H), 7.36 – 7.29 (m, 2H), 7.03 – 6.96 (m, 2H), 6.38 (dd, *J* = 15.9, 1.3 Hz, 1H), 6.22 (dd, *J* = 15.9, 6.8 Hz, 1H), 3.74 – 3.61 (m, 1H), 1.47 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.3 (d, $J_{C–F}$ = 246.6 Hz), 147.0, 133.4 (d, $J_{C–F}$ = 2.2 Hz), 133.2 (d, $J_{C–F}$ = 3.3 Hz), 132.1, 131.1, 130.2, 129.5, 128.7, 127.8 (d, $J_{C–F}$ = 8.0 Hz), 119.2, 115.6 (d, $J_{C–F}$ = 21.5 Hz), 112.7, 42.3, 21.1; FTIR (NaCl/thin film) 3038, 2968, 2228, 1600, 1507, 1481 1227, 1157, 967, 816, 693 cm^{-1}; HRMS (EI+) $[M]^+$ calculated for $C_{17}H_{14}FN$: 251.1110, found: 251.1115.

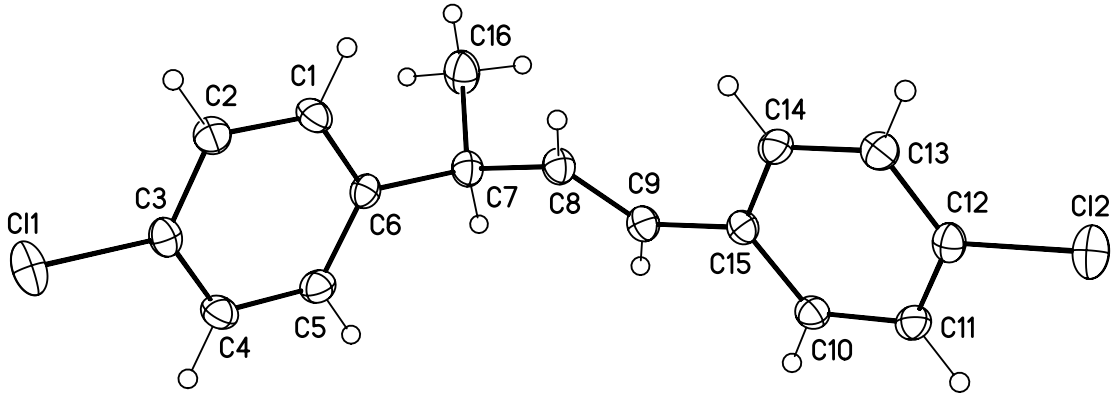


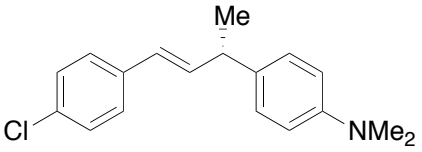
(*S*,*E*)-4,4'-(But-1-ene-1,3-diyl)bis(chlorobenzene) (17). Compound **17** was prepared via the General Procedure using pivalate **1d** (prepared in 96% ee). The crude material was purified by silica gel chromatography (100% hexanes) to give compound **17** (run 1: 72.1 mg, 87%; run 2: 70.8 mg, 85%) as a white solid (mp 69–73 °C). The enantiomeric excess was determined to be 90% (run 1: 90% ee; run 2: 90% ee) by chiral HPLC analysis (CHIRALCEL OD-H, 0.4 mL/min, 100% hexanes, λ=254 nm); t_R(major)=26.72 min, t_R(minor)=24.66 min. $[\alpha]_D^{24}$ = –11.7° (c 0.76, $CHCl_3$): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 – 7.22 (m, 6H), 7.23 – 7.15 (m, 2H), 6.41 – 6.24 (m, 2H), 3.61 (qd, *J* = 7.0, 4.8 Hz, 1H), 1.44 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 143.9, 136.0, 135.5, 132.9, 132.2, 128.83, 128.81, 128.79, 127.9, 127.5, 42.1, 21.2. The spectral data for this compound matches that reported in the literature.[10]

X-ray quality crystals were obtained from an Et_2O/hexanes mixture cooled to –18 °C. The crystal structure demonstrated that the absolute configuration is *S* (Fig S1).

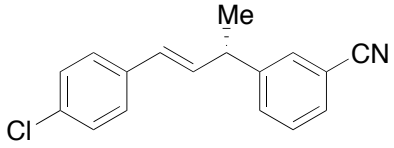
[10] Sanz, R.; Miguel, D.; Martinez, A.; Ålvarez-Gutierrez, J. M.; Rodriguez, F. *Org. Lett.* **2007**, 9, 2027.

Figure S1. Molecular diagram of (*S*)-**17** with ellipsoids at 30% probability. H-atoms depicted with arbitrary radii. (CCDC-973168)

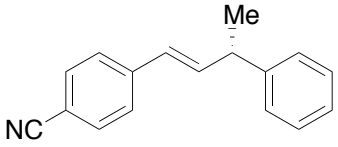




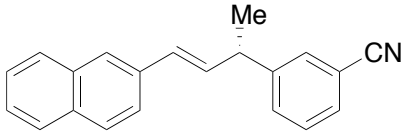
(*S*,*E*)-4-(4-(4-Chlorophenyl)but-3-en-2-yl)-*N*,*N*-dimethylaniline (18). Compound **18** was prepared via the General Procedure using pivalate **1d** (prepared in 96% ee). The crude material was purified by silica gel chromatography (5% Et_2O/hexanes) to give compound **18** (run 1: 71.8 mg, 84%; run 2: 70.2 mg, 82%) as a colorless oil. The enantiomeric excess was determined to be 89% (run 1: 89% ee; run 2: 88% ee) by chiral HPLC analysis (CHIRALPAK IA, 0.8 mL/min, 0.5% *i*-PrOH/hexane, λ=254 nm); t_R(major)=10.18 min, t_R(minor)=8.99 min. $[\alpha]_D^{24} = -14.8°$ (c 0.74, $CHCl_3$): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 – 7.20 (m, 4H), 7.19 – 7.10 (m, 2H), 6.78 – 6.69 (m, 2H), 6.40 – 6.29 (m, 2H), 3.56 (qd, *J* = 7.0, 4.4 Hz, 1H), 2.93 (s, 6H), 1.43 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 149.5, 137.0, 136.4, 133.4, 132.5, 128.7, 128.0, 127.5, 126.8, 113.1, 41.7, 41.0, 21.3; FTIR (NaCl/thin film) 2962, 2926, 2799, 1895, 1646, 1521, 1489 1403, 1090, 1011, 947, 809 cm^{-1}; HRMS (EI+) $[M]^+$ calculated for $C_{18}H_{20}ClN$: 285.1284, found: 285.1260.



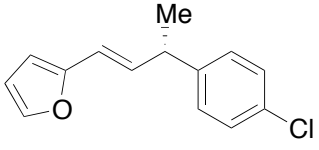
(*S*,*E*)-3-(4-(4-Chlorophenyl)but-3-en-2-yl)benzonitrile (19). Compound **19** was prepared via the General Procedure using pivalate **1d** (prepared in 96% ee). The crude material was purified by silica gel chromatography (5% Et_2O/hexanes) to give compound **19** (run 1: 69.6 mg, 87%; run 2: 68.3mg, 85%) as a colorless oil. The enantiomeric excess was determined to be 90% ee (run 1: 90% ee; run 2: 90% ee) by chiral HPLC analysis (CHIRALCEL OD-H, 0.8 mL/min, 1.0% *i*-PrOH/hexane, λ=254 nm); t_R(major)=15.06 min, t_R(minor)=17.46 min. $[\alpha]_D^{24}$ = –22.1° (c 2.76, $CHCl_3$): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56 – 7.48 (m, 3H), 7.45 – 7.40 (m, 1H), 7.27 (s, 4H), 6.37 (dd, *J* = 15.9, 1.1 Hz, 1H), 6.28 (dd, *J* = 15.9, 6.5 Hz, 1H), 3.71 – 3.63 (m, 1H), 1.47 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 146.9, 135.6, 134.3, 133.2, 132.1, 131.1, 130.3, 129.5, 128.9, 128.7, 127.6, 119.2, 112.7, 42.3, 21.0; FTIR (NaCl/thin film) 3028, 2967, 2871, 2229, 1896, 1581, 1490, 1091, 968 cm^{-1}; HRMS (EI+) $[M]^+$ calculated for $C_{17}H_{14}ClN$: 267.0815, found: 267.0807.



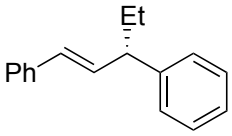
(*S*,*E*)-4-(3-Phenylbut-1-en-1-yl)benzonitrile (20). Compound **20** was prepared via the General Procedure using pivalate **1e** (prepared in 80% ee). The crude material was purified by silica gel chromatography (5% Et_2O/hexanes) to give compound **20** (run 1: 60.2 mg, 86%; run 2: 58.6 mg, 84%) as a colorless oil. The enantiomeric excess was determined to be 76% (run 1: 76% ee; run 2: 76% ee) by chiral HPLC analysis (CHIRALPAK IA, 0.8 mL/min, 1.0% *i*-PrOH/hexane, λ=254 nm); t_R(major)=15.73 min, t_R(minor)=14.13 min. $[\alpha]_D^{24}$ = –51.7° (c 2.28, $CHCl_3$): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57 (d, *J* = 8.4 Hz, 2H), 7.42 (d, *J* = 8.3 Hz, 2H), 7.38 – 7.31 (m, 2H), 7.27 – 7.21 (m, 3H), 6.54 (dd, *J* = 15.9, 6.7 Hz, 1H), 6.41 (dd, *J* = 15.8, 1.3 Hz, 1H), 3.75 – 3.63 (m, 1H), 1.49 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 144.8, 142.2, 139.6, 132.5, 128.8, 127.4, 127.3, 126.8, 126.7, 119.3, 110.3, 42.9, 21.1; FTIR (NaCl/thin film) 3060, 3027, 2967, 2225, 1645, 1581, 1492, 1299, 1012, 969, 819 cm^{-1}; HRMS (EI+) $[M]^+$ calculated for $C_{17}H_{15}N$: 233.1204, found: 233.1230.



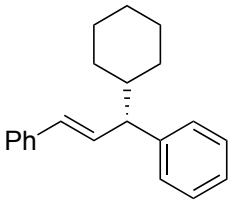
(*S*,*E*)-3-(4-(Naphthalen-2-yl)but-3-en-2-yl)benzonitrile (21). Compound **21** was prepared via the General Procedure using pivalate **1f** (prepared in 96% ee). The crude material was purified by silica gel chromatography (6% Et_2O/hexanes) to give compound **21** (run 1: 66.5 mg, 78%; run 2: 64.2 mg, 76%) as a white solid (mp 90–92 °C). The enantiomeric excess was determined to be 91% (run 1: 90% ee; run 2: 91% ee) by chiral HPLC analysis (CHIRALPAK IB, 1.0 mL/min, 1.0% *i*-PrOH/hexane, λ=254 nm); t_R(major)=18.77 min, t_R(minor)=23.33 min. $[\alpha]_D^{24}$ = –17.8° (c 0.56, $CHCl_3$): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 – 7.75 (m, 3H), 7.71 (s, 1H), 7.63 – 7.51 (m, 4H), 7.50 – 7.40 (m, 3H), 6.59 (d, *J* = 15.9 Hz, 1H), 6.44 (dd, *J* = 15.9, 6.8 Hz, 1H), 3.78 – 3.69 (m, 1H), 1.52 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 147.1, 134.6, 134.1, 133.7, 133.0, 132.2, 131.2, 130.3, 130.0, 129.5, 128.4, 128.1, 127.8, 126.5, 126.2, 126.0, 123.6, 119.2, 112.7, 42.5, 21.2; FTIR (NaCl/thin film) 3057, 2967, 2927, 2228, 1627, 1597, 1481, 1267, 934, 813, 747, 694 cm^{-1}; HRMS (EI+) $[M]^+$ calculated for $C_{21}H_{17}N$: 283.1361, found: 283.1335.



(*S*,*E*)-2-(3-(4-chlorophenyl)but-1-en-1-yl)furan (22). Compound **22** was prepared via the General Procedure using pivalate **1g** (prepared in 97% ee). The crude material was purified by silica gel chromatography (100% hexanes) to give compound **22** (56.8mg mg, 82%) as a colorless oil. The enantiomeric excess was determined to be 86% by chiral HPLC analysis (CHIRALCEL OD-H, 0.8 mL/min, 100% hexanes, λ=254 nm); t_R(major)=10.25 min, t_R(minor)=11.07 min. $[\alpha]_D^{24}$ = –48.5° (c 1.38, $CHCl_3$): ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.31 – 7.30 (m, 1H), 7.29 – 7.26 (m, 2H), 7.19 – 7.16 (m, 2H), 6.34 (dd, *J* = 3.3, 1.8 Hz, 1H), 6.30 (dd, *J* = 15.9, 6.7 Hz, 1H), 6.20 – 6.13 (m, 2H), 3.65 – 3.53 (m, 1H), 1.42 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 153.0, 144.0, 141.7, 133.7, 132.1, 128.9, 128.8, 117.9, 111.4, 107.1, 41.9, 21.2; FTIR (NaCl/thin film) 2966, 2928, 2871, 1897, 1491 1454, 1150, 1094, 1012, 963, 826, 733 cm^{-1}; HRMS (EI+) $[M]^+$ calculated for $C_{14}H_{13}ClO$: 232.0655, found: 232.0667.



(*S*,*E*)-Pent-1-ene-1,3-diyldibenzene (23). Compound **23** was prepared via the General Procedure using pivalate **1h** (prepared in 90% ee). The crude material was purified by silica gel chromatography (100% hexanes) to give compound **23** (run 1: 52.7 mg, 79%; run 2: 54.7 mg, 82%) as a colorless oil. The enantiomeric excess was determined to be 82% (run 1: 82% ee; run 2: 81% ee) by chiral HPLC analysis (CHIRALCEL OD-H, 0.4 mL/min, 0.1% *i*-PrOH/hexane, λ=254 nm); t_R(major)=13.87min, t_R(minor)=14.76 min. $[\alpha]_D^{24}$ = –15.1° (c 1.45, $CHCl_3$): ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.37 – 7.33 (m, 2H), 7.33 – 7.30 (m, 2H), 7.29 – 7.25 (m, 4H), 7.24 – 7.18 (m, 2H), 6.41 (d, *J* = 15.9 Hz, 1H), 6.35 (dd, *J* = 15.9, 7.6 Hz, 1H), 3.32 (q, *J* = 7.5 Hz, 1H), 1.91 – 1.78 (m, 2H), 0.93 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (101 MHz, $(CD_3)_2CO$) δ 145.7, 138.6, 135.1, 130.2, 129.30, 129.27 128.4, 127.8, 126.94, 126.90, 52.0, 29.7, 12.5. The spectral data for this compound matches that reported in the literature.[11]

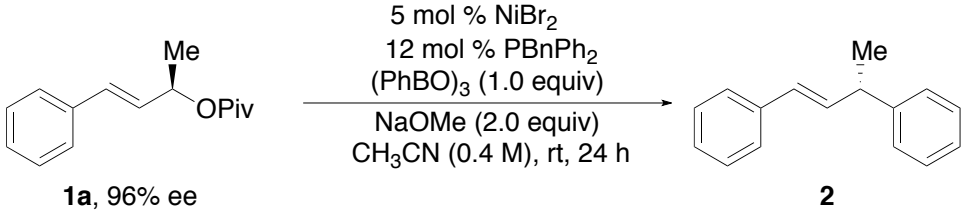


(*R*,*E*)-(3-Cyclohexylpent-1-en-1-yl)benzene (24). Compound **24** was prepared via the General Procedure using pivalate **1i** (prepared in 87% ee). The crude material was purified by silica gel chromatography (0.5% Et_2O/hexanes) to give compound **24** (run 1: 73.8 mg, 89%; run 2: 76.2 mg, 92%) as a colorless oil. The enantiomeric excess was determined to be 62% (run 1: 62% ee; run 2: 61% ee) by chiral HPLC analysis (CHIRALCEL OD-H, 0.8 mL/min, 100% hexanes, λ=254 nm); t_R(major)=8.46min, t_R(minor)=8.04min. $[\alpha]_D^{24}$ = –6.8° (c 1.89, $CHCl_3$): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 – 7.25 (m, 5H), 7.24 – 7.15 (m, 5H), 6.49 – 6.19 (m, 2H), 3.08 (dt, *J* = 8.8, 4.5 Hz, 1H), 2.00 – 1.88 (m, 1H), 1.79 – 1.60 (m, 4H), 1.52 – 1.42 (m, 1H), 1.31 – 1.08 (m, 3H), 1.06 – 0.78 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 144.2, 137.8, 133.4,

11 Sarkar, S. M.; Uozumi, Y.; Yamada, Y. M. A. *Angew. Chem. Int. Ed.* **2011**, 50, 9437.

130.3, 128.59, 128.58, 128.1, 127.1, 126.3, 126.2, 56.9, 42.8, 33.2, 31.7, 31.6, 26.8, 26.5. The spectral data for this compound matches that reported in the literature.[12]

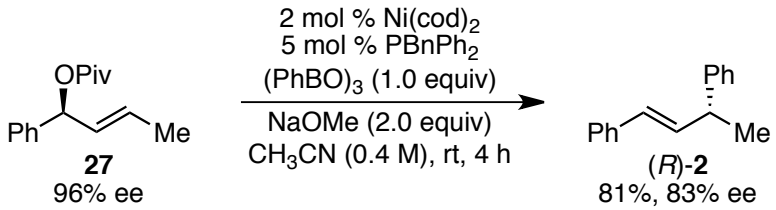
Benchtop Experiment: Preparation of (*S*,*E*)-But-1-ene-1,3-diyldibenzene (2)



1a, 96% ee **2**

This experiment, including weighing of reagents, was conducted entirely on the benchtop without the use of a glovebox. $NiBr_2$ (5.5 mg, 0.025 mmol, 5 mol %), benzyldiphenyl phosphine (16.6 mg, 0.06 mmol, 12 mol %) and NaOMe (54.03 mg, 1.0 mmol, 2.0 equiv) were weighed into a 1-dram vial. Pivalate **1a** (116.2 mg, 0.5 mmol, 1.0 equiv, 96% ee), phenylboroxine (156 mg, 0.5 mmol, 1.0 equiv) and zinc (32.6 mg, 0.5 mmol, 1.0 equiv) were added. Capped with a Teflon-lined cap, the vial was evacuated and backfilled with N_2 three times. Acetonitrile (1.25 mL, 0.4 M) was then added. The mixture was stirred for 24 h at room temperature. The reaction mixture was then diluted with Et_2O (3.0 mL) and filtered through a plug of silica gel, which was rinsed with Et_2O (15 mL). The filtrate was concentrated and purified by silica gel chromatography (100% hexanes) to give compound **2** (54.4 mg, 53%) as a colorless oil. The enantiomeric excess of the product was determined to be 93% by chiral HPLC analysis.

Cross Coupling of Pivalate 27



27
96% ee

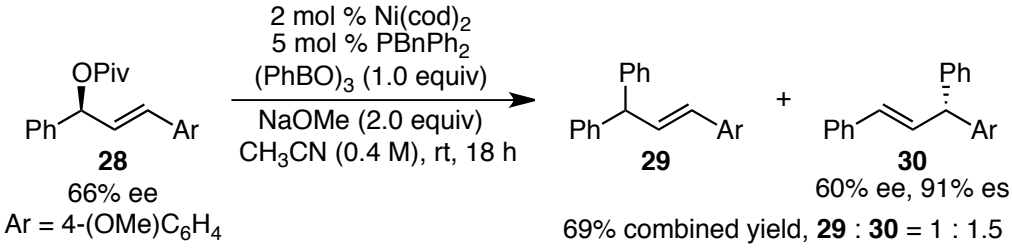
(*R*)-**2**
81%, 83% ee

The arylation of **27** was performed according to the General Procedure, but on a 0.2-mmol scale. In a N_2-atmosphere glovebox, $Ni(cod)_2$ (1.1 mg, 0.04 mmol, 2 mol %),

[12] Miranda, M. A.; Pérez-Prieto, J.; Font-Sanchis, E.; Kónya, K.; Scaiano, J. C. *J. Org. Chem.* **1997**, 62 , 5713.

benzyl diphenyl phosphine (2.8 mg, 0.010 mmol, 5 mol %) and NaOMe (21.7 mg, 0.40 mmol, 2.0 equiv) were weighed into a 1-dram vial. Allylic pivalate **27** (46.5 mg, 96% ee, 0.20 mmol, 1.0 equiv) and phenylboroxine (62.2 mg, 0.20 mmol, 1.0 equiv) were added, followed by acetonitrile (0.5 mL, 0.4 M). The vial was capped with a Teflon-lined cap and removed from the glovebox. The mixture was stirred at room temperature for 4 h. 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 (1% Et_2O/hexanes) to afford (*R*)-**2** (33.8 mg, 81%) as a colorless oil. The absolute configuration was determined by comparison to the HPLC trace and optical rotation of (*S*)**-2.** The enantiomeric excess was determined by chiral HPLC analysis (CHIRALCEL OD-H, 1.0 mL/min, 0.1% *i*-PrOH/hexane λ=254 nm); t_R(major)=6.59 min, t_R(minor)=6.18 min. $[\alpha]_D^{24}$ = +12.9° (c 1.39, $CHCl_3$).

Cross Coupling of Pivalate 28



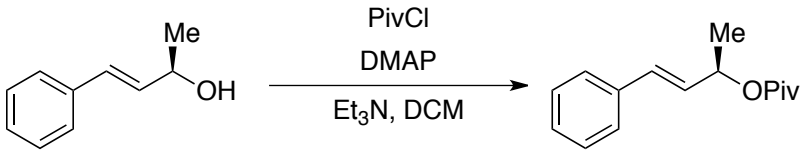
The arylation of **28** was performed according to the General Procedure, but on a 0.2-mmol scale. In a N_2-atmosphere glovebox, $Ni(cod)_2$ (1.1 mg, 0.04 mmol, 2 mol %), benzyl diphenyl phosphine (2.8 mg, 0.010 mmol, 5 mol %) and NaOMe (21.7 mg, 0.40 mmol, 2.0 equiv) were weighed into a 1-dram vial. Allylic pivalate **28** (64.9 mg, 66% ee, 0.20 mmol, 1.0 equiv) and phenylboroxine (62.2 mg, 0.20 mmol, 1.0 equiv) were added, followed by acetonitrile (0.5 mL, 0.4 M). The vial was capped with a Teflon-lined cap and removed from the glovebox. The mixture was stirred at room temperature for 18 h. 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 (5% Et_2O/hexanes) to afford a 1: 1.5 mixture of **29**[13] and **30**[14] (41.7 mg, 69% combined yield) as colorless oil. The enantiomeric excess of

[13] Baba, A.; Kajioka, M.; Nishimoto, Y.; Saito, T.; Yasuda, M. *Chem. Comm.* , **2008** , 47 , 6396.

30 was determined by chiral HPLC analysis (CHIRALCEL OD-H, 0.4 mL/min, 0.1% *i*-PrOH/hexane λ=254 nm); t_R(major)=43.72 min, t_R(minor)=37.96 min. $[\alpha]_D^{24}$ = +22.6° (c 0.66, $CHCl_3$). The spectral data of each compound in the mixture matches that reported in the literature.[13,14]

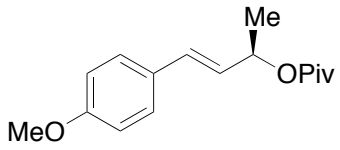
[14] Ji, J.-X.; Li, M.-M.; Wei, W.; Yang, Y.-R.; Yue, H.-L. *Adv. Synth. Catal.* , **2011**, 1 353 , 3139.

General Procedure for the Preparation of Allylic Pivalates: Preparation of (*R*)-4-Phenylbutan-2-yl Pivalate (1a)



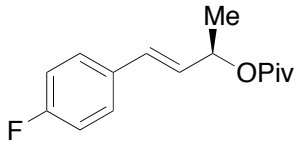
1a

(*R*)-4-Phenyl-3-buten-2-ol (1.80 g, 12.2 mmol, 1.0 equiv, 96% ee) and DMAP (149.5 mg, 1.22 mmol, 0.10 equiv) were dissolved in CH_2Cl_2 (20 mL, 0.61 M). Et_3N (3.5 mL, 24.5 mmol, 2.0 equiv) and pivaloyl chloride (1.80 mL, 14.7 mmol, 1.2 equiv) were then added. The reaction mixture was then stirred for 15 h at room temperature, before H_2O (20 mL) was added. The organic layer was extracted with CH_2Cl_2 (2 x 40 mL). The combined organic layers were washed with aq. KOH (2.0 M, 40 mL), dried ($MgSO_4$), filtered and concentrated. The resulting residue was purified by silica gel chromatography (5% Et_2O/hexanes) to give compound **1a** (2.20 g, 77%) as a white solid (mp 47–50 °C). The enantiomeric excess was determined to be 96% by chiral HPLC analysis (CHIRALPAK IC, 0.8 mL/min, 1.0% *i*-PrOH/hexane, λ=254 nm); t_R(major)=6.80 min, t_R(minor)=8.01 min. $[\alpha]_D^{24}$ = +106.1° (c 2.78, $CHCl_3$): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 – 7.35 (m, 2H), 7.34 – 7.28 (m, 2H), 7.25 – 7.21 (m, 1H), 6.59 (d, *J* = 16.0 Hz, 1H), 6.19 (dd, *J* = 16.0, 6.5 Hz, 1H), 5.54 – 5.46 (m, 1H), 1.39 (d, *J* = 6.5 Hz, 3H), 1.21 (s, 9H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 178.0, 136.6, 131.2, 129.3, 128.73, 128.70, 126.7, 70.7, 38.9, 27.3, 20.5; FTIR (NaCl/thin film) 2977, 2932, 1727, 1479, 1279, 1159, 1041, 965, 747, 692 cm^{-1}; HRMS (EI) $[M]^+$ calculated for $C_{15}H_{20}O_2$: 232.1463, found: 232.1479.

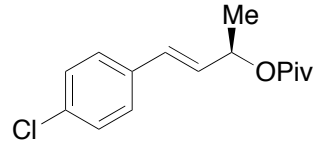


(*R*,*E*)-4-(4-Methoxyphenyl)but-3-en-2-yl pivalate (1b). Prepared according to the General Procedure on a 2.48 mmol scale to give **1b** (500 mg, 77%) as a white solid (mp 76–80 °C). The enantiomeric excess was determined to be 82% by chiral HPLC analysis (CHIRALPAK IC, 0.7 mL/min, 2.0% *i*-PrOH/hexane,

λ=254 nm); t_R(major)=9.23 min, t_R(minor)=10.47 min. $[\alpha]_D^{24}$ = +116.1° (c 1.08, $CHCl_3$): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 – 7.27 (m, 2H), 6.89 – 6.81 (m, 2H), 6.53 (d, *J* = 15.9 Hz, 1H), 6.05 (dd, *J* = 15.9, 6.7 Hz, 1H), 5.52 – 5.43 (m, 1H), 3.81 (s, 3H), 1.37 (d, *J* = 6.5 Hz, 3H), 1.20 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 178.0, 159.5, 130.8, 129.4, 127.9, 127.1, 114.1, 70.9, 55.5, 38.9, 27.3, 20.6; FTIR (NaCl/thin film) 2830, 2821, 1714, 1573, 1380, 1080, 754 cm^{-1}; HRMS (EI+) $[M]^+$ calculated for $C_{16}H_{22}O_3$: 262.1569, found: 262.1569.

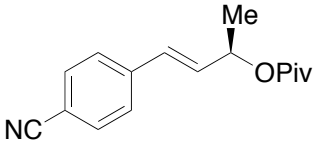


(*R*,*E*)-4-(4-Fluorophenyl)but-3-en-2-yl pivalate (1c). Prepared according to the General Procedure on a 3.32 mmol scale to give **1c** (660 mg, 79%) as a colorless oil. The enantiomeric excess was determined to be 98% by chiral HPLC analysis (CHIRALPAK IA, 1.0 mL/min, 2.0% *i*-PrOH/hexane, λ=254 nm); t_R(major)=4.66 min, t_R(minor)=5.49 min. $[\alpha]_D^{24}$ = +134.2° (c 0.92, $CHCl_3$): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 – 7.30 (m, 2H), 7.05 – 6.96 (m, 2H), 6.55 (d, *J* = 16.0 Hz, 1H), 6.10 (dd, *J* = 16.0, 6.5 Hz, 1H), 5.53 – 5.44 (m, 1H), 1.38 (d, *J* = 6.5 Hz, 3H), 1.24 – 1.19 (m, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 177.9, 162.6 (d, *J* = 247.1 Hz), 132.76 (d, *J* = 3.3 Hz), 130.05, 129.02 (d, *J* = 2.3 Hz), 128.2 (d, *J* = 8.0 Hz), 115.6 (d, *J* = 21.6 Hz), 70.6, 38.9, 27.3, 20.5; FTIR (NaCl/thin film) 2977, 2935, 2873, 1727, 1509, 1231, 1157, 1041 cm^{-1}; HRMS (EI+) $[M]^+$ calculated for $C_{15}H_{19}FO_2$: 250.1369, found: 250.1388.

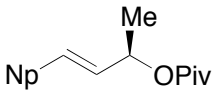


(*R*,*E*)-4-(4-Chlorophenyl)but-3-en-2-yl pivalate (1d). Prepared according to the General Procedure on a 5.40 mmol scale to give **1d** (1.3 g, 92%) as a white solid (mp 72–74 °C). The enantiomeric excess was determined to be 96% by chiral HPLC analysis (CHIRALPAK IA, 0.8 mL/min, 1.0% *i*-PrOH/hexane, λ=254 nm); t_R(major)=6.09 min, t_R(minor)=7.06 min. $[\alpha]_D^{24}$ = +150.8° (c 0.68, $CHCl_3$): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 – 7.24 (m, 4H), 6.53 (dd, *J* = 15.9, 1.2 Hz, 1H), 6.16 (dd, *J* = 16.0, 6.4 Hz, 1H), 5.53 – 5.44 (m, 1H), 1.38 (d, *J* = 6.5 Hz, 3H), 1.21 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 177.9, 135.1, 133.6, 130.0, 129.9, 128.9, 127.9, 70.5, 39.0,

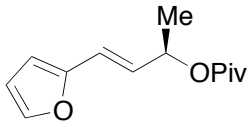
27.3, 20.4; FTIR (NaCl/thin film) 2923, 2840, 1824, 1713, 1569, 1417, 1380, 1340, 1080 cm^{-1}; HRMS (EI+) $[M]^+$ calculated for $C_{15}H_{19}ClO_2$: 266.1074, found: 266.1060.



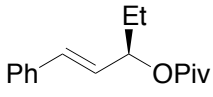
(*R*,*E*)-4-(4-Cyanophenyl)but-3-en-2-yl pivalate (1e). Prepared according to the General Procedure on a 2.49 mmol scale to give **1e** (425 mg, 66%) as a white solid (mp 84–86 °C). The enantiomeric excess was determined to be 80% by chiral HPLC analysis (CHIRALPAK IC, 1.0 mL/min, 2.0% *i*-PrOH/hexane, λ=254 nm); t_R(major)=24.46 min, t_R(minor)=32.11 min. $[\alpha]_D^{24}$ = +115.0° (c 1.00, $CHCl_3$): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 – 7.56 (m, 2H), 7.49 – 7.42 (m, 2H), 6.58 (d, *J* = 16.0 Hz, 1H), 6.31 (dd, *J* = 16.0, 6.1 Hz, 1H), 5.55 – 5.47 (m, 1H), 1.40 (d, *J* = 6.5 Hz, 3H), 1.22 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 177.8, 141.1, 133.3, 132.7, 129.3, 127.2, 119.1, 111.2, 70.1, 39.0, 27.3, 20.3; FTIR (NaCl/thin film) 2978, 2935, 2872, 2227, 1717, 1604, 1480, 1282, 1173, 1039, 968 cm^{-1}; HRMS (EI+) $[M]^+$ calculated for $C_{16}H_{19}NO_2$: 257.1416, found: 257.1408.



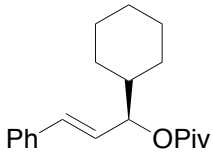
(*R*,*E*)-4-(Naphthalen-2-yl)but-3-en-2-yl pivalate (1f). Prepared according to the General Procedure on a 2.36 mmol scale to give **1f** (515 mg, 77%) as a white solid (mp 103–105 °C). The enantiomeric excess was determined to be 96% by chiral HPLC analysis (CHIRALPAK IA, 1.0 mL/min, 1.0% *i*-PrOH/hexane, λ=254 nm); t_R(major)=6.12 min, t_R(minor)=6.92 min. $[\alpha]_D^{24}$ = +141.9° (c 0.82, $CHCl_3$): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 – 7.71 (m, 4H), 7.59 (dd, *J* = 8.6, 1.8 Hz, 1H), 7.51 – 7.40 (m, 2H), 6.75 (d, *J* = 15.7 Hz, 1H), 6.32 (dd, *J* = 16.0, 6.4 Hz, 1H), 5.60 – 5.52 (m, 1H), 1.43 (d, *J* = 6.5 Hz, 3H), 1.24 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 178.0, 134.1, 133.7, 133.2, 131.2, 129.6, 128.3, 128.1, 127.8, 126.8, 126.4, 126.1, 123.7, 70.7, 39.0, 27.3, 20.5; FTIR (NaCl/thin film) 2974, 1718, 1653, 1368, 1278, 1164, 1035, 972, 743 cm^{-1}; HRMS (EI+) $[M]^+$ calculated for $C_{19}H_{22}O_2$: 282.1620, found: 282.1634.



(*R*,*E*)-4-(Furan-2-yl)but-3-en-2-yl pivalate (1g). Prepared according to the General Procedure on a 1.66 mmol scale to give **1g** (320 mg, 74%) as a colorless oil. The enantiomeric excess was determined to be 97% by chiral HPLC analysis (CHIRALPAK IA, 0.6 mL/min, 0.1% *i*-PrOH/hexane, λ=254 nm); t_R(major)=8.98 min, t_R(minor)=9.95 min. $[\alpha]_D^{24}$ = +124.1° (c 1.25, $CHCl_3$): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.32 (m, 1H), 6.44 – 6.34 (m, 2H), 6.25 (d, *J* = 3.3 Hz, 1H), 6.12 (dd, *J* = 15.9, 6.4 Hz, 1H), 5.50 – 5.42 (m, 1H), 1.36 (d, *J* = 6.4 Hz, 3H), 1.21 (s, 9H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 177.9, 152.3, 142.3, 127.9, 119.5, 111.5, 108.7, 70.3, 39.0, 27.3, 20.4; FTIR (NaCl/thin film) 2976, 2933, 2872, 1727, 1480, 1280, 1161, 1040, 959, 734 cm^{-1}; HRMS (EI+) $[M]^+$ calculated for $C_{13}H_{18}O_3$: 222.1256, found: 222.1262.



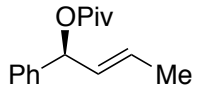
(*R*,*E*)-1-Phenylpent-1-en-3-yl pivalate (1h). Prepared according to the General Procedure on a 1.50 mmol scale to give **1h** (295 mg, 70%) as a pale yellow oil. The enantiomeric excess was determined to be 90% by chiral HPLC analysis (CHIRALCEL OD-H, 0.8 mL/min, 1.0% i-PrOH/hexane, λ=254 nm); t_R(major)=7.37min, t_R(minor)=5.84min. $[\alpha]_D^{24}$ = +87.1° (c 2.41, $CHCl_3$): ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.40 – 7.35 (m, 2H), 7.31 (t, *J* = 7.6 Hz, 2H), 7.27 – 7.21 (m, 1H), 6.59 (d, *J* = 15.9 Hz, 1H), 6.13 (dd, *J* = 16.0, 6.9 Hz, 1H), 5.37 – 5.30 (m, 1H), 1.78 – 1.69 (m, 2H), 1.23 (s, 9H), 0.95 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 178.0, 136.7, 132.1, 128.7, 128.0, 127.9, 126.7, 75.5, 39.10, 27.9, 27.4, 9.7. The spectral data for this compound matches that reported in the literature.[15]



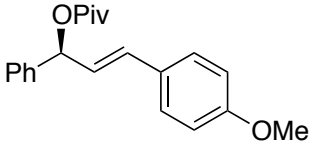
(*R*,*E*)-1-Cyclohexyl-3-phenylallyl pivalate (1i). Prepared according to the General Procedure on a 1.50 mmol scale to give **1i** (295 mg, 70%) as a white solid

15 Phipps, R. J.; McMurray, L.; Ritter, S.; Duong, H. A.; Gaunt, M. J. *J. Am. Chem. Soc.* **2012**, 134, 10773.

(mp 69–71 °C). The enantiomeric excess was determined to be 87% by chiral HPLC analysis (CHIRALCEL OD-H, 0.8 mL/min, 1.0% i-PrOH/hexane, λ=254 nm); t_R(major)=6.89 min, t_R(minor)=5.32 min. $[\alpha]_D^{24}$ = +29.2° (c 0.82, $CHCl_3$): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 – 7.19 (m, 5H), 6.55 (d, *J* = 15.9 Hz, 1H), 6.12 (dd, *J* = 15.9, 7.4 Hz, 1H), 5.23 – 5.14 (m, 1H), 1.85 – 1.59 (m, 6H), 1.22 (s, 10H), 1.22 – 0.99 (m, 4H);^{13}C NMR (101 MHz, $CDCl_3$) δ 177.9, 136.8, 132.8, 128.7, 127.9, 127.0, 126.7, 78.4, 42.3, 39.2, 29.07, 28.6, 27.4, 26.6, 26.2, 26.1; FTIR (NaCl/thin film) 3026, 2929, 2854, 1728, 1479, 1280, 1158, 966, 748 cm^{-1}; HRMS (EI+) $[M]^+$ calculated for $C_{20}H_{28}O_2$: 300.2089, found: 300.2066.



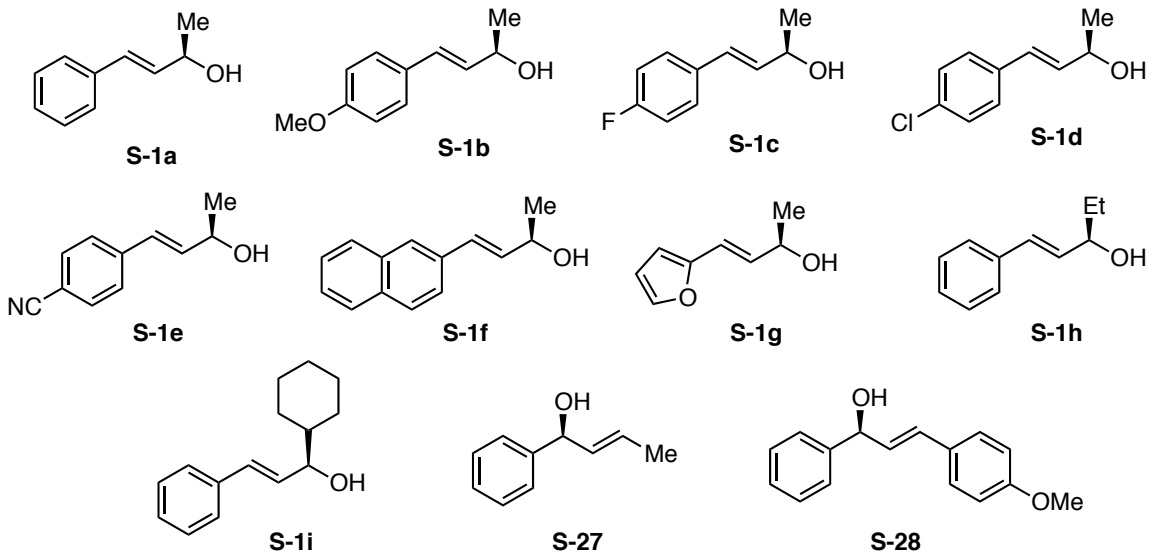
***(S,E)*-1-Phenylbut-2-en-1-yl pivalate (27).** Prepared according to the General Procedure on a 0.6 mmol scale to give **27** (103.5 mg, 78%) as a colorless oil. The enantiomeric excess was assumed to be unchanged from allylic alcohol **S-27** (96% ee). $[\alpha]_D^{24}$ = –10.2° (c 2.15, $CHCl_3$): ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.39 – 7.26 (m, 5H), 6.17 (d, *J* = 6.9 Hz, 1H), 5.80 – 5.70 (m, 1H), 5.67 – 5.60 (m, 1H), 1.71 (d, *J* = 6.6 Hz, 3H), 1.22 (s, 9H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 177.5, 140.3, 130.0, 129.2, 128.6, 127.8, 126.7, 76.0, 39.0, 27.3, 17.9; FTIR (NaCl/thin film) 2972, 1732, 1480, 1455, 1278, 1149, 963 cm^{-1}; HRMS (EI+) [M]+ calculated for $C_{21}H_{24}O_3$: 232.1463, found: 232.1470.



***(S,E)*-3-(4-Methoxyphenyl)-1-phenylallyl pivalate (28).** Compound **28** was prepared via the General Procedure a 1.0 mmol scale. The crude material was purified by silica gel chromatography (5–7.5% Et_2O/hexanes, silica gel pre-saturated with Et_3N) to give **28** (167 mg, 54%) as a colorless oil. The enantiomeric excess was determined to be 66% by chiral HPLC analysis (CHIRALPAK IA, 1.0 mL/min, 5% *i*-PrOH/hexane, λ=210 nm); t_R(major)=7.21 min, t_R(minor)=6.26 min. $[\alpha]_D^{24}$ = +4.1° (c 4.83, $CHCl_3$): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 – 7.29 (m, 7H), 6.84 (d, *J* = 8.8 Hz, 2H), 6.46 (d, *J* = 15.6 Hz, 1H), 6.37 (d, *J* = 7.2 Hz, 1H), 6.17 (dd, *J* = 15.6, 6.8 Hz, 1H), 3.80 (s, 3H), 1.24 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 177.4, 159.5, 139.9, 131.9,

129.0, 128.6, 128.0, 127.9, 126.7, 125.6, 113.9, 76.0, 55.3, 38.9, 27.1; FTIR (NaCl/thin film) 2971, 2933, 1732, 1608, 1512, 1277,1252, 1149, 699 cm^{-1}; HRMS (LIFDI) [M]+ calculated for $C_{21}H_{24}O_3$: 324.1720, found: 324.1725.

Preparation of Enantioenriched Allylic Alcohols

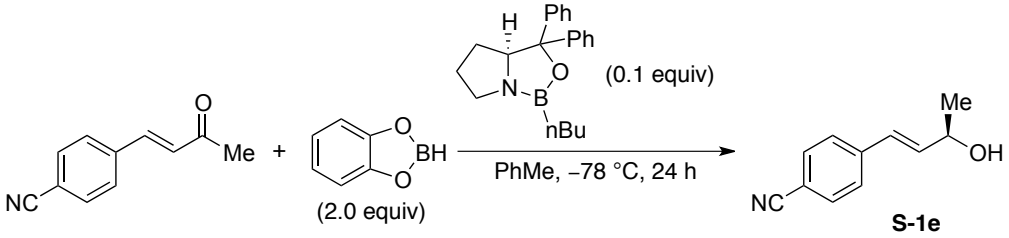


Enantioenriched allylic alcohols (**S-1a** to **S-1i)** were prepared via CBS reduction of the corresponding ketones according to the procedure reported in the literature.[16] These reductions are known to give the stereochemistry shown. A detailed example of the synthesis of **S-1e** obtained by this method is given below. **S-27** and **S-28** were prepared according to a reported procedure.[17]

16 (a) Corey, E. J.; Bakshi, R. K. *Tetrahedron Lett.* **1990**, 31, 611. (b) Corey, E. J.; Helal, C. J. *Tetrahedron Lett*. **1995**, 36, 9153.

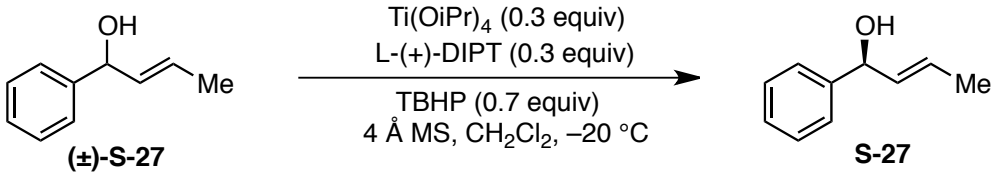
17 Hidaka, I.; Michiko, S.; Kei, T.; Masatoshi, K; Kentaro, Y. *Chem. Eur. J.* **2009**, 15, 4663.

General Procedure for the Preparation of Allylic Alcohols S-1a to S-1i: Preparation of (*R*)-4-(4-Cyanophenyl)-3-buten-2-ol (S-1e)

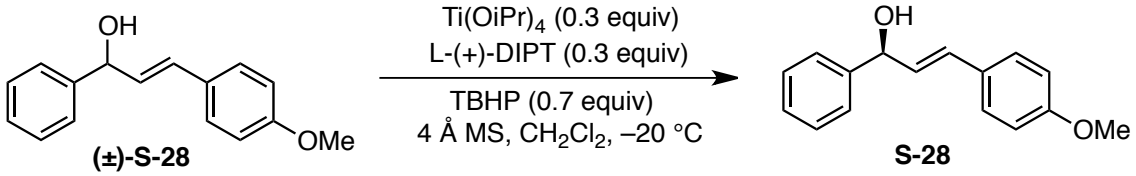


In an oven-dried, 100-mL round-bottomed flask, (*E*)-4-(4-cyanophenyl)-3-buten-2-one (1.08 g, 5.84 mmol, 1.0 equiv) was dissolved in 10 mL PhMe. Under N_2, (*S*)-(–)-2-butyl-CBS-oxazaborolidine (0.58 mL, 0.58 mmol, 1.0 M in PhMe, 0.1 equiv) was added. After stirring at room temperature for 15 min, the mixture was cooled to –78 °C, and catecholborane (1.25 mL, 11.68 mmol, 2.0 equiv) was added slowly. The mixture was stirred at –78 °C for additional 24 h. Sat. $NaHCO_3$ (10 mL) was added, and the crude product was extracted with EtOAc (3 x 20 mL). Tthe combined organic layers were washed with aq. NaOH (1.5 M) until the color of the solution was light yellow, indicating the complete removal of residual catecholborane. The organic layers were then treated with sat. NaCl, dried ($MgSO_4$), filtered and concentrated. The resulting residue was purified by silica gel chromatography (50% Et_2O/hexanes) to give compound **S-1e** (920 mg, 92%) as a yellow oil. The enantiomeric excess was determined to be 80% by chiral HPLC analysis (CHIRALPAK IA, 1.0 mL/min, 6.0% *i*-PrOH/hexane, λ =254 nm); t_R(major)=21.77 min, t_R(minor)=19.56 min. $[\alpha]_D^{24}$ = –9.2° (c 2.39, $CHCl_3$): ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.60 (d, J = 8.2 Hz, 2H), 7.45 (d, J = 8.2 Hz, 2H), 6.60 (d, J = 16.0 Hz, 1H), 6.39 (dd, J = 15.9, 5.8 Hz, 1H), 4.57 – 4.50 (m, 1H), 1.65 (brs, 1H), 1.39 (d, J = 6.4 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 141.5, 137.6, 132.57, 127.6, 127.1, 119.1, 111.0, 68.6, 23.6; FTIR (NaCl/thin film) 3421(brs), 2973, 2226, 1604, 1504, 1142, 971 cm^{-1}; HRMS (EI) $[M]^+$ calculated for $C_{11}H_{11}NO$: 173.0841, found: 173.0867.

Preparation of Allylic Alcohols S-27 and S-28



In an oven-dried, 100-mL round-bottomed flask, L-(+)-DIPT (0.74 mL, 3.54 mmol, 0.3 equiv) was added to a suspension of (*E*)-1-phenyl-2-buten-1-ol (1.76g, 11.8 mmol, 1.0 equiv), 4 Å MS (0.85 g, crushed before use), and CH_2Cl_2 (47 mL). The suspension was then cooled to −20 °C, and $Ti(O^iPr)_4$ (1.06 mL, 3.54 mmol, 0.3 equiv) and TBHP (1.5 mL, 8.25 mmol, 5.5 M in decane, 0.7 equiv) were added. The mixture was stirred for 3 h at –20 °C. $FeSO_4 \cdot 7H_2O$ (6.5 g) and H_2O (40 mL) were then added, followed by tartaric acid (2.2 g), H_2O (20 mL), and aq. HCl (1.0 M, 30 mL) to dissolve the precipitate. The layers were separated. The organic layer was then washed with sat. NaCl, dried (Na_2SO_4), filtered, and concentrated. The resulting residue was purified by silica gel chromatography (15% Et_2O/hexanes) to give compound **S-27** (240 mg, 27%) as colorless oil. The enantiomeric excess was determined to be 96% by chiral GC analysis (Agilent 19091C-133/HP-225, 50% cyanopropyl, 50% phenylmethyl polysiloxane capillary column, initial temperature of 40 °C, ramp 1.0 °C/min to 180 °C, hold for 10 min); t_R(major)=105.67 min, t_R(minor)=106.02 min. $[\alpha]_D^{24}$ = +16.2° (c 3.29, $CHCl_3$). The absolute configuration was determined to be *S* by comparison of the optical rotation to that reported in the literature.[18]

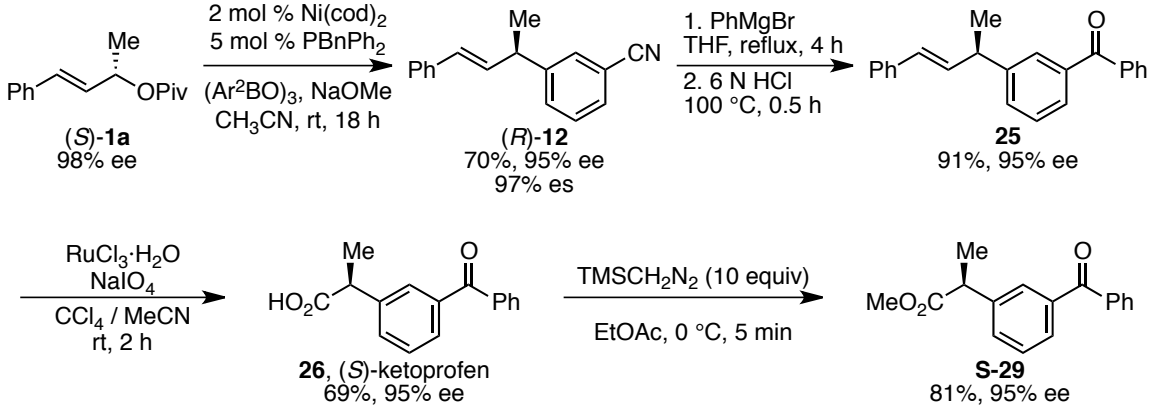


S-28 was prepared on a 8.0-mmol scale from (±)-**S-28** according to the procedure described for **S-27** above. **S-28** (662 mg, 70%) was obtained as a sticky yellow oil from the crude residue by silica gel chromatography (33% Et_2O/hexanes). The enantiomeric

[18] Hayashi, T.; Yamamoto, A.; Hagishita, T. *J. Org. Chem.* **1986**, 51, 723.

excess was determined to be 67% by chiral HPLC analysis (CHIRALPAK IA, 1.0 mL/min, 8.0% *i*-PrOH/hexane, λ =254 nm); t_R(major)=20.38 min, t_R(minor)=17.82 min. [mi$_D^{24}$ = −5.1° (c 4.26, $CHCl_3$). The spectral data for this compound matches that reported in the literature.[19]

Preparation of *(S)*-Ketoprofen



Compound (*R*)-**12** was prepared in 95% ee via the General Procedure using pivalate (*S*)-**1a** (prepared in 98% ee), as described above.

The conversion of (*R*)-**12** to **25** followed a procedure adapted from the literature.[20] In a 25-mL, three-neck round-bottomed flask equipped with a reflux condenser, **12** (180 mg, 0.77 mmol, 95% ee, 1.0 equiv) and THF (1.6 mL) were combined. PhMgBr (1.0 M in THF, 3.9 mL, 3.9 mmol, 5.0 equiv) was added slowly at room temperature. Upon completion of addition, the mixture was refluxed for 4 h. After cooling to room temperature, HCl (6 N, 2 mL) was added. The mixture was heated at 100 °C for 30 min. After cooling to room temperature, H_2O (20 mL) was added. The crude product was extracted with Et_2O (3 x 10 mL), dried ($MgSO_4$), filtered, and concentrated. The resulting material was purified by silica gel chromatography (5% Et_2O/hexanes) to give **25** (219 mg, 91%) as a light yellow oil. The enantiomeric excess was determined to be 95% by chiral HPLC analysis (CHIRALPAK IA, 1.0 mL/min, 1.0% *i*-PrOH/hexane, λ=220 nm); t_R(major)=13.43 min, t_R(minor)=14.41 min. [α]$_D^{24}$ = +35.2° (c 1.7, $CHCl_3$):

[19] Xu, W.; Zhou, Y.; Wang, R.; Wu, G.; Chen, P. *Org. Biomol. Chem.* **2012**, 10, 367.

[20] Riva, E.;Gagliardi, M.; Martinelli, M.; Passarella, D.; Vigo, D.; Rencurosi, A. *Tetrahedron* **2010**, 66, 3242.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.84 – 7.79 (m, 2H), 7.75 (s, 1H), 7.64 – 7.61 (m, 1H), 7.60 – 7.56 (m, 1H), 7.53 – 7.45 (m, 3H), 7.42 (t, *J* = 7.6 Hz, 1H), 7.38 – 7.34 (m, 2H), 7.32 – 7.27 (m, 2H), 7.23 – 7.18 (m, 1H), 6.48 – 6.34 (m, 2H), 3.76 –3.69 (m, 1H), 1.50 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 197.0, 146.2, 138.0, 137.8, 137.5, 134.7, 132.5, 131.6, 130.2, 129.2, 129.0, 128.7, 128.5, 128.39, 128.38, 127.4, 126.3, 42.7, 21.3; FTIR (NaCl/thin film) 3024, 2965, 1658, 1597, 1493, 1447, 1317,1178, 966 cm^{-1}; HRMS (EI) $[M]^+$ calculated for $C_{23}H_{20}O$: 312.1514, found: 312.1534.

The conversion of **25** to **26** followed a procedure adapted from the literature.[21] A suspension of $RuCl_3 \cdot H_2O$ (7.3 mg, 0.035 mmol, 0.05 equiv) in H_2O (7.3 mL) was added to a solution of **25** (219 mg, 0.7 mmol, 1.0 equiv), $NaIO_4$ (749 mg, 3.5 mmol, 5.0 equiv), and CCl_4/MeCN (v/v = 1/1, 9.4 mL) at 0 °C. The mixture was then stirred at room temperature for 2 h. Et_2O (30 mL) was added, and the crude product was then extracted with sat. $NaHCO_3$ (5 x 15 mL). The combined aqueous layers were then cooled to 0 °C, and HCl (6 N) was carefully added until the pH ≤ 1. The crude product was then extracted from aqueous layer with CH_2Cl_2 (5 x 15 mL). The combined organic layers were dried (Na_2SO_4), filtered, and concentrated. The crude material was purified by silica gel chromatography (0–3% MeOH/CH_2Cl_2) to give (*S*)-ketoprofen (**26**) as an off-white solid (124 mg, 69%). The spectral data of **26** matches that reported in the literature.[22]

The enantiomeric excess of **26** was determined to be 95% based on the corresponding methyl ester **S-29**, which was prepared via a reported procedure on a 0.17 mmol scale.[22a] The spectral data of **S-29** matches that reported in the literature.[22] The enantiomeric excess of **S-29** was determined to be 95% by chiral HPLC analysis (CHIRALPAK IB, 0.8 mL/min, 0.5% EtOH/hexane, λ=254 nm); t_R(major)=39.65 min, t_R(minor)=37.39 min. $[\alpha]_D^{24}$ = +43.5° (c 0.80, $CHCl_3$). The absolute configuration of **S-29** was determined to be *S* by comparing the optical rotation with reported data.[23]

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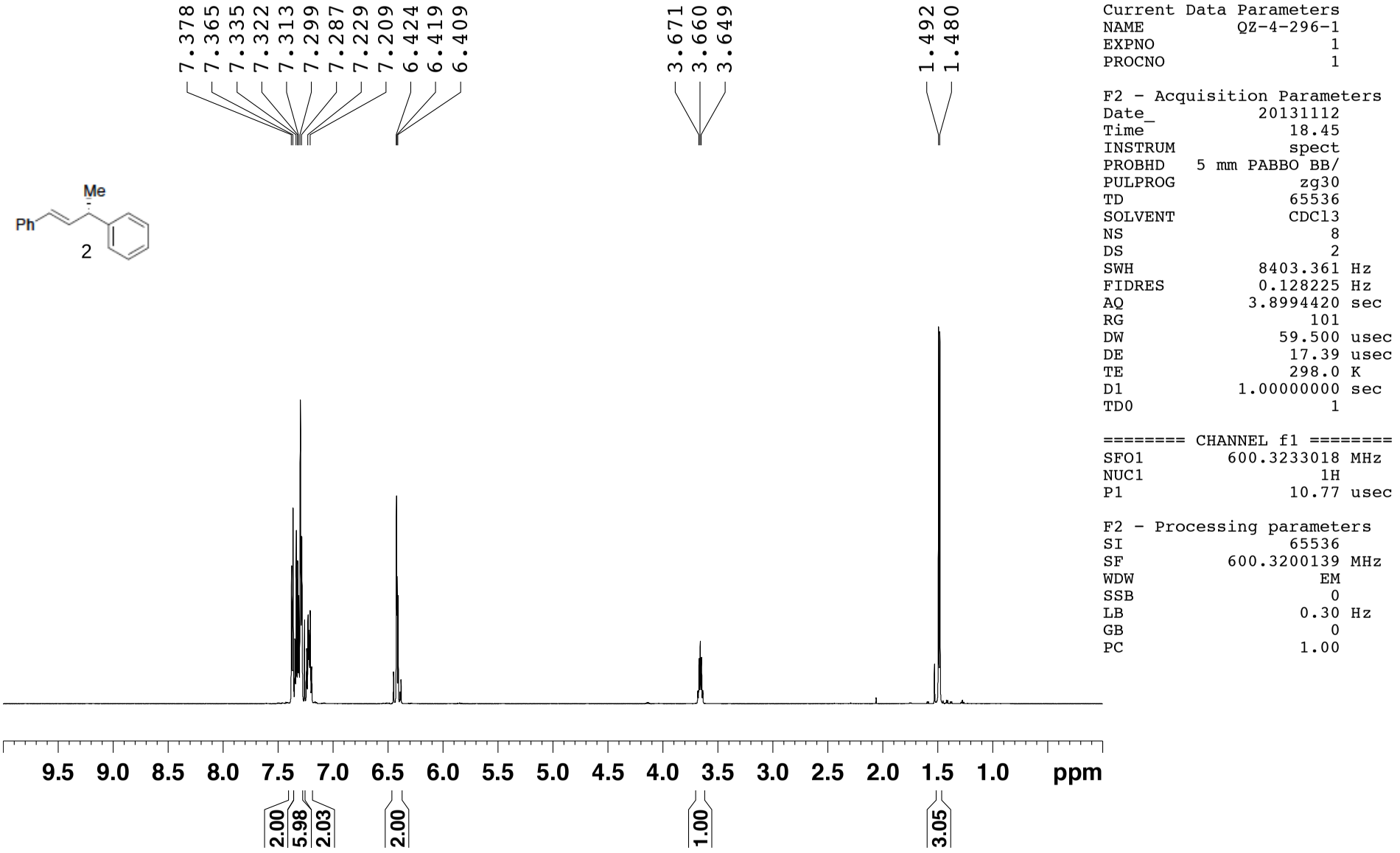
[21] Norinder, J.; Bogár, K.; Kanupp, L.; Bäckvall, J.-E. *Org. Lett.* **2007**, 9, 5095.

[22] (a)Allen, A. E.; MacMillan, D. W. C. *J. Am. Chem. Soc.*, **2011**, 133, 4260; (b) Shiina, I.; Nakata, K.; Onda, Y.-S. *Eur. J. Org. Chem.* **2008**, 5887; (c) Fadel, A. *Synlett* **1992**, 48.

[23] Neumann, H.; Brennführer, A.; Beller, M. *Adv. Synth. Catal.* **2008**, 350, 2437.

compound 2

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TE                298.0 K
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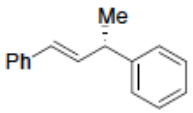
compound 2



2

S31

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PCPD2            90.00 usec
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PL13W        0.08120718 W
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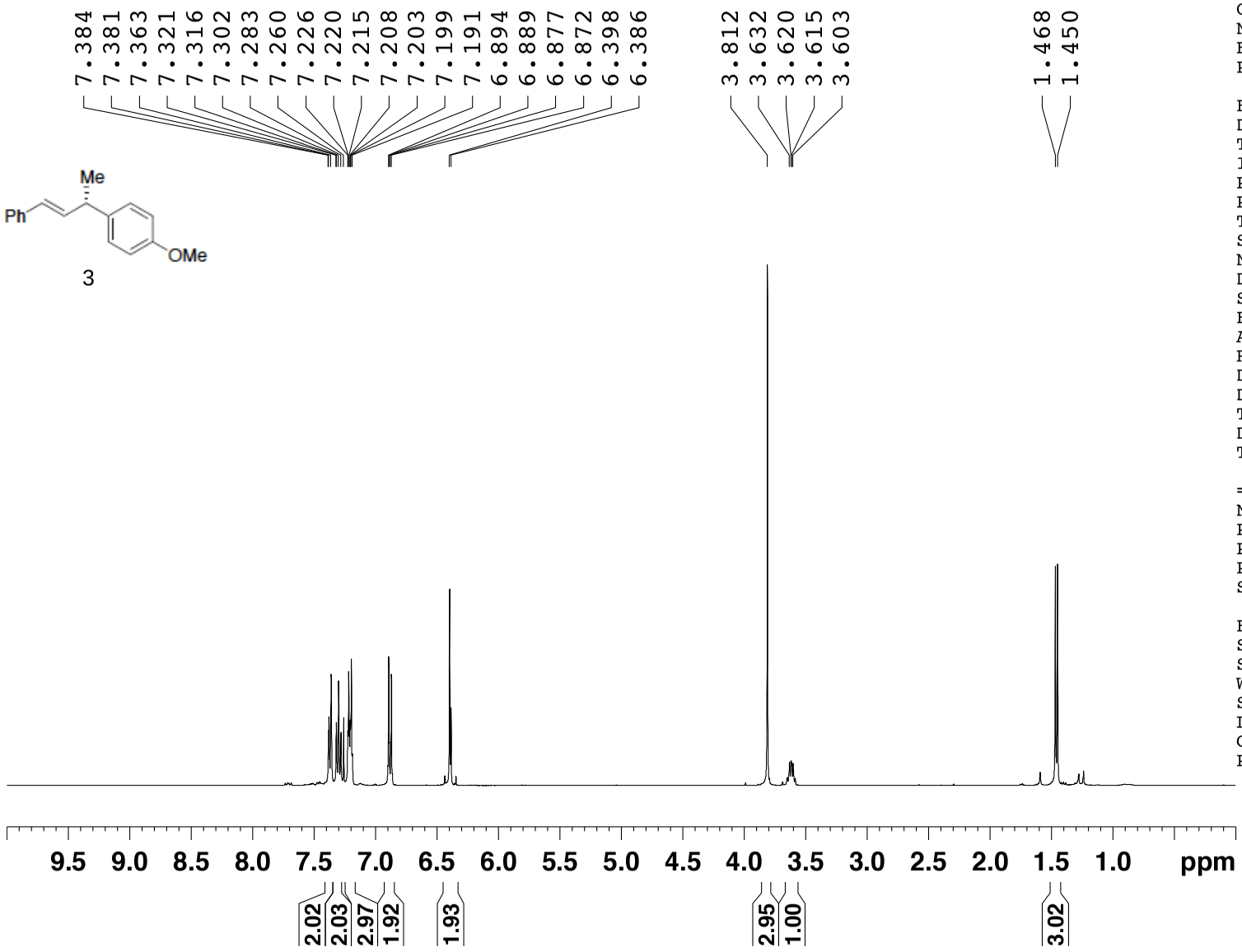
compound 3

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RG                           10.1
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TD0                           1

======== CHANNEL f1 ========
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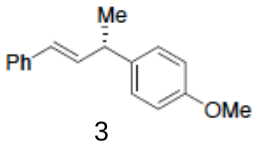
compound 3



3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DW               20.850 usec
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TE               298.2 K
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======== CHANNEL f1 ========
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F2 - Processing parameters
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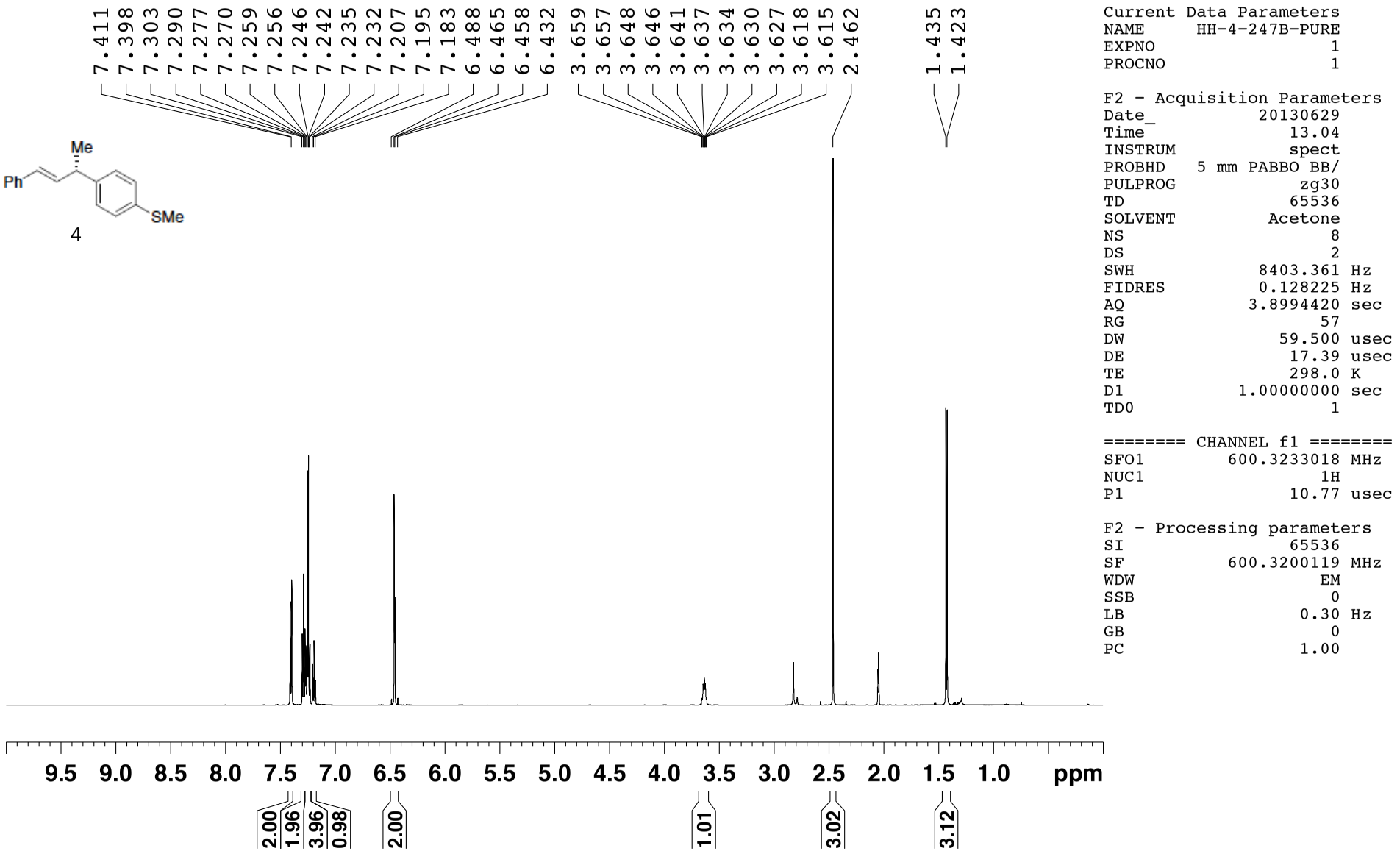
compound 4

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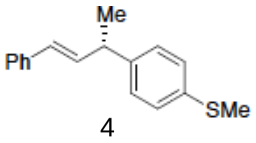
compound 4



142.76
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135.05
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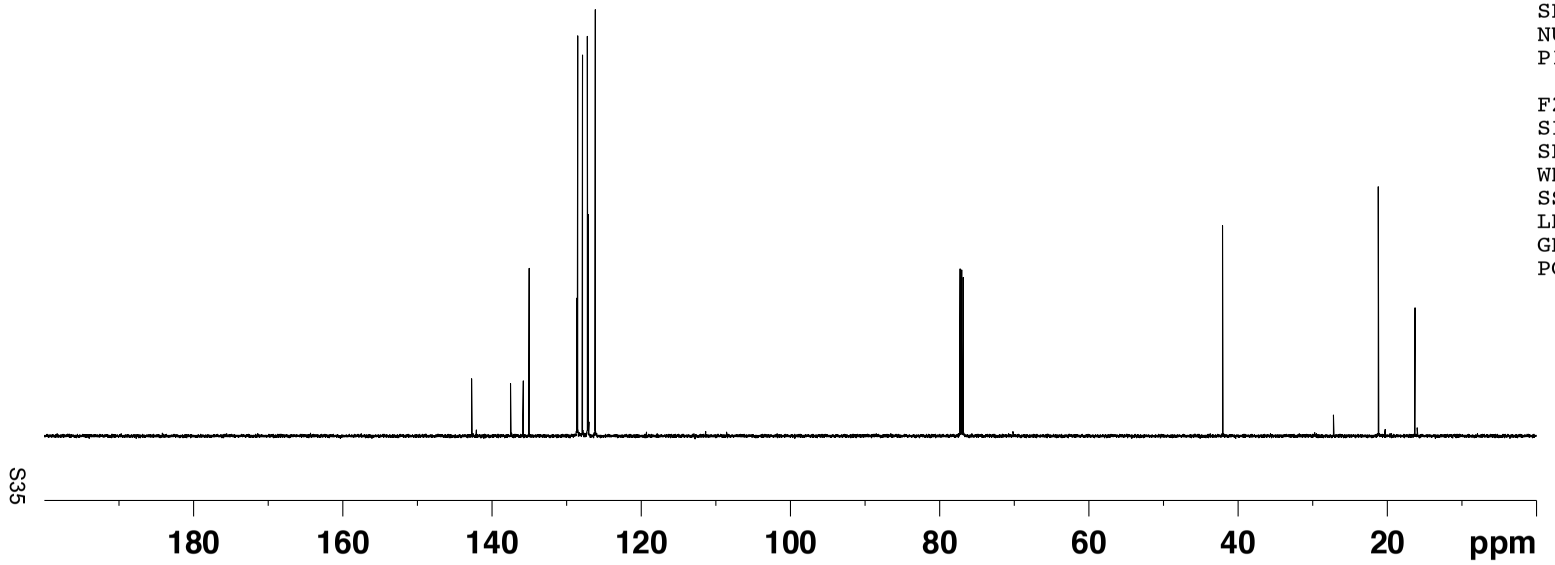
128.54
127.90
127.23
127.13
126.18

42.07

21.18
16.30

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PROCNO                  1

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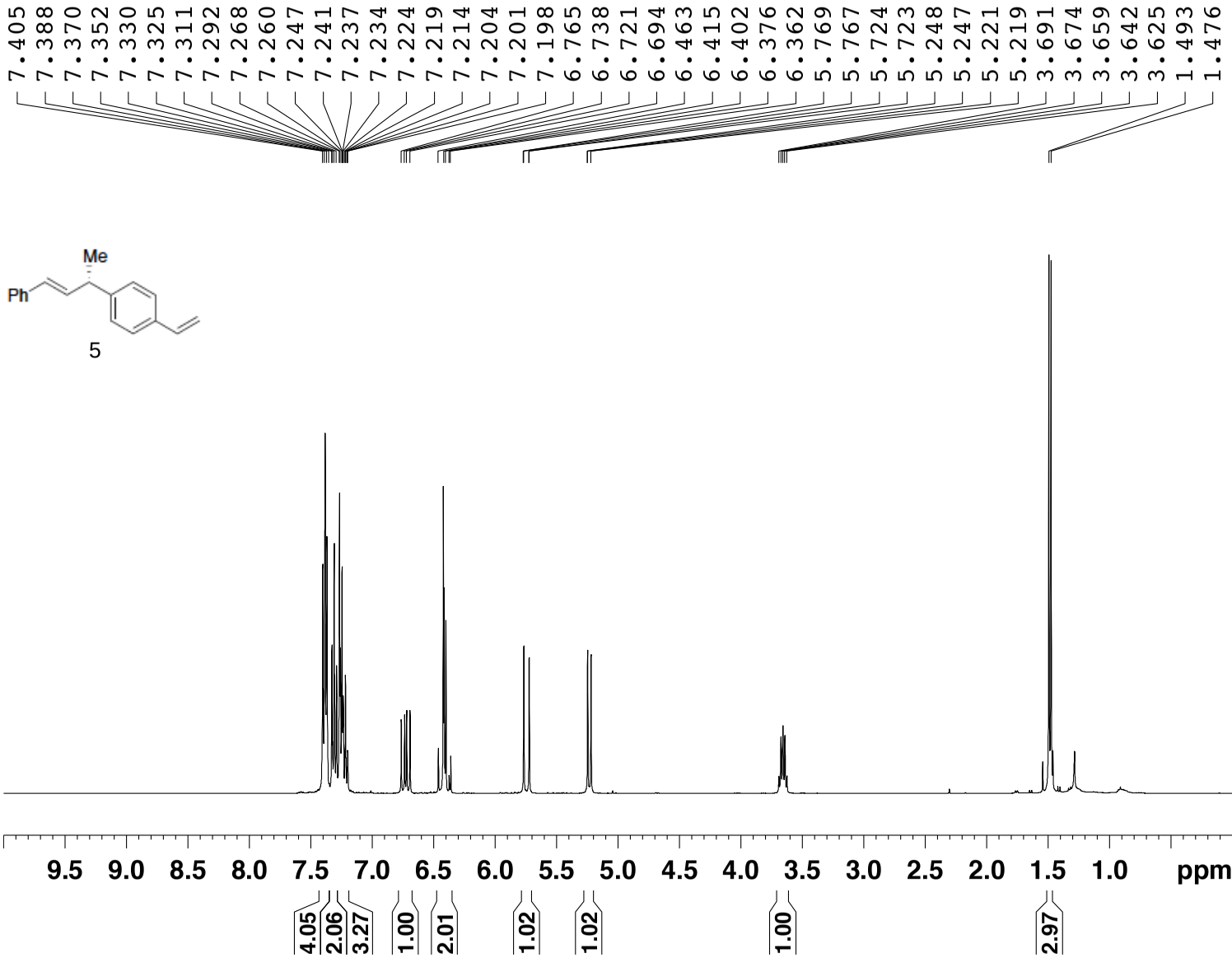
PULPROG           zgpg55
TD                 65536
SOLVENT            CDCl3
NS                   256
DS                     4
SWH            34722.223 Hz
FIDRES          0.529819 Hz
AQ             0.9437684 sec
RG                  2050
DW                14.400 usec
DE                 19.34 usec
TE                 298.0 K
D1            1.10000002 sec
D11           0.03000000 sec
TD0                    1

======== 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                     0
PC                  1.40
```

compound 5



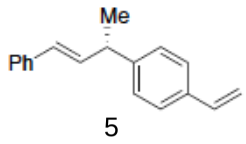

```
Current Data Parameters
NAME                  QZ-3-240a
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20130415
Time                      15.55
INSTRUM                   spect
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                    1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                         1H
P1                        15.00 usec
PL1                        4.90 dB
PL1W                  3.30822015 W
SFO1                400.1324710 MHz

F2 - Processing parameters
SI                        32768
SF                  400.1300096 MHz
WDW                          EM
SSB                           0
LB                         0.30 Hz
GB                            0
PC                         1.00
```

compound 5



5


```
Current Data Parameters
NAME          QZ-3-240a
EXPNO                 2
PROCNO                1

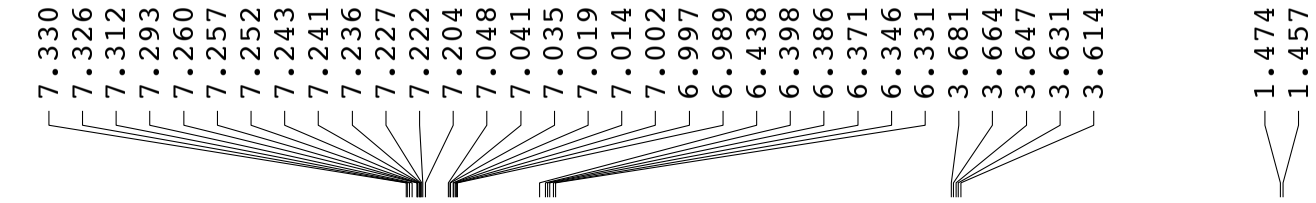
F2 - Acquisition Parameters
Date_          20130415
Time              22.07
INSTRUM           spect
PROBHD   5 mm CPQNP 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ            1.3664756 sec
RG                  512
DW               20.850 usec
DE               18.00 usec
TE               298.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

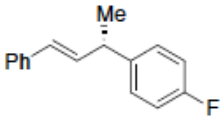
======== CHANNEL f1 ========
NUC1                13C
P1                9.25 usec
PL1               0.55 dB
PL1W       35.18820572 W
SFO1       100.6228298 MHz

======== 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

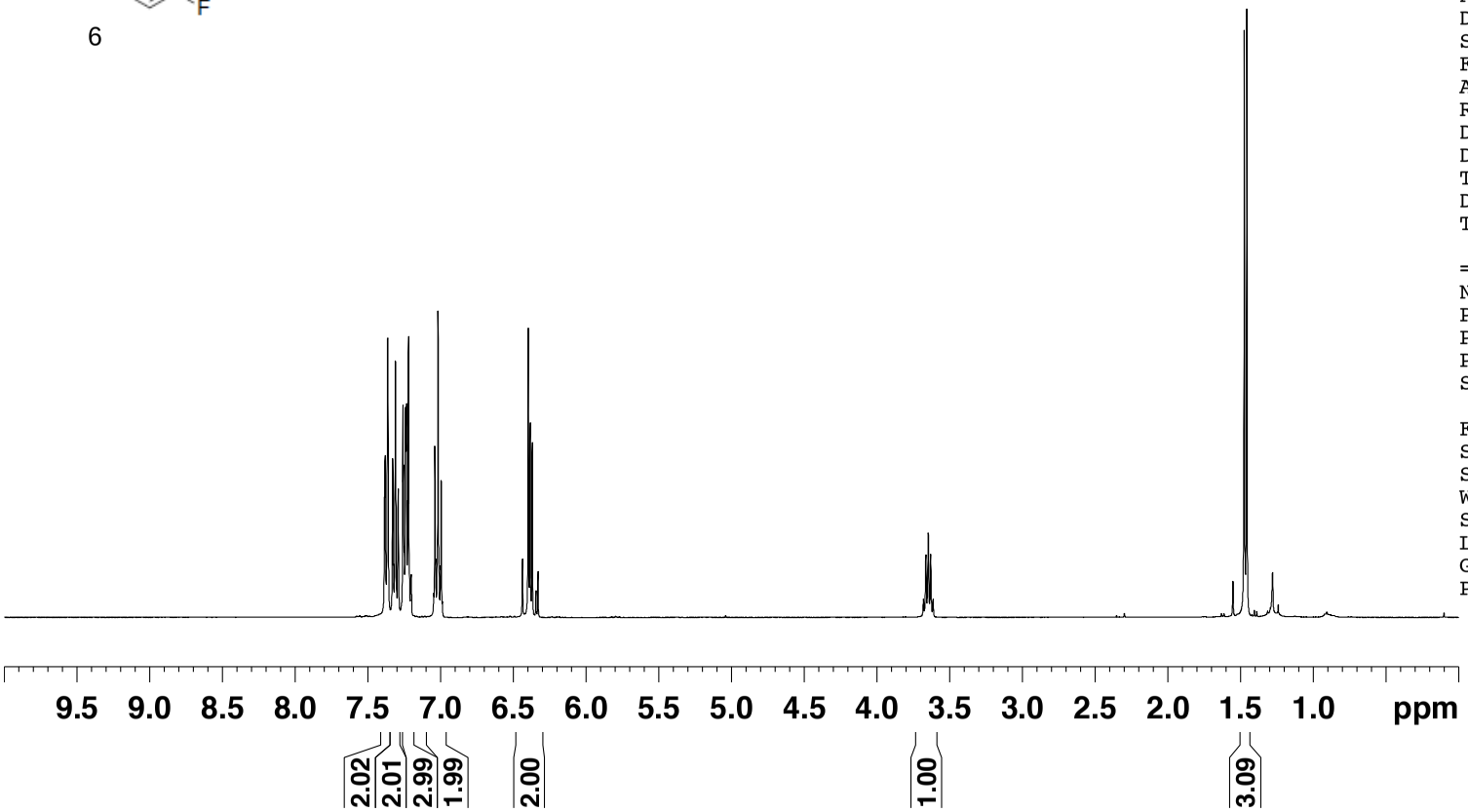
F2 - Processing parameters
SI                32768
SF         100.6127561 MHz
WDW                EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

compound 6





6



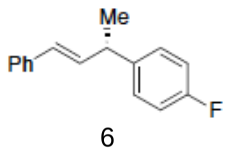

```
Current Data Parameters
NAME            HH-4-242
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130413
Time                5.33
INSTRUM            spect
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                298.1 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 15.00 usec
PL1                 4.90 dB
PL1W          3.30822015 W
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF          400.1300092 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

compound 6



6


```
Current Data Parameters
NAME            HH-4-242
EXPNO                  2
PROCNO                 1

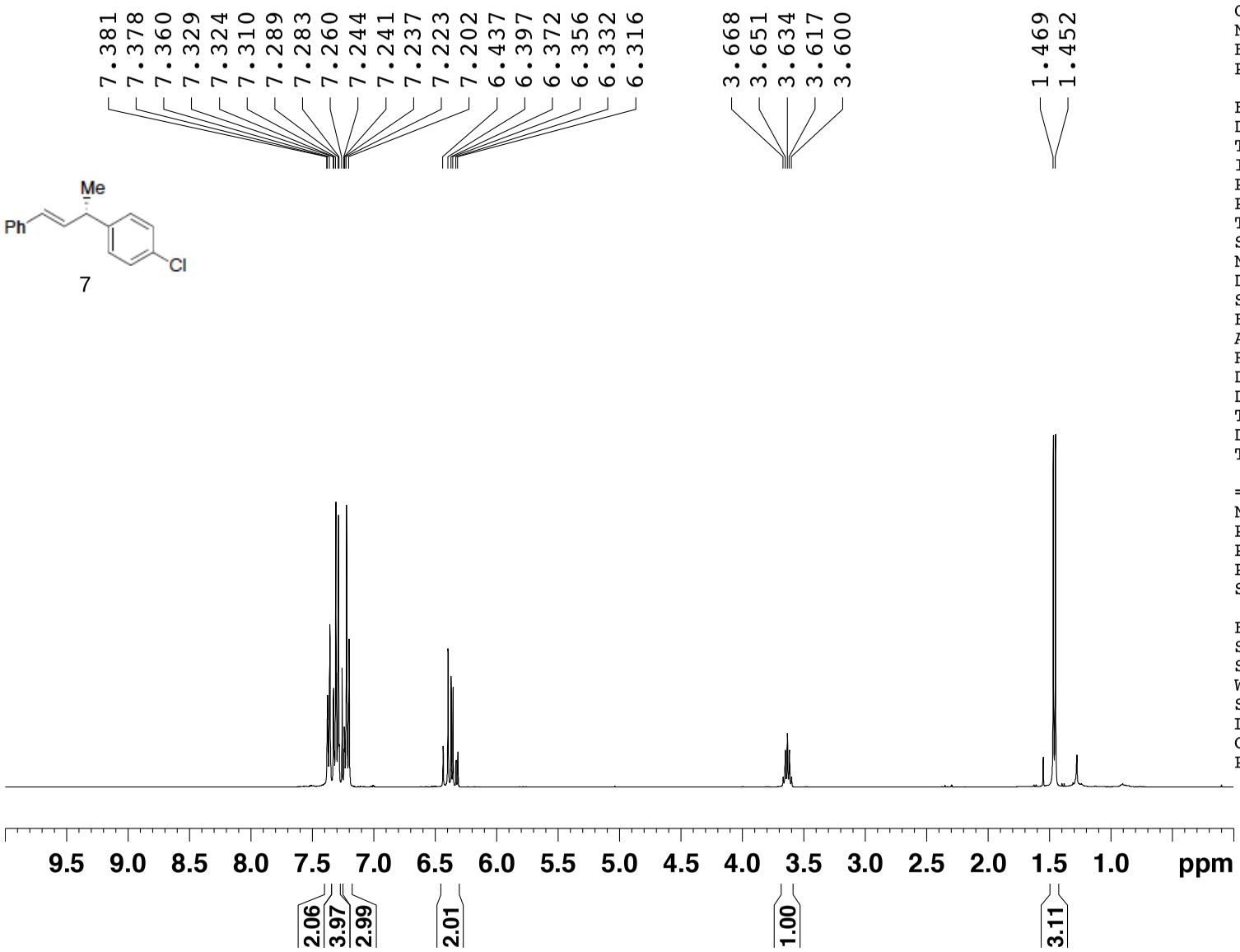
F2 - Acquisition Parameters
Date_           20130413
Time                6.34
INSTRUM            spect
PROBHD   5 mm CPQNP 1H/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ             1.3664756 sec
RG                   512
DW                20.850 usec
DE                18.00 usec
TE                298.2 K
D1            2.00000000 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
SF          100.6127555 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

compound 7



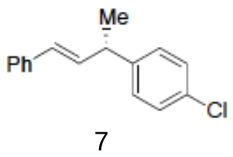

```
Current Data Parameters
NAME                  HH-4-243
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20130413
Time                      6.40
INSTRUM                  spect
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                      298.1 K
D1                 1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                      15.00 usec
PL1                      4.90 dB
PL1W               3.30822015 W
SFO1             400.1324710 MHz

F2 - Processing parameters
SI                      32768
SF               400.1300097 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00
```

compound 7



7

S41

Current Data Parameters
NAME HH-4-243
EXPNO 2
PROCNO 1

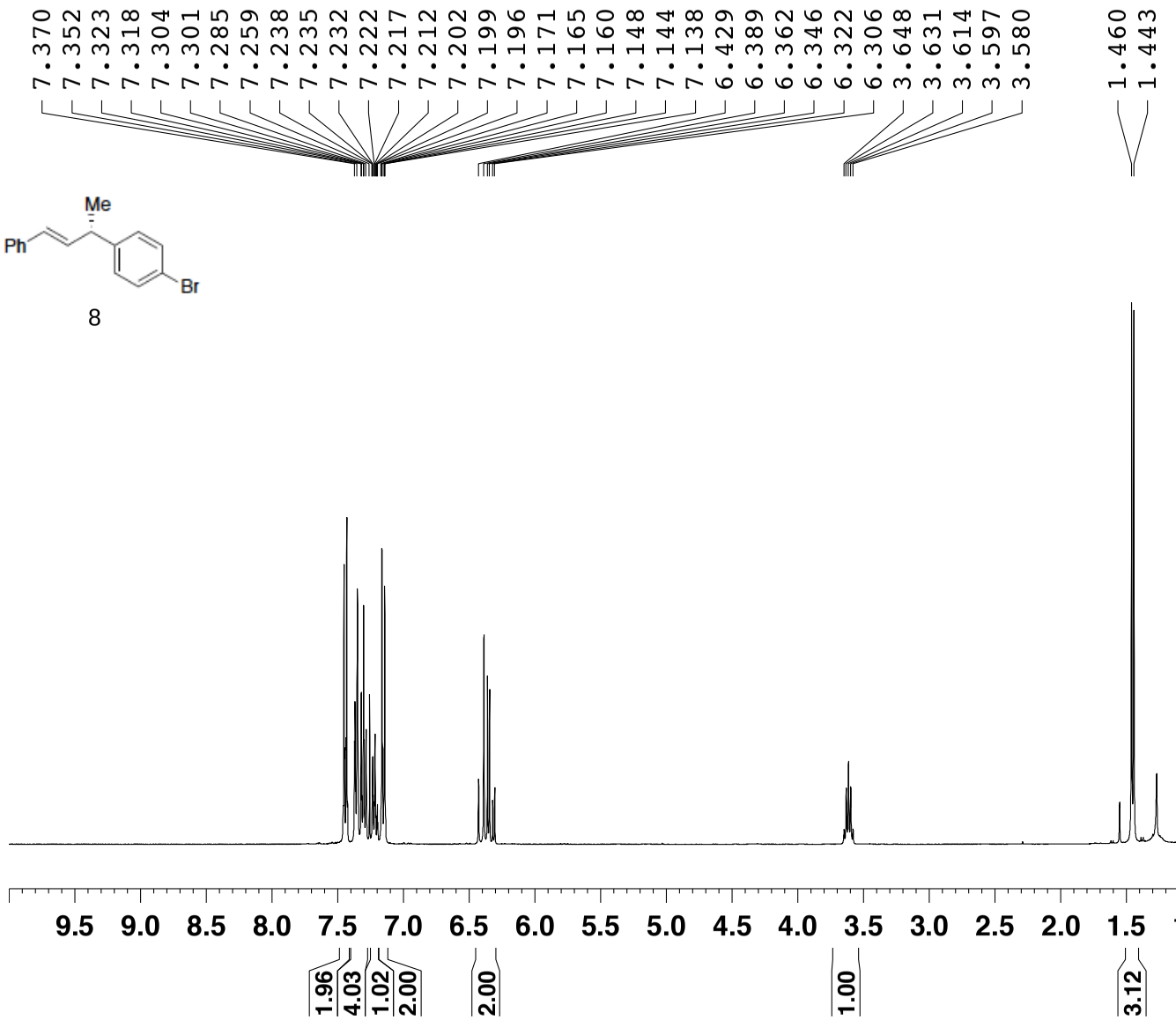
F2 - Acquisition Parameters
Date_ 20130413
Time 7.40
INSTRUM spect
PROBHD 5 mm CPQNP 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 512
DW 20.850 usec
DE 18.00 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== 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
SF 100.6127555 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

compound 8



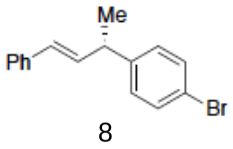

```
Current Data Parameters
NAME         QZ-3-240(1)
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130417
Time               17.29
INSTRUM            spect
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                     9
DW                60.400 usec
DE                 6.00 usec
TE                298.2 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 15.00 usec
PL1                 4.90 dB
PL1W          3.30822015 W
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF           400.1300099 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

compound 8



8


```
Current Data Parameters
NAME         QZ-3-240(1)
EXPNO                  2
PROCNO                 1

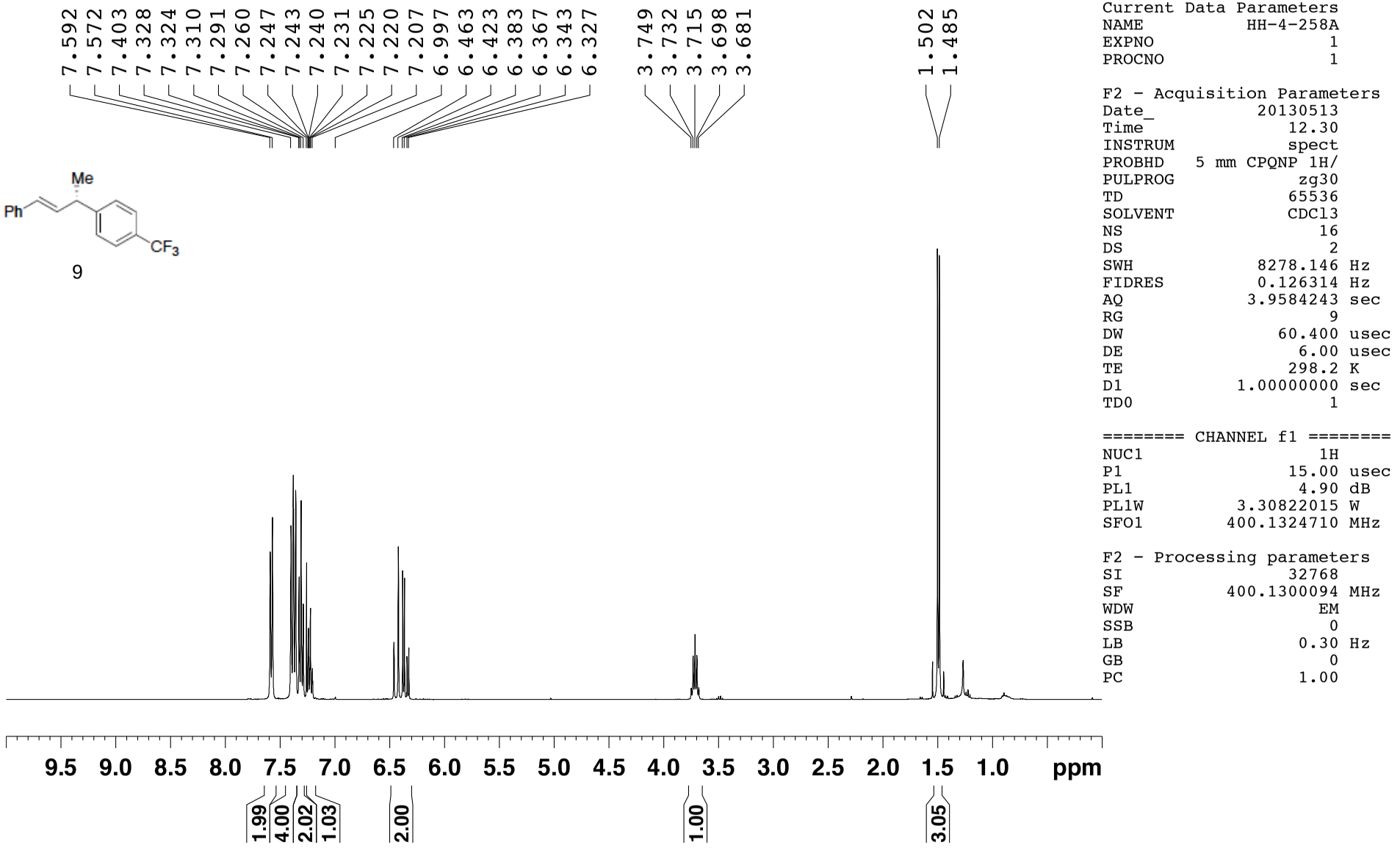
F2 - Acquisition Parameters
Date_           20130417
Time              23.11
INSTRUM           spect
PROBHD   5 mm CPQNP 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ            1.3664756 sec
RG                  512
DW               20.850 usec
DE               18.00 usec
TE               298.1 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
SF         100.6127551 MHz
WDW                EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

compound 9



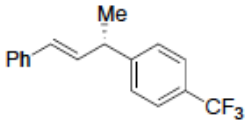

```
Current Data Parameters
NAME          HH-4-258A
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20130513
Time              12.30
INSTRUM           spect
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                    9
DW               60.400 usec
DE                6.00 usec
TE                298.2 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                15.00 usec
PL1                4.90 dB
PL1W         3.30822015 W
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                32768
SF          400.1300094 MHz
WDW                  EM
SSB                   0
LB                0.30 Hz
GB                    0
PC                1.00
```

compound 9



9


```
Current Data Parameters
NAME          HH-4-258A
EXPNO                 2
PROCNO                1

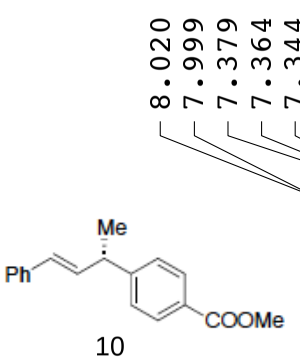
F2 - Acquisition Parameters
Date_          20130513
Time              22.23
INSTRUM           spect
PROBHD   5 mm CPQNP 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ            1.3664756 sec
RG                  512
DW               20.850 usec
DE               18.00 usec
TE               298.2 K
D1           2.00000000 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
SF         100.6127547 MHz
WDW                EM
SSB                   0
LB                1.00 Hz
GB                    0
PC                1.40
```

compound 10



10

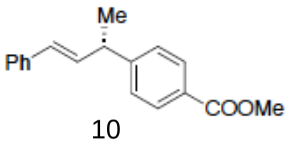

```
Current Data Parameters
NAME          HH-4-241A
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20130413
Time               4.26
INSTRUM           spect
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                   5.7
DW                60.400 usec
DE                 6.00 usec
TE                298.1 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                15.00 usec
PL1                4.90 dB
PL1W         3.30822015 W
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                32768
SF          400.1300093 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

compound 10



10


```
Current Data Parameters
NAME          HH-4-241A
EXPNO                 2
PROCNO                1

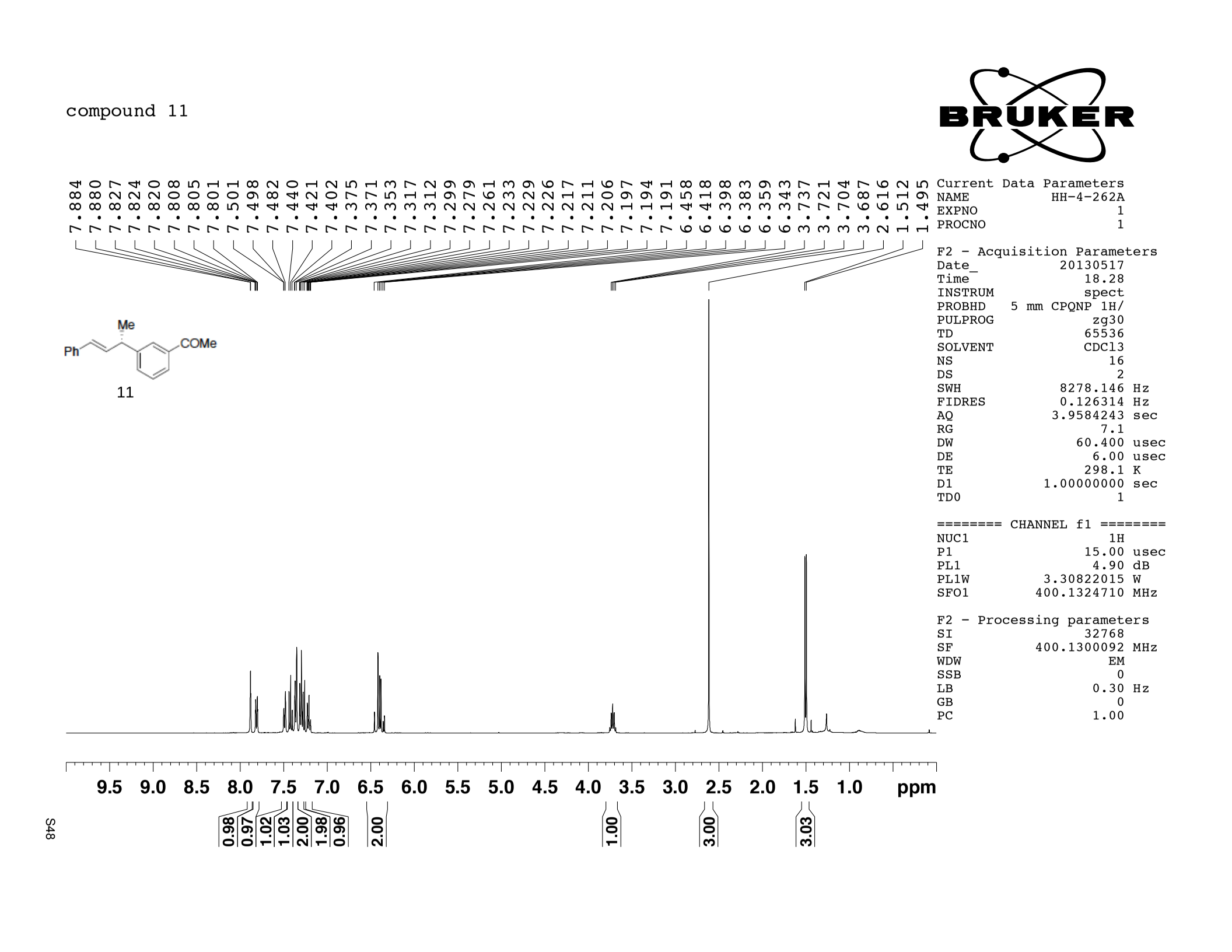
F2 - Acquisition Parameters
Date_          20130413
Time               5.26
INSTRUM           spect
PROBHD   5 mm CPQNP 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ             1.3664756 sec
RG                  512
DW               20.850 usec
DE               18.00 usec
TE               298.2 K
D1            2.00000000 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
SF         100.6127587 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC                1.40
```

compound 11



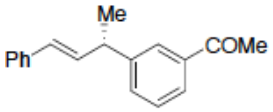

```
Current Data Parameters
NAME            HH-4-262A
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20130517
Time                18.28
INSTRUM             spect
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                     7.1
DW                60.400 usec
DE                 6.00 usec
TE                 298.1 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 15.00 usec
PL1                 4.90 dB
PL1W          3.30822015 W
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                  32768
SF           400.1300092 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                   1.00
```

compound 11



11


```
Current Data Parameters
NAME          HH-4-262A
EXPNO                 2
PROCNO                1

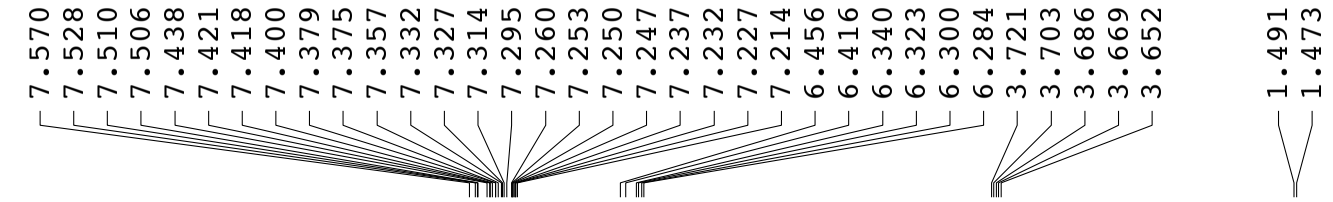
F2 - Acquisition Parameters
Date_          20130517
Time              18.32
INSTRUM           spect
PROBHD   5 mm CPQNP 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   32
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ             1.3664756 sec
RG                  512
DW                20.850 usec
DE                18.00 usec
TE                298.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

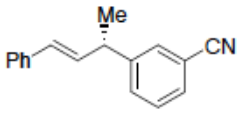
======== CHANNEL f1 ========
NUC1                 13C
P1                  9.25 usec
PL1                 0.55 dB
PL1W        35.18820572 W
SFO1        100.6228298 MHz

======== 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

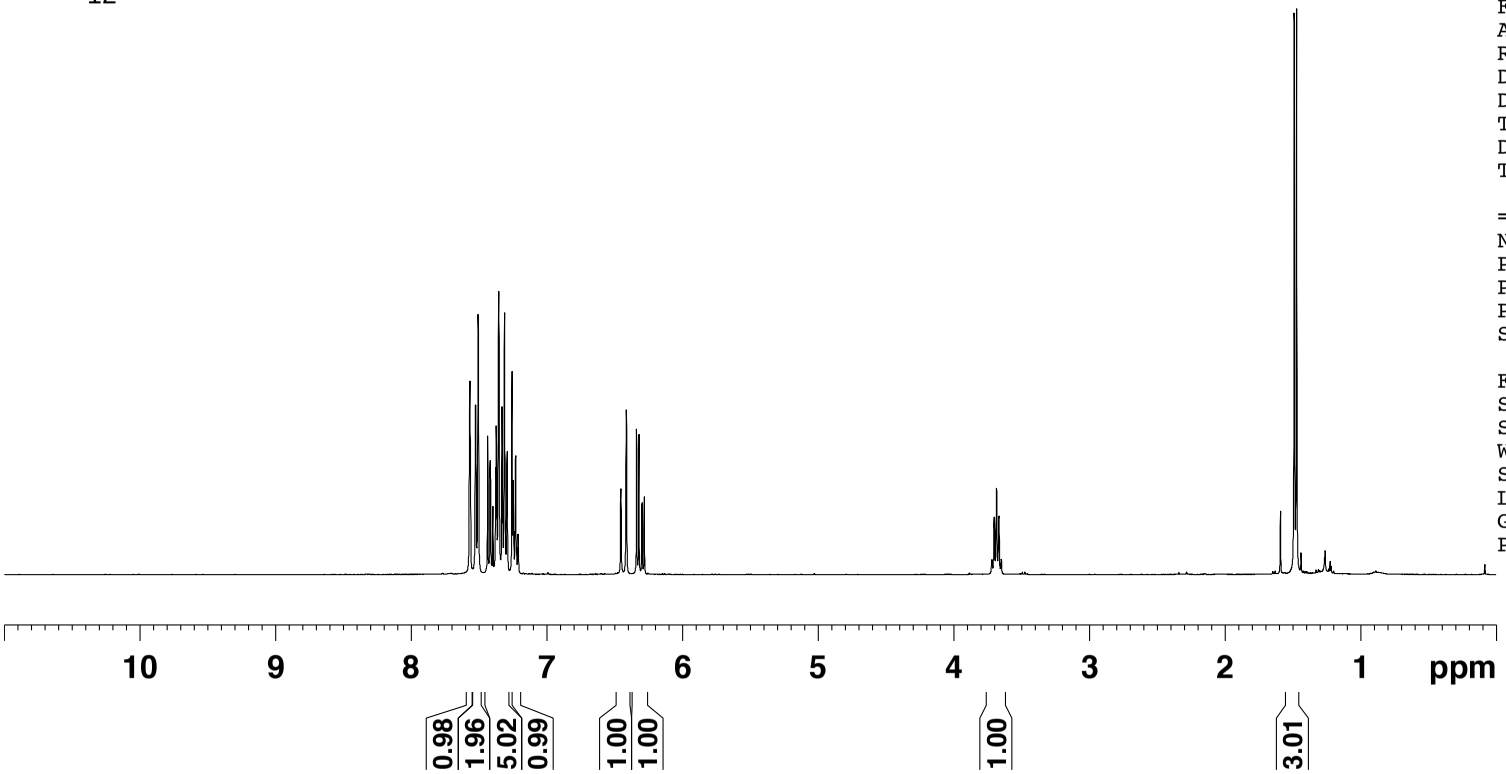
F2 - Processing parameters
SI                 32768
SF          100.6127558 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

compound 12





12



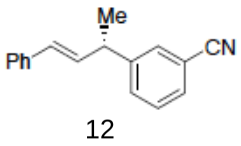

```
Current Data Parameters
NAME            HH-4-240
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130411
Time                18.37
INSTRUM             spect
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                    10.1
DW                 60.400 usec
DE                  6.00 usec
TE                 298.1 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 15.00 usec
PL1                 4.90 dB
PL1W          3.30822015 W
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF           400.1300097 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

compound 12



12

S51

```
Current Data Parameters
NAME            HH-4-240
EXPNO                  2
PROCNO                 1

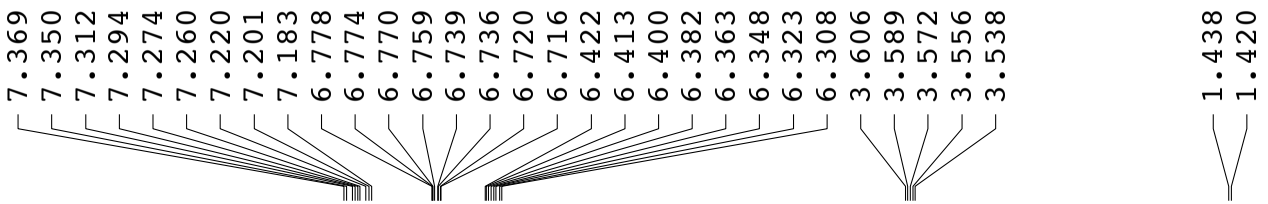
F2 - Acquisition Parameters
Date_           20130412
Time                4.48
INSTRUM            spect
PROBHD   5 mm CPQNP 1H/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ            1.3664756 sec
RG                   512
DW                20.850 usec
DE                18.00 usec
TE                298.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

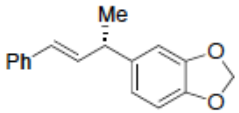
======== CHANNEL f1 ========
NUC1                 13C
P1                  9.25 usec
PL1                 0.55 dB
PL1W        35.18820572 W
SFO1        100.6228298 MHz

======== 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

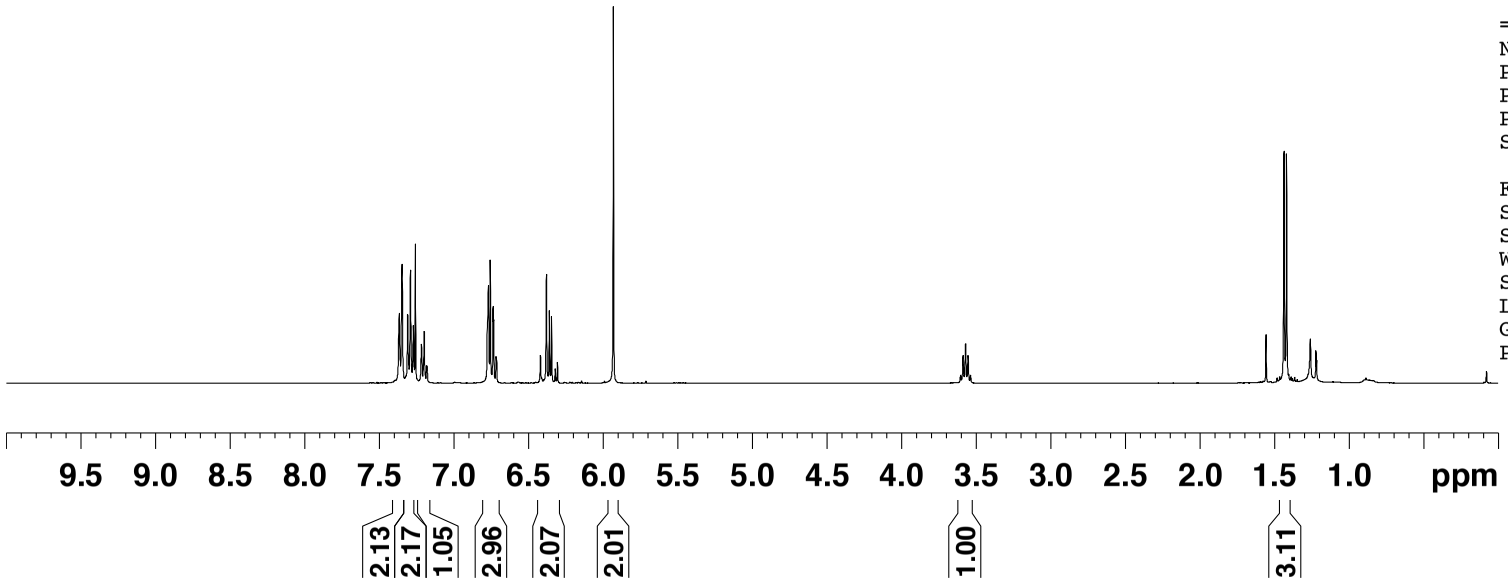
F2 - Processing parameters
SI                 32768
SF          100.6127574 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

compound 13





13



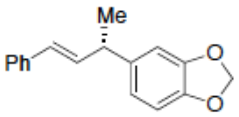

```
Current Data Parameters
NAME          HH-4-249B
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20130429
Time              17.21
INSTRUM           spect
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                11.3
DW              60.400 usec
DE               6.00 usec
TE              298.1 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                15.00 usec
PL1                4.90 dB
PL1W         3.30822015 W
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                32768
SF          400.1300095 MHz
WDW                  EM
SSB                   0
LB                0.30 Hz
GB                    0
PC                1.00
```

compound 13



13

S53

```
Current Data Parameters
NAME                  HH-4-249B
EXPNO                         2
PROCNO                        1

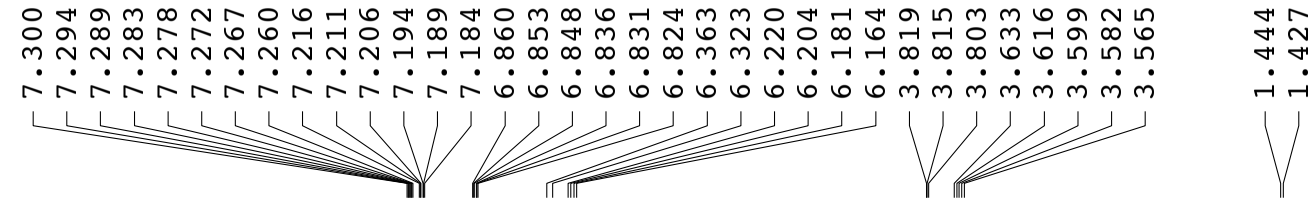
F2 - Acquisition Parameters
Date_                  20130429
Time                      17.24
INSTRUM                   spect
PROBHD    5 mm CPQNP 1H/
PULPROG                 zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                           32
DS                            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                    2.00000000 sec
D11                   0.03000000 sec
TD0                           1

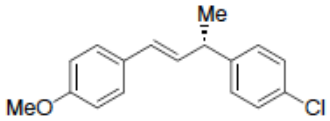
======== CHANNEL f1 ========
NUC1                        13C
P1                        9.25 usec
PL1                       0.55 dB
PL1W                35.18820572 W
SFO1                100.6228298 MHz

======== 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

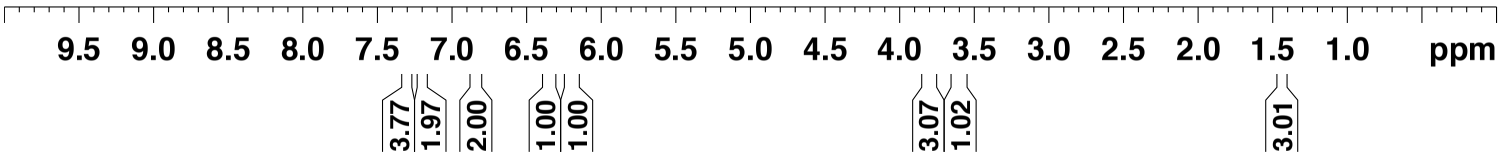
F2 - Processing parameters
SI                        32768
SF                  100.6127554 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```

compound 14





14



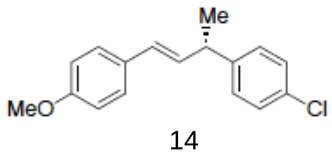

```
Current Data Parameters
NAME         HH-4-264Brs
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130522
Time              13.42
INSTRUM           spect
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                298.1 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                15.00 usec
PL1                4.90 dB
PL1W         3.30822015 W
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                32768
SF          400.1300094 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

compound 14



14


```
Current Data Parameters
NAME         HH-4-264Brs
EXPNO                  2
PROCNO                 1

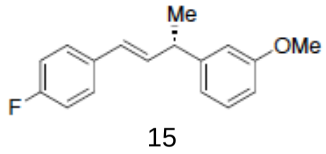
F2 - Acquisition Parameters
Date_           20130522
Time              13.45
INSTRUM           spect
PROBHD   5 mm CPQNP 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   32
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ             1.3664756 sec
RG                  512
DW               20.850 usec
DE               18.00 usec
TE               298.2 K
D1            2.00000000 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
SF          100.6127547 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

compound 15



15

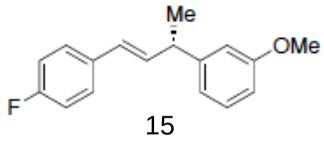

```
Current Data Parameters
NAME            HH-4-252A
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20130502
Time                17.32
INSTRUM             spect
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              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  15.00 usec
PL1                  4.90 dB
PL1W          3.30822015 W
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                  32768
SF           400.1300094 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

compound 15



15


```
Current Data Parameters
NAME          HH-4-252A
EXPNO                 2
PROCNO                1

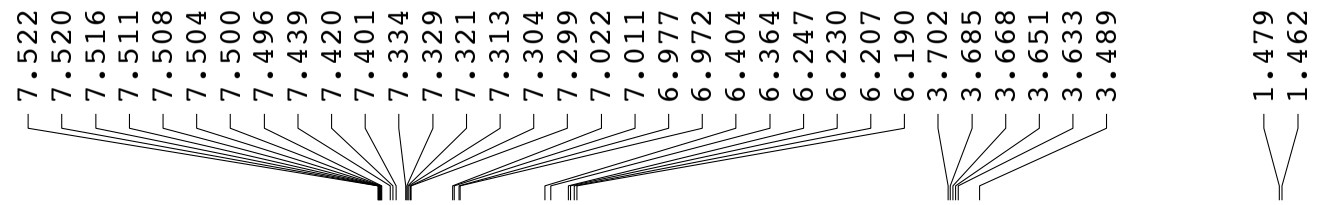
F2 - Acquisition Parameters
Date_          20130502
Time              22.12
INSTRUM           spect
PROBHD   5 mm CPQNP 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ             1.3664756 sec
RG                  512
DW               20.850 usec
DE               18.00 usec
TE               298.1 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

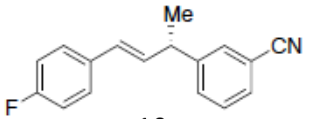
======== CHANNEL f1 ========
NUC1                13C
P1                9.25 usec
PL1               0.55 dB
PL1W        35.18820572 W
SFO1       100.6228298 MHz

======== 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

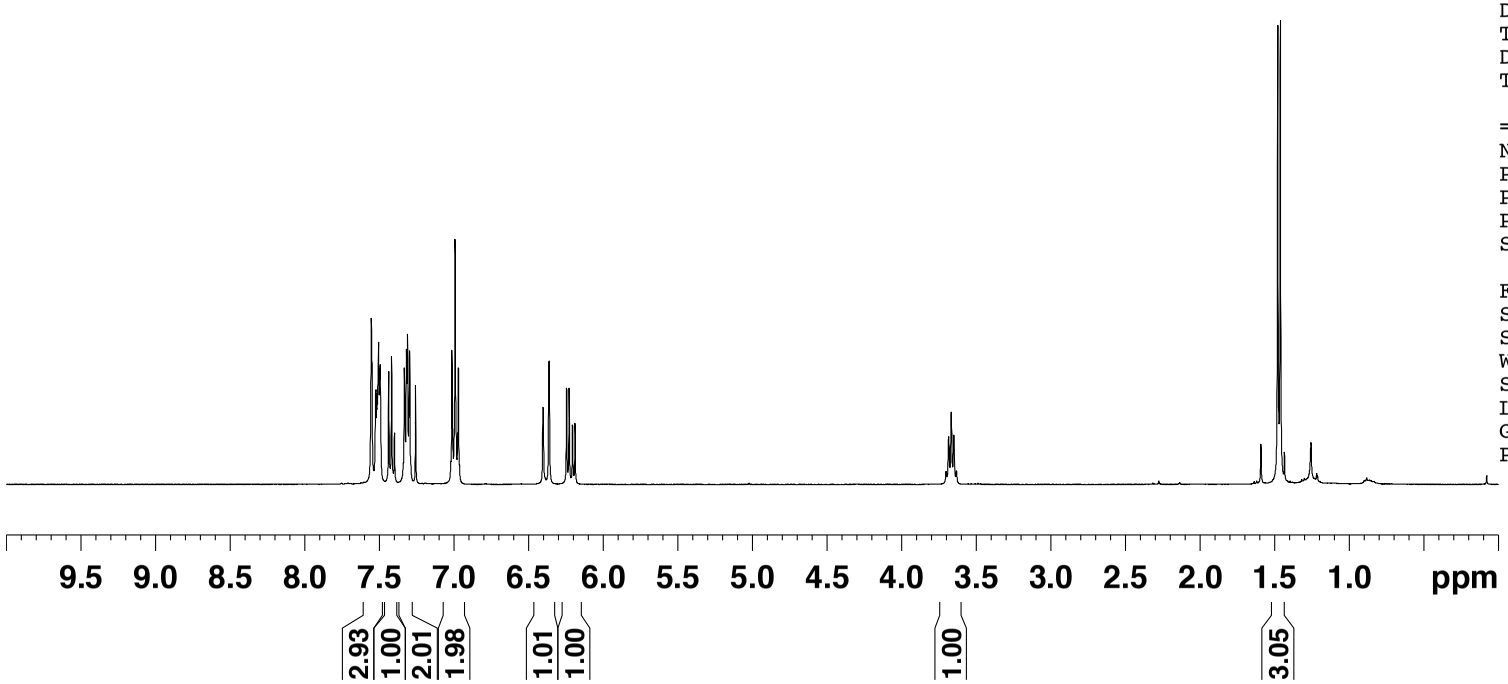
F2 - Processing parameters
SI                32768
SF         100.6127562 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC                1.40
```

compound 16





16



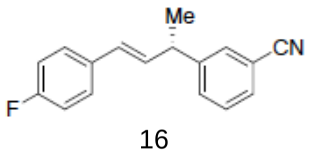

```
Current Data Parameters
NAME          HH-4-246B
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20130504
Time               8.06
INSTRUM           spect
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                    4
DW               60.400 usec
DE                6.00 usec
TE                298.2 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                15.00 usec
PL1                4.90 dB
PL1W         3.30822015 W
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                32768
SF          400.1300098 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

compound 16



16

S59

```
Current Data Parameters
NAME            HH-4-246B
EXPNO                   2
PROCNO                  1

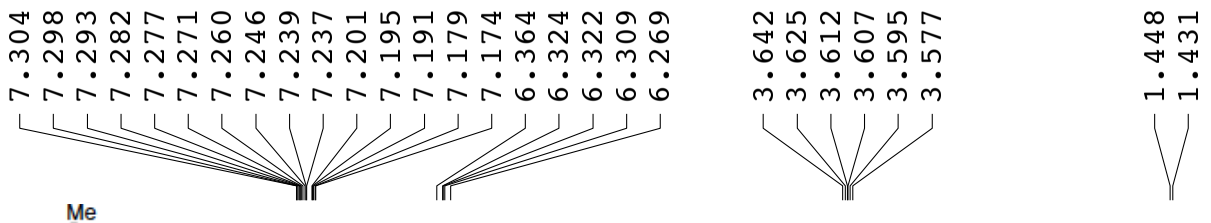
F2 - Acquisition Parameters
Date_            20130504
Time                21.03
INSTRUM             spect
PROBHD   5 mm CPQNP 1H/
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                   1024
DS                      4
SWH            23980.814 Hz
FIDRES          0.365918 Hz
AQ             1.3664756 sec
RG                    512
DW                20.850 usec
DE                18.00 usec
TE                298.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                     1

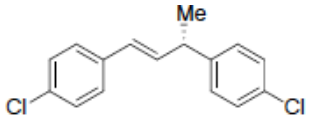
======== CHANNEL f1 ========
NUC1                  13C
P1                   9.25 usec
PL1                  0.55 dB
PL1W         35.18820572 W
SFO1         100.6228298 MHz

======== 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

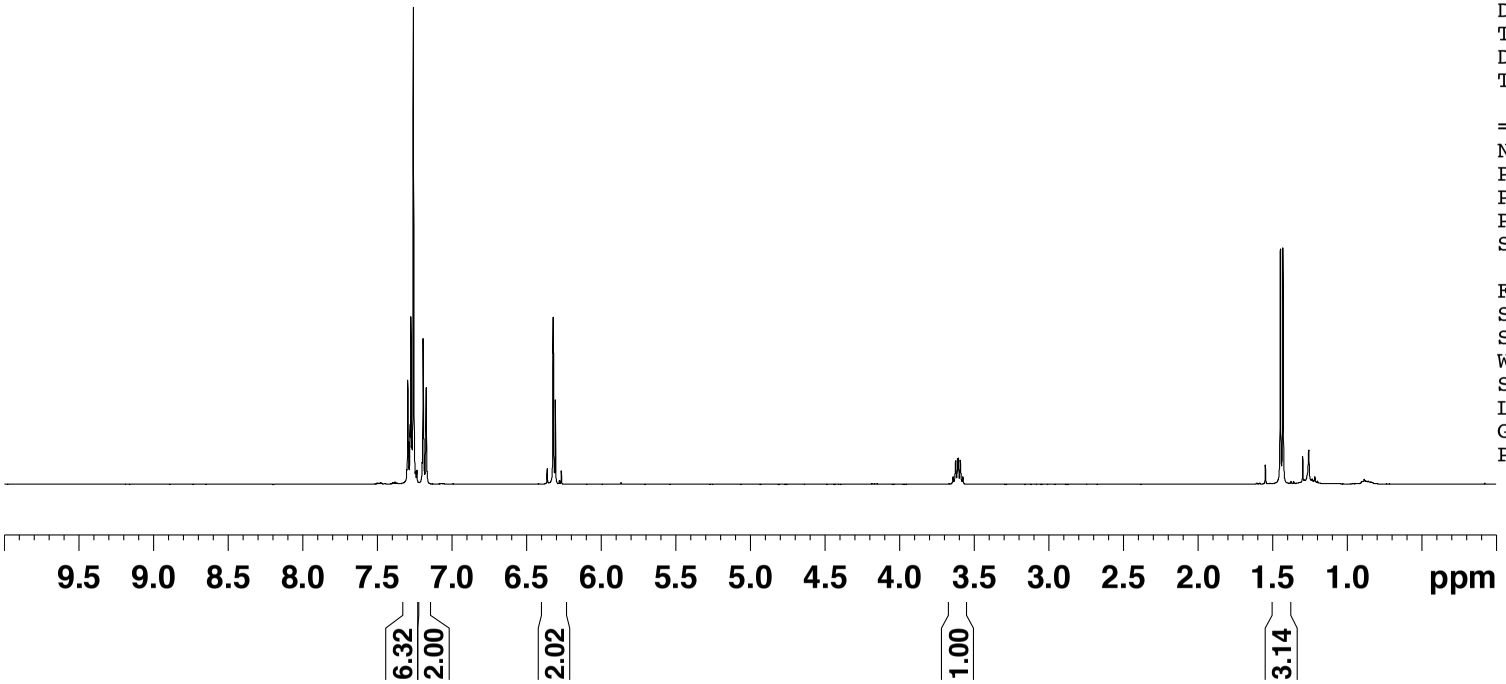
F2 - Processing parameters
SI                  32768
SF           100.6127567 MHz
WDW                    EM
SSB                     0
LB                 1.00 Hz
GB                      0
PC                   1.40
```

compound 17





17



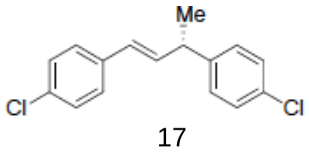

```
Current Data Parameters
NAME            HH-4-257A
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20130513
Time                2.34
INSTRUM            spect
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                  11.3
DW                60.400 usec
DE                 6.00 usec
TE                298.1 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 15.00 usec
PL1                 4.90 dB
PL1W          3.30822015 W
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF          400.1300105 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

compound 17



17

S61

```
Current Data Parameters
NAME          HH-4-257A
EXPNO                 2
PROCNO                1

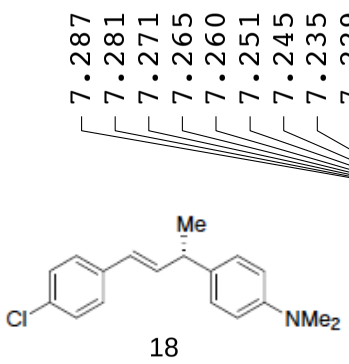
F2 - Acquisition Parameters
Date_          20130513
Time               3.34
INSTRUM           spect
PROBHD   5 mm CPQNP 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ             1.3664756 sec
RG                  512
DW               20.850 usec
DE               18.00 usec
TE               298.1 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
SF         100.6127538 MHz
WDW                EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

compound 18



18

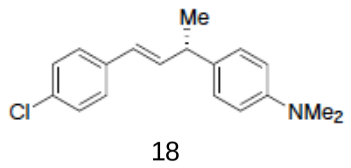

```
Current Data Parameters
NAME                  HH-4-276A
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20130531
Time                       7.45
INSTRUM                   spect
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                         10.1
DW                        60.400 usec
DE                         6.00 usec
TE                        298.2 K
D1                    1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                         1H
P1                        15.00 usec
PL1                        4.90 dB
PL1W                 3.30822015 W
SFO1                400.1324710 MHz

F2 - Processing parameters
SI                        32768
SF                  400.1300095 MHz
WDW                          EM
SSB                           0
LB                         0.30 Hz
GB                            0
PC                         1.00
```

compound 18



18


```
Current Data Parameters
NAME                  HH-4-276A
EXPNO                         2
PROCNO                        1

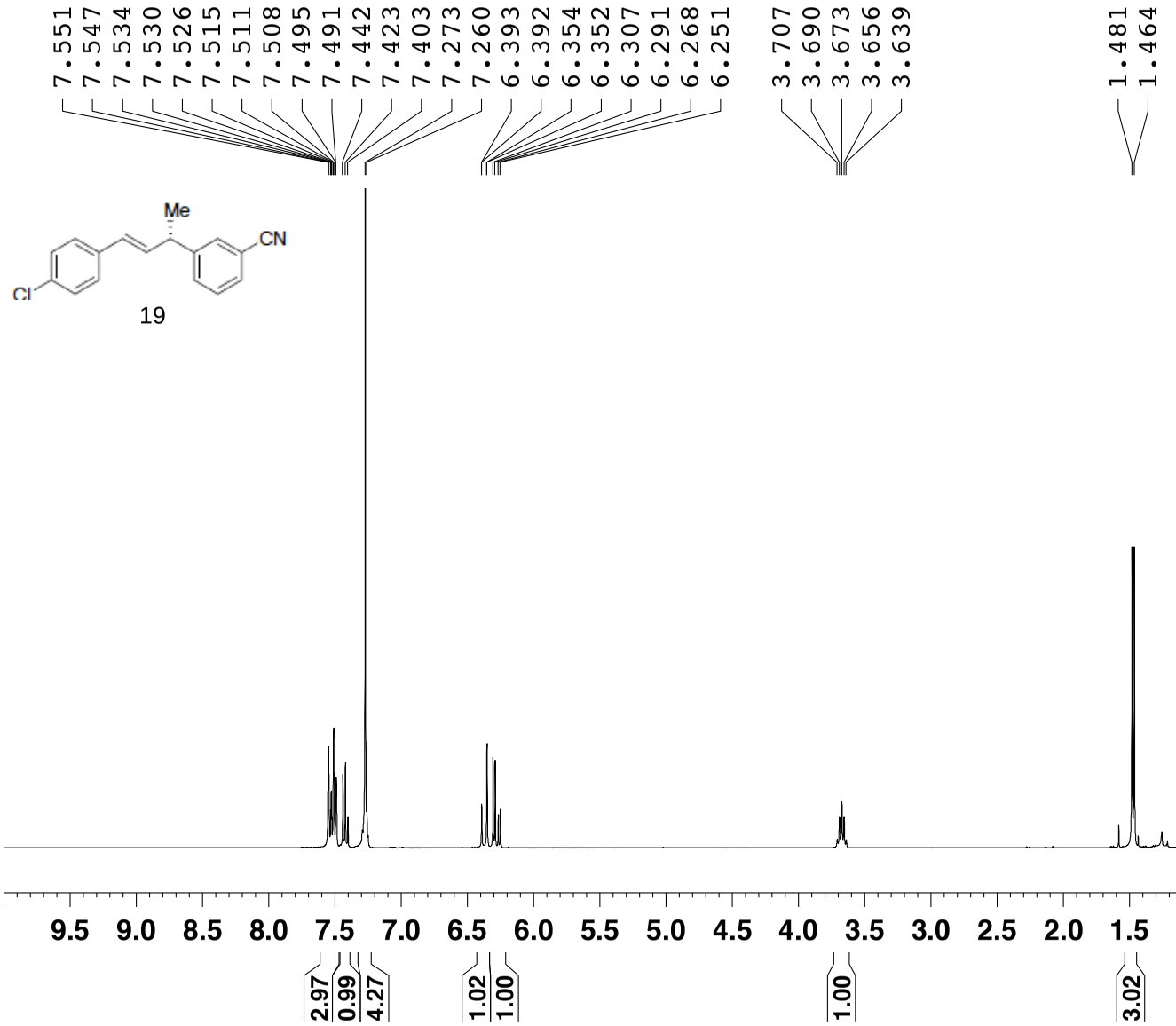
F2 - Acquisition Parameters
Date_                  20130601
Time                       1.40
INSTRUM                   spect
PROBHD    5 mm CPQNP 1H/
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                        1024
DS                           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                   2.00000000 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
SF                 100.6127550 MHz
WDW                           EM
SSB                            0
LB                        1.00 Hz
GB                             0
PC                        1.40
```

compound 19



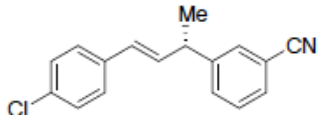

```
Current Data Parameters
NAME                  HH-4-256B
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20130513
Time                       1.28
INSTRUM                   spect
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                         11.3
DW                       60.400 usec
DE                        6.00 usec
TE                       298.2 K
D1                  1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                         1H
P1                       15.00 usec
PL1                       4.90 dB
PL1W                3.30822015 W
SFO1              400.1324710 MHz

F2 - Processing parameters
SI                        32768
SF                400.1300093 MHz
WDW                          EM
SSB                           0
LB                       0.30 Hz
GB                            0
PC                        1.00
```

compound 19



19

S65

```
Current Data Parameters
NAME          HH-4-256B
EXPNO                 2
PROCNO                1

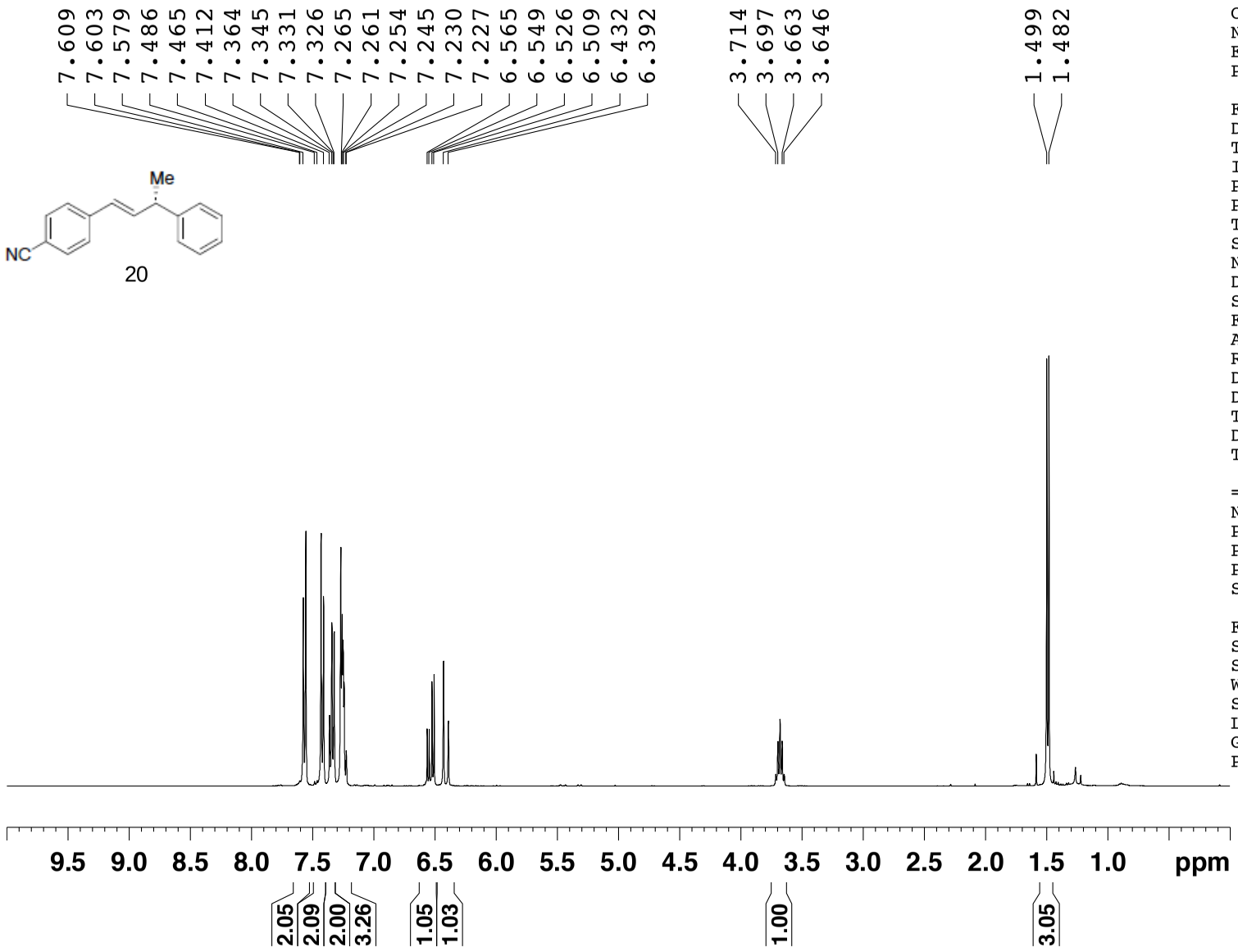
F2 - Acquisition Parameters
Date_          20130513
Time               2.28
INSTRUM           spect
PROBHD   5 mm CPQNP 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ             1.3664756 sec
RG                  512
DW               20.850 usec
DE               18.00 usec
TE               298.2 K
D1            2.00000000 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
SF          100.6127558 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC                1.40
```

compound 20



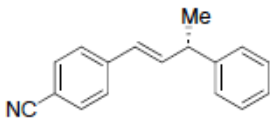

```
Current Data Parameters
NAME                  HH-4-263A
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20130518
Time                       1.11
INSTRUM                   spect
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                           6.3
DW                        60.400 usec
DE                         6.00 usec
TE                        298.2 K
D1                    1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                         1H
P1                        15.00 usec
PL1                        4.90 dB
PL1W                 3.30822015 W
SFO1               400.1324710 MHz

F2 - Processing parameters
SI                        32768
SF                 400.1300093 MHz
WDW                          EM
SSB                           0
LB                         0.30 Hz
GB                            0
PC                         1.00
```

compound 20



20

S67

```
Current Data Parameters
NAME          HH-4-263A
EXPNO                 2
PROCNO                1

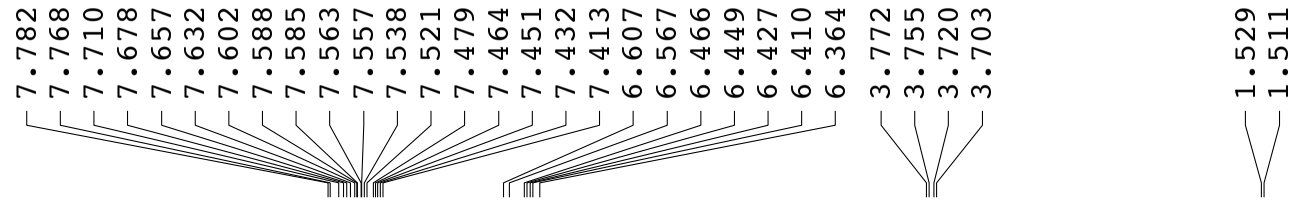
F2 - Acquisition Parameters
Date_          20130518
Time               2.11
INSTRUM           spect
PROBHD   5 mm CPQNP 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ            1.3664756 sec
RG                  512
DW               20.850 usec
DE               18.00 usec
TE               298.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

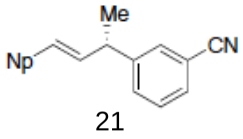
======== CHANNEL f1 ========
NUC1                13C
P1                9.25 usec
PL1               0.55 dB
PL1W       35.18820572 W
SFO1       100.6228298 MHz

======== 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

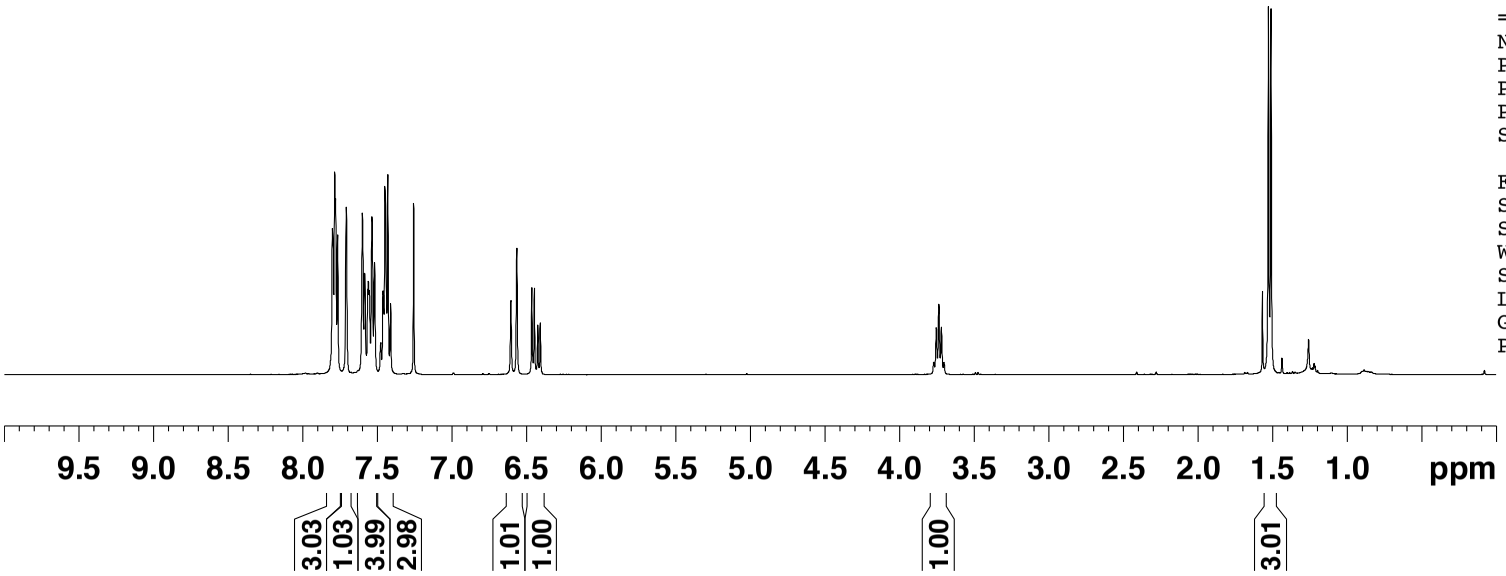
F2 - Processing parameters
SI                32768
SF         100.6127549 MHz
WDW                EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

compound 21





21



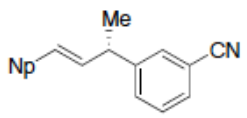

```
Current Data Parameters
NAME          HH-4-245A
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20130425
Time               7.04
INSTRUM           spect
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                 10.1
DW               60.400 usec
DE                6.00 usec
TE                298.1 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                15.00 usec
PL1                4.90 dB
PL1W         3.30822015 W
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                32768
SF          400.1300101 MHz
WDW                  EM
SSB                   0
LB                0.30 Hz
GB                    0
PC                1.00
```

compound 21



21

S69

```
Current Data Parameters
NAME          HH-4-245A
EXPNO                 2
PROCNO                1

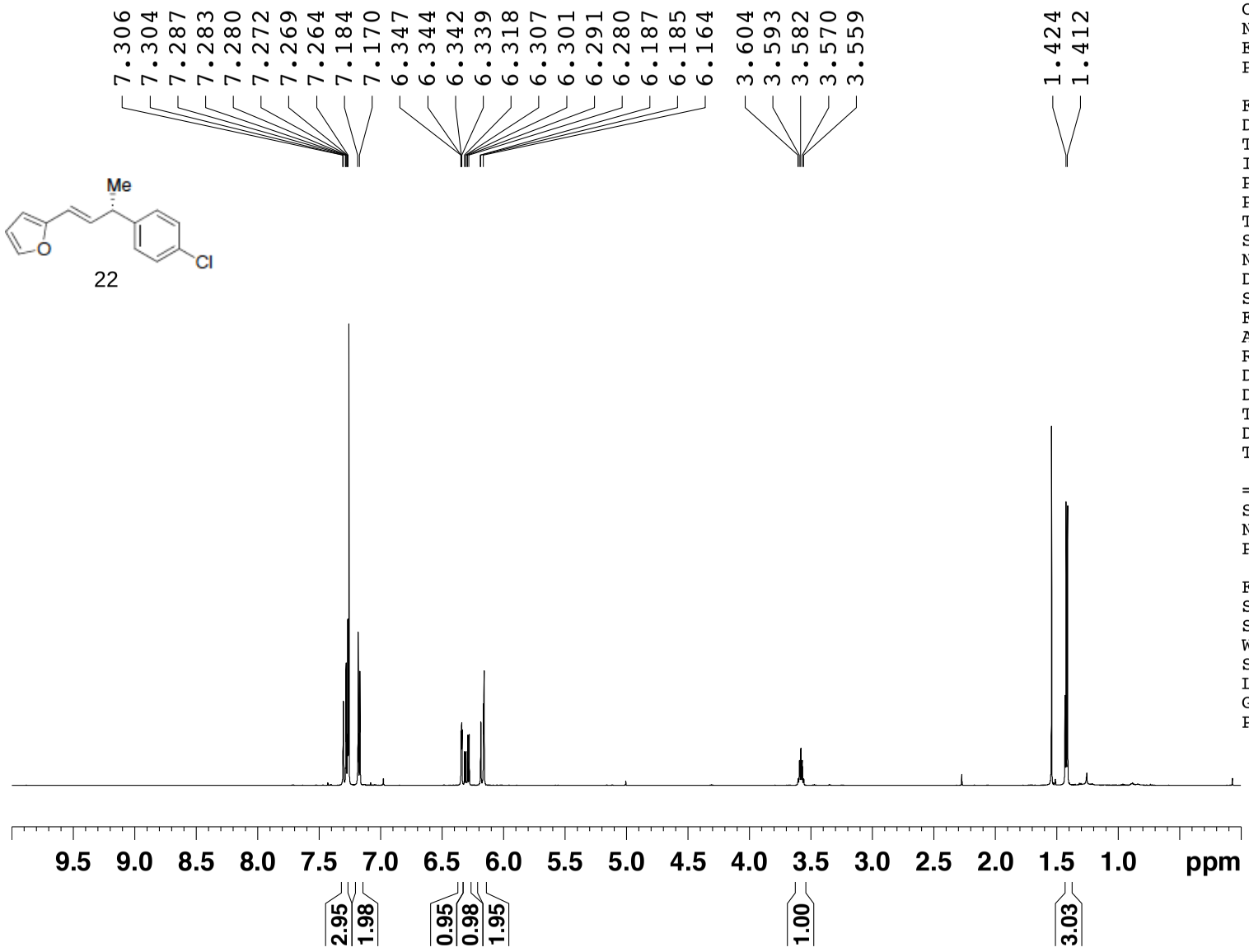
F2 - Acquisition Parameters
Date_          20130425
Time             21.08
INSTRUM          spect
PROBHD   5 mm CPQNP 1H/
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                1024
DS                   4
SWH          23980.814 Hz
FIDRES        0.365918 Hz
AQ            1.3664756 sec
RG                 512
DW              20.850 usec
DE              18.00 usec
TE              298.1 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
SF         100.6127549 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40
```

compound 22



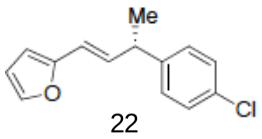

```
Current Data Parameters
NAME      QZ-4-038-PURE
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20130629
Time              13.01
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                  228
DW                59.500 usec
DE                17.39 usec
TE                298.0 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        600.3233018 MHz
NUC1                1H
P1                10.77 usec

F2 - Processing parameters
SI                65536
SF          600.3200163 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

compound 22



22

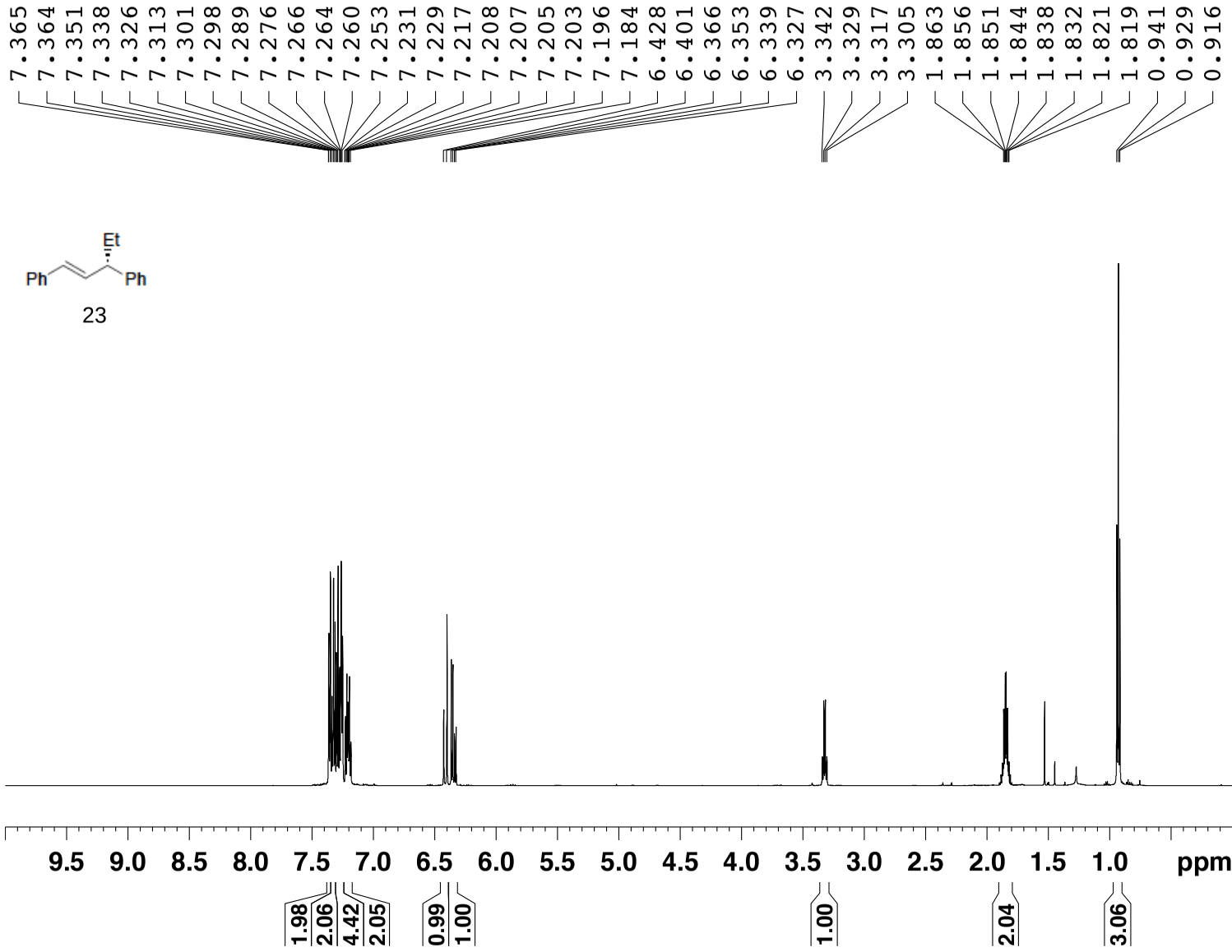

```
Current Data Parameters
NAME                  QZ-4-038
EXPNO                        2
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20130628
Time                     16.10
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                      19.34 usec
TE                      298.0 K
D1                   1.10000002 sec
D11                  0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1               150.9656784 MHz
NUC1                       13C
P1                      10.63 usec

F2 - Processing parameters
SI                       32768
SF                 150.9505603 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```

compound 23



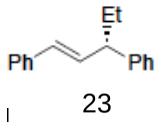

```
Current Data Parameters
NAME          QZ-4-022-2
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130617
Time               10.04
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                   114
DW                59.500 usec
DE                17.39 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         600.3233018 MHz
NUC1                   1H
P1                 10.77 usec

F2 - Processing parameters
SI                 65536
SF           600.3200161 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                 1.00
```

compound 23



23


```
Current Data Parameters
NAME      QZ-4-022-2-Acetone
EXPNO                      2
PROCNO                     1

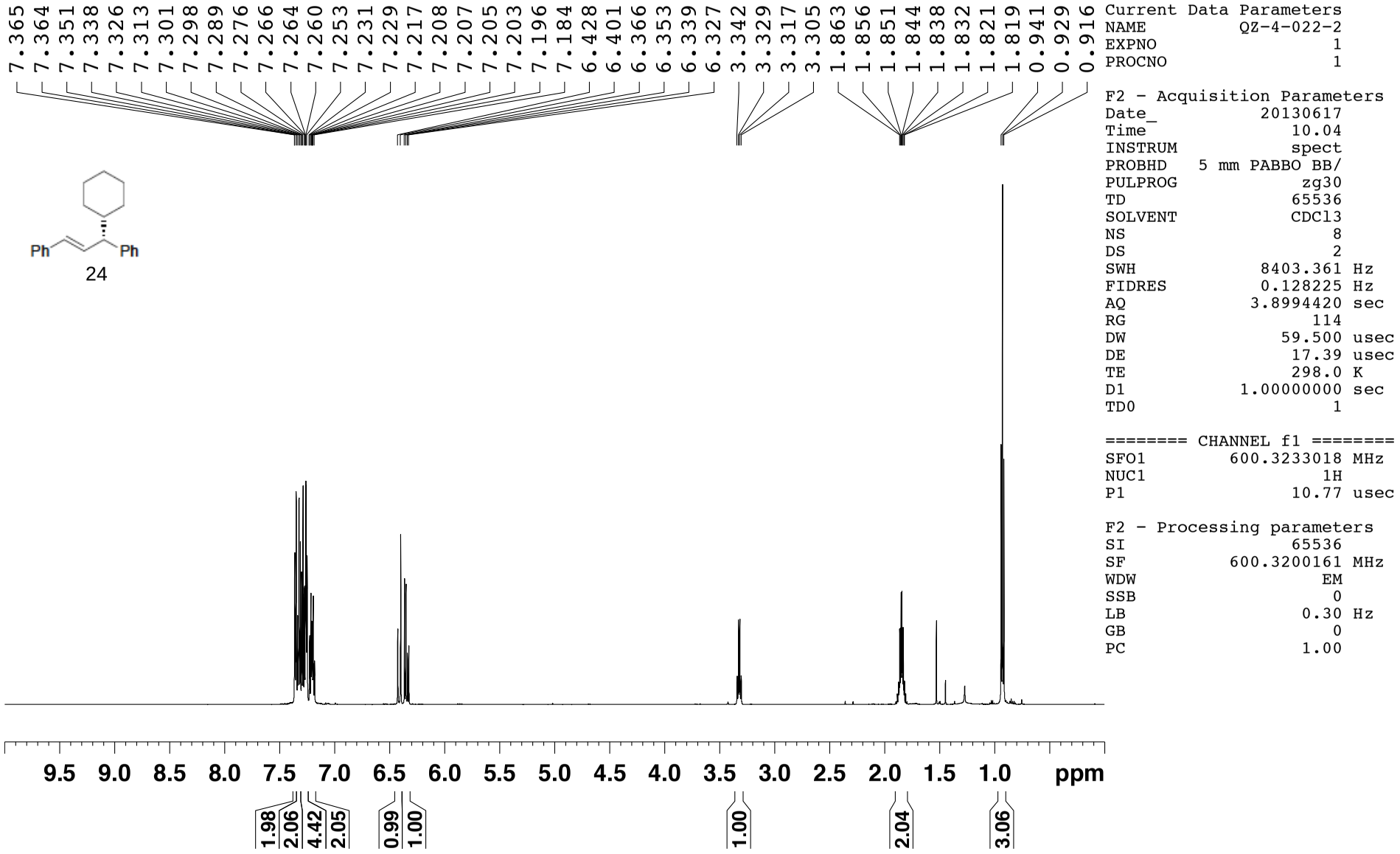
F2 - Acquisition Parameters
Date_              20130620
Time                 17.34
INSTRUM              spect
PROBHD   5 mm CPQNP 1H/
PULPROG             zgpg30
TD                   65536
SOLVENT            Acetone
NS                     256
DS                       4
SWH              23980.814 Hz
FIDRES            0.365918 Hz
AQ                1.3664756 sec
RG                     512
DW                  20.850 usec
DE                  18.00 usec
TE                  298.2 K
D1               2.00000000 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
SF            100.6126839 MHz
WDW                       EM
SSB                        0
LB                    1.00 Hz
GB                         0
PC                    1.40
```

compound 24



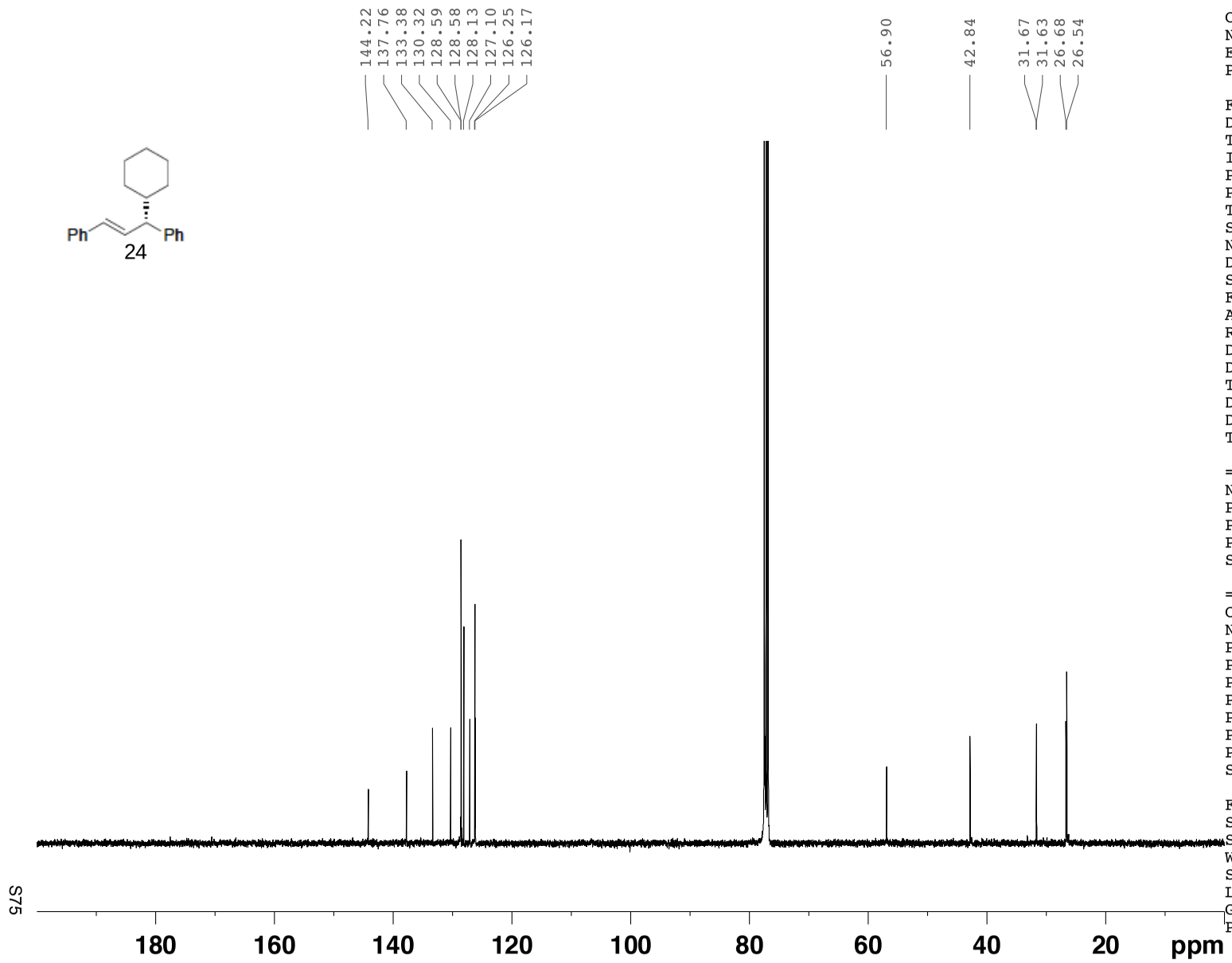

```
Current Data Parameters
NAME          QZ-4-022-2
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130617
Time               10.04
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                   114
DW                59.500 usec
DE                17.39 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         600.3233018 MHz
NUC1                   1H
P1                 10.77 usec

F2 - Processing parameters
SI                 65536
SF           600.3200161 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

compound 24




```
Current Data Parameters
NAME            QZ-4-017
EXPNO                  2
PROCNO                 1

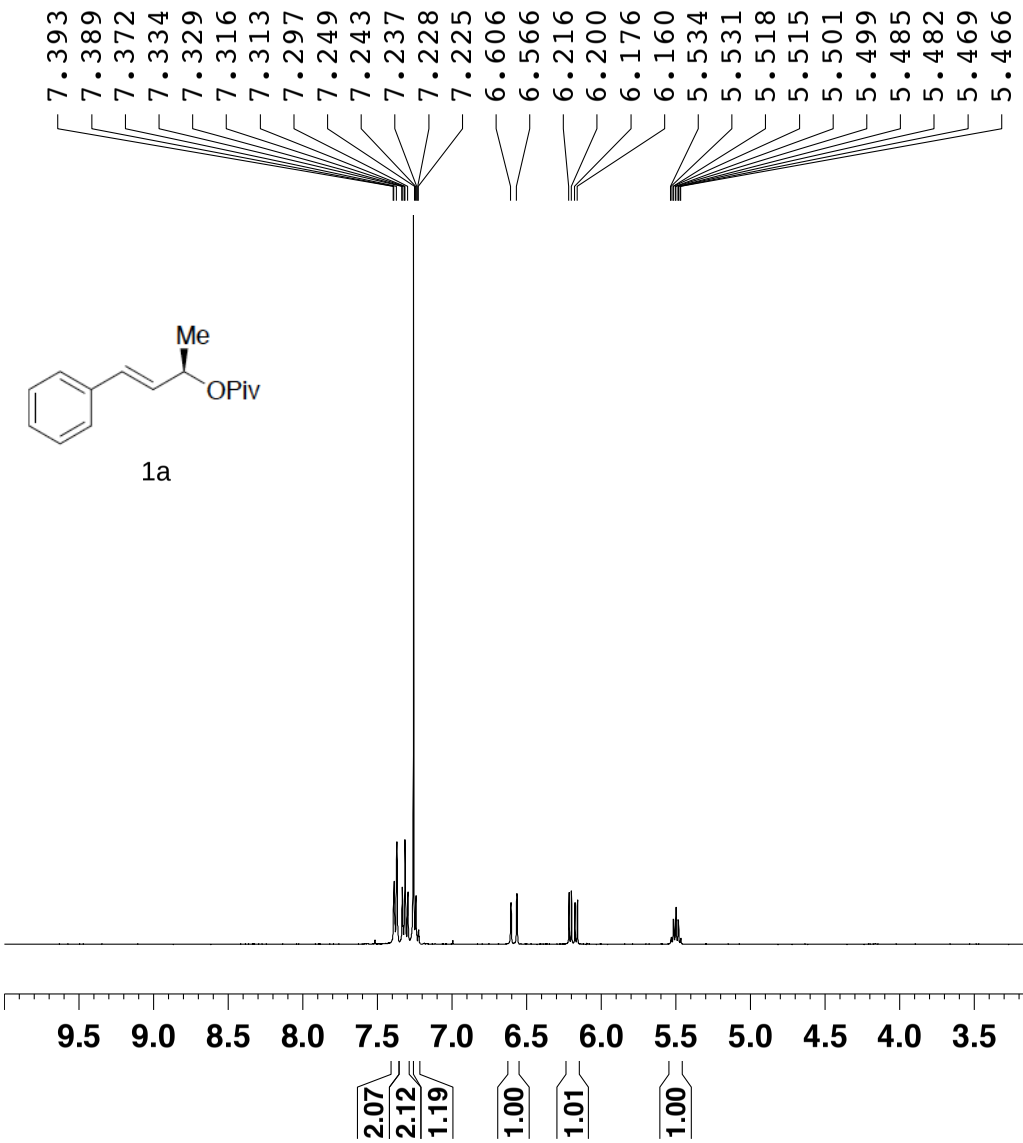
F2 - Acquisition Parameters
Date_           20130628
Time               10.50
INSTRUM            spect
PROBHD   5 mm CPQNP 1H/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   256
DS                     4
SWH            23980.814 Hz
FIDRES          0.365918 Hz
AQ              1.3664756 sec
RG                   512
DW                20.850 usec
DE                18.00 usec
TE                298.2 K
D1             2.00000000 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
SF          100.6127549 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                 1.40
```

compound 1a



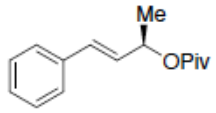

```
Current Data Parameters
NAME                  HH-4-248
EXPNO                        3
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20130522
Time                      7.55
INSTRUM                  spect
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                          9
DW                     60.400 usec
DE                      6.00 usec
TE                      298.1 K
D1                1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                      15.00 usec
PL1                      4.90 dB
PL1W               3.30822015 W
SFO1             400.1324710 MHz

F2 - Processing parameters
SI                      32768
SF               400.1300098 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00
```

compound 1a



1a


```
Current Data Parameters
NAME            HH-4-248
EXPNO                  2
PROCNO                 1

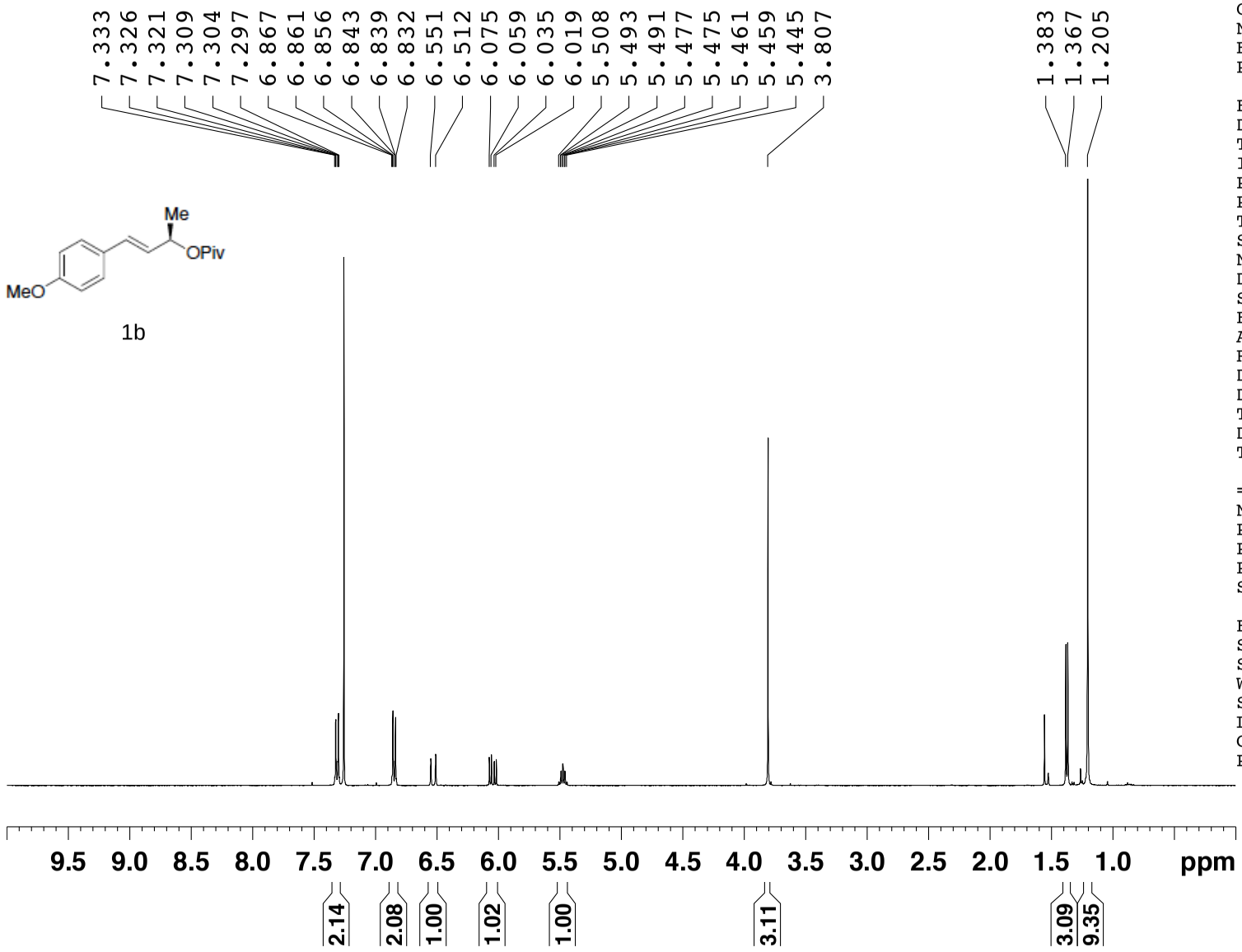
F2 - Acquisition Parameters
Date_           20130426
Time                0.18
INSTRUM            spect
PROBHD   5 mm CPQNP 1H/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ            1.3664756 sec
RG                   512
DW                20.850 usec
DE                18.00 usec
TE                298.2 K
D1           2.00000000 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
SF          100.6127538 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

compound 1b



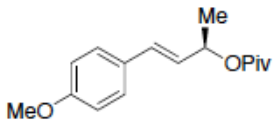

```
Current Data Parameters
NAME            HH-4-148
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130201
Time                2.32
INSTRUM            spect
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                  11.3
DW                60.400 usec
DE                 6.00 usec
TE                298.1 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                15.00 usec
PL1                4.90 dB
PL1W          3.30822015 W
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF           400.1300107 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                 1.00
```

compound 1b



1b


```
Current Data Parameters
NAME            HH-4-148
EXPNO                  2
PROCNO                 1

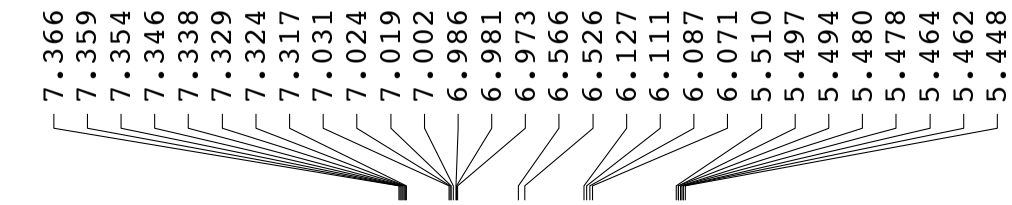
F2 - Acquisition Parameters
Date_           20130201
Time                3.32
INSTRUM            spect
PROBHD   5 mm CPQNP 1H/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            23980.814 Hz
FIDRES          0.365918 Hz
AQ             1.3664756 sec
RG                   512
DW                20.850 usec
DE                18.00 usec
TE                298.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

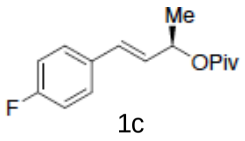
======== CHANNEL f1 ========
NUC1                 13C
P1                 9.25 usec
PL1                0.55 dB
PL1W        35.18820572 W
SFO1       100.6228298 MHz

======== 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

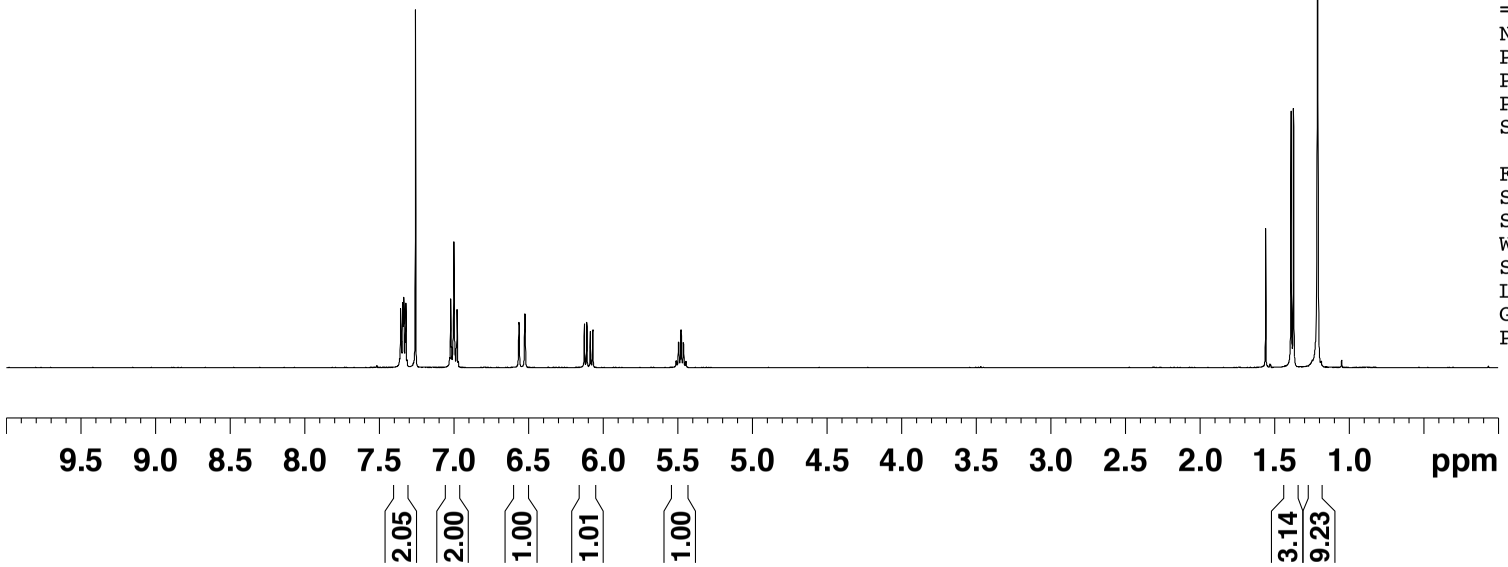
F2 - Processing parameters
SI                 32768
SF         100.6127536 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                 1.40
```

compound 1c





1c



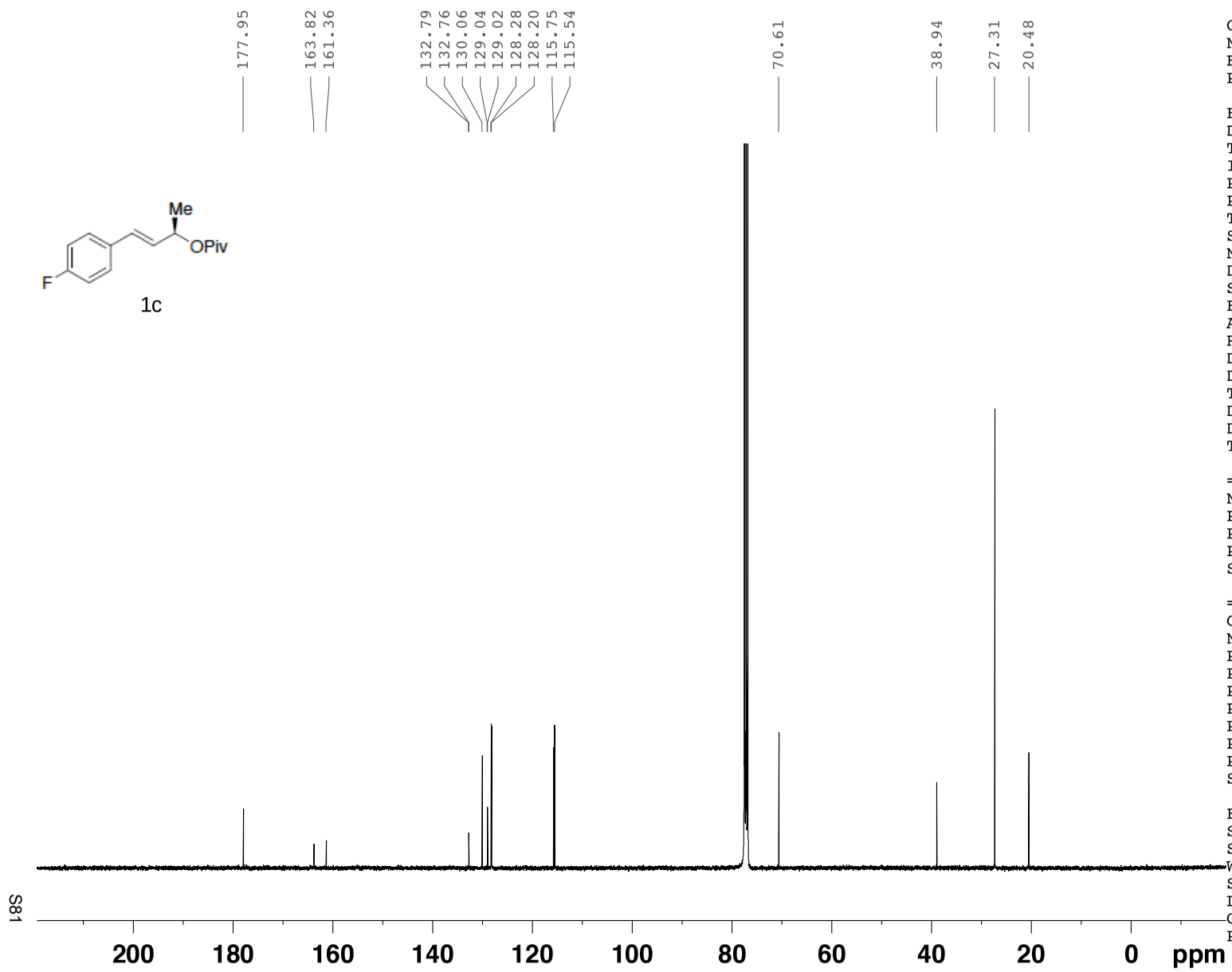

```
Current Data Parameters
NAME            HH-4-202
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130316
Time                17.41
INSTRUM             spect
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                   7.1
DW                60.400 usec
DE                 6.00 usec
TE                298.2 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 15.00 usec
PL1                 4.90 dB
PL1W          3.30822015 W
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF          400.1300099 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```

compound 1c



S81

```
Current Data Parameters
NAME            HH-4-202
EXPNO                  2
PROCNO                 1

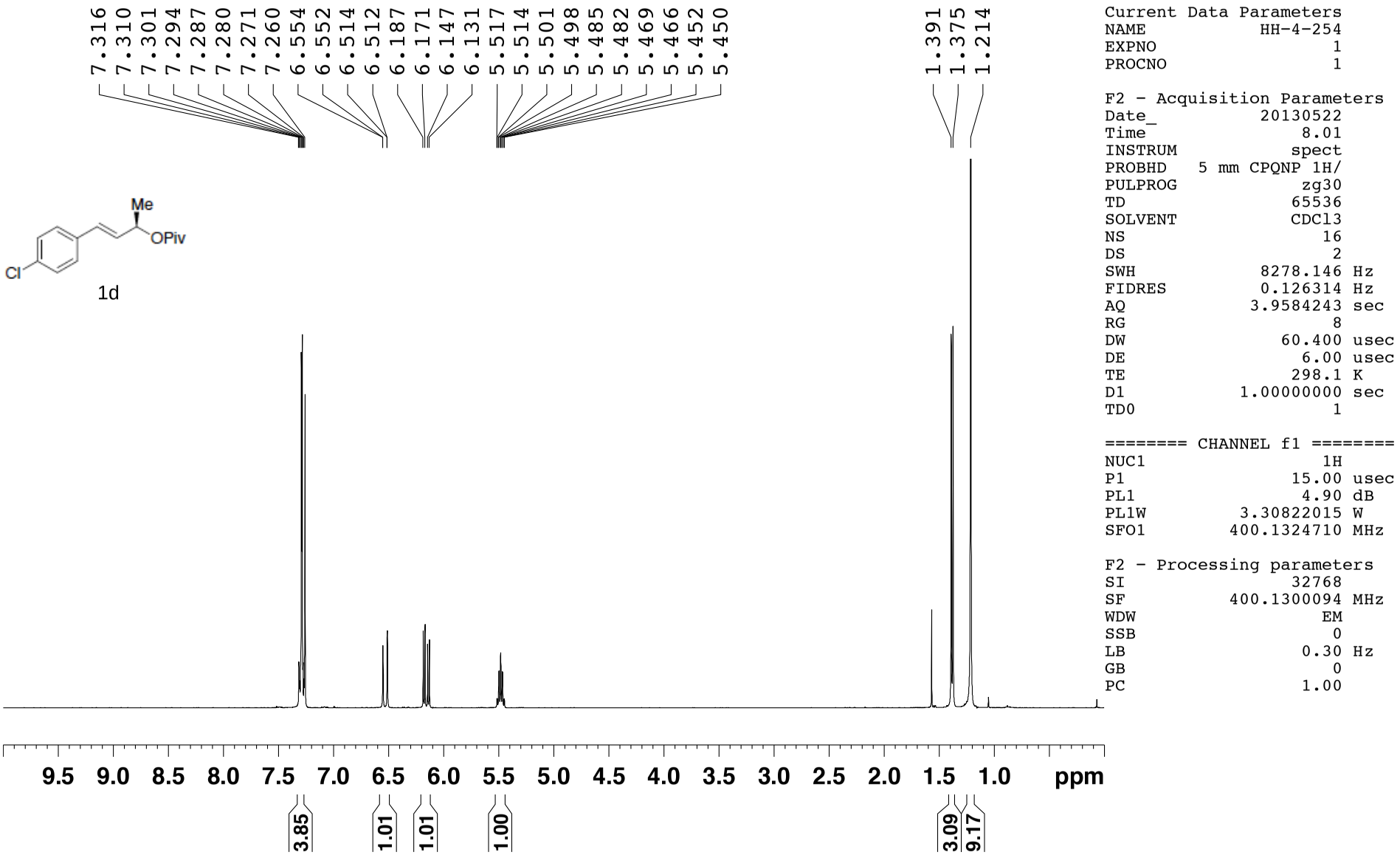
F2 - Acquisition Parameters
Date_           20130317
Time                0.29
INSTRUM            spect
PROBHD   5 mm CPQNP 1H/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ             1.3664756 sec
RG                   512
DW                20.850 usec
DE                18.00 usec
TE                298.1 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
SF          100.6127540 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

compound 1d



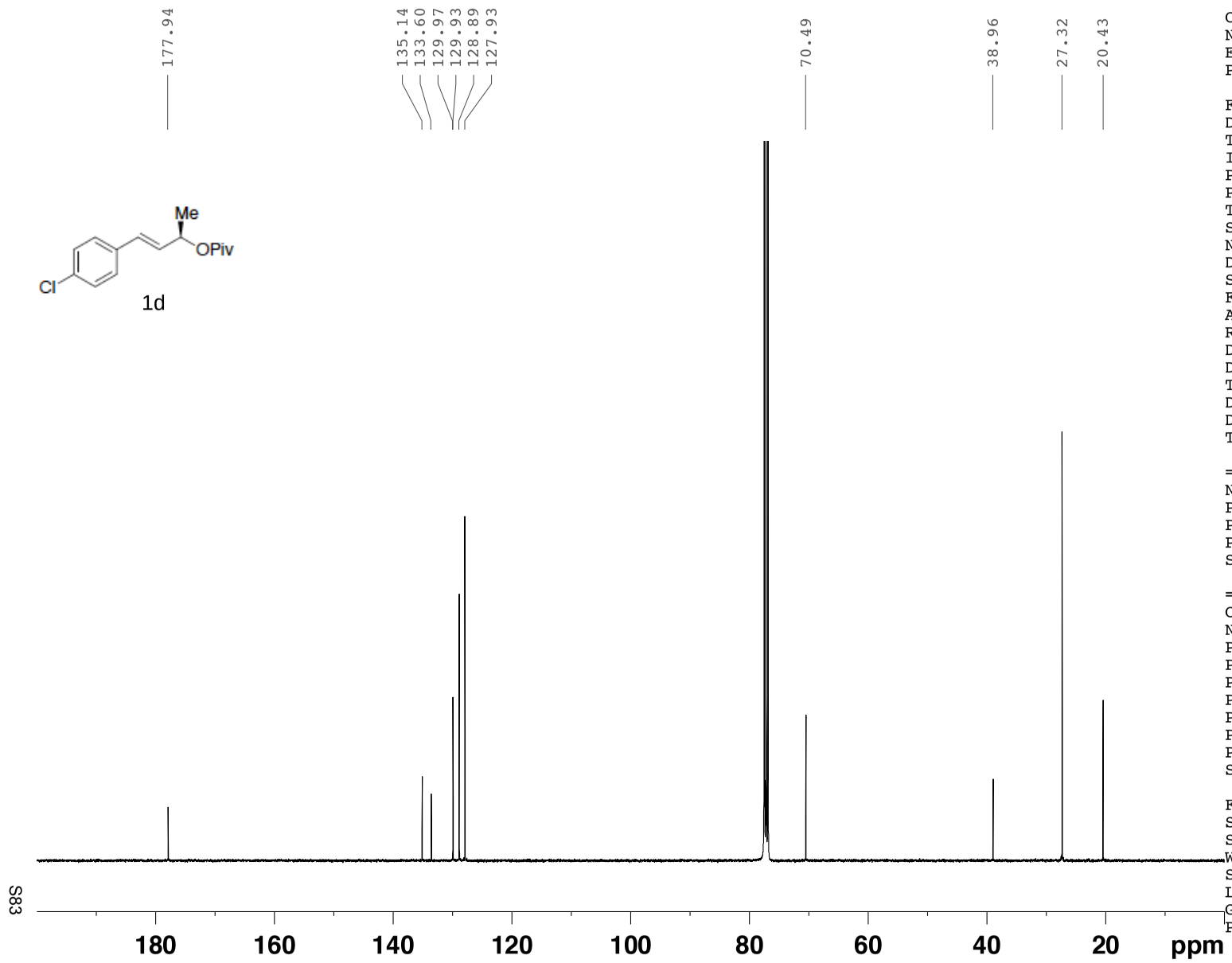

```
Current Data Parameters
NAME            HH-4-254
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130522
Time                8.01
INSTRUM            spect
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                298.1 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                15.00 usec
PL1                4.90 dB
PL1W          3.30822015 W
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF           400.1300094 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```

compound 1d



S83

```
Current Data Parameters
NAME            HH-4-254
EXPNO                  2
PROCNO                 1

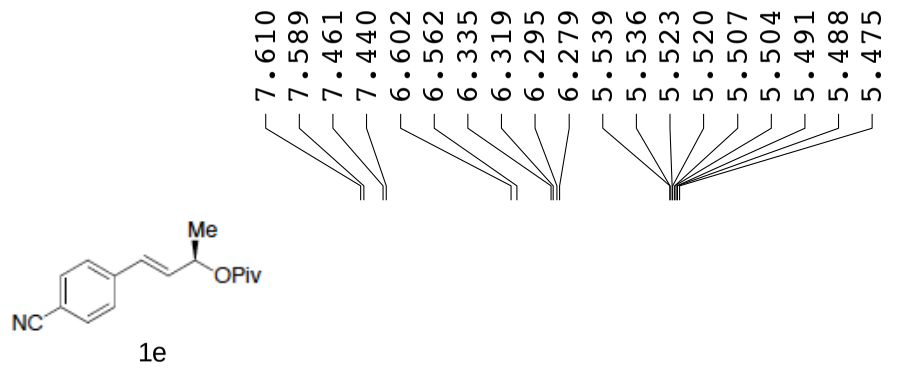
F2 - Acquisition Parameters
Date_           20130522
Time              22.06
INSTRUM           spect
PROBHD   5 mm CPQNP 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ             1.3664756 sec
RG                  512
DW               20.850 usec
DE               18.00 usec
TE               298.1 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
SF          100.6127529 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC                1.40
```

compound 1e



1e

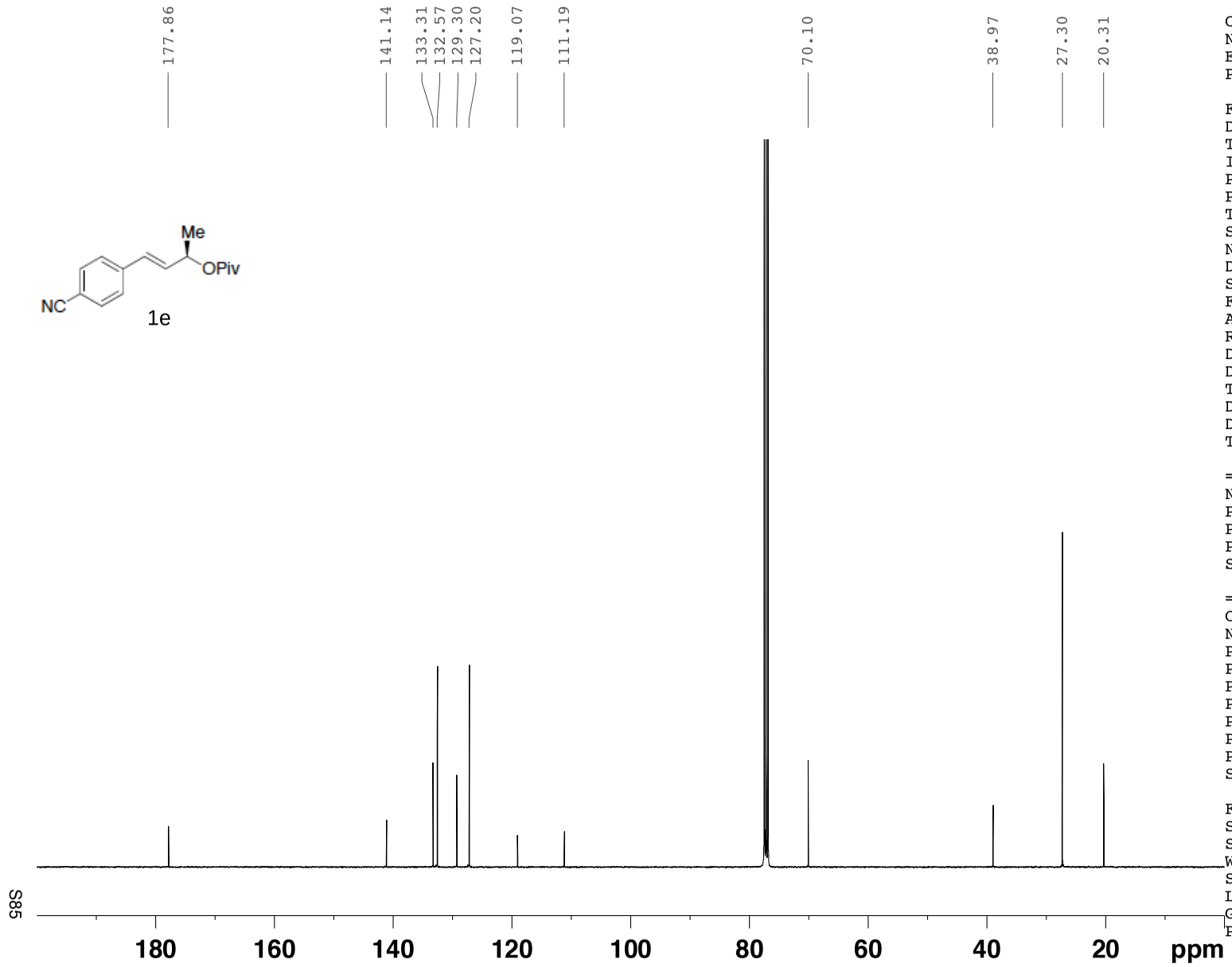

```
Current Data Parameters
NAME                  HH-4-227
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20130522
Time                      5.43
INSTRUM                  spect
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                          9
DW                     60.400 usec
DE                      6.00 usec
TE                      298.1 K
D1                1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                      15.00 usec
PL1                      4.90 dB
PL1W               3.30822015 W
SFO1             400.1324710 MHz

F2 - Processing parameters
SI                       32768
SF               400.1300094 MHz
WDW                         EM
SSB                          0
LB                      0.30 Hz
GB                           0
PC                      1.00
```

compound 1e




```
Current Data Parameters
NAME            HH-4-227
EXPNO                  2
PROCNO                 1

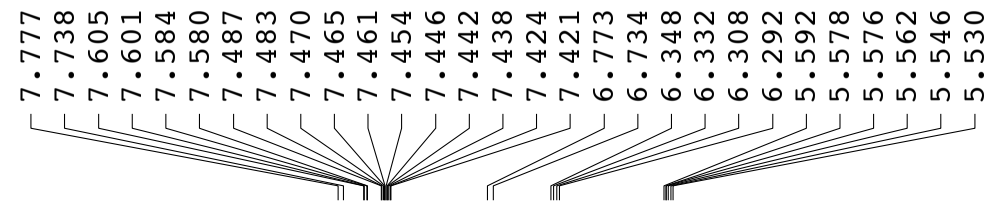
F2 - Acquisition Parameters
Date_           20130522
Time                6.43
INSTRUM            spect
PROBHD   5 mm CPQNP 1H/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ             1.3664756 sec
RG                   512
DW                20.850 usec
DE                18.00 usec
TE                298.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

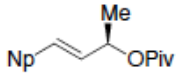
======== CHANNEL f1 ========
NUC1                 13C
P1                  9.25 usec
PL1                 0.55 dB
PL1W        35.18820572 W
SFO1        100.6228298 MHz

======== 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

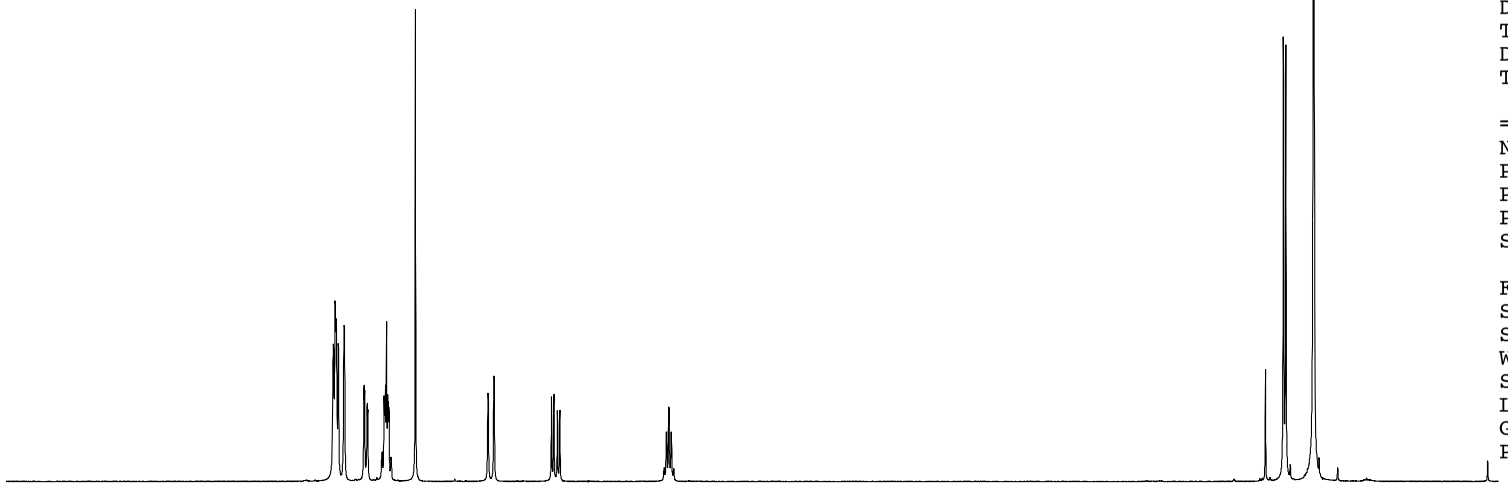
F2 - Processing parameters
SI                 32768
SF          100.6127541 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

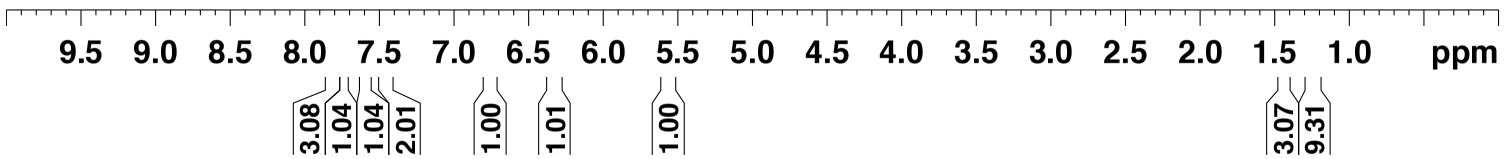
compound 1f





1f





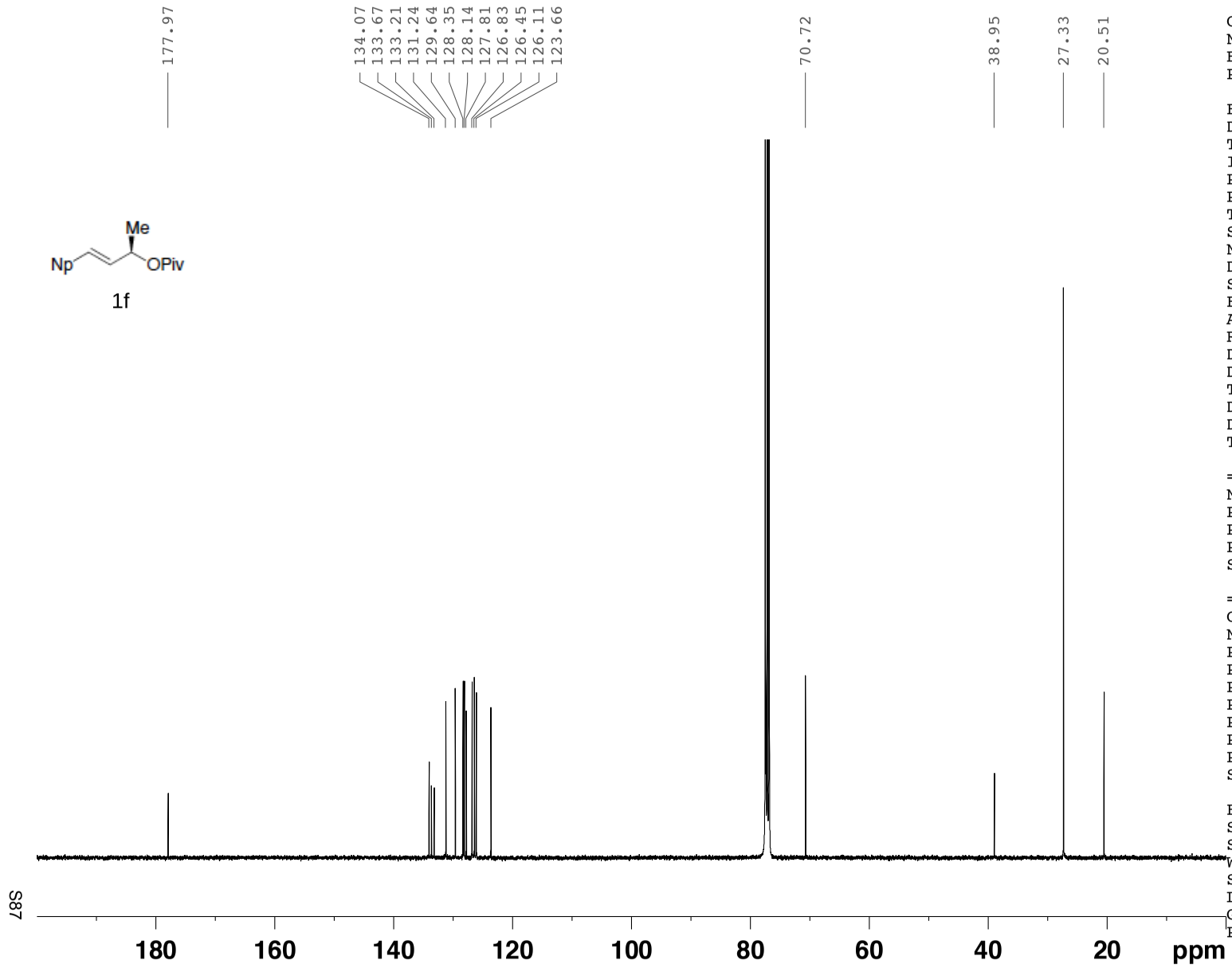

```
Current Data Parameters
NAME          HH-4-203rs
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130522
Time                4.37
INSTRUM            spect
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             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                15.00 usec
PL1                4.90 dB
PL1W         3.30822015 W
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF          400.1300095 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```

compound 1f




```
Current Data Parameters
NAME          HH-4-203rs
EXPNO                  2
PROCNO                 1

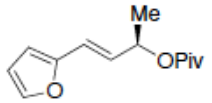
F2 - Acquisition Parameters
Date_           20130522
Time               5.37
INSTRUM           spect
PROBHD   5 mm CPQNP 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ            1.3664756 sec
RG                  512
DW               20.850 usec
DE               18.00 usec
TE               298.2 K
D1            2.00000000 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
SF          100.6127560 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

compound 1g



1g

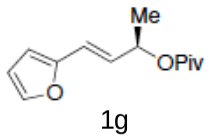

```
Current Data Parameters
NAME      QZ-3-242-1-RAC
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20130422
Time                13.33
INSTRUM             spect
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                    5.7
DW                60.400 usec
DE                 6.00 usec
TE                298.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                15.00 usec
PL1                4.90 dB
PL1W         3.30822015 W
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF          400.1300095 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                 1.00
```

compound 1g



1g


```
Current Data Parameters
NAME          QZ-3-242-1
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130620
Time              15.33
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               19.34 usec
TE               298.0 K
D1            1.10000002 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1        150.9656784 MHz
NUC1               13C
P1               10.63 usec

F2 - Processing parameters
SI                 32768
SF          150.9505582 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

compound 1h

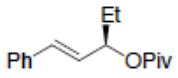

```
Current Data Parameters
NAME                  QZ-4-015
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20130612
Time                     16.23
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                         114
DW                    59.500 usec
DE                    17.39 usec
TE                    298.0 K
D1                 1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1               600.3233018 MHz
NUC1                       1H
P1                    10.77 usec

F2 - Processing parameters
SI                      65536
SF                 600.3200161 MHz
WDW                        EM
SSB                         0
LB                    0.30 Hz
GB                          0
PC                     1.00
```

compound 1h



1h

S91

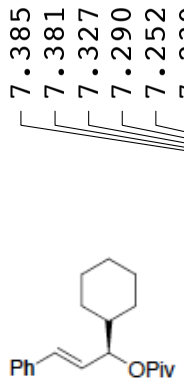
```
Current Data Parameters
NAME            QZ-4-015
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130612
Time              16.33
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               19.34 usec
TE               298.0 K
D1            1.10000002 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1        150.9656784 MHz
NUC1                13C
P1                10.63 usec

F2 - Processing parameters
SI                 32768
SF          150.9505582 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

compound 1i



1i

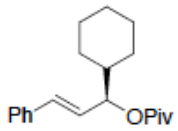

```
Current Data Parameters
NAME                  HH-4-291
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20130610
Time                     17.27
INSTRUM                  spect
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                         7.1
DW                    60.400 usec
DE                     6.00 usec
TE                     298.2 K
D1                1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                    15.00 usec
PL1                    4.90 dB
PL1W             3.30822015 W
SFO1           400.1324710 MHz

F2 - Processing parameters
SI                      32768
SF               400.1300099 MHz
WDW                        EM
SSB                         0
LB                    0.30 Hz
GB                          0
PC                     1.00
```

compound 1i



1i


```
Current Data Parameters
NAME            HH-4-291
EXPNO                  2
PROCNO                 1

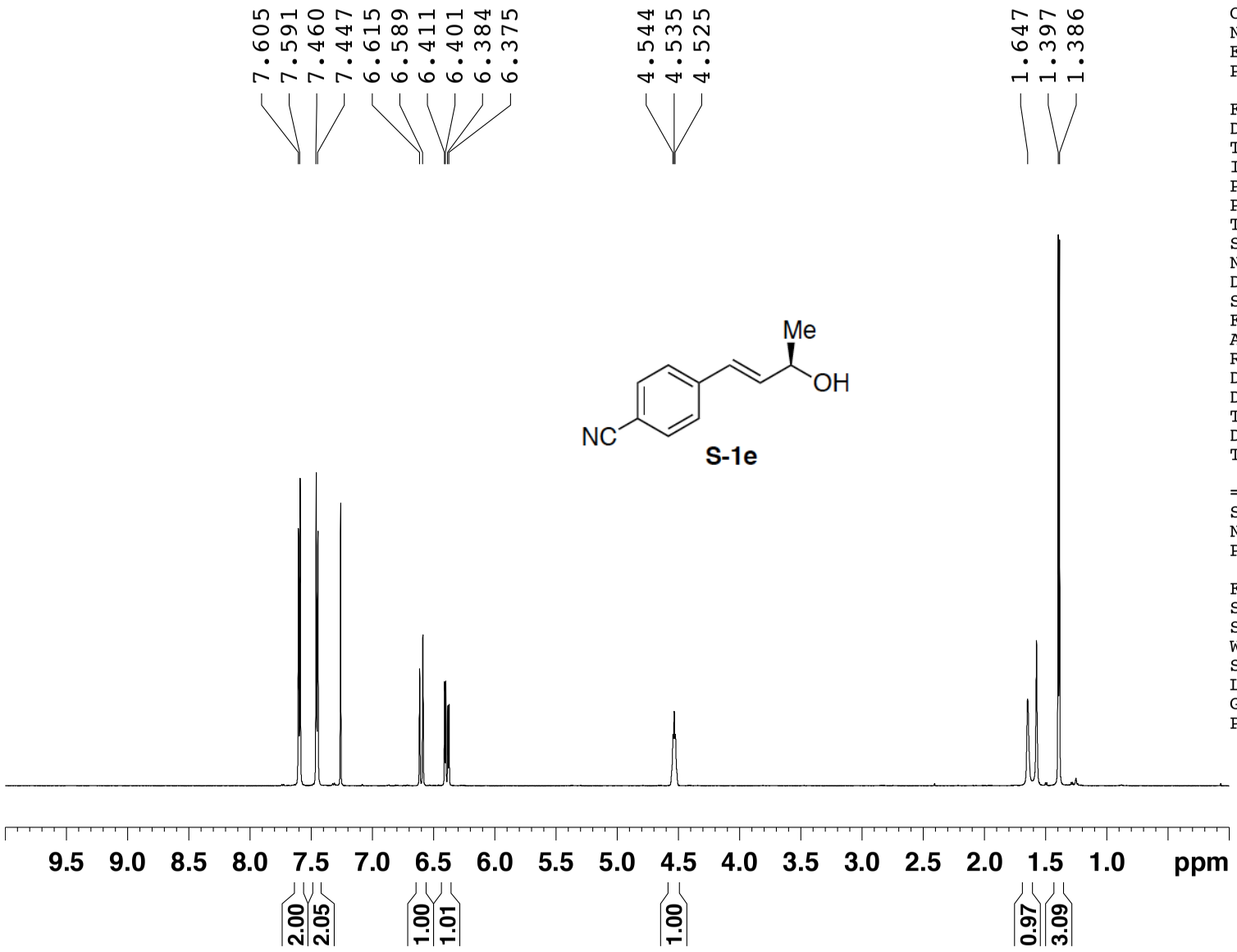
F2 - Acquisition Parameters
Date_           20130611
Time                0.35
INSTRUM            spect
PROBHD   5 mm CPQNP 1H/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ            1.3664756 sec
RG                   512
DW                20.850 usec
DE                18.00 usec
TE                298.2 K
D1           2.00000000 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
SF          100.6127524 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

compound S-1e 1HNMR



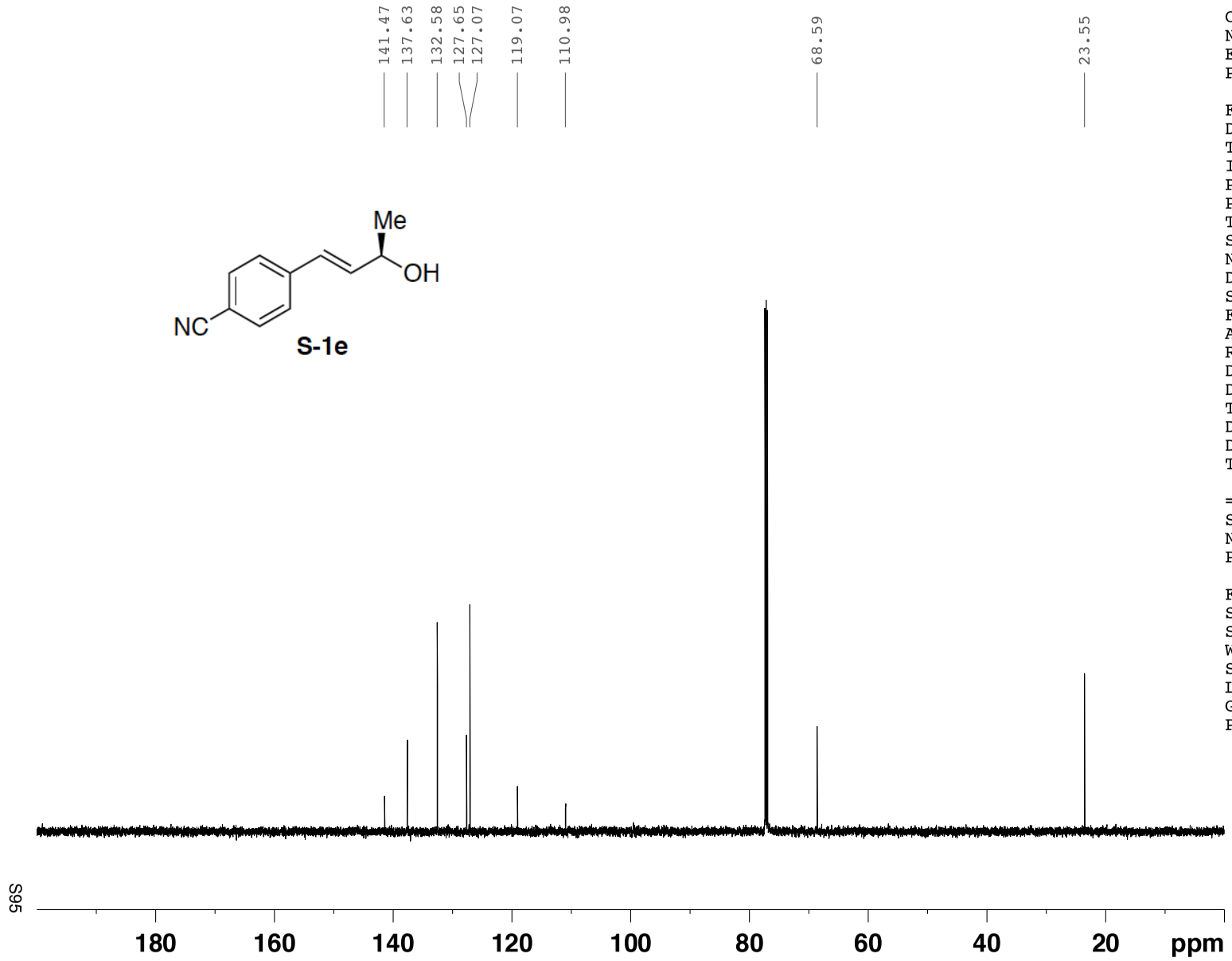

```
Current Data Parameters
NAME      QZ-HH-5-090-2
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20140220
Time              16.42
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                  181
DW                59.500 usec
DE                17.39 usec
TE                298.0 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        600.3233018 MHz
NUC1                1H
P1                10.77 usec

F2 - Processing parameters
SI                65536
SF          600.3200135 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

compound S-1e 13CNMR



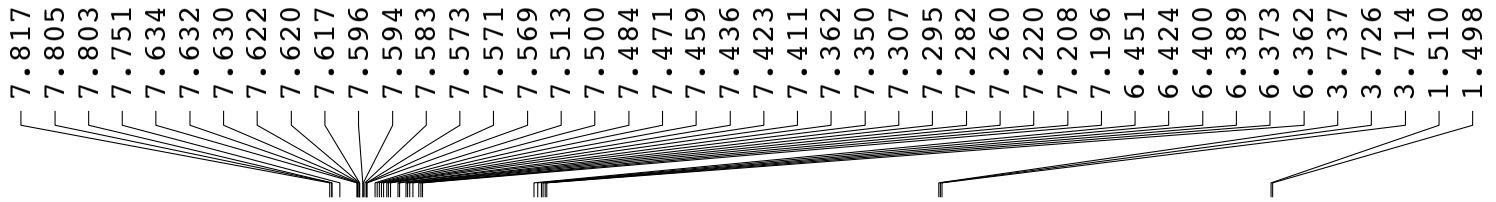

```
Current Data Parameters
NAME       QZ-HH-5-090-2
EXPNO                  2
PROCNO                 1

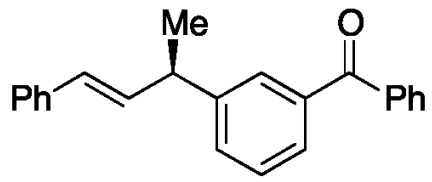
F2 - Acquisition Parameters
Date_           20140220
Time              17.00
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg55
TD                65536
SOLVENT           CDCl3
NS                  256
DS                    4
SWH           34722.223 Hz
FIDRES         0.529819 Hz
AQ            0.9437684 sec
RG                 2050
DW               14.400 usec
DE               19.34 usec
TE               298.0 K
D1            1.10000002 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        150.9656784 MHz
NUC1               13C
P1               10.63 usec

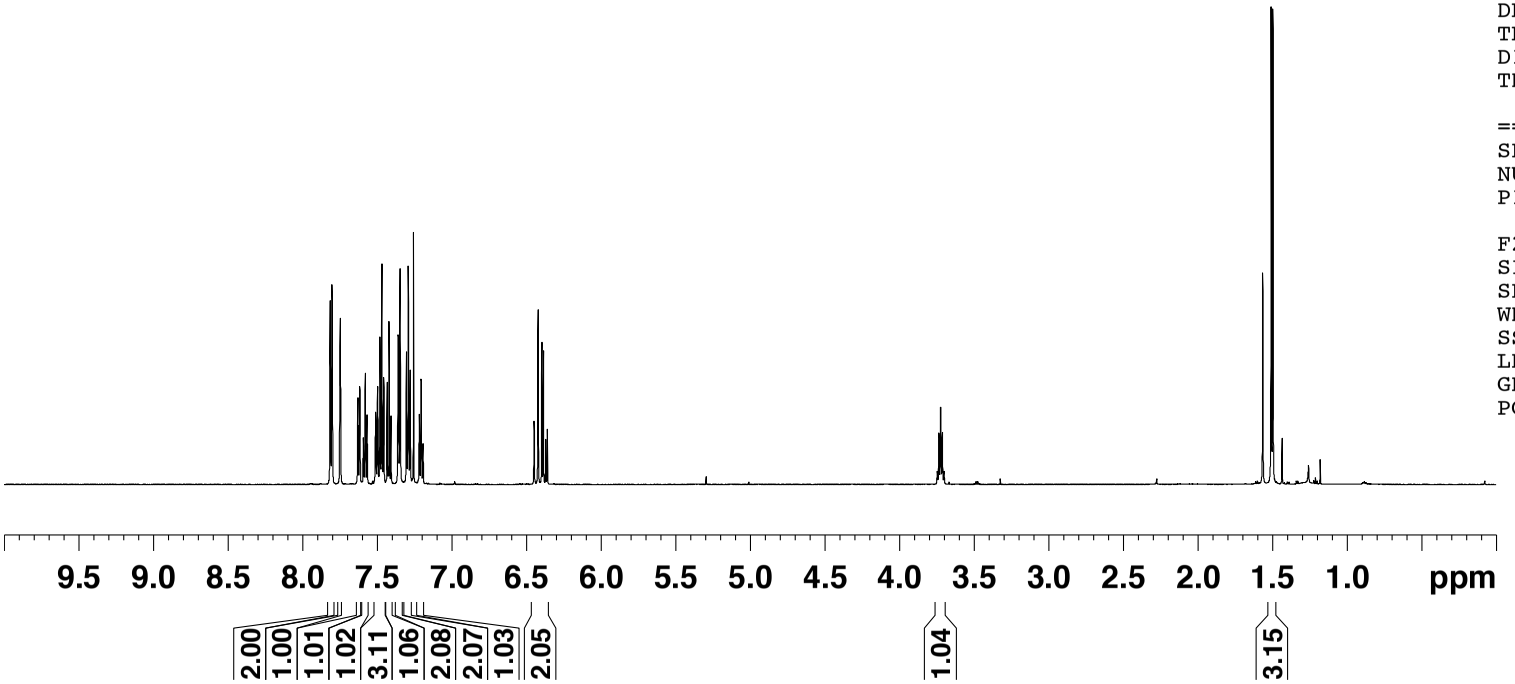
F2 - Processing parameters
SI                32768
SF          150.9505634 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40
```

compound 25 1HNMR





25



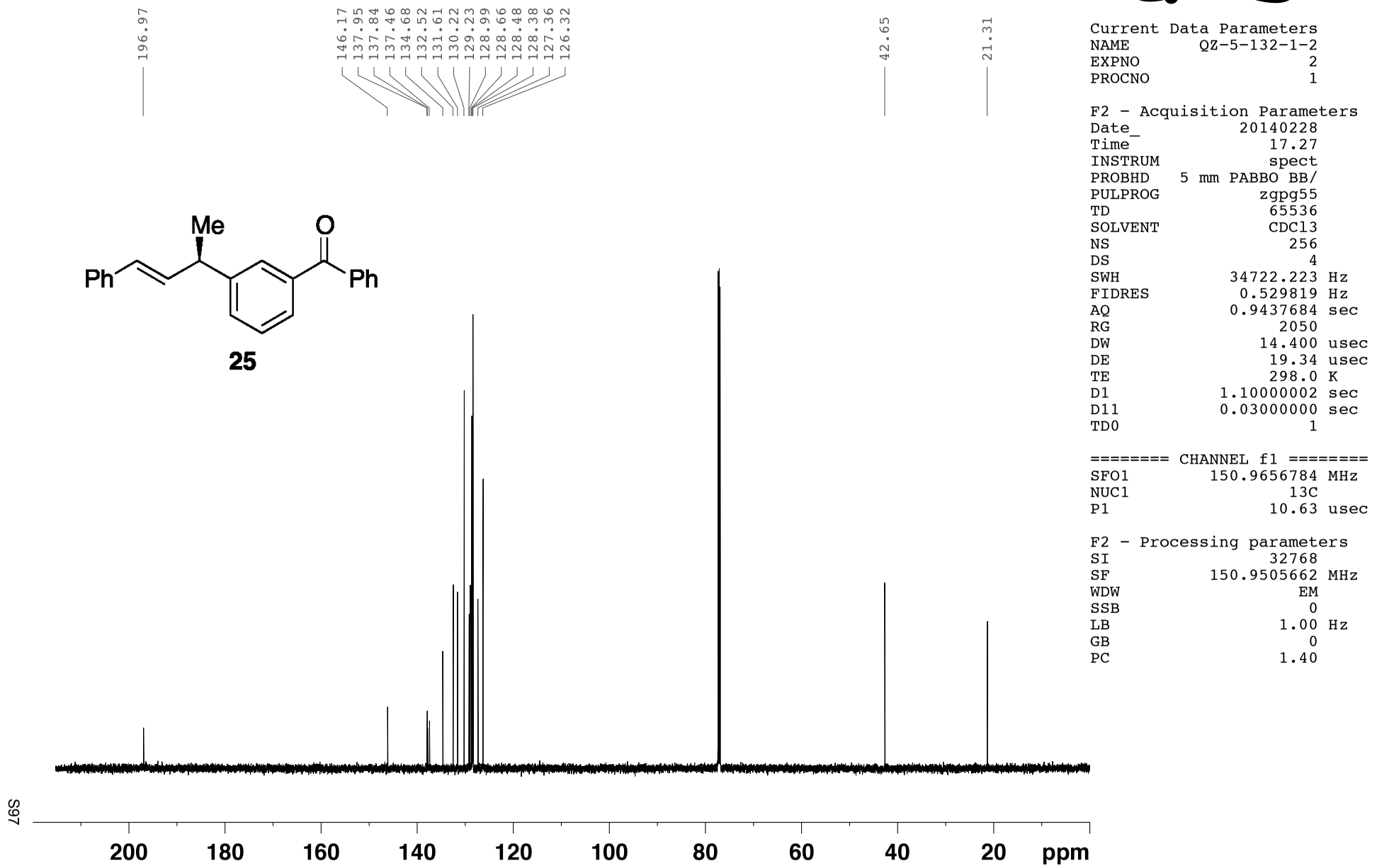
Current Data Parameters
NAME QZ-5-132-1-2
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140228
Time 17.17
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 128
DW 41.600 usec
DE 16.81 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 600.3240221 MHz
NUC1 1H
P1 10.77 usec

F2 - Processing parameters
SI 65536
SF 600.3200143 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

compound 25 13CNMR



Current Data Parameters

NAME	QZ-5-132-1-2
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20140228
Time	17.27
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	19.34 usec
TE	298.0 K
D1	1.10000002 sec
D11	0.03000000 sec
TD0	1

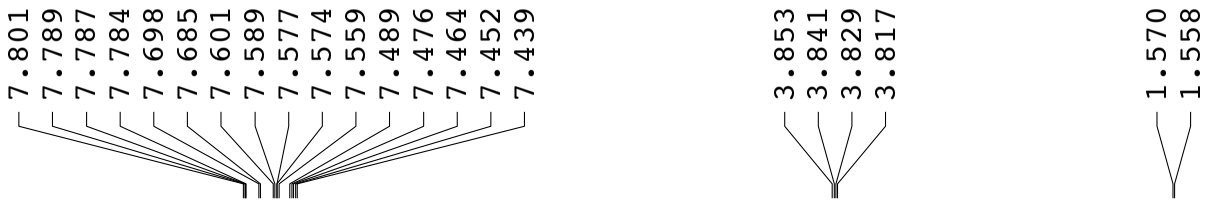
======== CHANNEL f1 ========

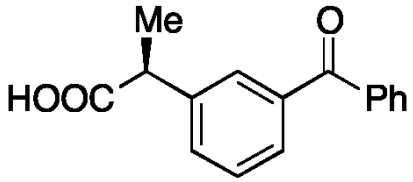
SFO1	150.9656784 MHz
NUC1	13C
P1	10.63 usec

F2 - Processing parameters

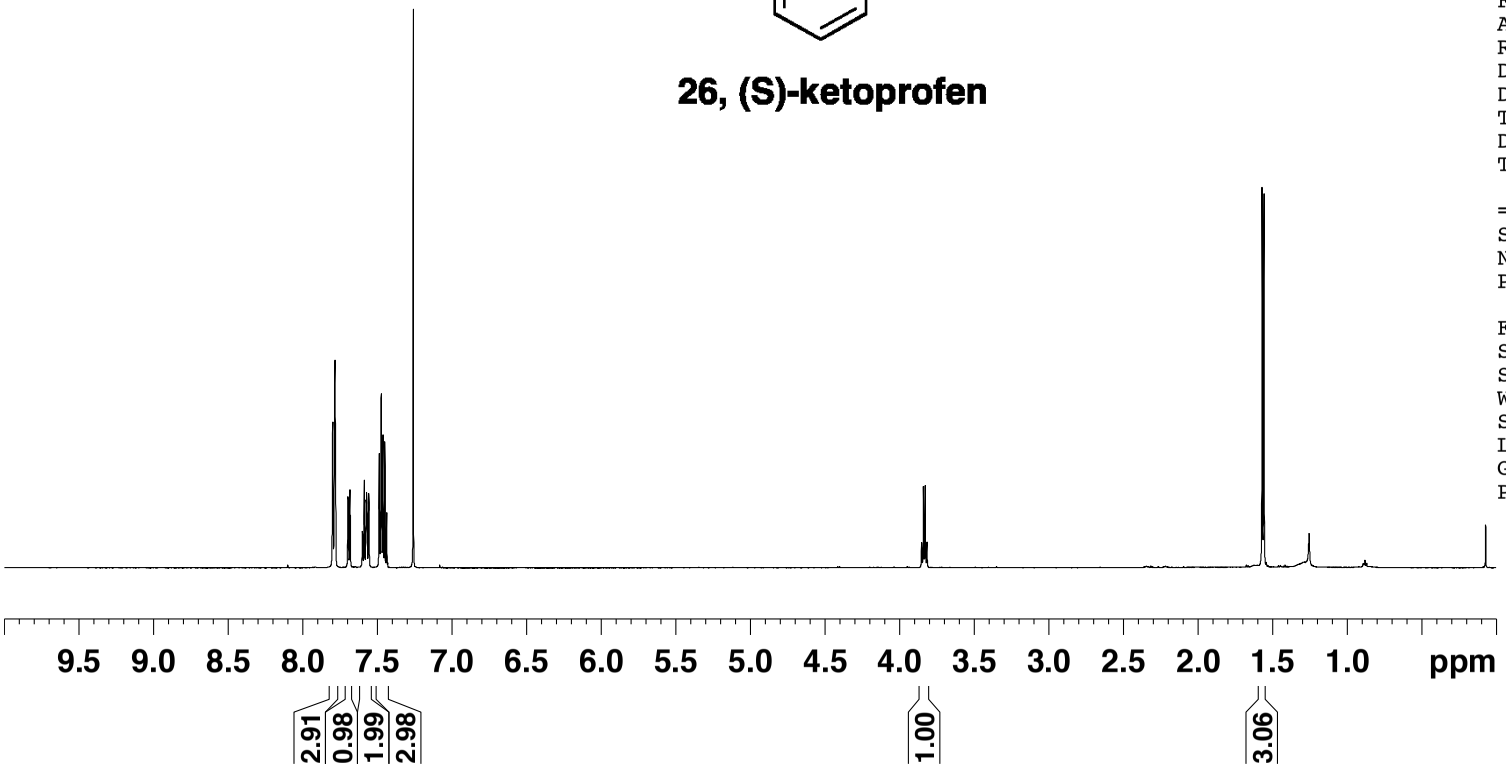
SI	32768
SF	150.9505662 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

compound 26 1HNMR (*S*)-ketoprofen





26, (S)-ketoprofen



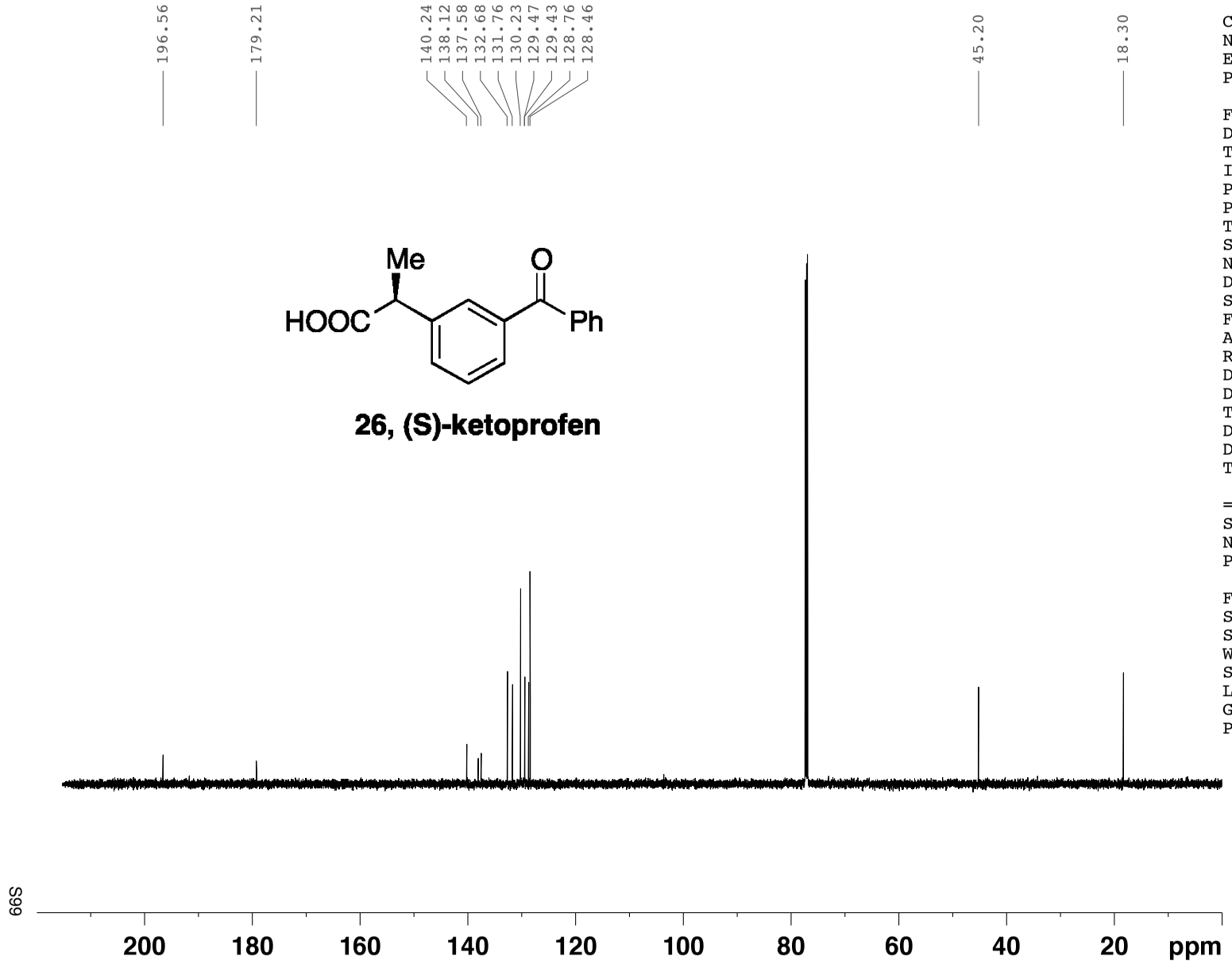

```
Current Data Parameters
NAME        QZ-5-135-2-8
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140305
Time               14.47
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH            12019.230 Hz
FIDRES          0.183399 Hz
AQ             2.7263477 sec
RG                   203
DW                41.600 usec
DE                16.81 usec
TE                298.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         600.3240221 MHz
NUC1                   1H
P1                 10.77 usec

F2 - Processing parameters
SI                 65536
SF           600.3200135 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                 1.00
```

compound 26 13CNMR (*S*)-ketoprofen



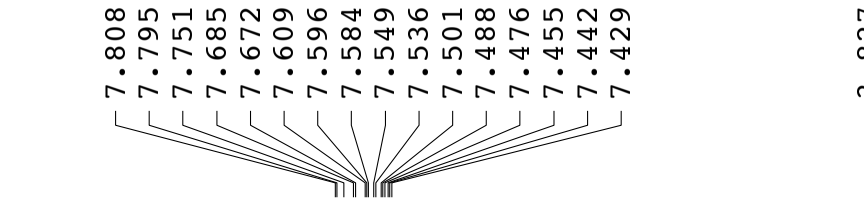

```
Current Data Parameters
NAME        QZ-5-135-2-8
EXPNO                  2
PROCNO                 1

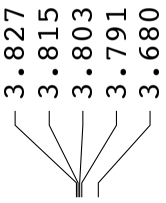
F2 - Acquisition Parameters
Date_           20140305
Time               15.00
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg55
TD                65536
SOLVENT           CDCl3
NS                  256
DS                    4
SWH           34722.223 Hz
FIDRES         0.529819 Hz
AQ            0.9437684 sec
RG                 2050
DW               14.400 usec
DE               19.34 usec
TE               298.0 K
D1            1.10000002 sec
D11           0.03000000 sec
TD0                   1

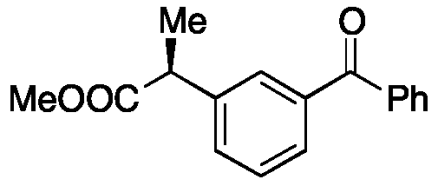
======== CHANNEL f1 ========
SFO1        150.9656784 MHz
NUC1                13C
P1                10.63 usec

F2 - Processing parameters
SI                32768
SF          150.9505633 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

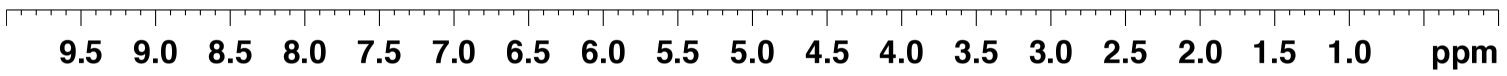
compound S-29 1HNMR







S-29



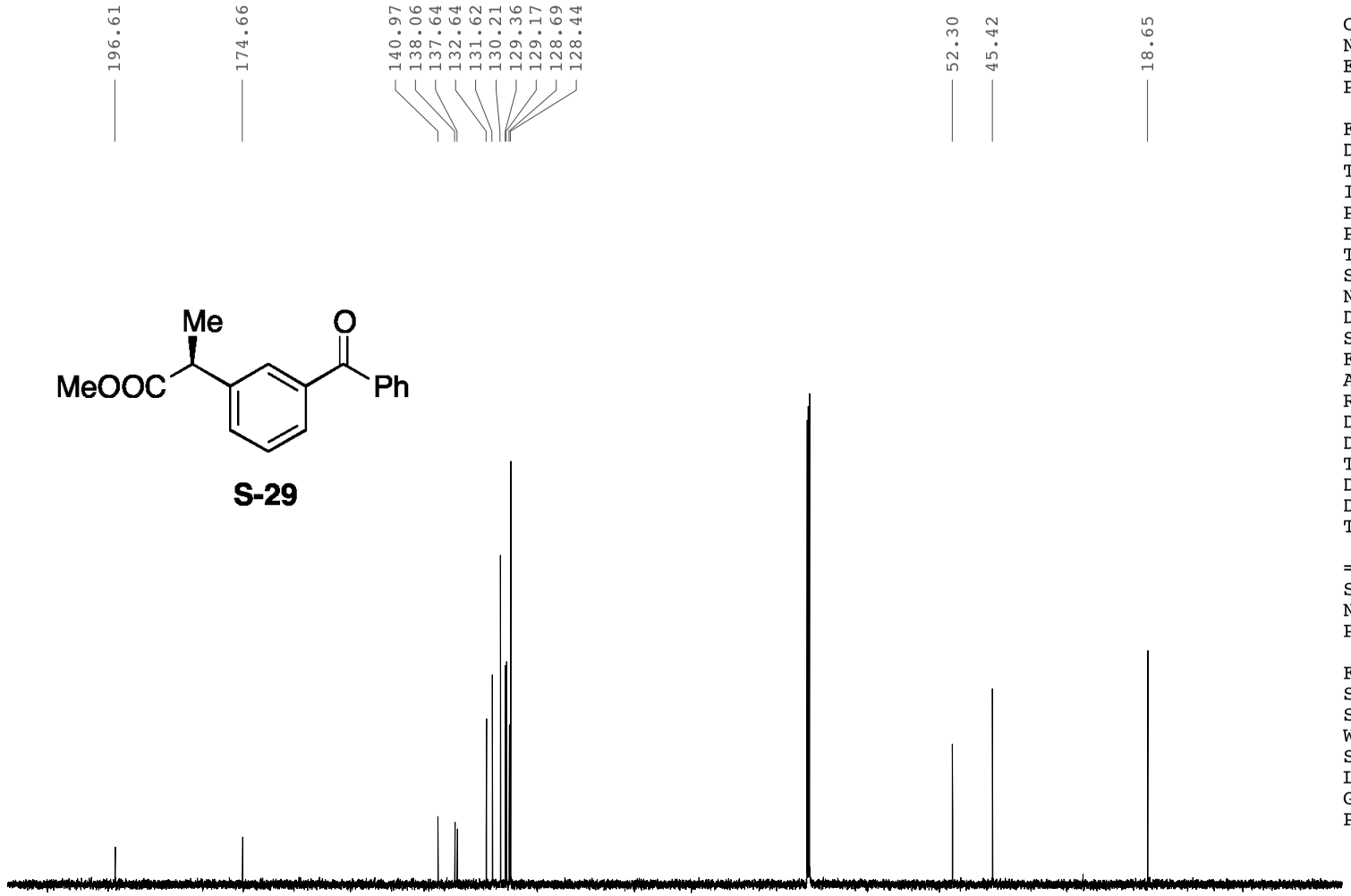

```
Current Data Parameters
NAME      QZ-5-137-1NEW
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20140318
Time              10.55
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    8
DS                    2
SWH           12019.230 Hz
FIDRES         0.183399 Hz
AQ            2.7263477 sec
RG                  128
DW               41.600 usec
DE               16.81 usec
TE               298.0 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        600.3240221 MHz
NUC1                1H
P1                10.77 usec

F2 - Processing parameters
SI                65536
SF          600.3200135 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

compound 31 13CNMR



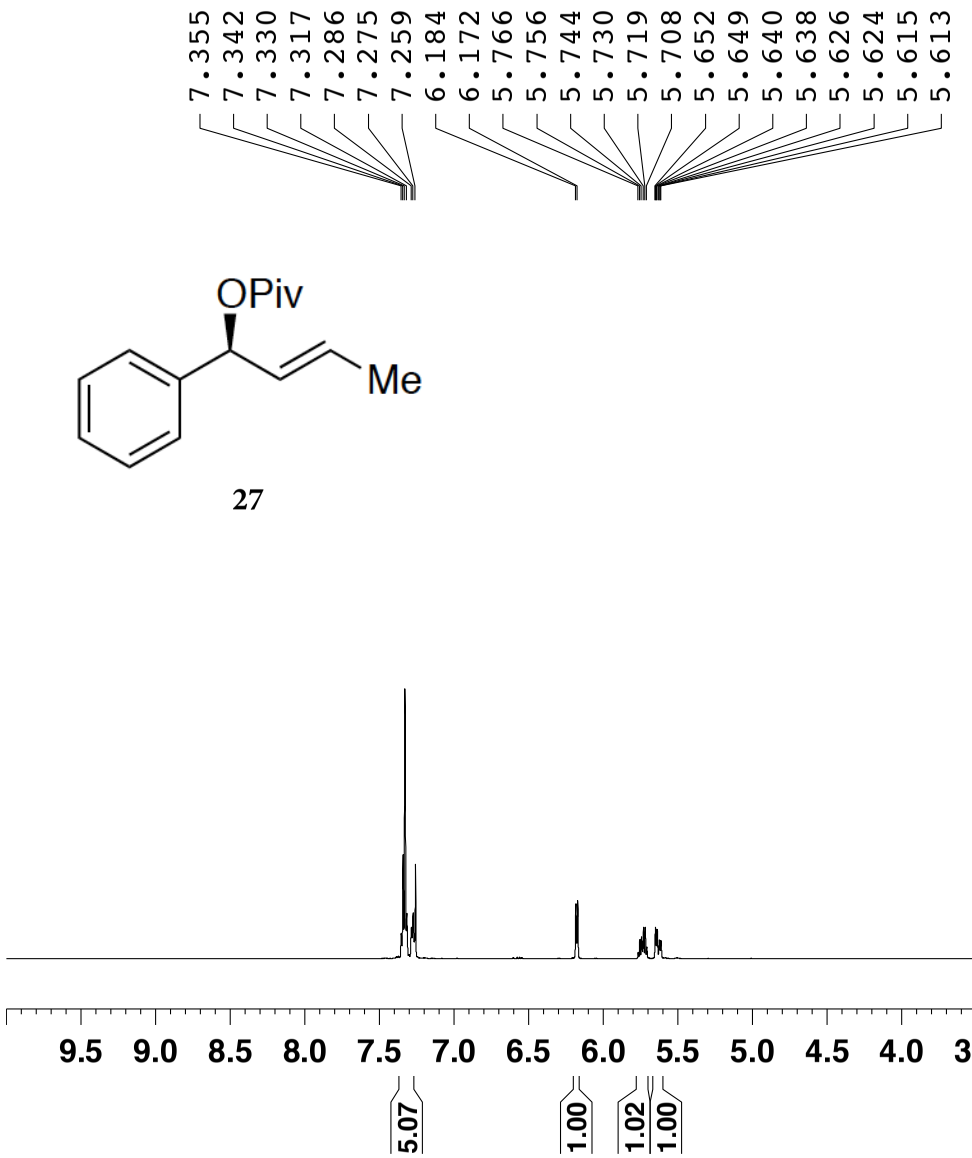
Current Data Parameters
NAME QZ-5-137-1NEW
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140318
Time 11.05
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 19.34 usec
TE 298.0 K
D1 1.10000002 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9656784 MHz
NUC1 13C
P1 10.63 usec

F2 - Processing parameters
SI 32768
SF 150.9505656 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

compound 27 1HNMR



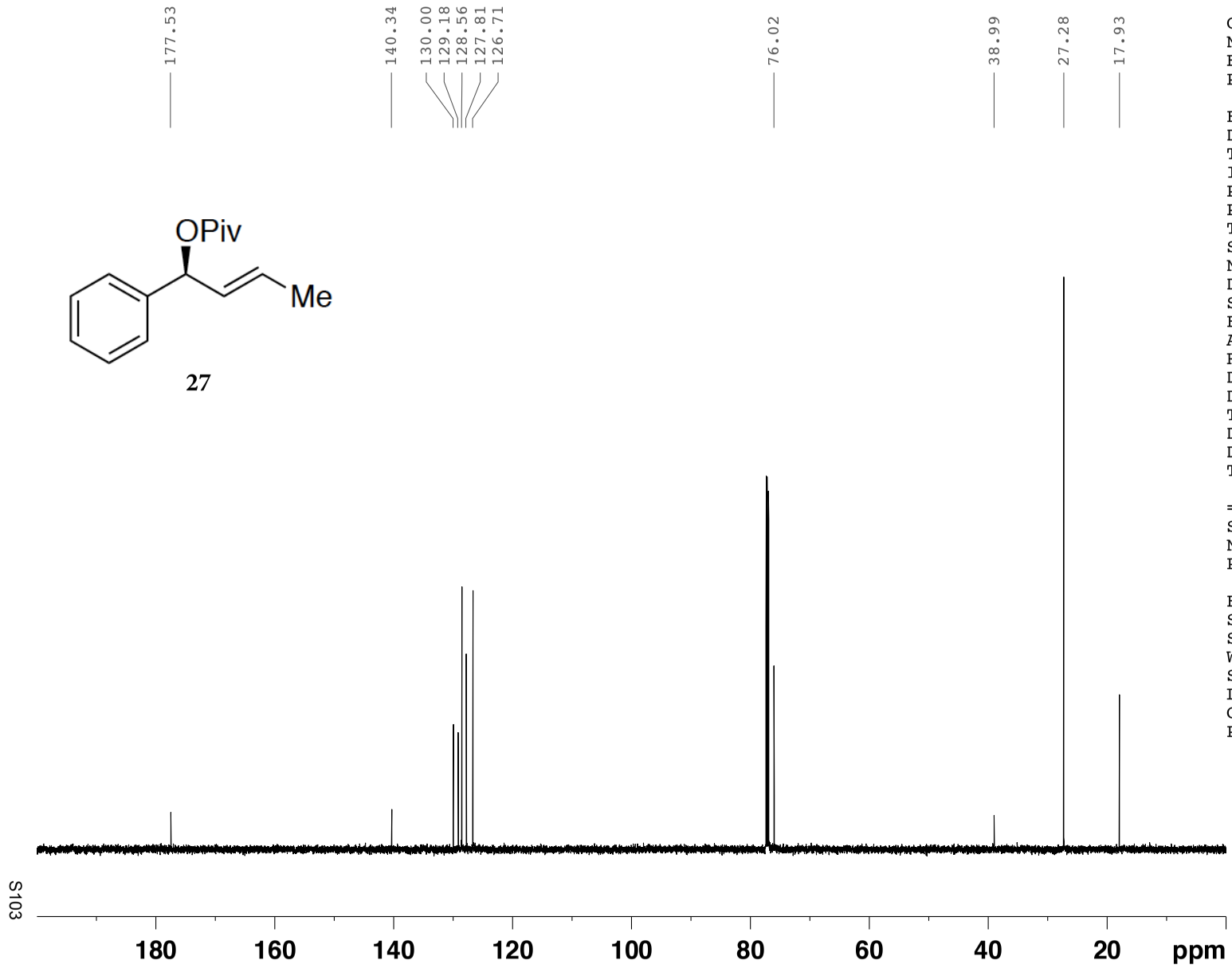

```
Current Data Parameters
NAME      QZ-3-239-1-NEW
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20140108
Time              12.21
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                  128
DW                59.500 usec
DE                17.39 usec
TE                298.0 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        600.3233018 MHz
NUC1                  1H
P1                10.77 usec

F2 - Processing parameters
SI                65536
SF          600.3200150 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

compound 27 13CNMR



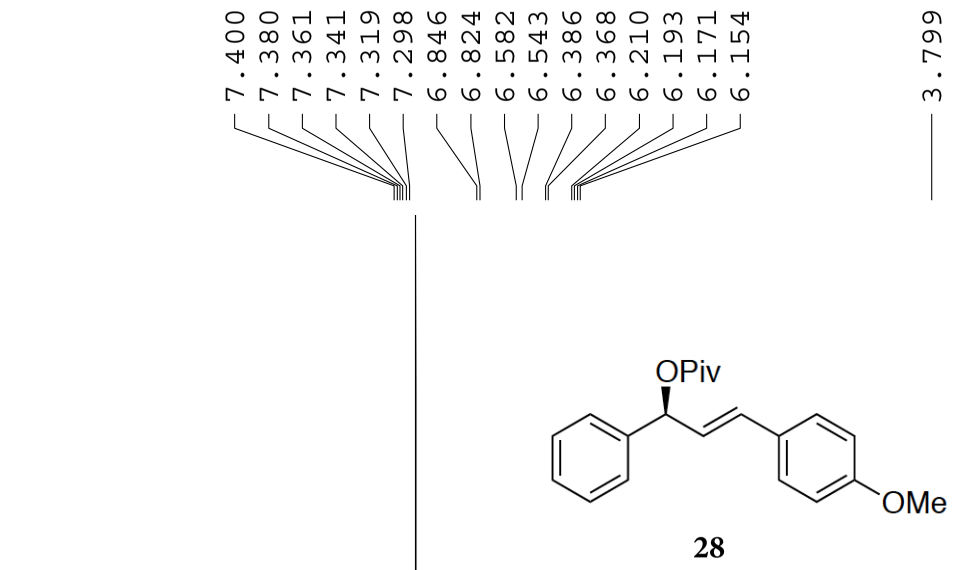

```
Current Data Parameters
NAME      QZ-3-239-1-NEW
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_            20140108
Time                12.32
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                19.34 usec
TE                298.0 K
D1            1.10000002 sec
D11           0.03000000 sec
TD0                    1

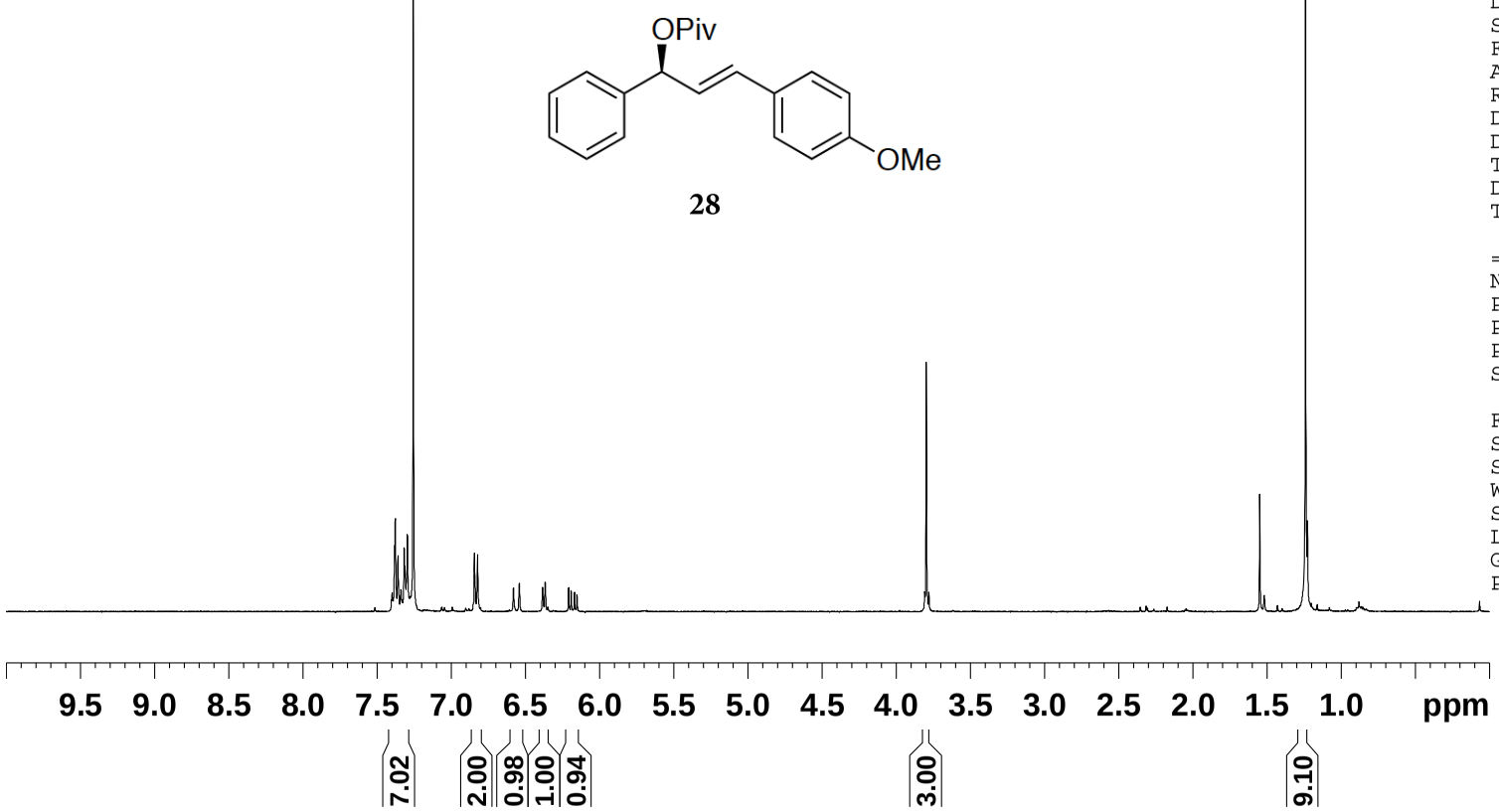
======== CHANNEL f1 ========
SFO1         150.9656784 MHz
NUC1                  13C
P1                 10.63 usec

F2 - Processing parameters
SI                 32768
SF           150.9505639 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

compound 28 1HNMR



28



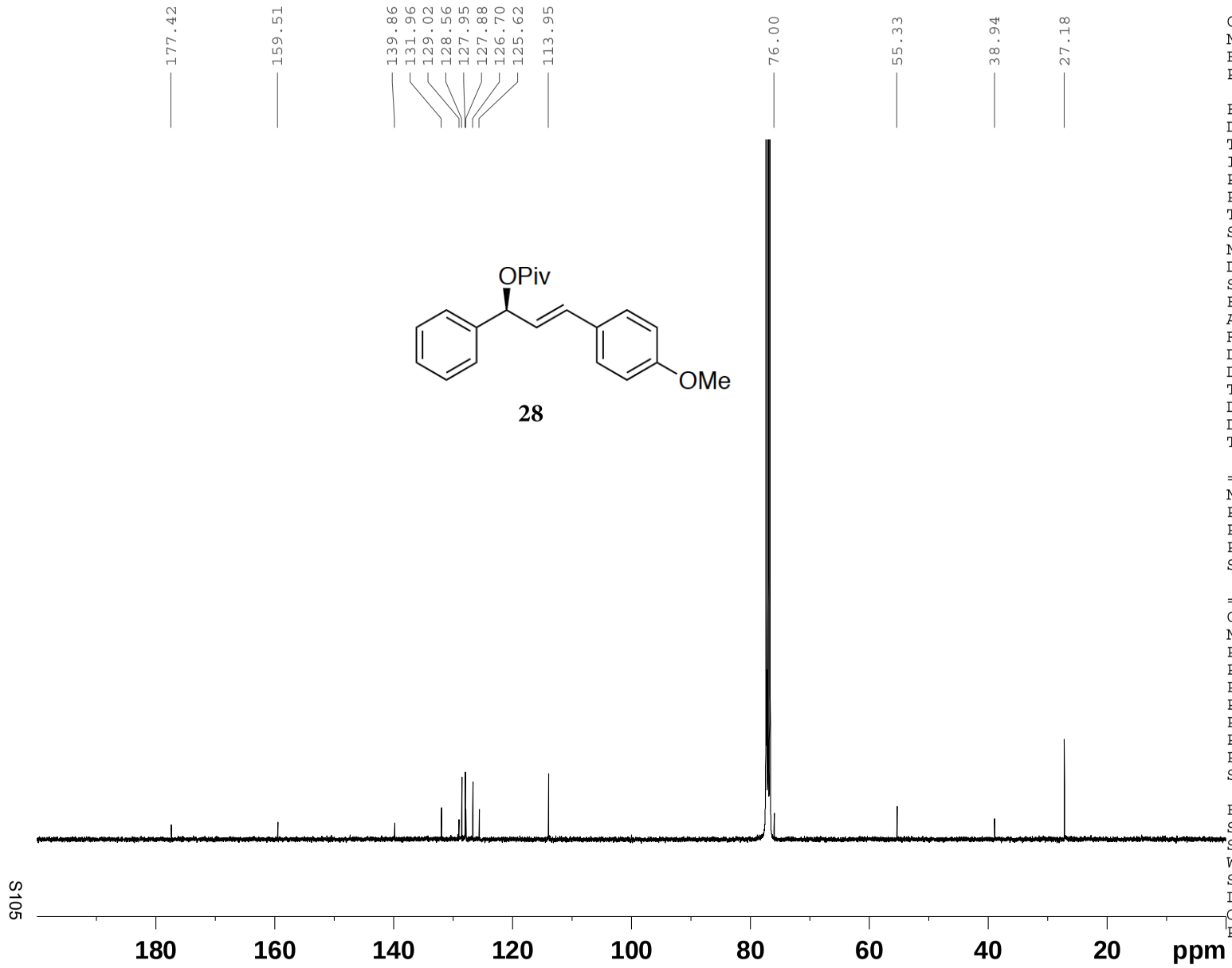

```
Current Data Parameters
NAME         QZ-5-009fr1
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20131121
Time               18.18
INSTRUM            spect
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                298.1 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                15.00 usec
PL1                4.90 dB
PL1W          3.30822015 W
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF           400.1300105 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```

compound 28 13CNMR




```
Current Data Parameters
NAME         QZ-5-009fr1
EXPNO                  2
PROCNO                 1

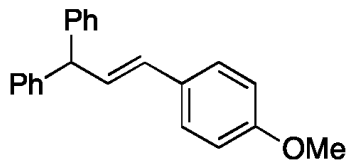
F2 - Acquisition Parameters
Date_           20131121
Time               22.06
INSTRUM            spect
PROBHD   5 mm CPQNP 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ             1.3664756 sec
RG                  512
DW               20.850 usec
DE               18.00 usec
TE                298.2 K
D1           2.00000000 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

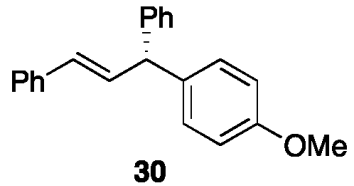
F2 - Processing parameters
SI                32768
SF          100.6127690 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC                1.40
```

29 and enantioenriched **30**



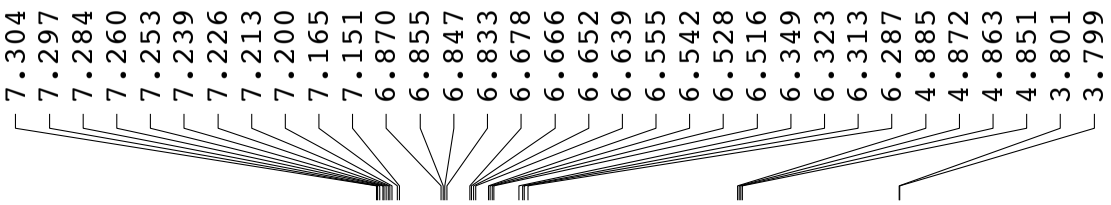
29

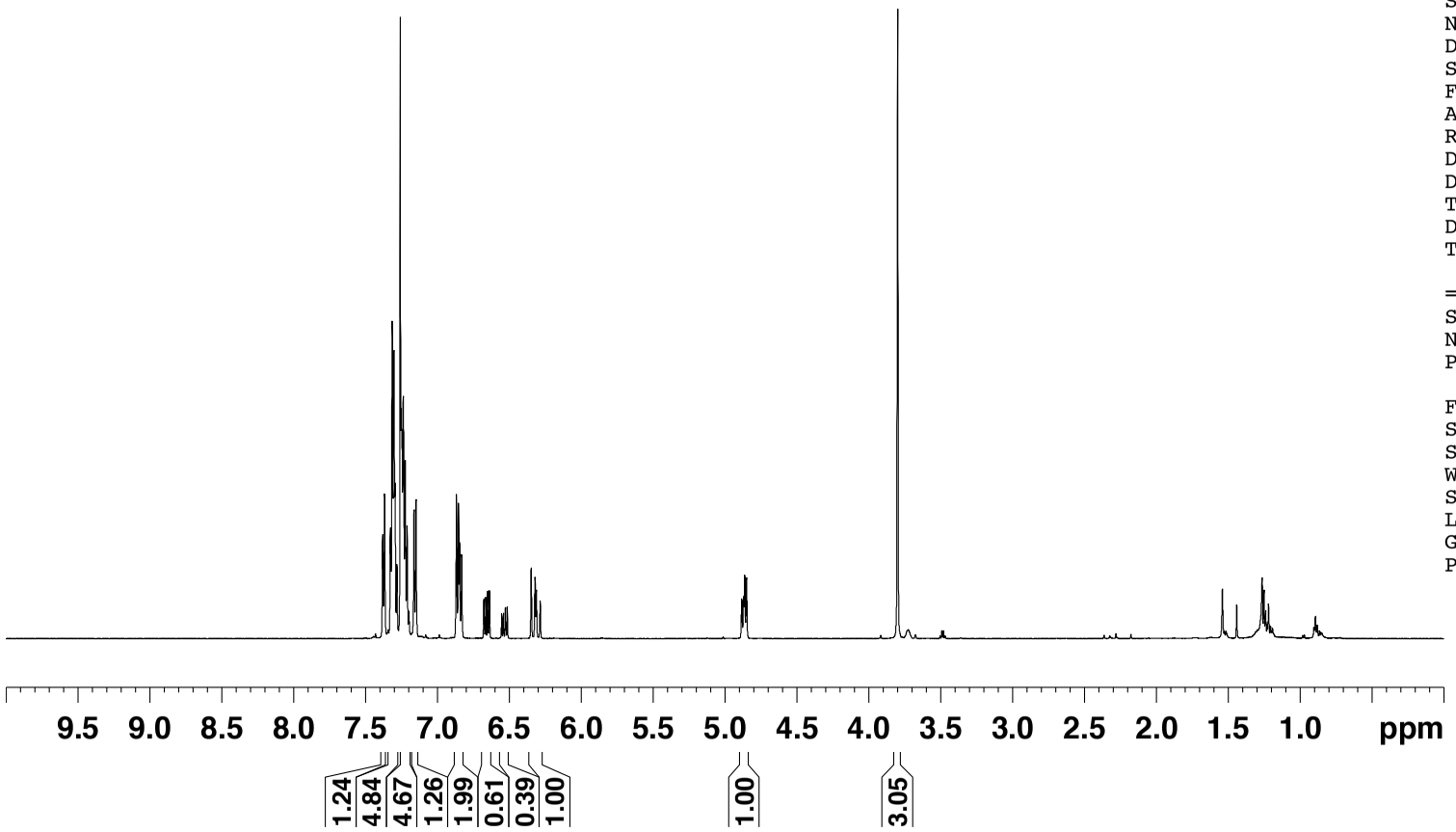
:



30

1 : 1.5





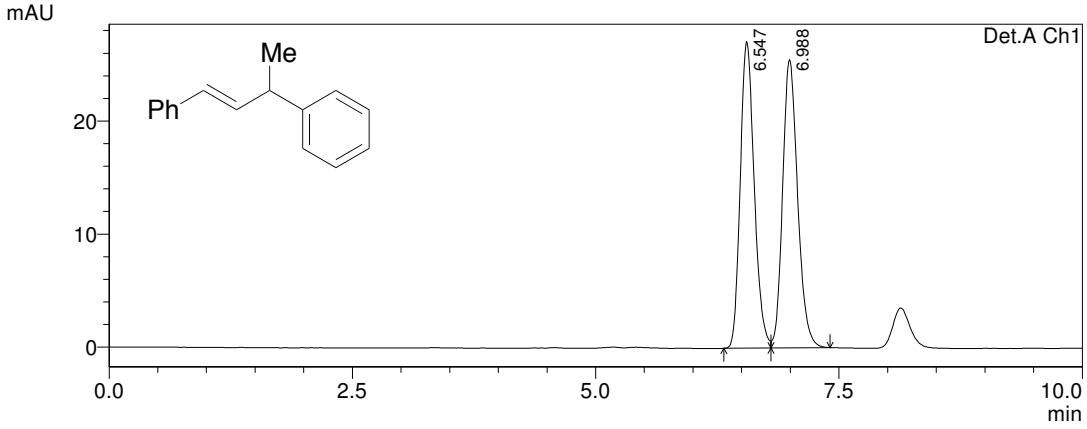

```
Current Data Parameters
NAME          QZ-5-123-2
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140214
Time               13.20
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                17.39 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         600.3233018 MHz
NUC1                   1H
P1                 10.77 usec

F2 - Processing parameters
SI                 65536
SF           600.3200141 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```

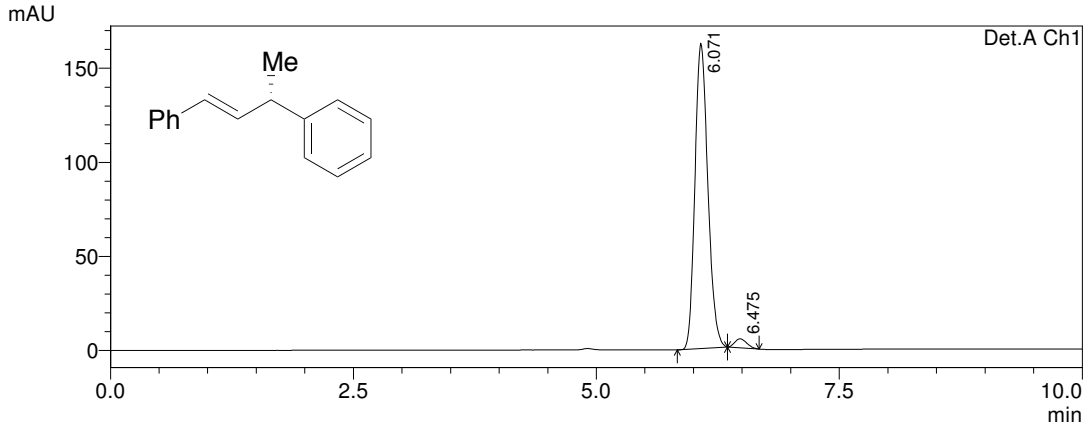
Racemic **2**



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.547	270789	27126	49.766	51.556
2	6.988	273337	25488	50.234	48.444
Total		544126	52614	100.000	100.000

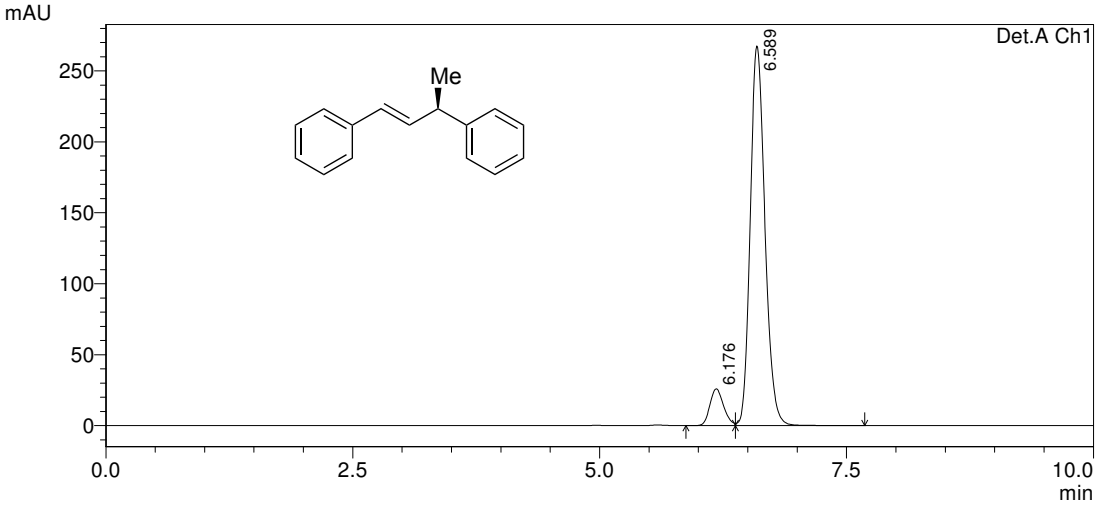
Enantioenriched **2**, ee = 95%



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.071	1497700	162221	97.383	97.181
2	6.475	40241	4706	2.617	2.819
Total		1537941	166928	100.000	100.000

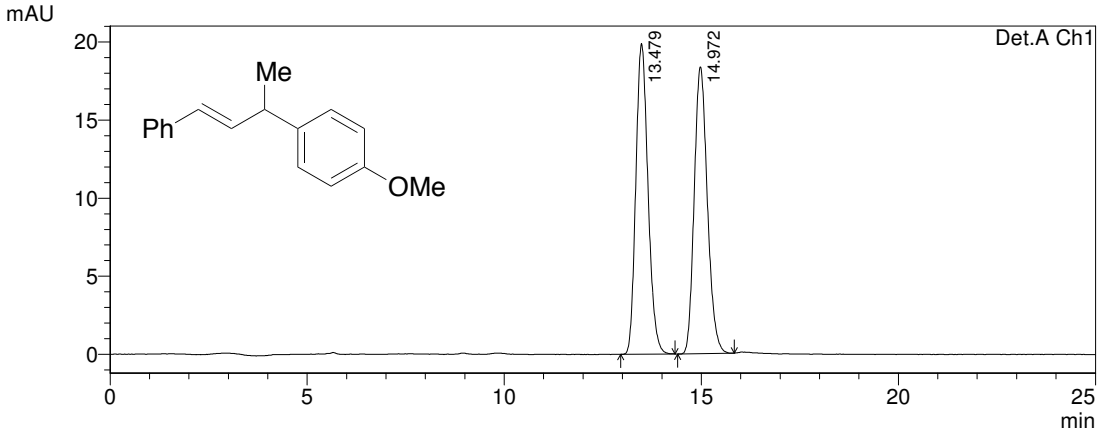
Enantioenriched *(R)*-**2**, prepared from **27**, as shown in Scheme 5, ee = 83%



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.176	249802	25887	8.275	8.818
2	6.589	2768940	267665	91.725	91.182
Total		3018741	293552	100.000	100.000

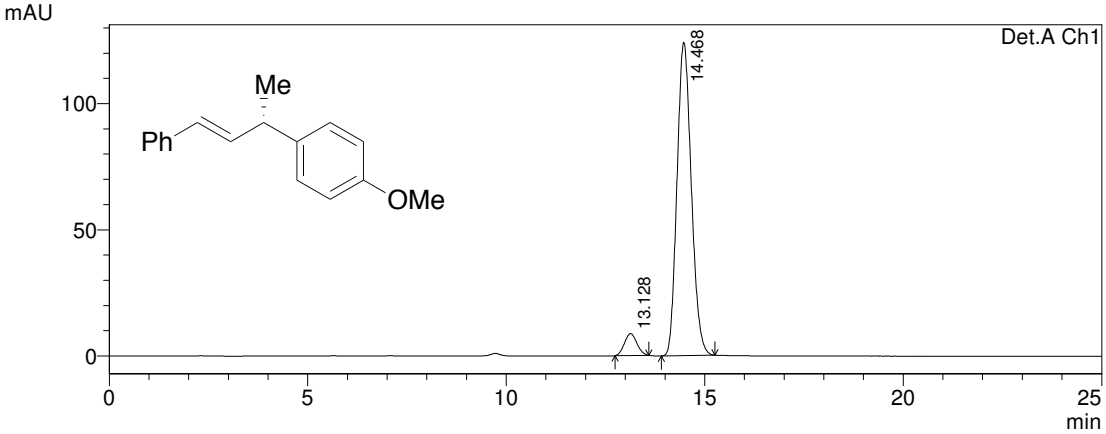
Racemic **3**



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	13.479	427846	19884	50.127	51.998
2	14.972	425680	18356	49.873	48.002
Total		853526	38240	100.000	100.000

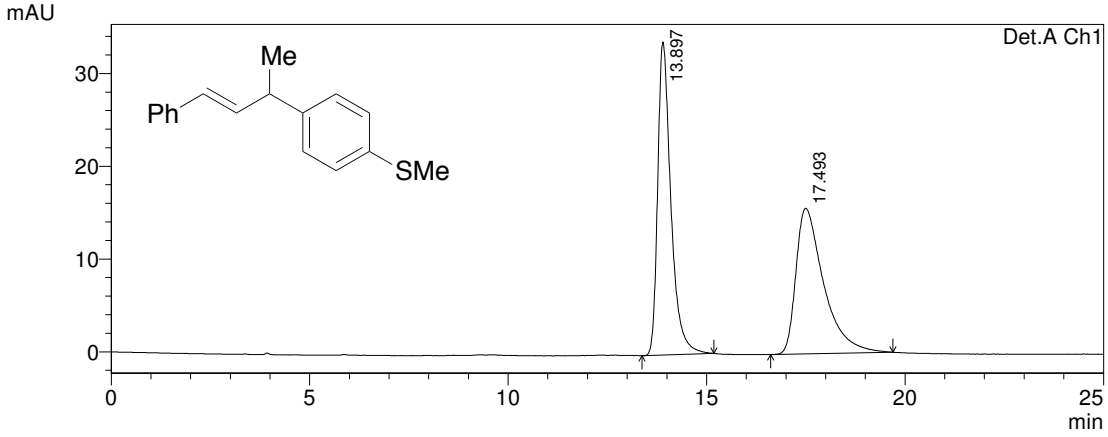
Enantioenriched **3**, ee = 89%



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	13.128	189418	8679	5.739	6.535
2	14.468	3111331	124131	94.261	93.465
Total		3300749	132811	100.000	100.000

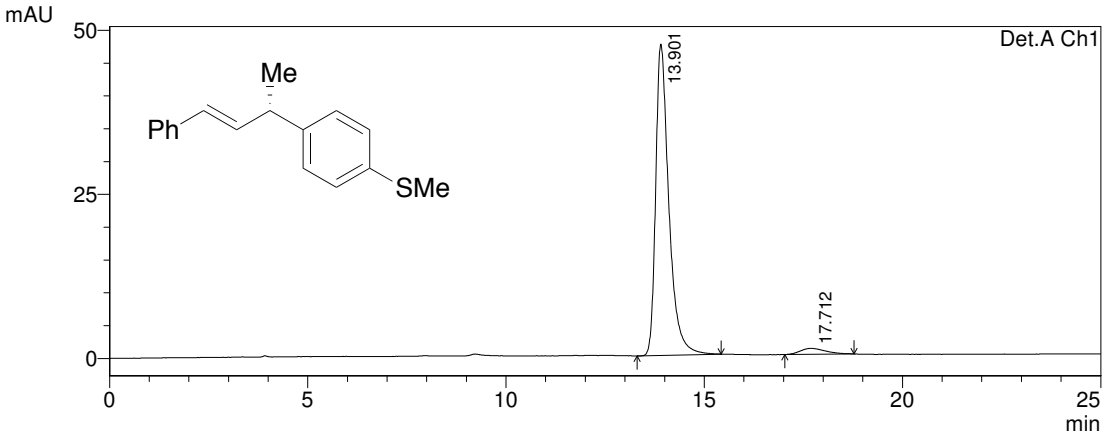
Racemic **4**



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	13.897	780775	33745	50.587	68.263
2	17.493	762670	15688	49.413	31.737
Total		1543445	49433	100.000	100.000

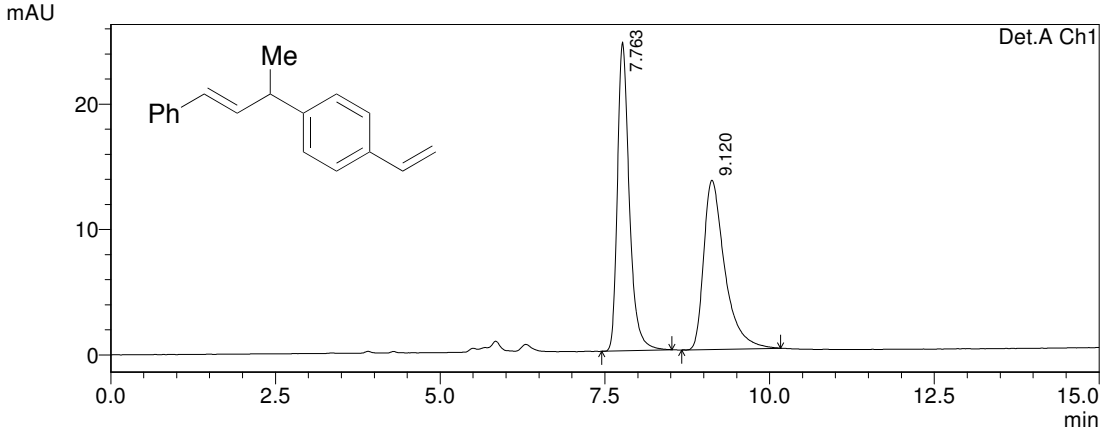
Enantioenriched **4**, ee = 93%



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	13.901	1093788	47430	96.408	98.119
2	17.712	40748	909	3.592	1.881
Total		1134537	48339	100.000	100.000

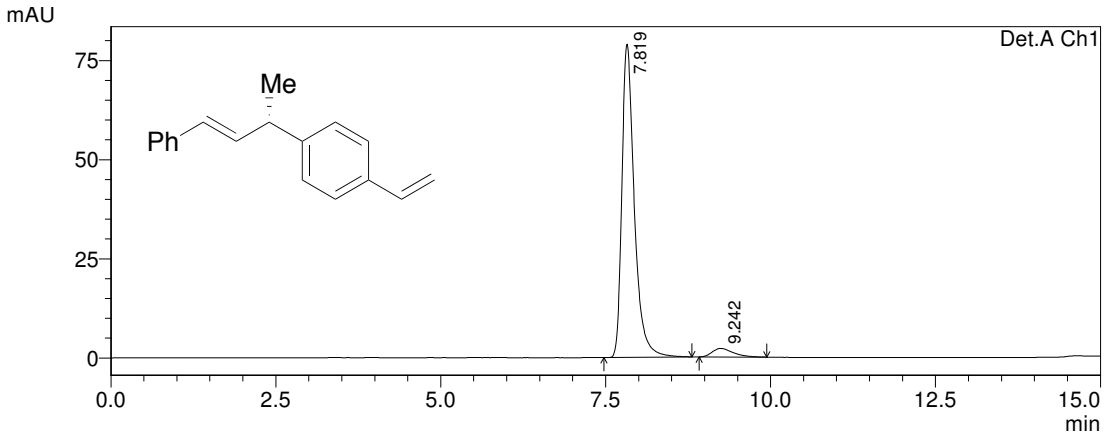
Racemic **5**



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.763	309122	24632	50.797	64.592
2	9.120	299424	13503	49.203	35.408
Total		608545	38135	100.000	100.000

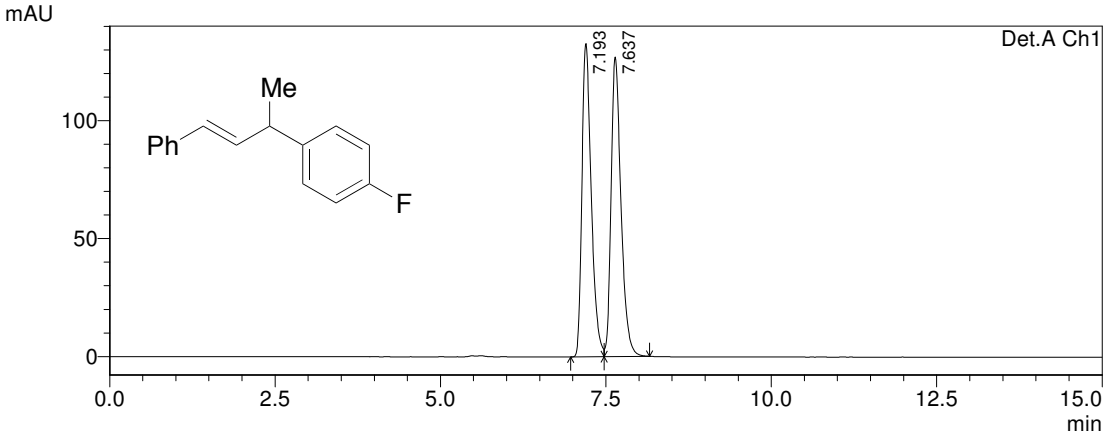
Enantioenriched **5**, ee = 91%



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.819	1036677	78979	95.516	97.324
2	9.242	48666	2171	4.484	2.676
Total		1085343	81150	100.000	100.000

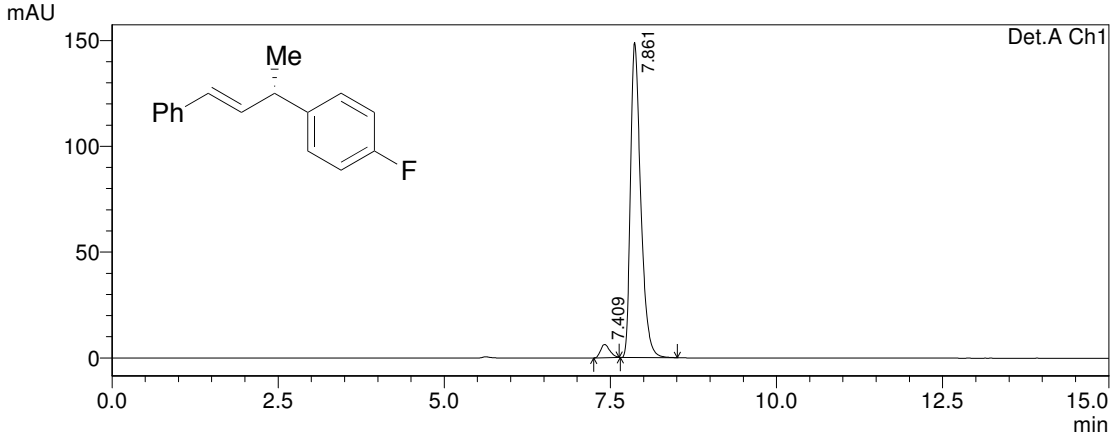
Racemic **6**



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.193	1303841	132756	49.668	51.098
2	7.637	1321281	127053	50.332	48.902
Total		2625122	259810	100.000	100.000

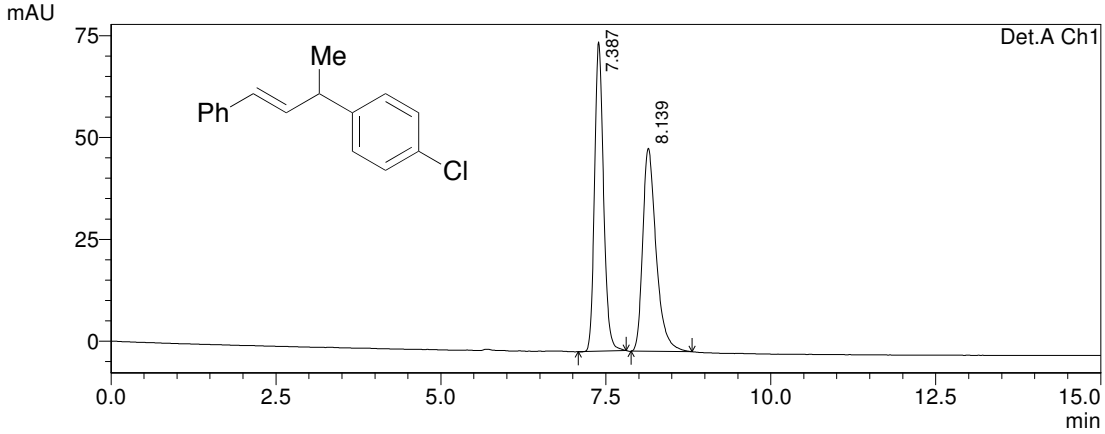
Enantioenriched **6**, ee = 93%



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.409	59690	6261	3.607	4.036
2	7.861	1595262	148864	96.393	95.964
Total		1654951	155125	100.000	100.000

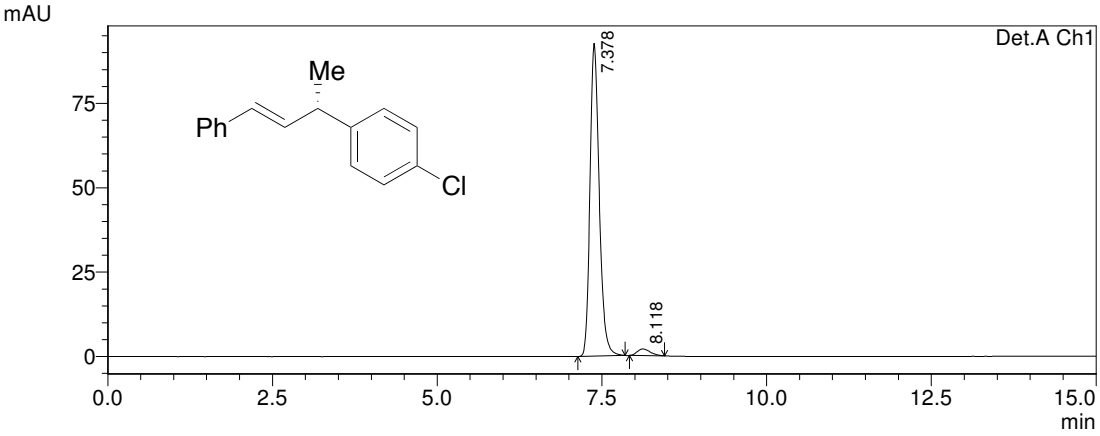
Racemic **7**



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.387	719087	75904	50.501	60.370
2	8.139	704825	49826	49.499	39.630
Total		1423912	125730	100.000	100.000

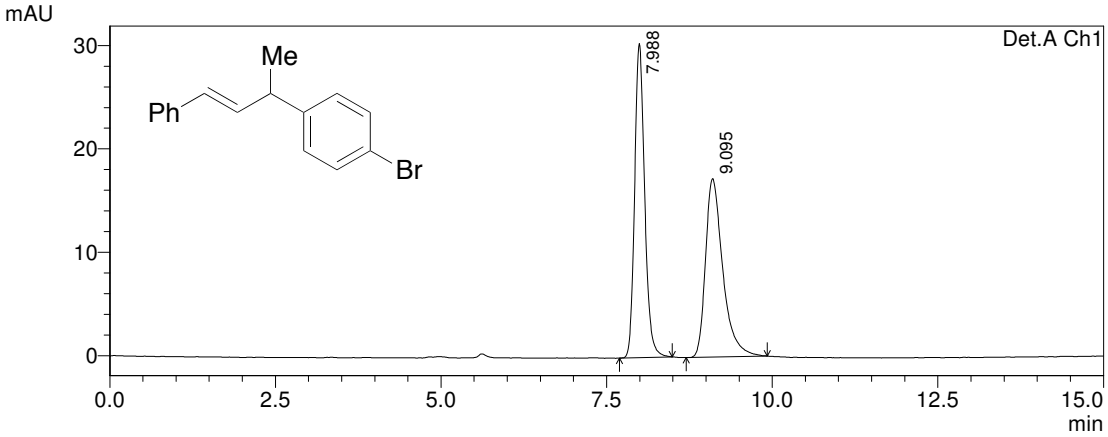
Enantioenriched **7**, ee = 94%



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.378	904758	92637	97.094	97.924
2	8.118	27077	1964	2.906	2.076
Total		931836	94601	100.000	100.000

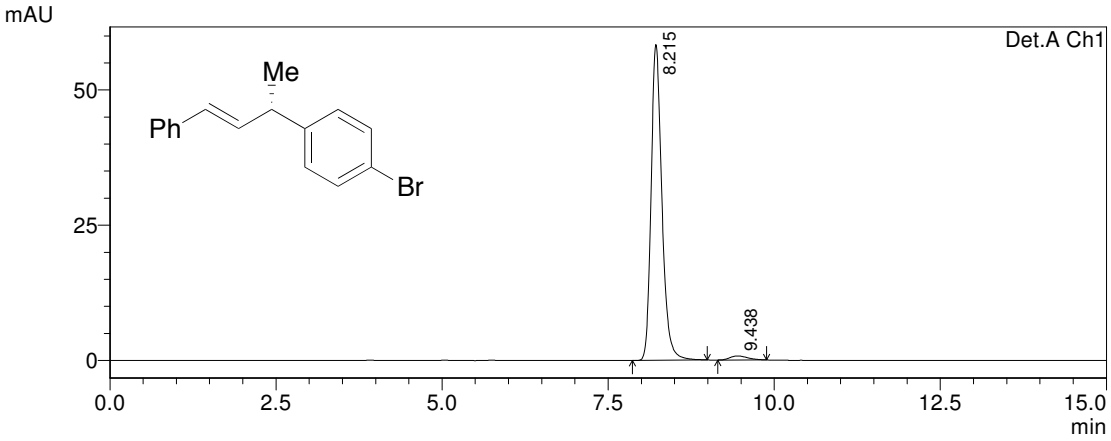
Racemic **8**



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.988	320442	30359	50.720	63.778
2	9.095	311345	17242	49.280	36.222
Total		631787	47601	100.000	100.000

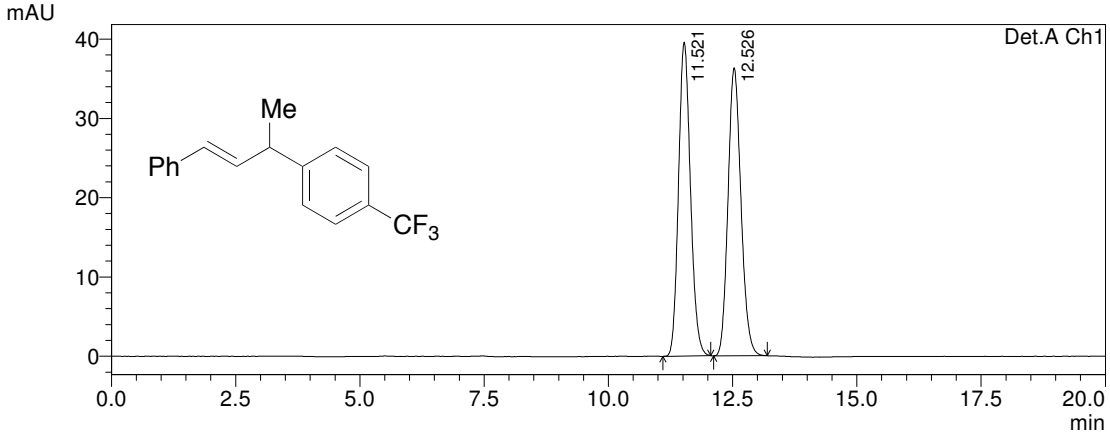
Enantioenriched **8**, ee = 96%



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.215	661539	58358	97.938	98.732
2	9.438	13930	749	2.062	1.268
Total		675469	59108	100.000	100.000

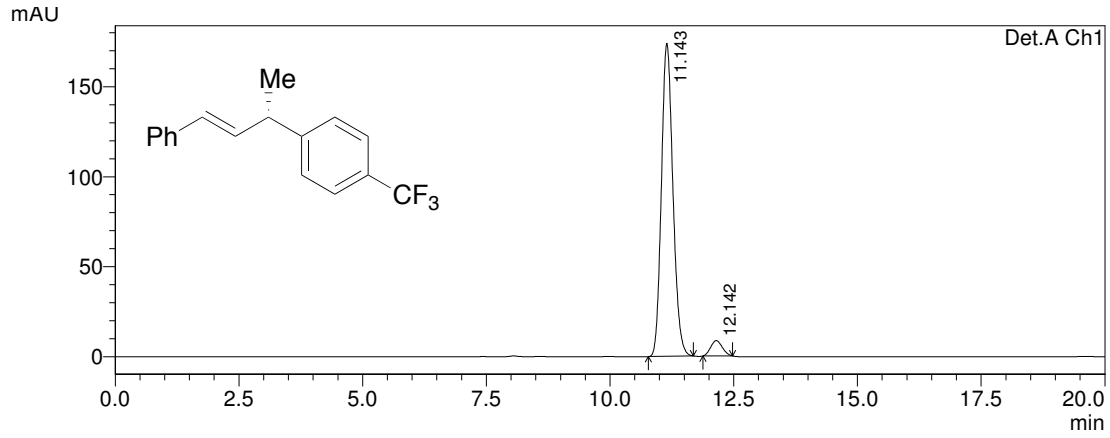
Racemic **9**



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	11.521	660610	39588	50.006	52.136
2	12.526	660455	36345	49.994	47.864
Total		1321065	75933	100.000	100.000

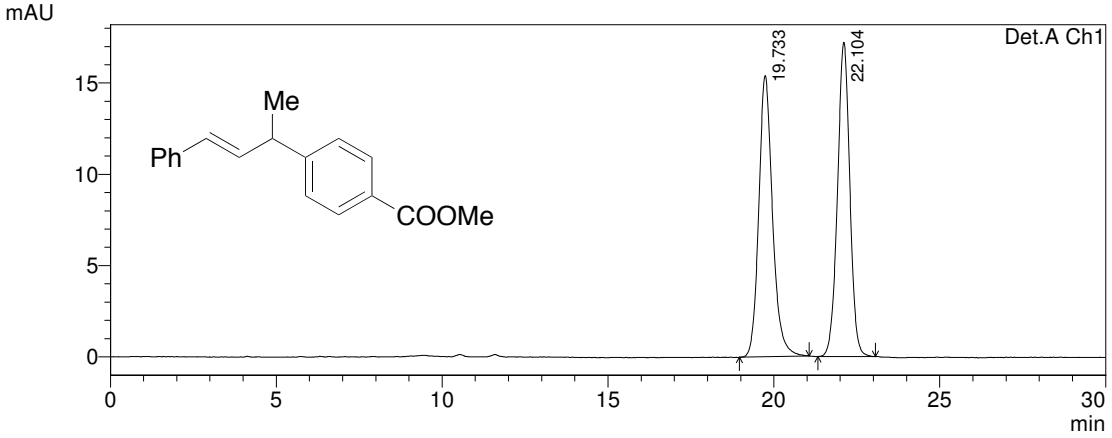
Enantioenriched **9**, ee = 91%



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	11.143	2817309	173908	95.290	95.325
2	12.142	139267	8530	4.710	4.675
Total		2956576	182438	100.000	100.000

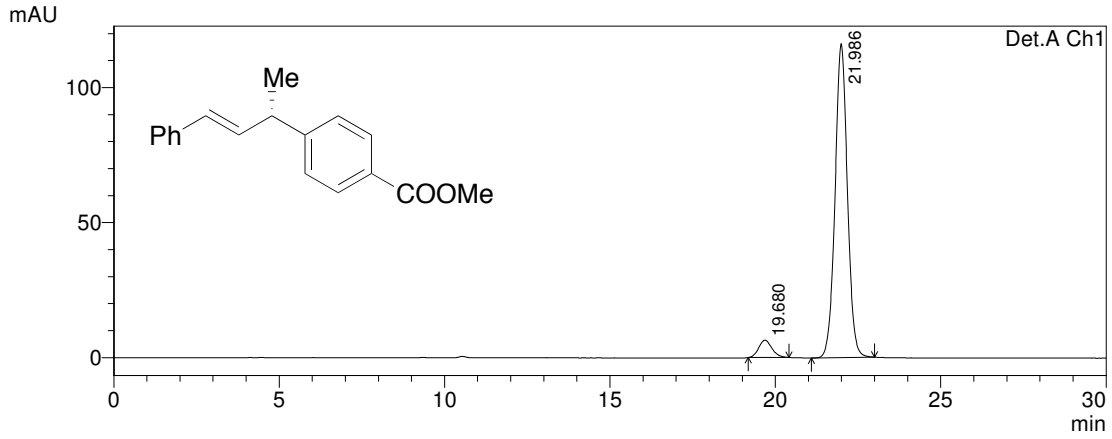
Racemic **10**



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	19.733	444003	15399	49.901	47.223
2	22.104	445768	17211	50.099	52.777
Total		889771	32610	100.000	100.000

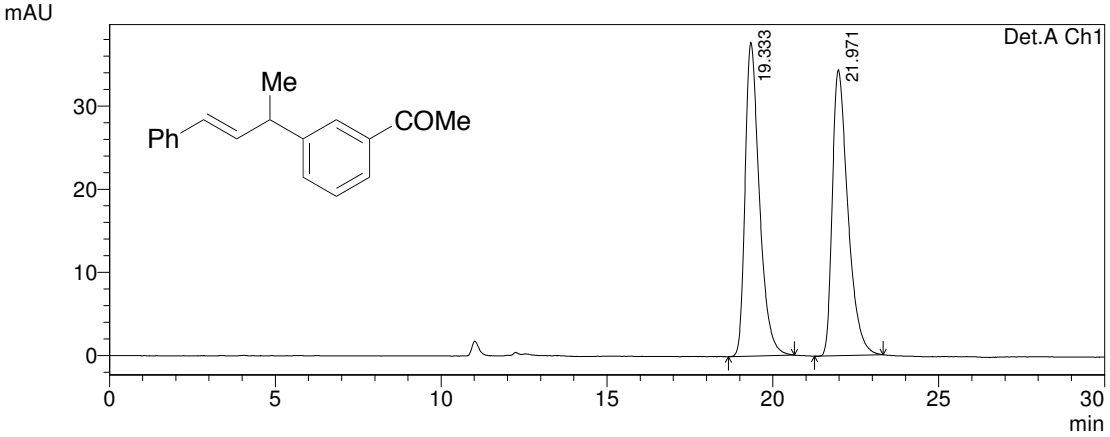
Enantioenriched **10**, ee = 89%



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	19.680	178540	6440	5.592	5.249
2	21.986	3014453	116234	94.408	94.751
Total		3192993	122673	100.000	100.000

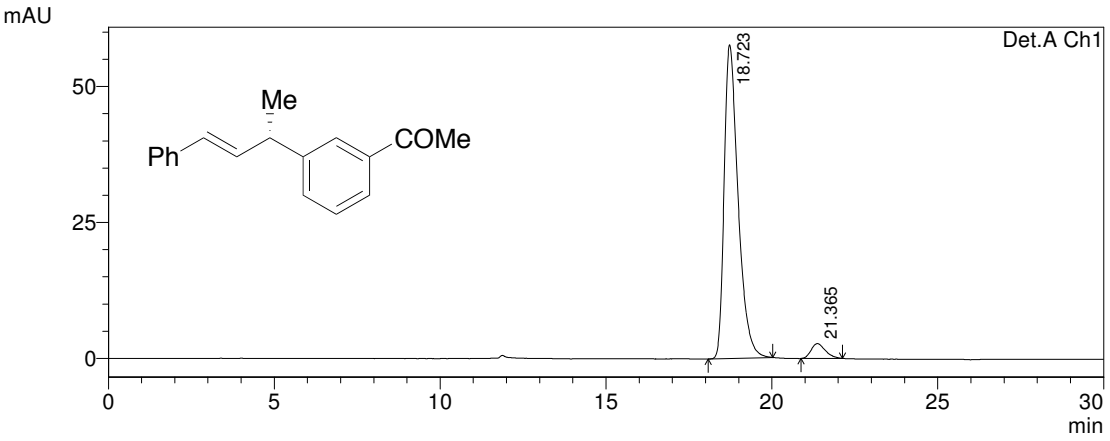
Racemic **11**



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	19.333	1126904	37748	50.126	52.323
2	21.971	1121253	34396	49.874	47.677
Total		2248157	72144	100.000	100.000

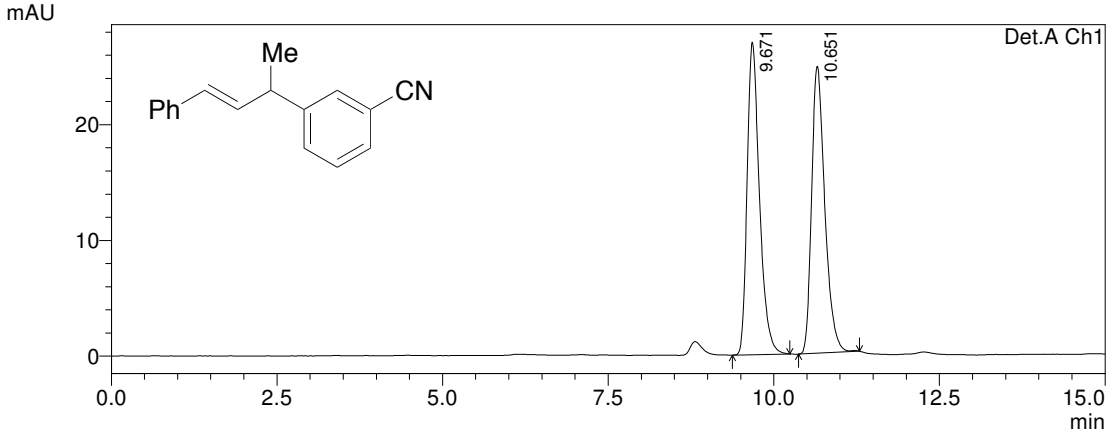
Enantioenriched **11**, ee = 91%



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	18.723	1683869	57676	95.364	95.458
2	21.365	81861	2744	4.636	4.542
Total		1765730	60420	100.000	100.000

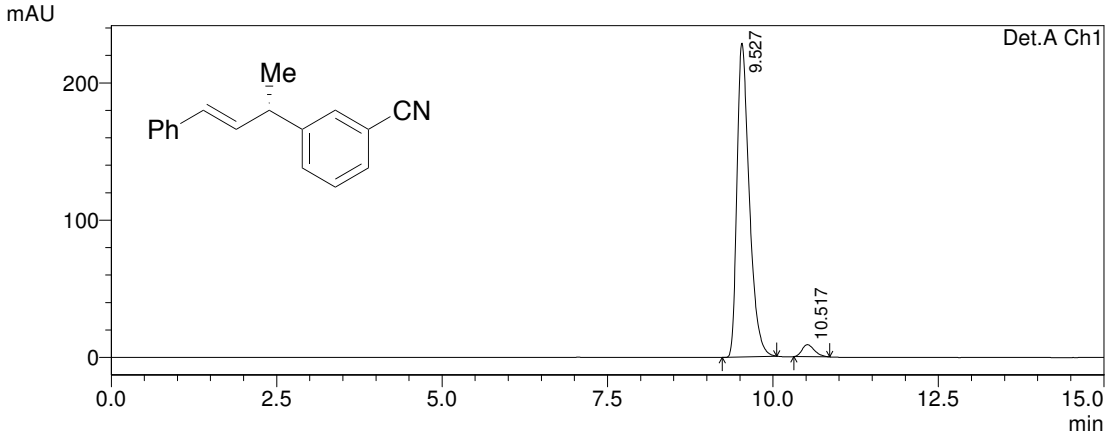
Racemic **12**



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.671	349082	27005	50.386	52.117
2	10.651	343730	24810	49.614	47.883
Total		692812	51815	100.000	100.000

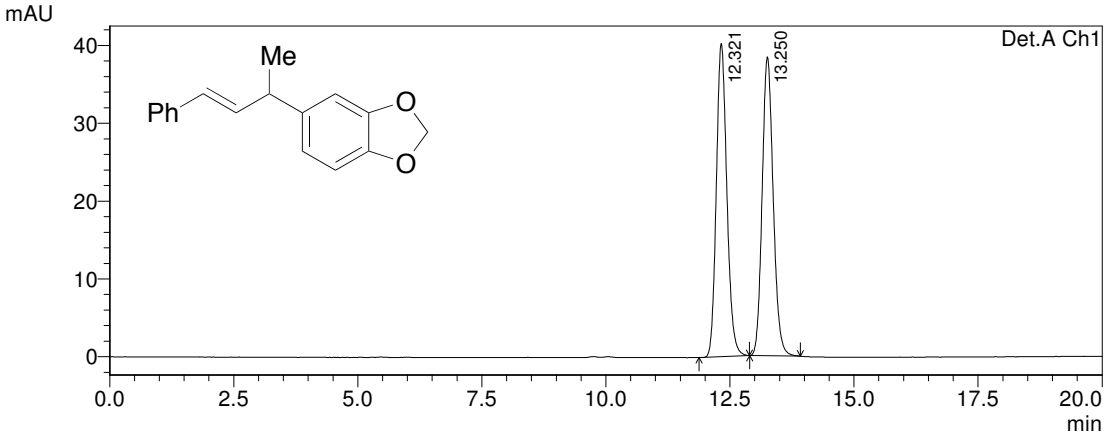
Enantioenriched **12**, ee = 93%



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.527	2955263	228575	96.263	96.292
2	10.517	114736	8801	3.737	3.708
Total		3069999	237376	100.000	100.000

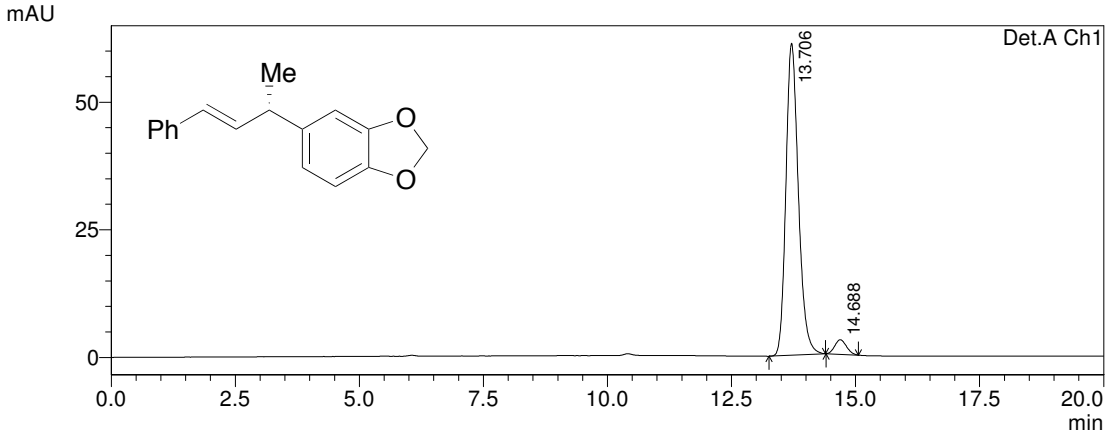
Racemic **13**



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	12.321	594232	40220	49.983	51.167
2	13.250	594630	38386	50.017	48.833
Total		1188862	78605	100.000	100.000

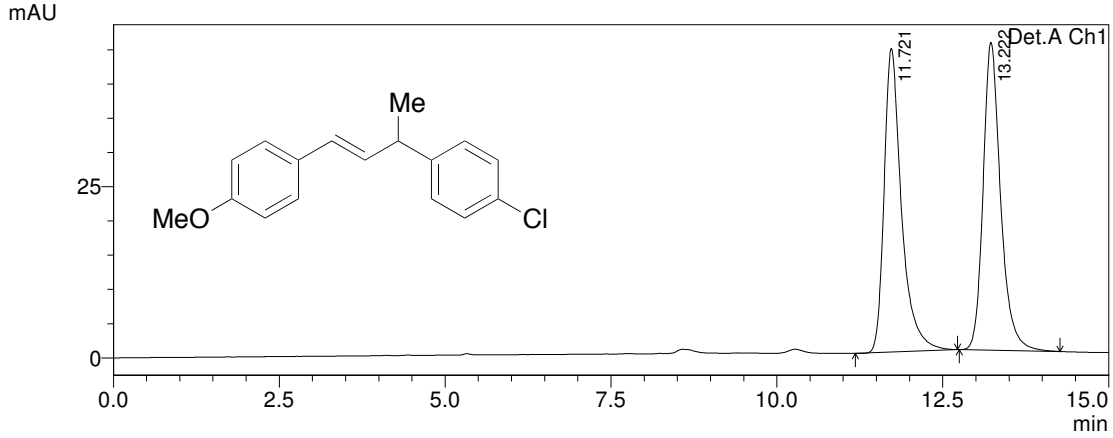
Enantioenriched **13**, ee = 92%



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	13.706	1067949	61099	95.807	95.549
2	14.688	46740	2846	4.193	4.451
Total		1114689	63945	100.000	100.000

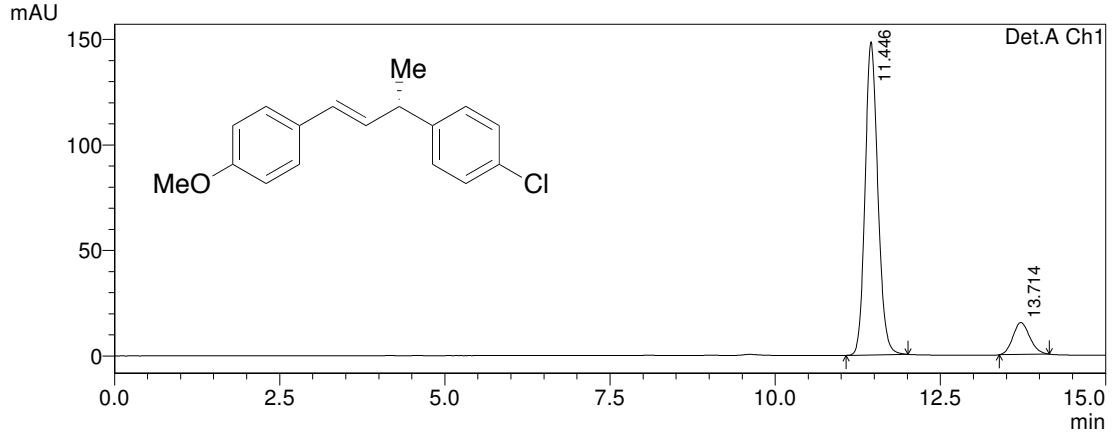
Racemic **14**



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	11.721	807561	44373	49.506	49.663
2	13.222	823662	44976	50.494	50.337
Total		1631223	89349	100.000	100.000

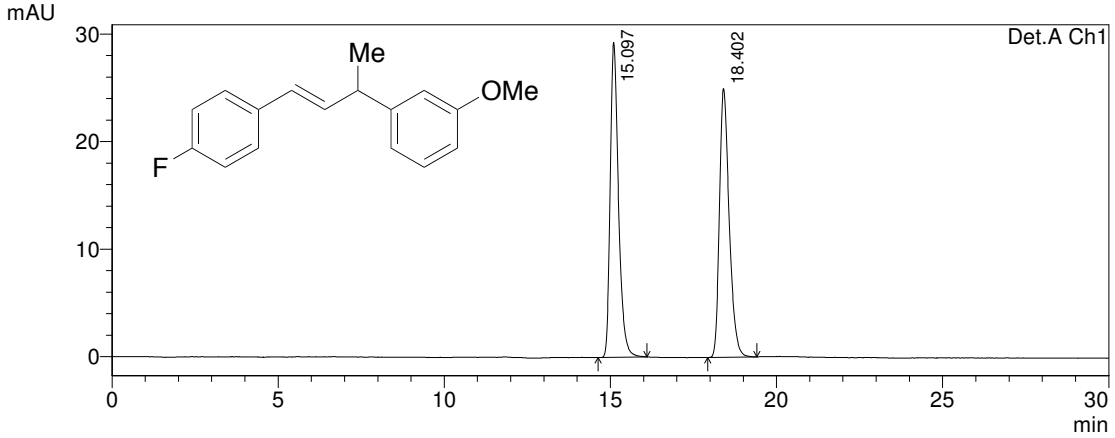
Enantioenriched **14**, ee = 76%



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	11.446	1995853	148409	88.115	90.733
2	13.714	269209	15157	11.885	9.267
Total		2265062	163566	100.000	100.000

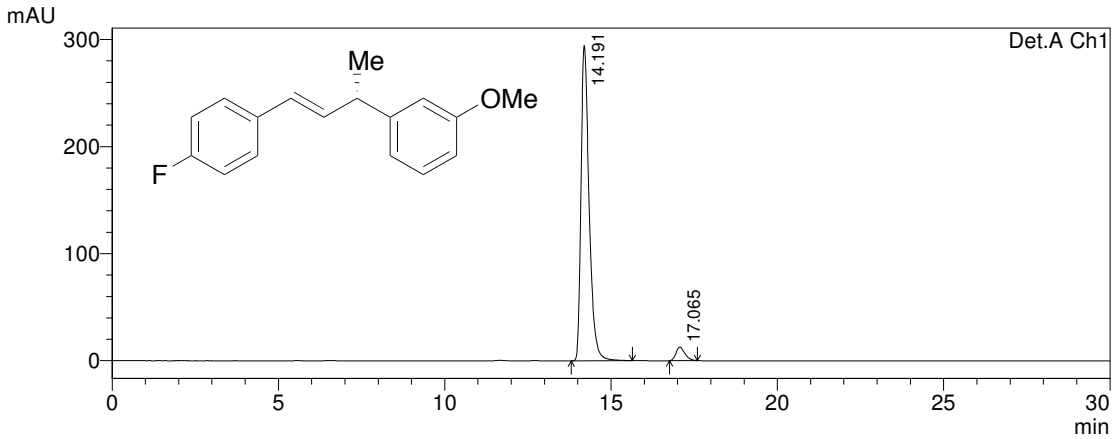
Racemic **15**



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	15.097	508175	29289	49.940	53.947
2	18.402	509391	25003	50.060	46.053
Total		1017566	54292	100.000	100.000

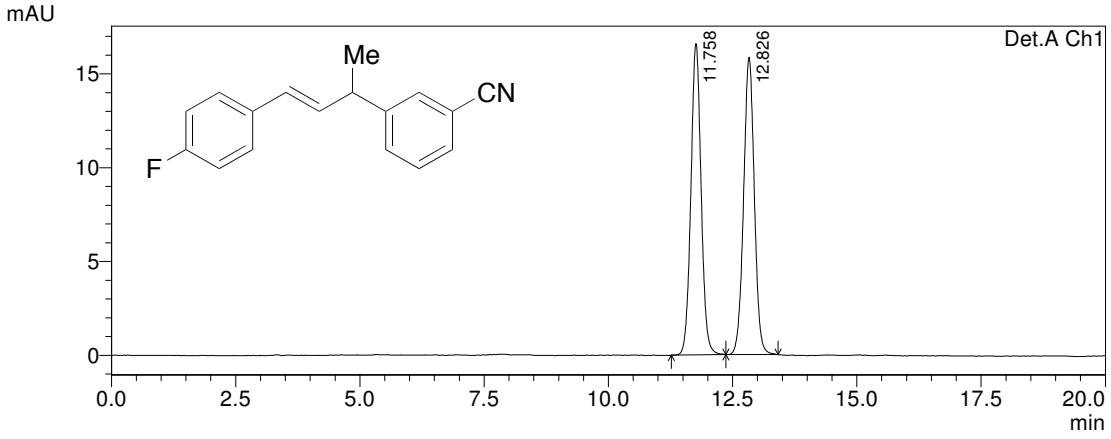
Enantioenriched **15**, ee = 91%



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	14.191	5000569	294197	95.503	95.887
2	17.065	235491	12621	4.497	4.113
Total		5236060	306818	100.000	100.000

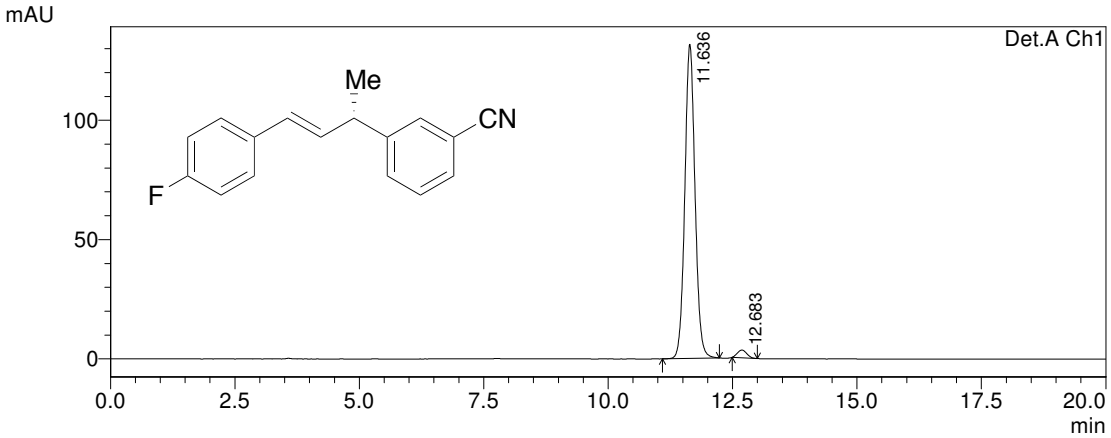
Racemic **16**



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	11.758	237836	16587	49.983	51.145
2	12.826	237998	15845	50.017	48.855
Total		475834	32432	100.000	100.000

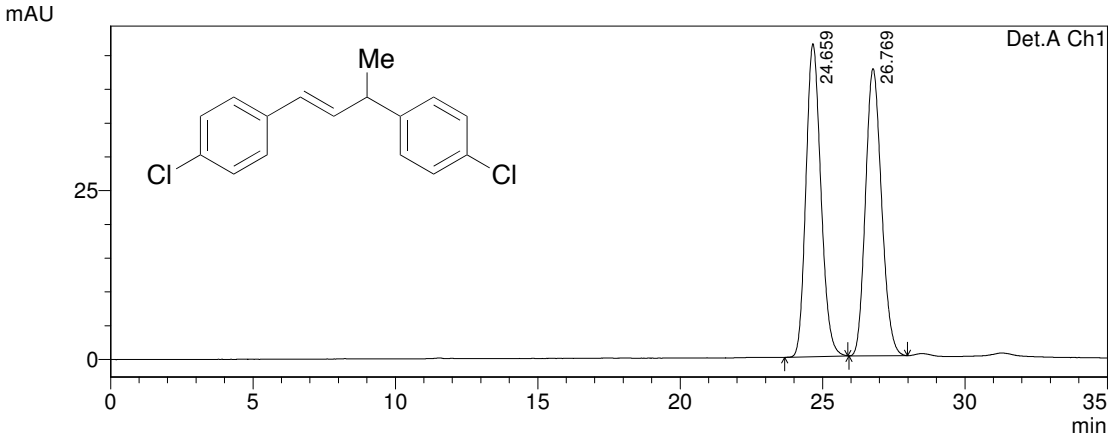
Enantioenriched **16**, ee = 96%



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	11.636	1878029	131646	97.804	97.592
2	12.683	42169	3248	2.196	2.408
Total		1920198	134894	100.000	100.000

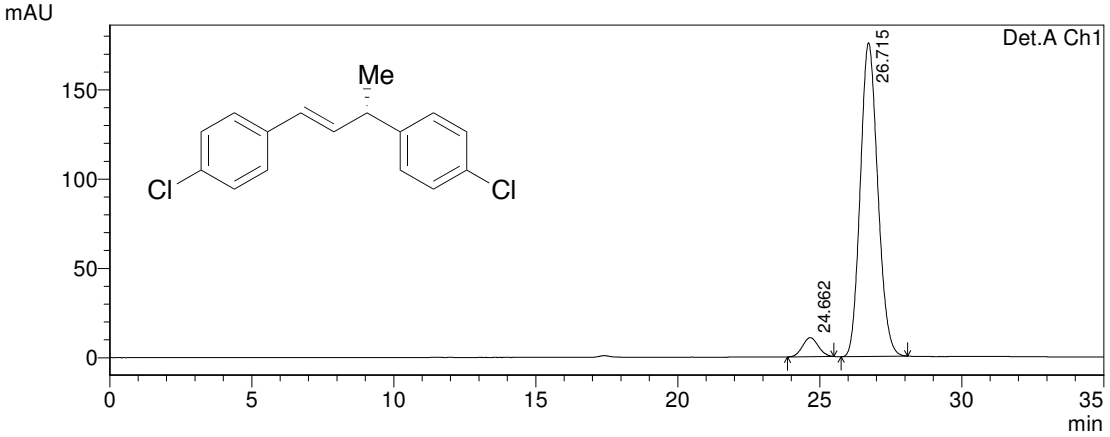
Racemic **17**



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	24.659	1689765	46329	50.349	52.130
2	26.769	1666345	42543	49.651	47.870
Total		3356110	88872	100.000	100.000

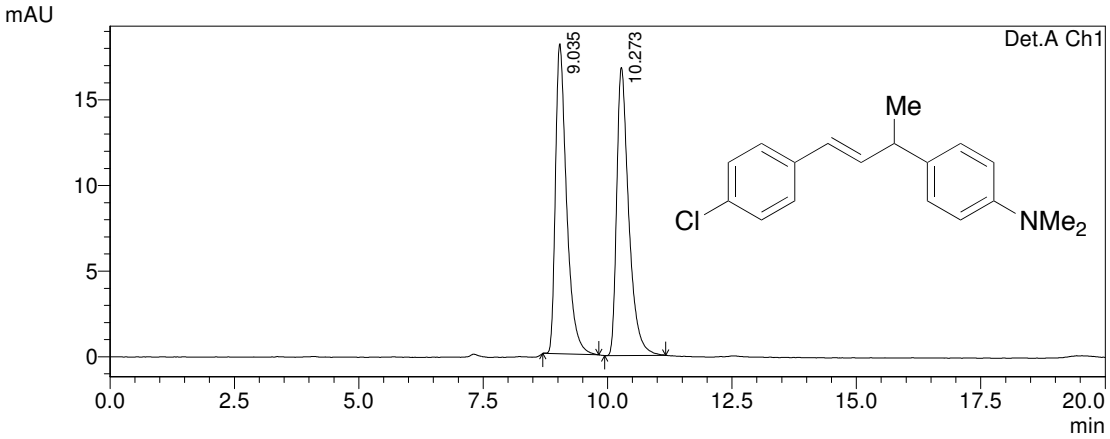
Enantioenriched **17**, ee = 90%



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	24.662	411573	10667	5.191	5.724
2	26.715	7516797	175693	94.809	94.276
Total		7928370	186360	100.000	100.000

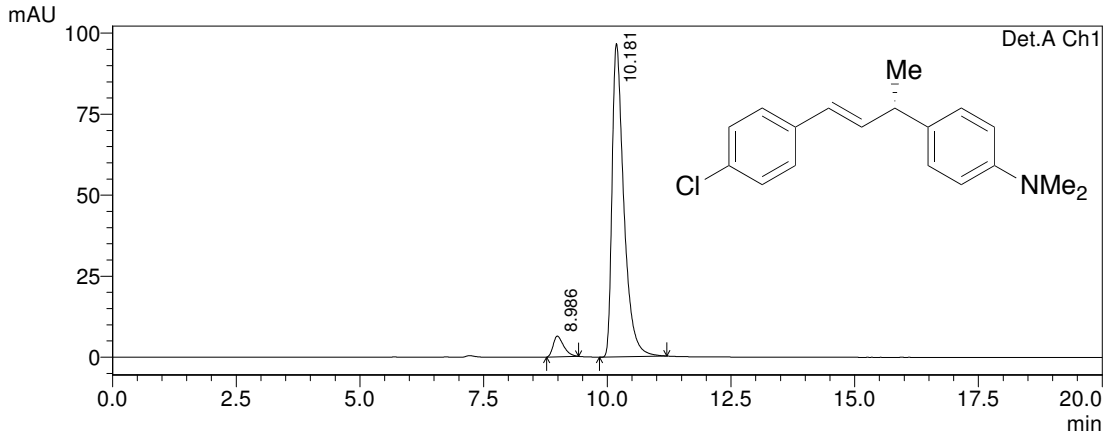
Racemic **18**



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.035	281831	18100	49.870	51.821
2	10.273	283301	16828	50.130	48.179
Total		565132	34927	100.000	100.000

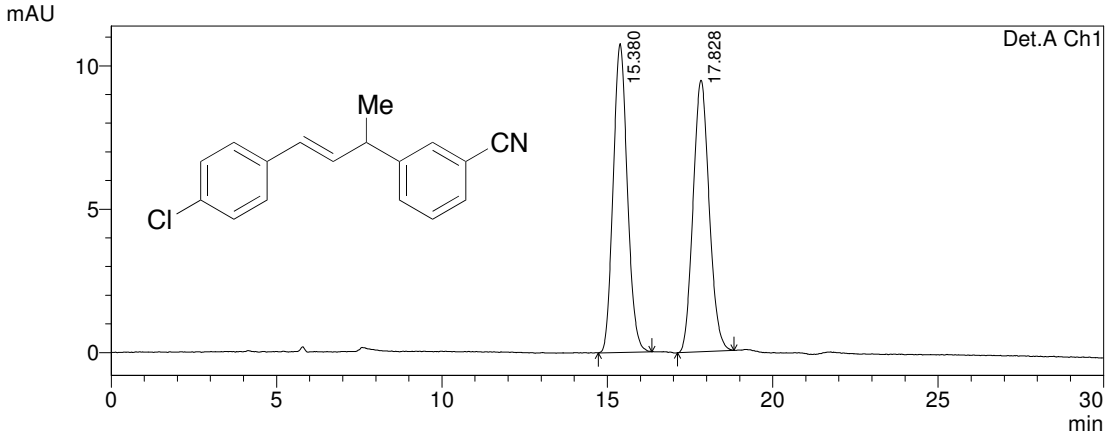
Enantioenriched **18**, ee = 89%



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.986	94596	6406	5.584	6.214
2	10.181	1599390	96673	94.416	93.786
Total		1693986	103078	100.000	100.000

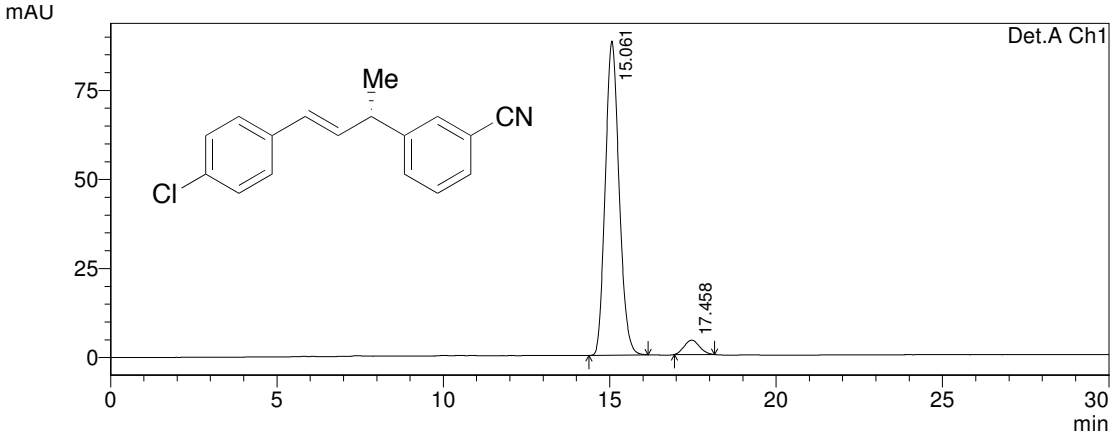
Racemic **19**



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	15.380	320415	10768	50.063	53.201
2	17.828	319614	9472	49.937	46.799
Total		640029	20239	100.000	100.000

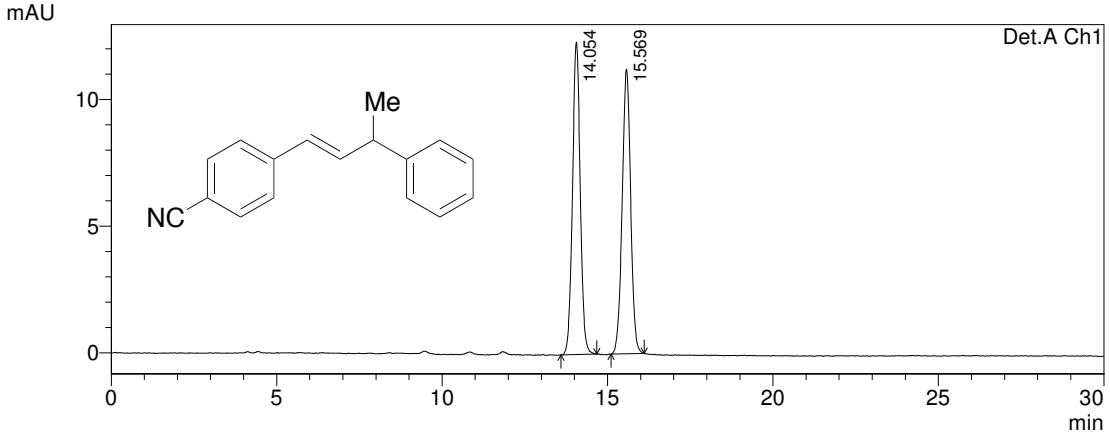
Enantioenriched **19**, ee = 90%



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	15.061	2546825	88146	95.154	95.602
2	17.458	129704	4055	4.846	4.398
Total		2676529	92201	100.000	100.000

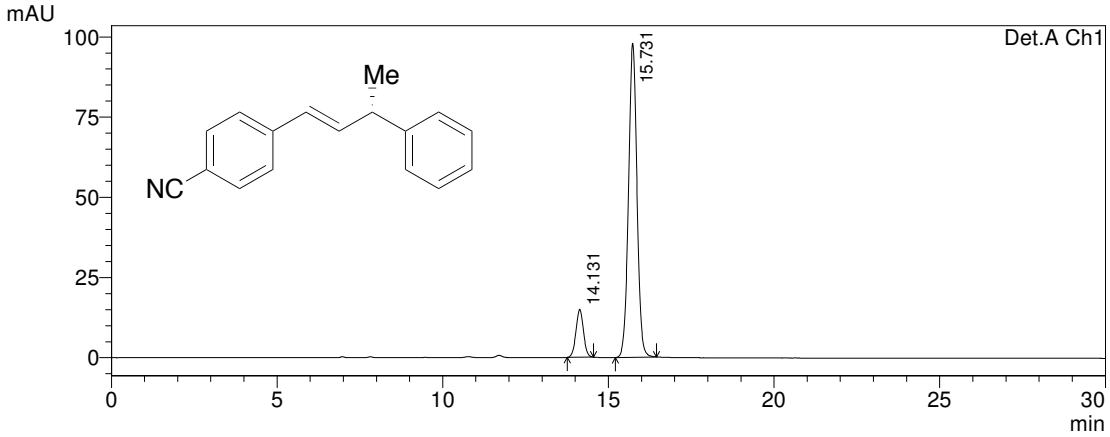
Racemic **20**



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	14.054	200511	12325	50.027	52.327
2	15.569	200293	11229	49.973	47.673
Total		400804	23554	100.000	100.000

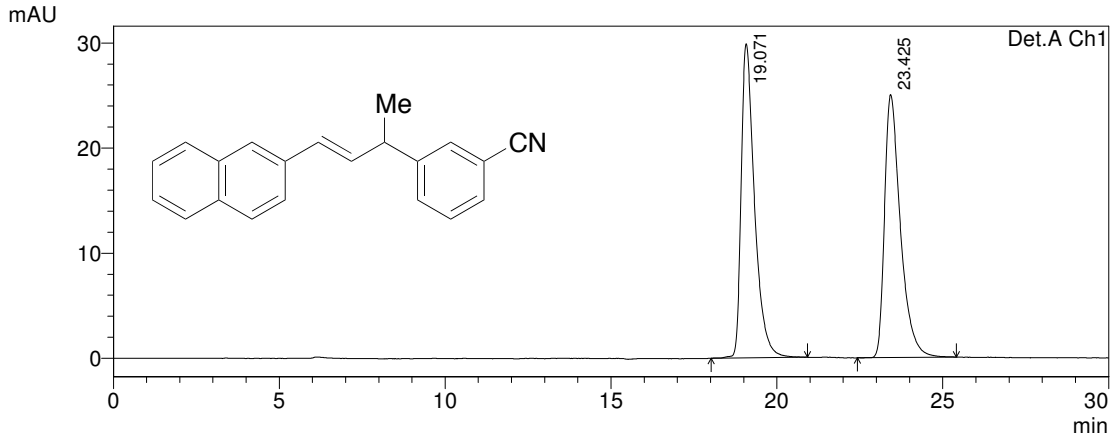
Enantioenriched **20**, ee = 76%



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	14.131	235932	14981	11.961	13.275
2	15.731	1736595	97870	88.039	86.725
Total		1972527	112851	100.000	100.000

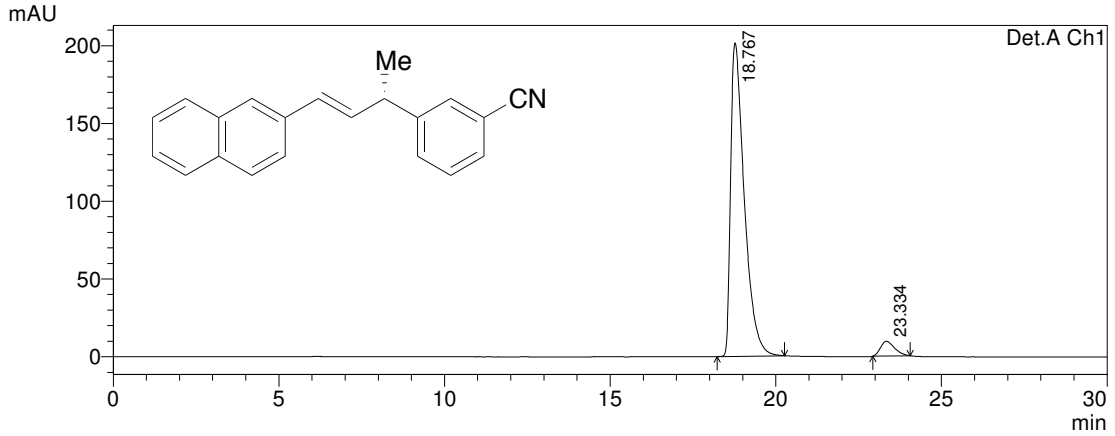
Racemic **21**



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	19.071	835245	29879	50.109	54.427
2	23.425	831604	25018	49.891	45.573
Total		1666849	54897	100.000	100.000

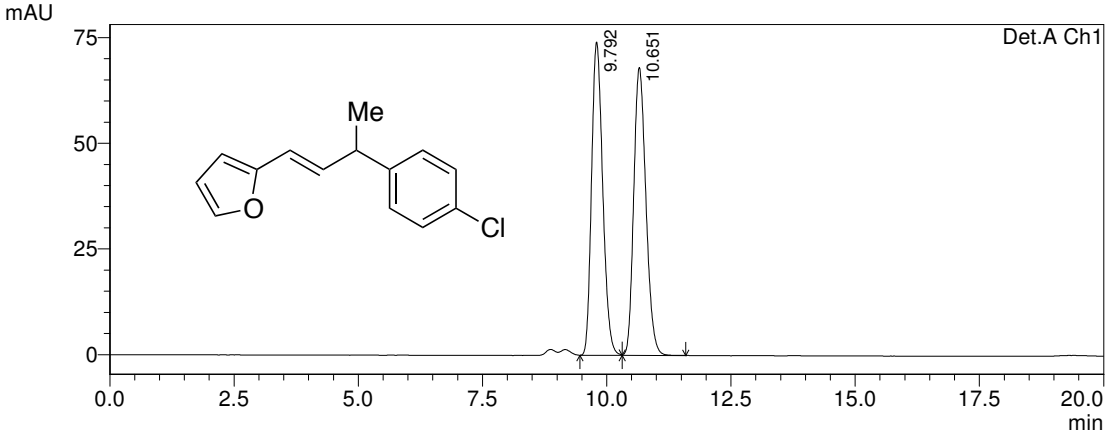
Enantioenriched **21**, ee = 91%



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	18.767	5778302	201515	95.422	95.541
2	23.334	277193	9405	4.578	4.459
Total		6055496	210920	100.000	100.000

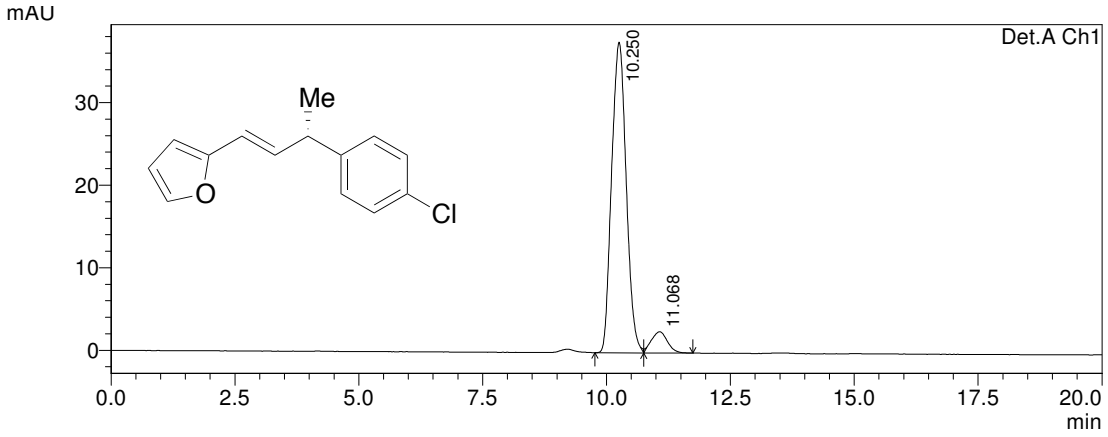
Racemic **22**



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.792	1156522	74059	49.899	52.103
2	10.651	1161221	68081	50.101	47.897
Total		2317742	142140	100.000	100.000

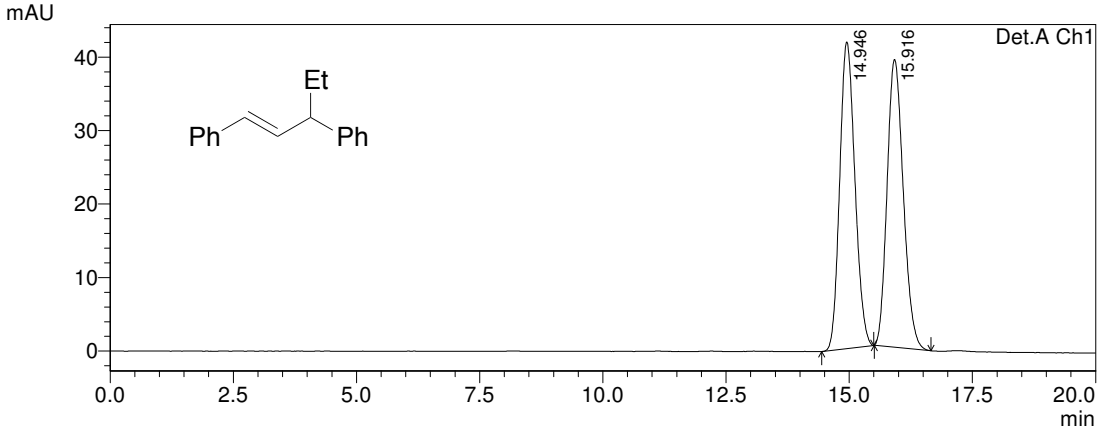
Enantioenriched **22**, ee = 86%



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	10.250	771297	37625	92.947	93.597
2	11.068	58527	2574	7.053	6.403
Total		829824	40198	100.000	100.000

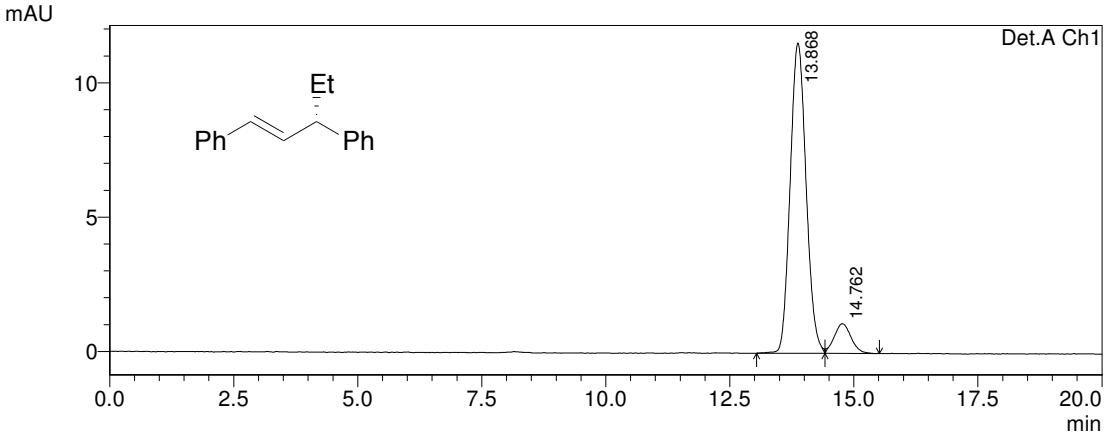
Racemic **23**



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	14.946	895104	41713	49.972	51.580
2	15.916	896105	39157	50.028	48.420
Total		1791209	80870	100.000	100.000

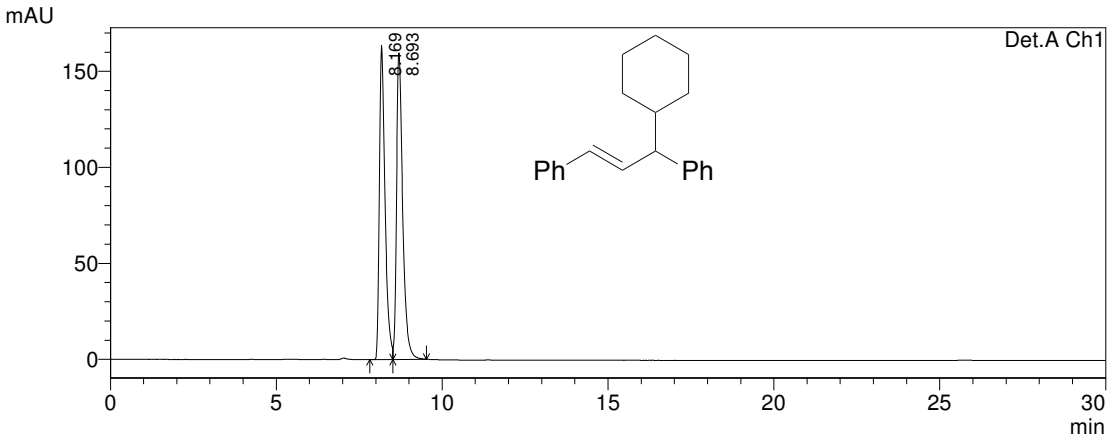
Enantioenriched **23**, ee = 82%



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	13.868	258330	11546	90.794	91.272
2	14.762	26194	1104	9.206	8.728
Total		284524	12650	100.000	100.000

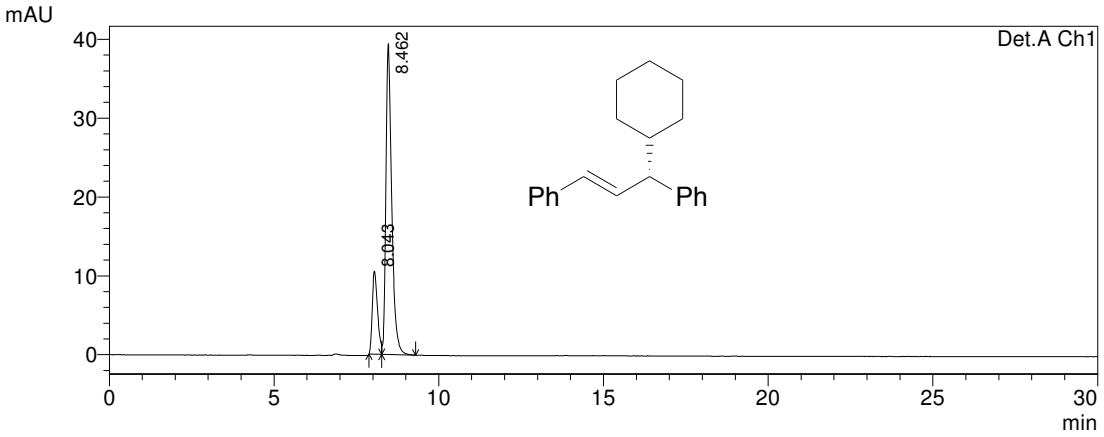
Racemic **24**



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.169	1940959	163447	48.814	50.644
2	8.693	2035273	159293	51.186	49.356
Total		3976232	322740	100.000	100.000

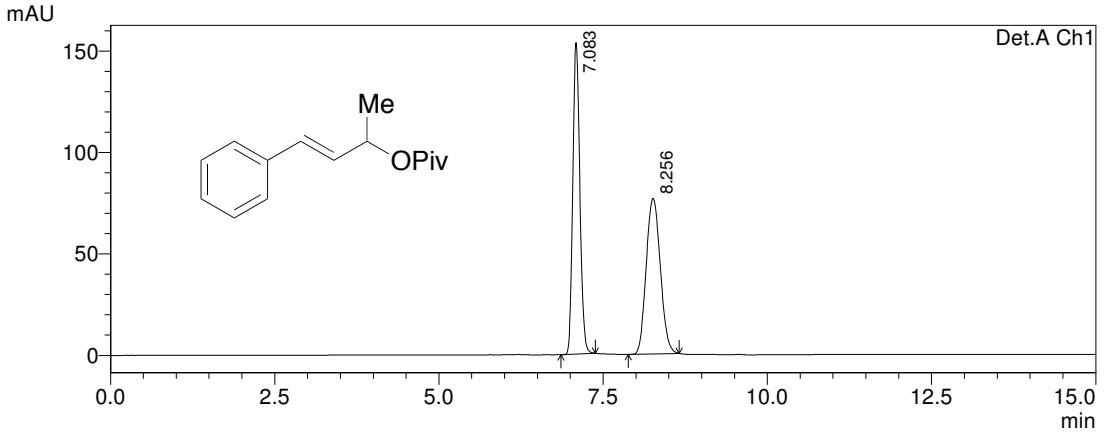
Enantioenriched **24**, ee = 61%



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.043	116720	10518	19.377	21.073
2	8.462	485645	39394	80.623	78.927
Total		602365	49912	100.000	100.000

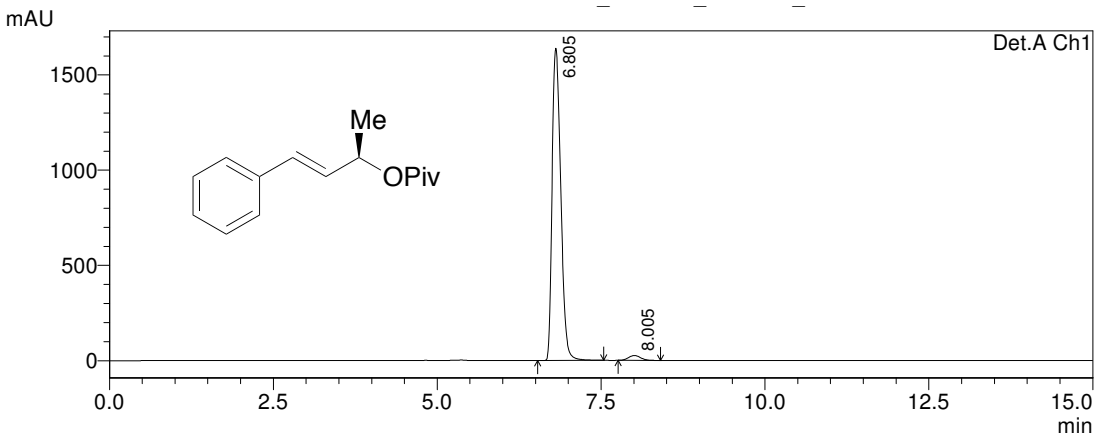
Racemic **1a**



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.083	1148930	153378	50.011	66.649
2	8.256	1148419	76750	49.989	33.351
Total		2297349	230128	100.000	100.000

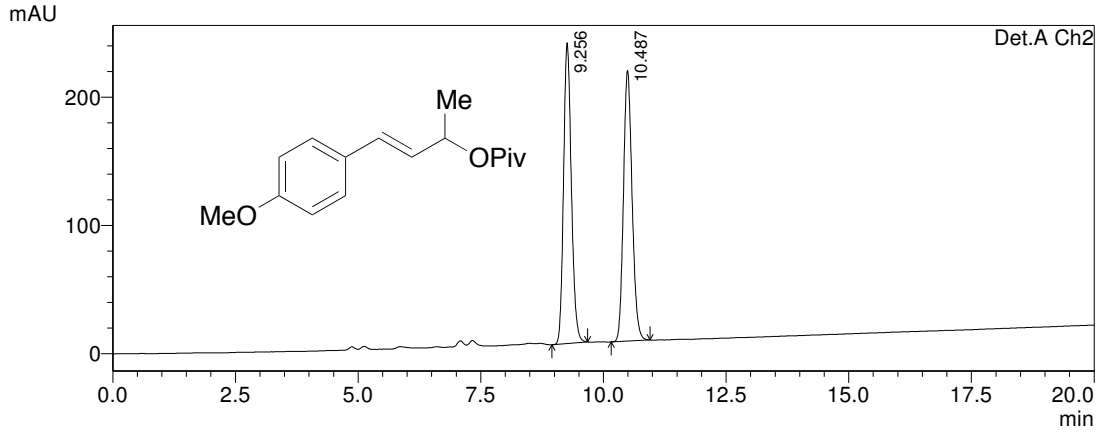
Enantioenriched **1a**, ee = 96%



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.805	15085485	1638394	97.867	98.461
2	8.005	328742	25601	2.133	1.539
Total		15414227	1663995	100.000	100.000

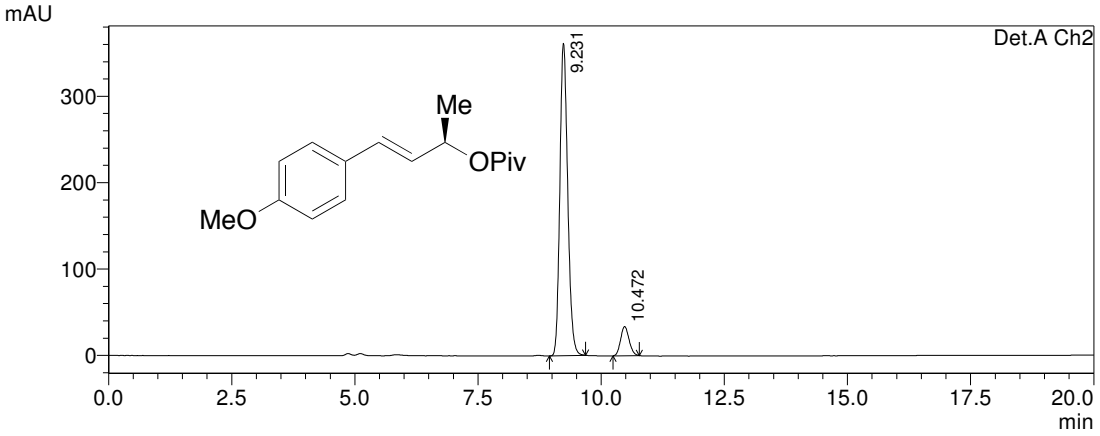
Racemic **1b**



Detector A Ch2 210nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.256	2587105	234367	50.020	52.612
2	10.487	2585032	211095	49.980	47.388
Total		5172137	445462	100.000	100.000

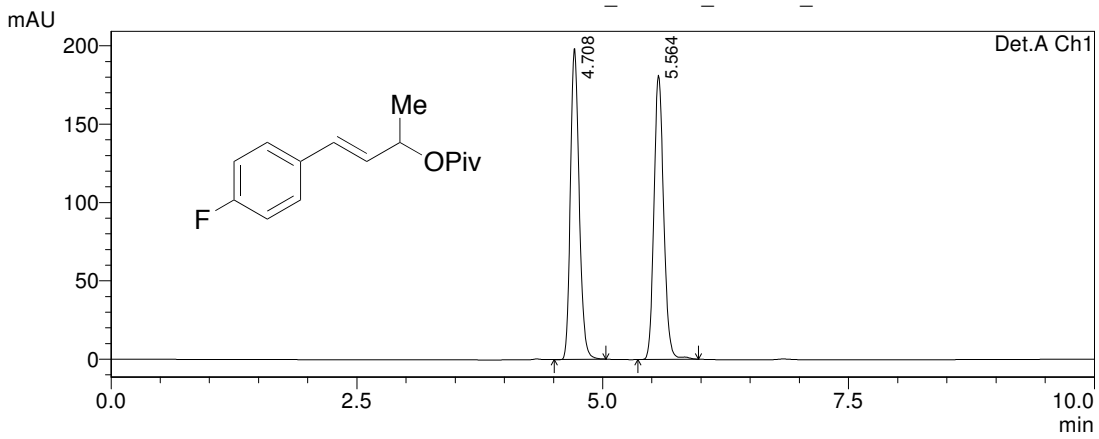
Enantioenriched **1b**, ee = 82%



Detector A Ch2 210nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.231	4008739	361390	90.806	91.434
2	10.472	405887	33855	9.194	8.566
Total		4414626	395245	100.000	100.000

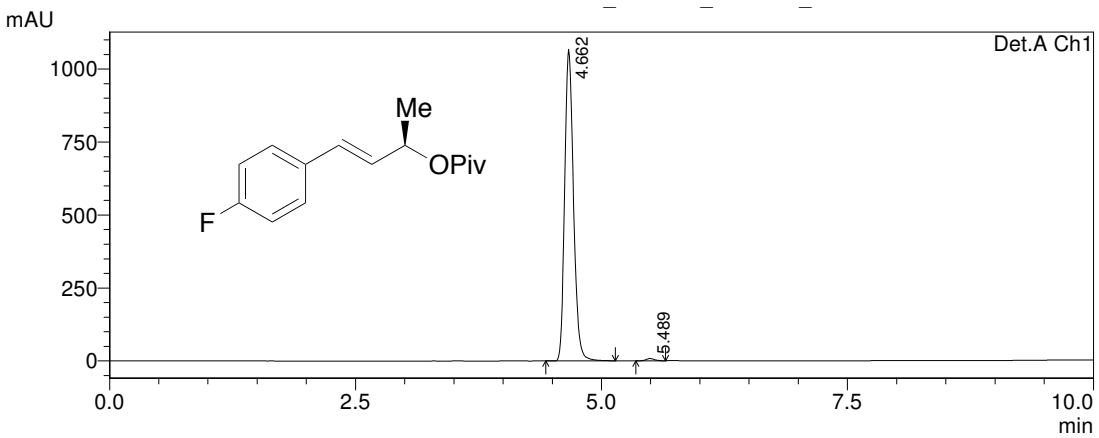
Racemic **1c**



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	4.708	1233223	198303	49.818	52.220
2	5.564	1242245	181443	50.182	47.780
Total		2475468	379746	100.000	100.000

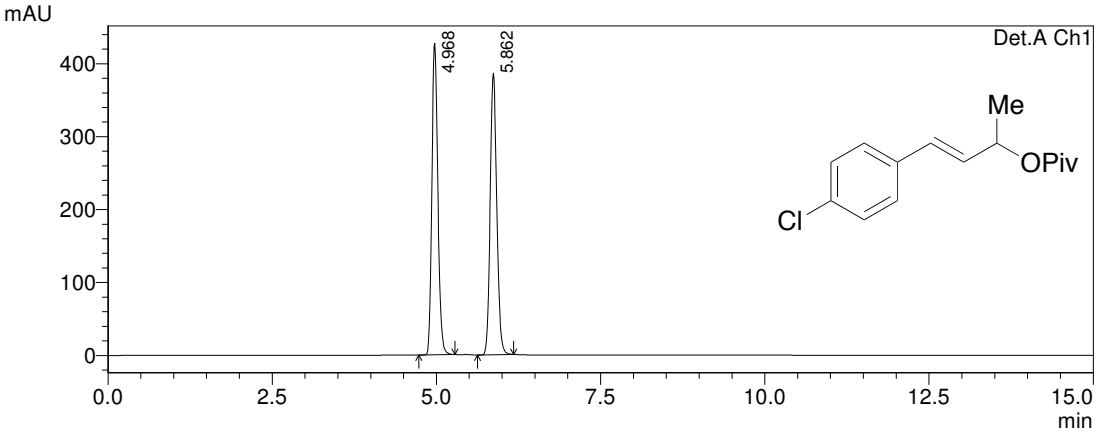
Enantioenriched **1c**, ee = 98%



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	4.662	6650232	1066854	99.298	99.295
2	5.489	47028	7574	0.702	0.705
Total		6697260	1074428	100.000	100.000

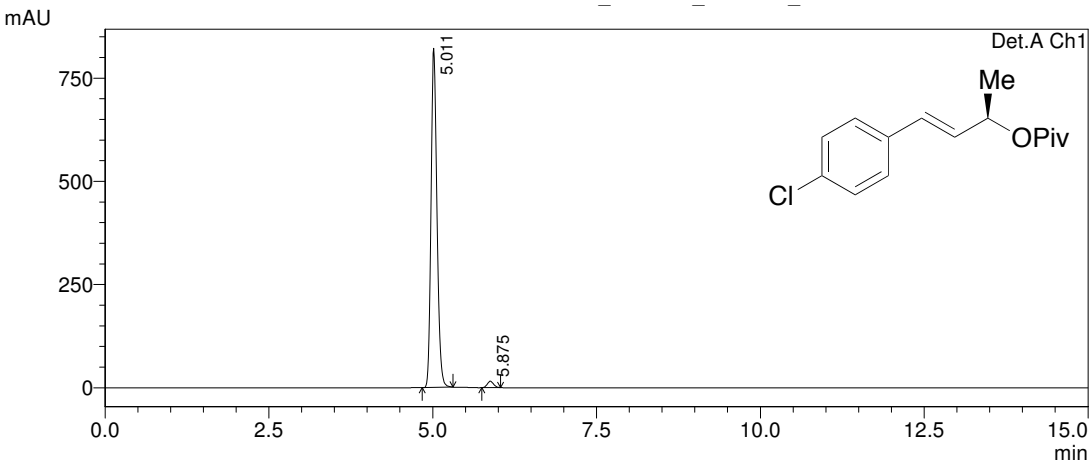
Racemic **1d**



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	4.968	2698912	427008	50.039	52.519
2	5.862	2694758	386051	49.961	47.481
Total		5393670	813059	100.000	100.000

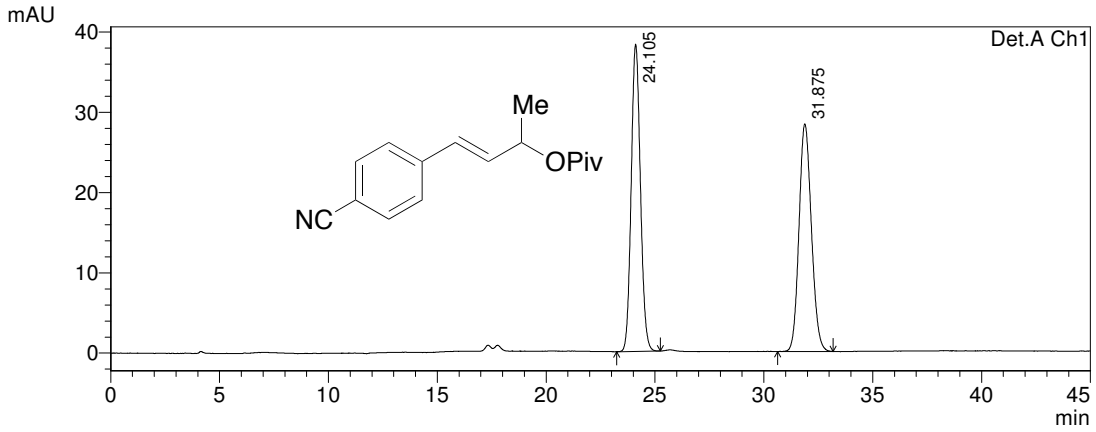
Enantioenriched **1d**, ee = 96%



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.011	5220879	821541	98.026	98.146
2	5.875	105126	15518	1.974	1.854
Total		5326005	837059	100.000	100.000

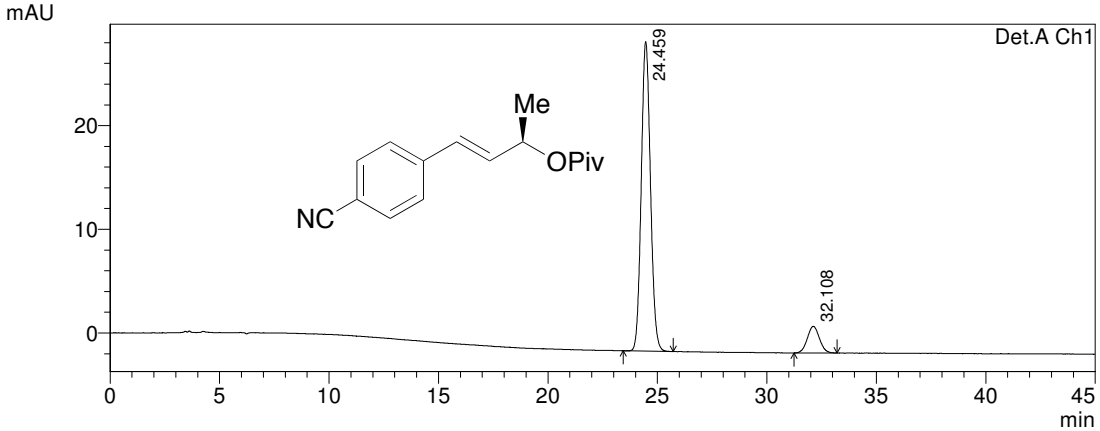
Racemic **1e**



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	24.105	1125881	38286	50.089	57.434
2	31.875	1121870	28374	49.911	42.566
Total		2247751	66661	100.000	100.000

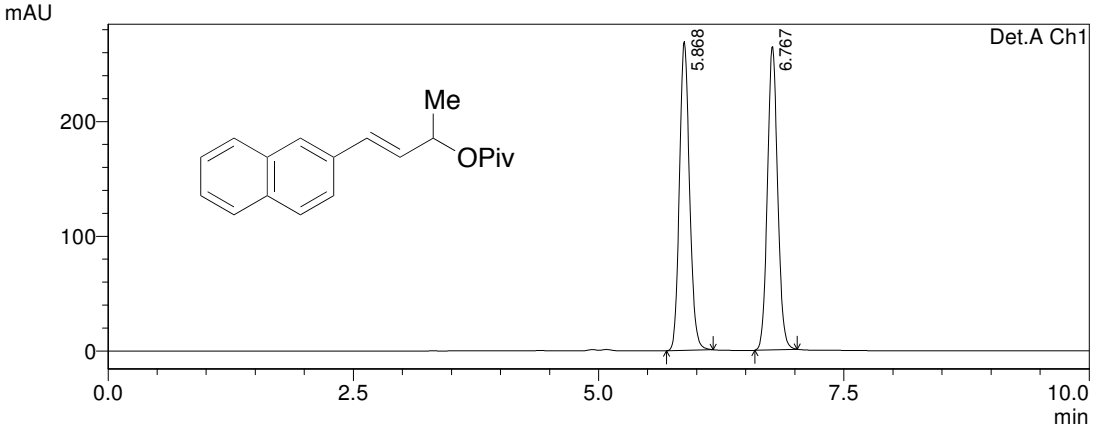
Enantioenriched **1e**, ee = 80%



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	24.459	874057	29841	89.821	92.069
2	32.108	99056	2571	10.179	7.931
Total		973112	32411	100.000	100.000

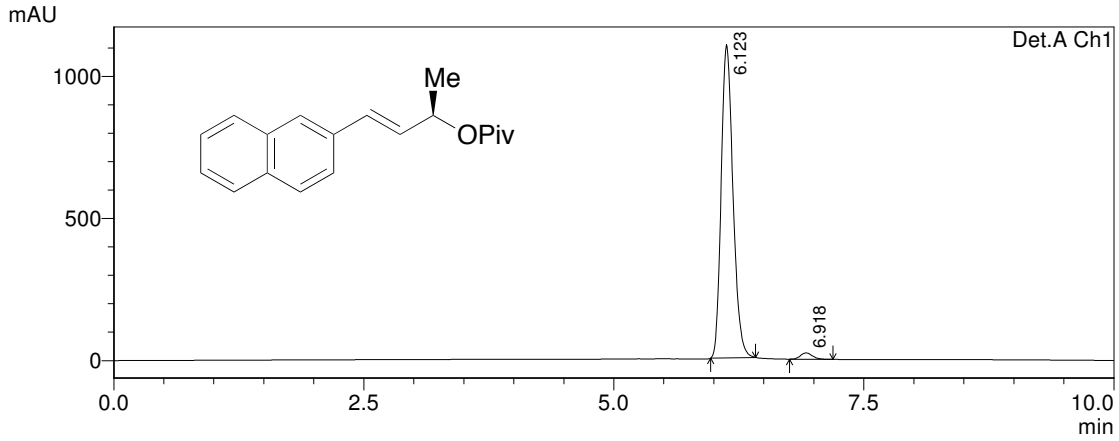
Racemic **1f**



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.868	1927376	269194	50.115	50.459
2	6.767	1918496	264295	49.885	49.541
Total		3845872	533490	100.000	100.000

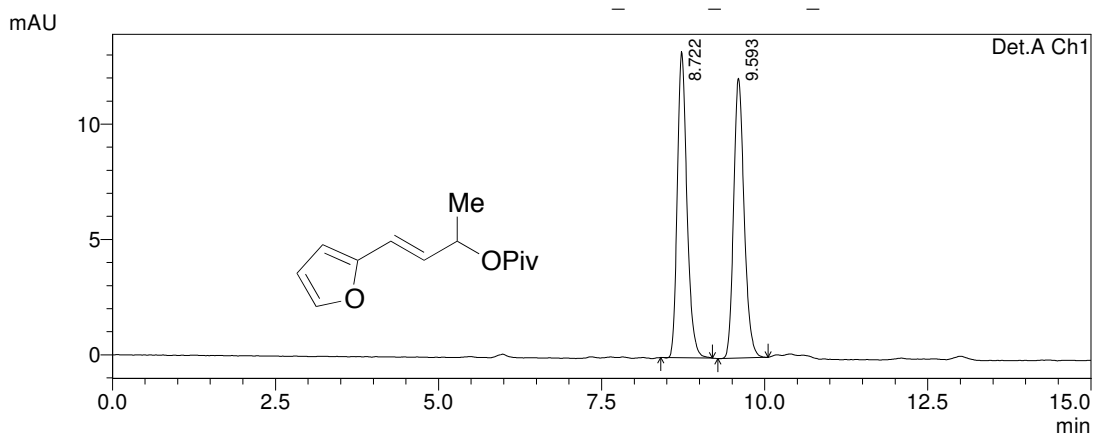
Enantioenriched **1f**, ee = 96%



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.123	9044130	1102126	97.972	98.006
2	6.918	187203	22422	2.028	1.994
Total		9231333	1124548	100.000	100.000

Racemic **1g**

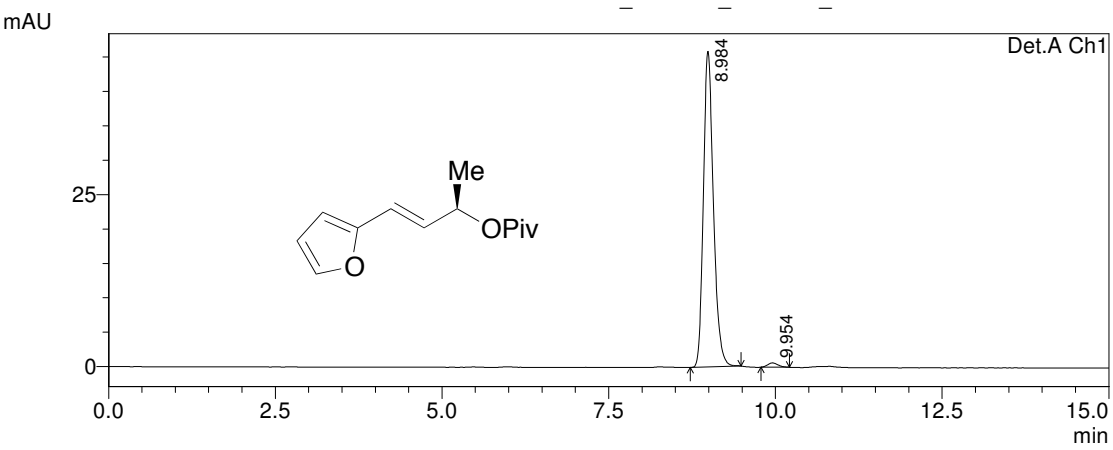


0

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.722	136201	13271	50.103	52.242
2	9.593	135642	12132	49.897	47.758
Total		271843	25403	100.000	100.000

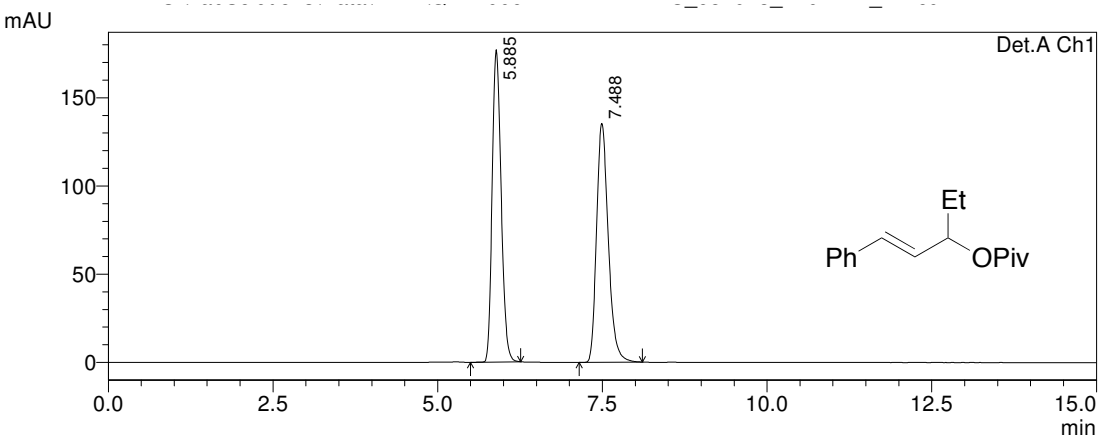
Enantioenriched **1g**, ee = 97%



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.984	480703	45863	98.617	98.634
2	9.954	6741	635	1.383	1.366
Total		487444	46498	100.000	100.000

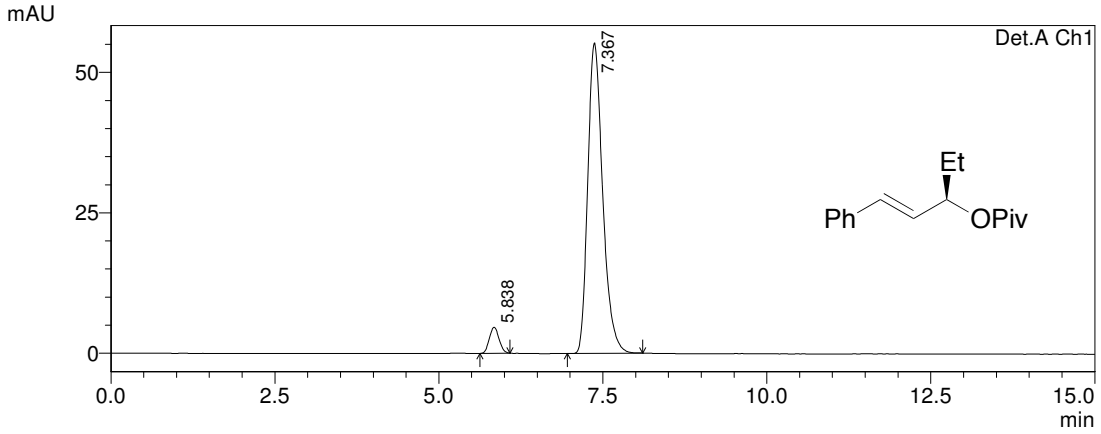
Racemic **1h**



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.885	1677000	176994	50.017	56.641
2	7.488	1675869	135488	49.983	43.359
Total		3352870	312481	100.000	100.000

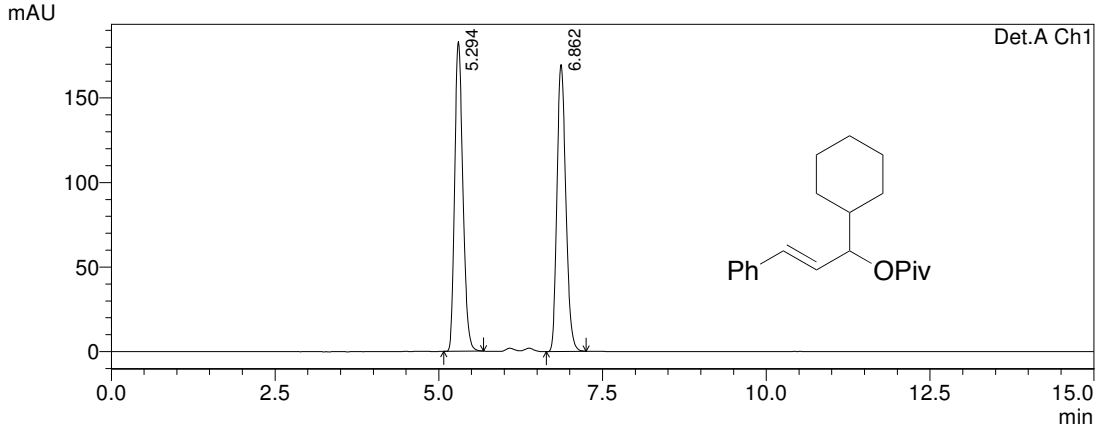
Enantioenriched **1h**, ee = 90%



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.838	47604	4634	5.158	7.735
2	7.367	875250	55270	94.842	92.265
Total		922854	59903	100.000	100.000

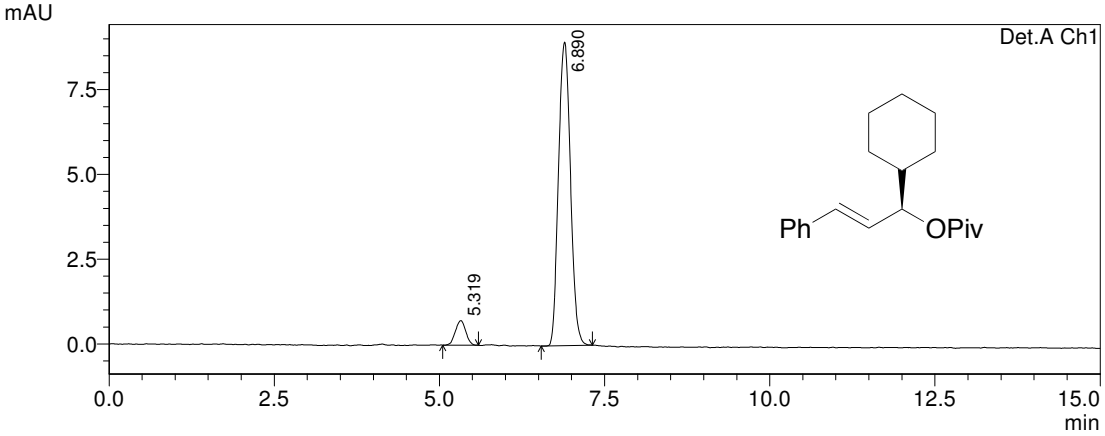
Racemic **1i**



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.294	1600071	183097	50.004	51.903
2	6.862	1599846	169669	49.996	48.097
Total		3199916	352766	100.000	100.000

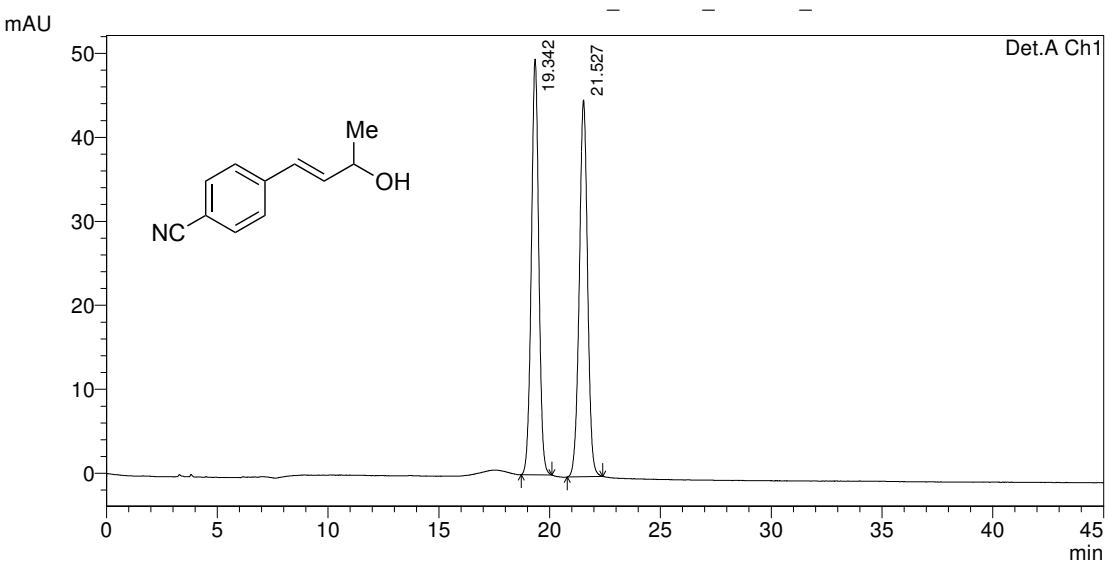
Enantioenriched **1i**, ee = 87%



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.319	7957	726	6.585	7.505
2	6.890	112885	8953	93.415	92.495
Total		120842	9680	100.000	100.000

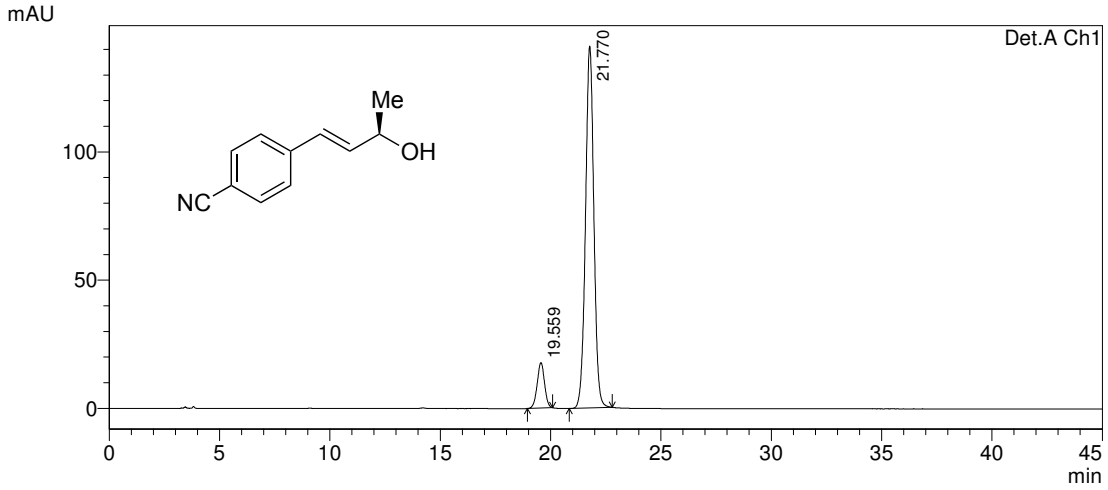
Racemic **S-1e**



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	19.342	1155111	49477	49.791	52.459
2	21.527	1164813	44838	50.209	47.541
Total		2319925	94314	100.000	100.000

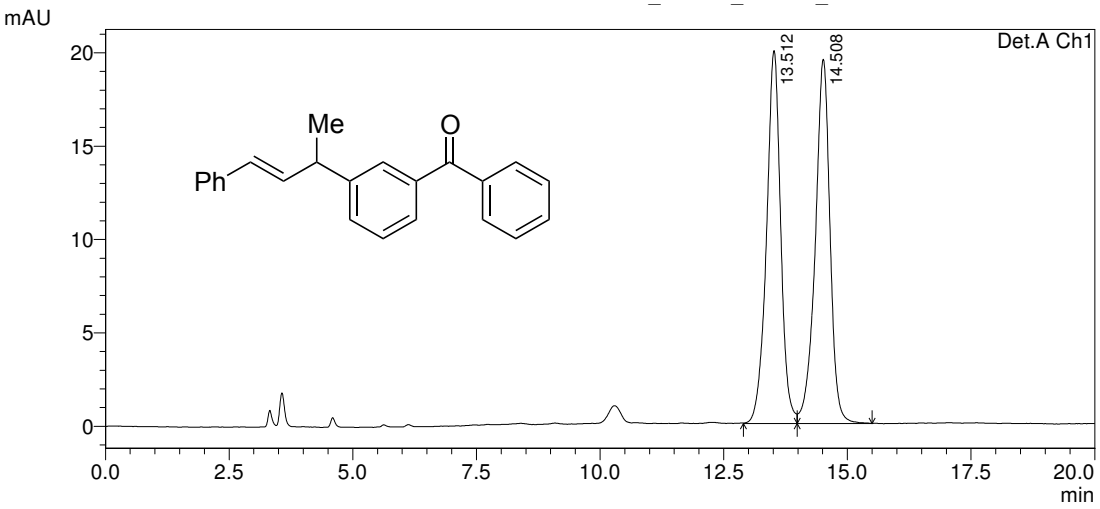
Enantioenriched **S-1e**, 80% ee



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	19.559	411243	17736	9.981	11.160
2	21.770	3708859	141186	90.019	88.840
Total		4120101	158922	100.000	100.000

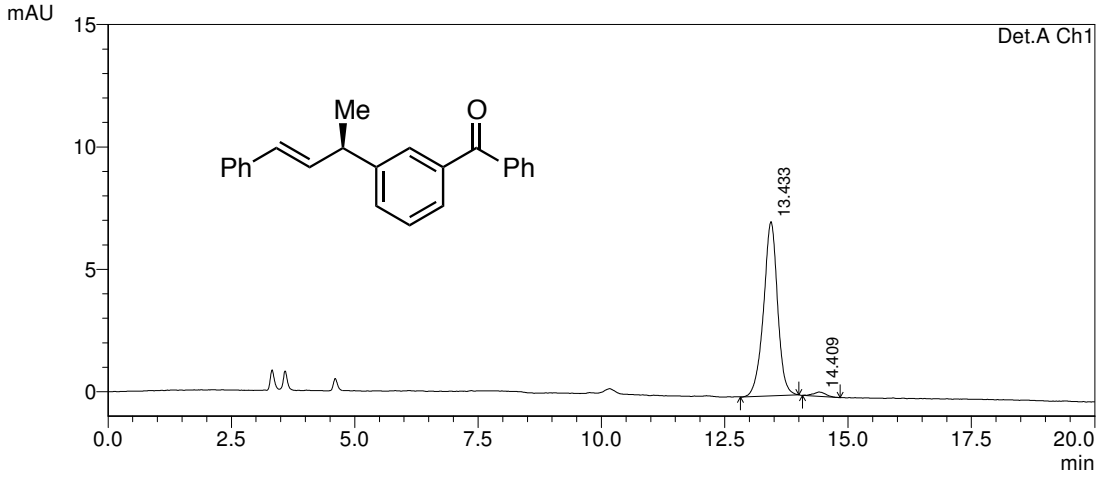
Racemic **25**



Detector A Ch1 220nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	13.512	399033	19975	49.585	50.590
2	14.508	405720	19509	50.415	49.410
Total		804753	39484	100.000	100.000

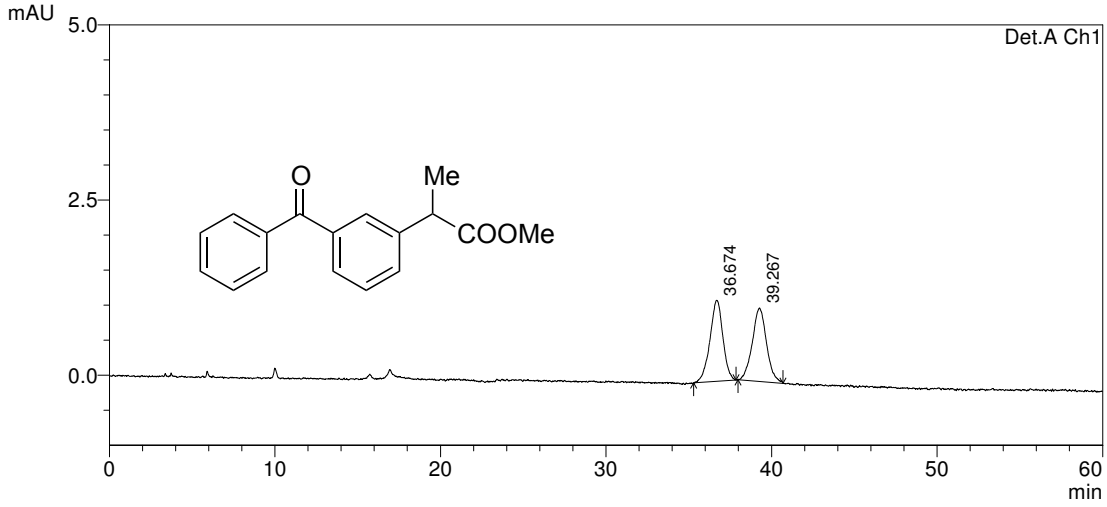
Enantioenriched **25**, 95% ee



Detector A Ch1 220nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	13.433	140135	7107	97.873	97.615
2	14.409	3045	174	2.127	2.385
Total		143180	7281	100.000	100.000

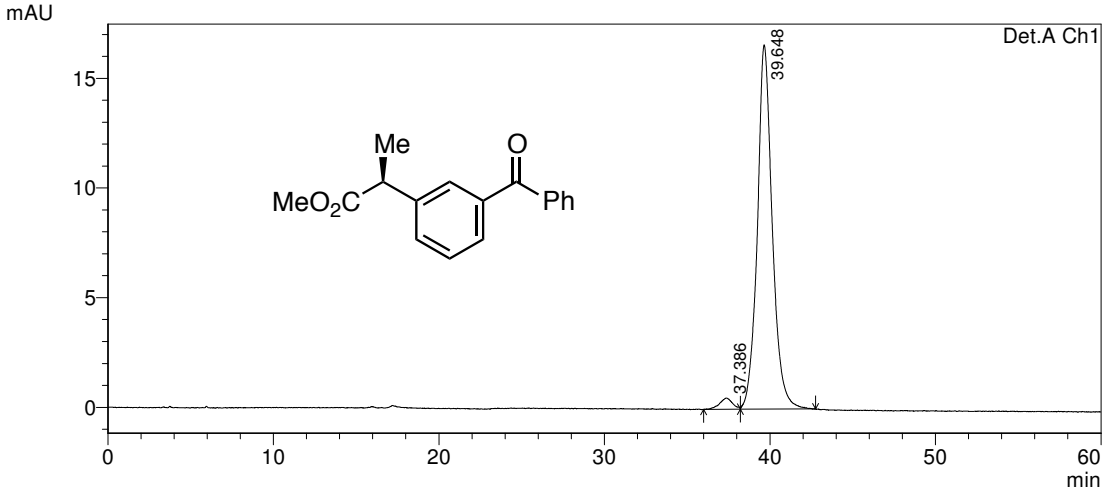
Racemic **S-29**



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	36.674	62988	1156	50.223	52.472
2	39.267	62430	1047	49.777	47.528
Total		125418	2203	100.000	100.000

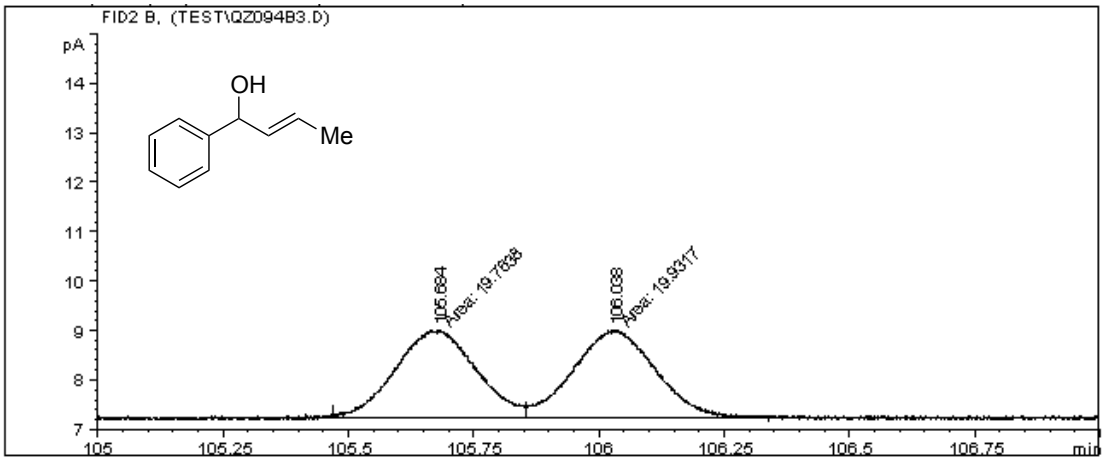
Enantioenriched **S-29**, 95% ee



Detector A Ch1 254nm

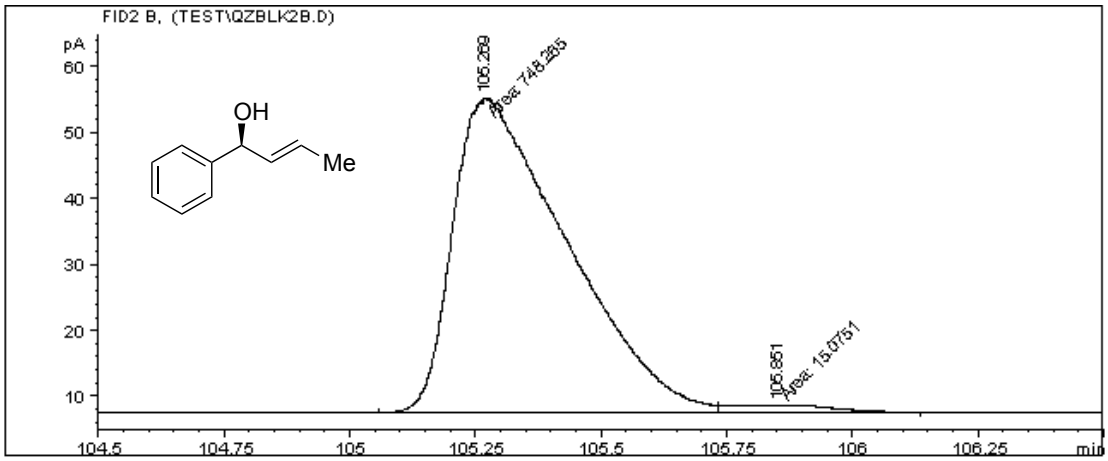
Peak#	Ret. Time	Area	Height	Area %	Height %
1	37.386	27762	513	2.592	2.995
2	39.648	1043427	16616	97.408	97.005
Total		1071188	17129	100.000	100.000

Racemic **S-27**



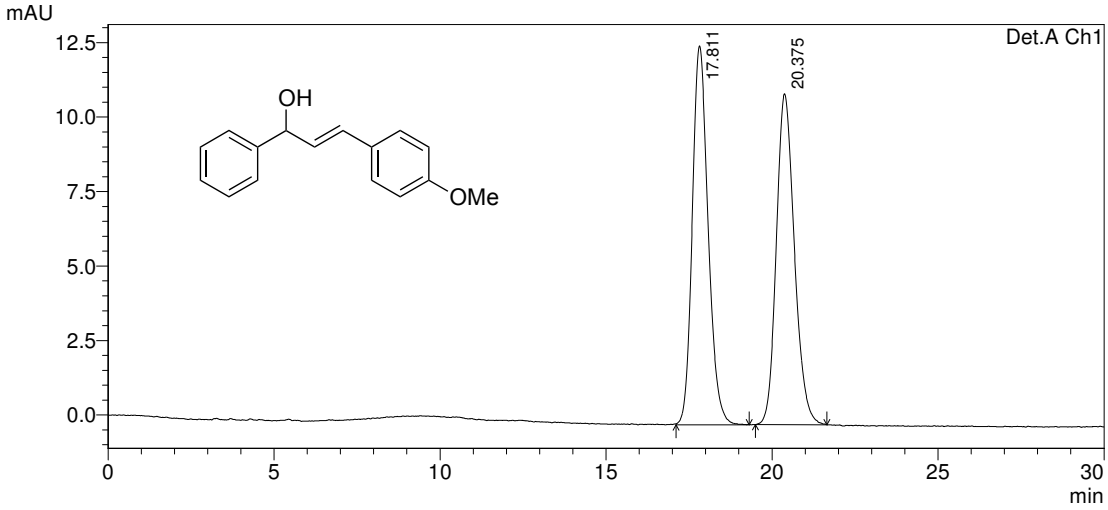
Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	105.684	FM	0.1869	19.76379	1.76283	49.78847
2	106.038	FM	0.1888	19.93172	1.75918	50.21153
Totals :				39.69551	3.52201	

Enantioenriched **S-27**, 96% ee



Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	105.269	MF	0.2624	748.26532	47.52031	98.02511
2	105.851	FM	0.2294	15.07510	1.09533	1.97489
Totals :				763.34042	48.61564	

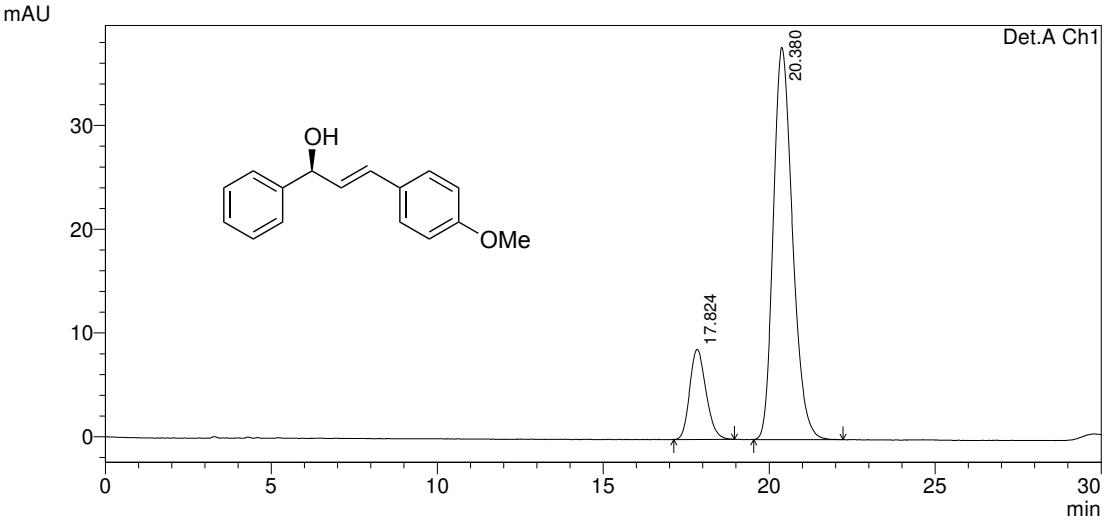
Racemic **S-28**



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	17.811	421028	12719	50.067	53.386
2	20.375	419895	11106	49.933	46.614
Total		840923	23825	100.000	100.000

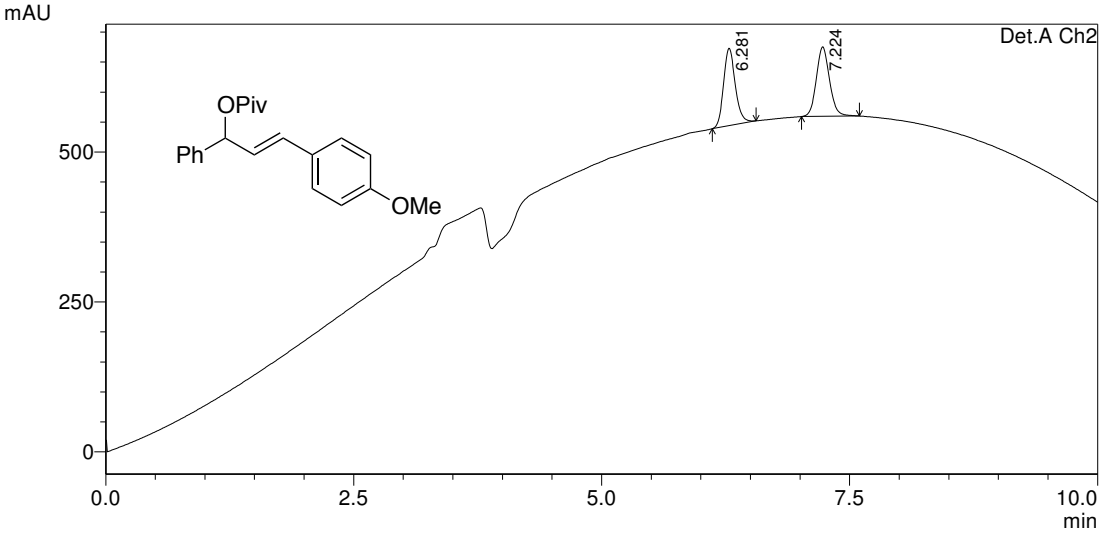
Enantioenriched **S-28**, 67% ee



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	17.824	297238	8687	16.342	18.689
2	20.380	1521674	37796	83.658	81.311
Total		1818913	46483	100.000	100.000

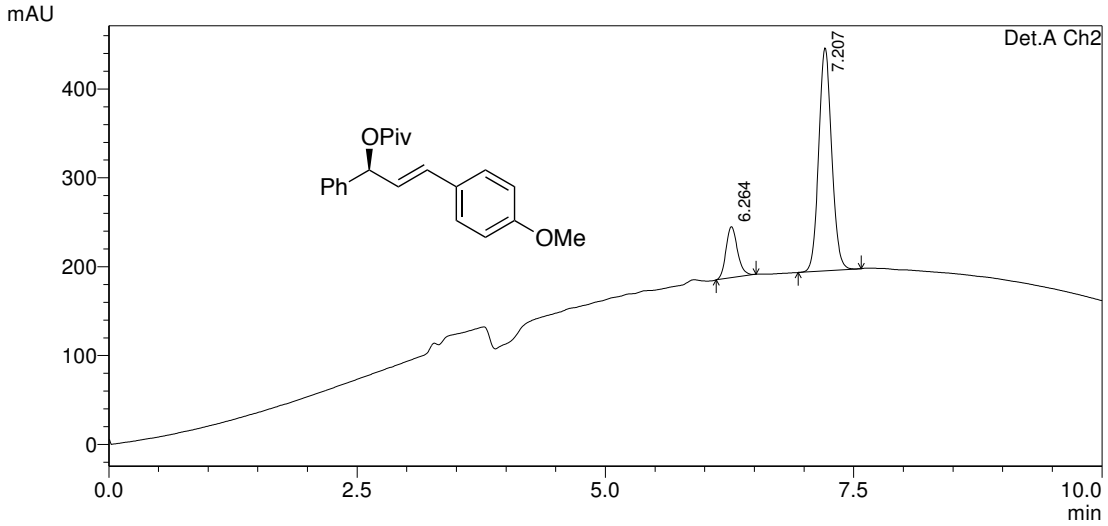
Racemic **28**



Detector A Ch2 210nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.281	1057077	129088	49.605	52.686
2	7.224	1073915	115925	50.395	47.314
Total		2130992	245013	100.000	100.000

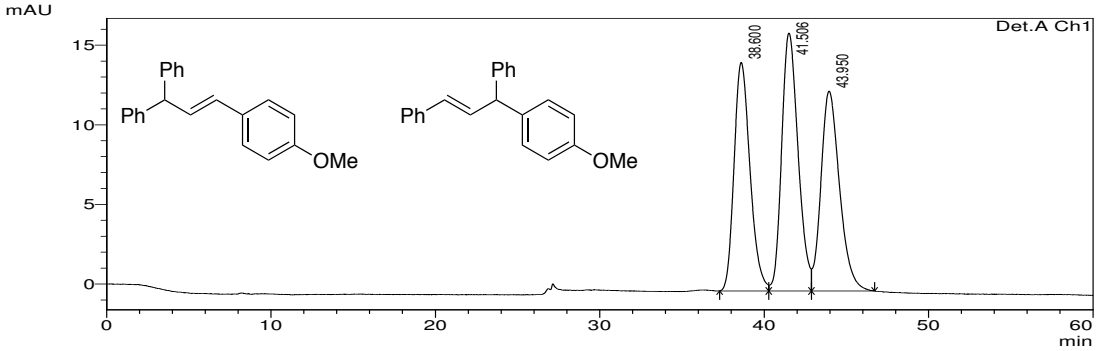
Enantioenriched **28**, 66% ee



Detector A Ch2 210nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.264	463024	57592	16.886	18.670
2	7.207	2279005	250877	83.114	81.330
Total		2742028	308469	100.000	100.000

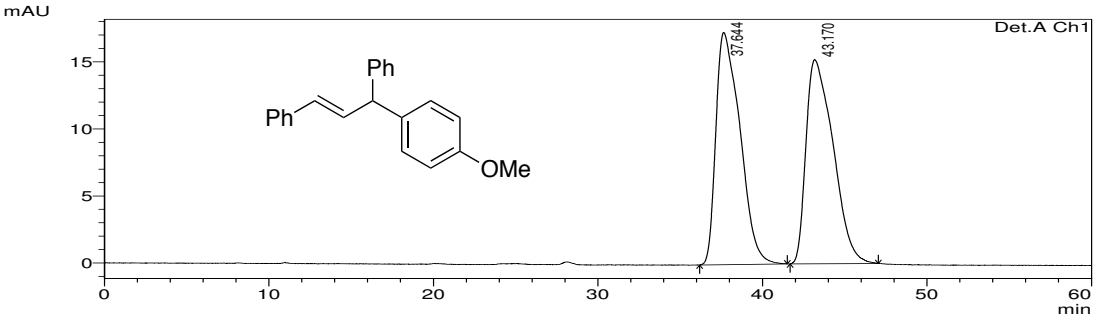
Mixture of **29** and Racemic **30**



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	38.600	968147	14354	31.460	33.306
2	41.506	1121651	16195	36.449	37.578
3	43.950	987549	12548	32.091	29.116
Total		3077347	43098	100.000	100.000

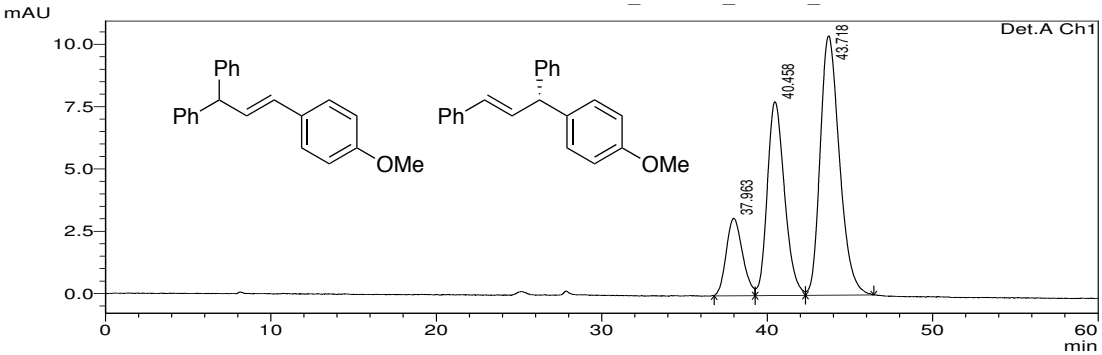
Racemic **30**



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	37.644	1763496	17331	50.233	53.226
2	43.170	1747151	15230	49.767	46.774
Total		3510647	32562	100.000	100.000

Mixture of **29** and Enantioenriched **30** (60% ee)



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	37.963	213435	3115	13.183	14.627
2	40.458	563166	7775	34.784	36.504
3	43.718	842453	10408	52.034	48.869
Total		1619054	21299	100.000	100.000

Crystal Structure Report for (*S*)-17 (CCDC-973168)

A specimen of $C_{16}H_{14}Cl_2$, approximate dimensions 0.270 mm x 0.400 mm x 0.440 mm, was used for the X-ray crystallographic analysis. The X-ray intensity data were measured.

The total exposure time was 9.95 hours. The frames were integrated with the Bruker SAINT software package using a narrow-frame algorithm. The integration of the data using an orthorhombic unit cell yielded a total of 20983 reflections to a maximum θ angle of 73.55° (0.80 Å resolution), of which 2806 were independent (average redundancy 7.478, completeness = 98.9%, R_{int} = 3.25%, R_{sig} = 2.06%) and 2788 (99.36%) were greater than $2\sigma(F^2)$. The final cell constants of a = 5.6849(8) Å, b = 7.6858(10) Å, c = 32.425(4) Å, volume = 1416.7(3) $Å^3$, are based upon the refinement of the XYZ-centroids of 9863 reflections above 20 σ(I) with 11.83° < 2θ < 147.1°. Data were corrected for absorption effects using the multi-scan method (SADABS). The ratio of minimum to maximum apparent transmission was 0.724. The calculated minimum and maximum transmission coefficients (based on crystal size) are 0.2748 and 0.4188.

The structure was solved and refined using the Bruker SHELXTL Software Package, using the space group P 21 21 21, with Z = 4 for the formula unit, $C_{16}H_{14}Cl_2$. The final anisotropic full-matrix least-squares refinement on F^2 with 164 variables converged at R1 = 2.88%, for the observed data and wR2 = 8.08% for all data. The goodness-of-fit was 1.025. The largest peak in the final difference electron density synthesis was 0.276 $e^-/Å^3$ and the largest hole was -0.220 $e^-/Å^3$ with an RMS deviation of 0.036 $e^-/Å^3$. On the basis of the final model, the calculated density was 1.299 g/cm^3 and F(000), 576 e^-.

Table 1. Sample and crystal data for (*S*)-17.

Identification code	mary015	
Chemical formula	$C_{16}H_{14}Cl_2$	
Formula weight	277.17	
Temperature	200(2) K	
Wavelength	1.54178 Å	
Crystal size	0.270 x 0.400 x 0.440 mm	
Crystal system	orthorhombic	
Space group	P 21 21 21	
Unit cell dimensions	a = 5.6849(8) Å	α = 90°
	b = 7.6858(10) Å	β = 90°
	c = 32.425(4) Å	γ = 90°
Volume	1416.7(3) $Å^3$	
Z	4	
Density (calculated)	1.299 g/cm^3	
Absorption coefficient	3.932 mm^{-1}	
F(000)	576	

Table 2. Data collection and structure refinement for (*S*)-17.

Theta range for data collection	2.73 to 73.55°	
Index ranges	-7<=h<=7, -9<=k<=9, -32<=l<=39	
Reflections collected	20983	
Independent reflections	2806 [R(int) = 0.0325]	
Coverage of independent reflections	98.9%	
Absorption correction	multi-scan	
Max. and min. transmission	0.4188 and 0.2748	
Structure solution technique	direct methods	
Structure solution program	SHELXS-97 (Sheldrick, 2008)	
Refinement method	Full-matrix least-squares on F^2	
Refinement program	SHELXL-97 (Sheldrick, 2008)	
Function minimized	$\Sigma w(F_o^2 - F_c^2)^2$	
Data / restraints / parameters	2806 / 0 / 164	
Goodness-of-fit on F^2	1.025	
Δ/σ_{max}	0.001	
Final R indices	2788 data; I>2σ(I)	R1 = 0.0288, wR2 = 0.0806
	all data	R1 = 0.0290, wR2 = 0.0808
Weighting scheme	$w=1/[\sigma^2(F_o^2)+(0.0478P)^2+0.3632P]$ where $P=(F_o^2+2F_c^2)/3$	
Absolute structure parameter	0.0(0)	
Largest diff. peak and hole	0.276 and -0.220 eÅ^{-3}	
R.M.S. deviation from mean	0.036 eÅ^{-3}	

Table 3. Atomic coordinates and equivalent isotropic atomic displacement parameters (Å^2) for (*S*)-17.

U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x/a	y/b	z/c	U(eq)
C1	0.4469(3)	0.1683(2)	0.21565(5)	0.0349(4)
C2	0.4286(3)	0.1699(2)	0.25857(5)	0.0356(4)
C3	0.2386(3)	0.25589(19)	0.27638(5)	0.0325(3)
C4	0.0703(3)	0.3383(2)	0.25260(6)	0.0374(4)
C5	0.0940(3)	0.3351(2)	0.20982(5)	0.0356(4)
C6	0.2809(3)	0.25149(19)	0.19077(5)	0.0295(3)
C7	0.3019(3)	0.2518(2)	0.14363(5)	0.0341(3)
C8	0.2461(3)	0.0744(2)	0.12590(5)	0.0339(4)

	x/a	y/b	z/c	U(eq)
C9	0.0853(3)	0.0478(2)	0.09675(5)	0.0321(3)
C10	0.8084(3)	0.8693(2)	0.05562(5)	0.0325(3)
C11	0.7382(3)	0.7147(2)	0.03709(5)	0.0340(3)
C12	0.8825(3)	0.5704(2)	0.03977(5)	0.0314(3)
C13	0.0957(3)	0.5769(2)	0.06084(5)	0.0347(4)
C14	0.1620(3)	0.7316(2)	0.07961(5)	0.0331(3)
C15	0.0207(3)	0.8804(2)	0.07753(4)	0.0293(3)
C16	0.5463(4)	0.3127(3)	0.12941(6)	0.0493(5)
Cl1	0.21302(11)	0.26192(6)	0.330252(13)	0.05065(14)
Cl2	0.79771(10)	0.37802(6)	0.015223(13)	0.04754(14)

Table 4. Bond lengths (Å) for (*S*)-17.

C1-C2	1.396(2)	C1-C6	1.396(2)
C1-H1	0.95	C2-C3	1.391(2)
C2-H2	0.95	C3-C4	1.383(2)
C3-Cl1	1.7535(16)	C4-C5	1.394(3)
C4-H4	0.95	C5-C6	1.387(2)
C5-H5	0.95	C6-C7	1.533(2)
C7-C8	1.513(2)	C7-C16	1.537(3)
C7-H7	1.0	C8-C9	1.331(2)
C8-H8	0.95	C9-C15	1.476(2)
C9-H9	0.95	C10-C11	1.390(2)
C10-C15	1.403(2)	C10-H10	0.95
C11-C12	1.382(2)	C11-H11	0.95
C12-C13	1.392(2)	C12-Cl2	1.7470(16)
C13-C14	1.388(2)	C13-H13	0.95
C14-C15	1.400(2)	C14-H14	0.95
C16-H16A	0.98	C16-H16B	0.98
C16-H16C	0.98		

Table 5. Bond angles (°) for (*S*)-17.

C2-C1-C6	121.47(16)	C2-C1-H1	119.3
C6-C1-H1	119.3	C3-C2-C1	118.40(16)
C3-C2-H2	120.8	C1-C2-H2	120.8
C4-C3-C2	121.56(15)	C4-C3-Cl1	119.07(13)
C2-C3-Cl1	119.37(13)	C3-C4-C5	118.69(16)
C3-C4-H4	120.7	C5-C4-H4	120.7

C6-C5-C4	121.69(16)	C6-C5-H5	119.2
C4-C5-H5	119.2	C5-C6-C1	118.18(15)
C5-C6-C7	120.21(14)	C1-C6-C7	121.60(14)
C8-C7-C6	111.17(13)	C8-C7-C16	110.51(15)
C6-C7-C16	111.72(14)	C8-C7-H7	107.8
C6-C7-H7	107.8	C16-C7-H7	107.8
C9-C8-C7	123.52(15)	C9-C8-H8	118.2
C7-C8-H8	118.2	C8-C9-C15	127.21(16)
C8-C9-H9	116.4	C15-C9-H9	116.4
C11-C10-C15	121.17(15)	C11-C10-H10	119.4
C15-C10-H10	119.4	C12-C11-C10	119.25(15)
C12-C11-H11	120.4	C10-C11-H11	120.4
C11-C12-C13	121.22(15)	C11-C12-Cl2	119.12(13)
C13-C12-Cl2	119.64(13)	C14-C13-C12	118.87(15)
C14-C13-H13	120.6	C12-C13-H13	120.6
C13-C14-C15	121.53(14)	C13-C14-H14	119.2
C15-C14-H14	119.2	C14-C15-C10	117.94(15)
C14-C15-C9	123.30(14)	C10-C15-C9	118.76(15)
C7-C16-H16A	109.5	C7-C16-H16B	109.5
H16A-C16-H16B	109.5	C7-C16-H16C	109.5
H16A-C16-H16C	109.5	H16B-C16-H16C	109.5

Table 6. Torsion angles (°) for (*S*)-17.

C6-C1-C2-C3	0.5(3)	C1-C2-C3-C4	0.0(3)
C1-C2-C3-Cl1	-179.32(14)	C2-C3-C4-C5	-0.3(3)
Cl1-C3-C4-C5	179.03(14)	C3-C4-C5-C6	0.1(3)
C4-C5-C6-C1	0.4(3)	C4-C5-C6-C7	-179.60(16)
C2-C1-C6-C5	-0.7(3)	C2-C1-C6-C7	179.30(16)
C5-C6-C7-C8	-107.64(17)	C1-C6-C7-C8	72.4(2)
C5-C6-C7-C16	128.40(18)	C1-C6-C7-C16	-51.6(2)
C6-C7-C8-C9	125.84(17)	C16-C7-C8-C9	-109.5(2)
C7-C8-C9-C15	179.54(16)	C15-C10-C11-C12	-1.1(2)
C10-C11-C12-C13	0.4(2)	C10-C11-C12-Cl2	-178.22(12)
C11-C12-C13-C14	0.4(2)	Cl2-C12-C13-C14	179.00(12)
C12-C13-C14-C15	-0.5(2)	C13-C14-C15-C10	-0.2(2)
C13-C14-C15-C9	-179.49(15)	C11-C10-C15-C14	1.1(2)
C11-C10-C15-C9	-179.66(15)	C8-C9-C15-C14	-17.4(3)

C8-C9-C15-C10 163.35(17)

Table 7. Anisotropic atomic displacement parameters (Å^2) for (*S*)-17.

The anisotropic atomic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2} U_{11} + ... + 2 h k a^* b^* U_{12}]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	0.0362(8)	0.0336(8)	0.0350(8)	-0.0031(7)	0.0045(7)	0.0081(7)
C2	0.0397(9)	0.0326(9)	0.0346(8)	0.0025(7)	-0.0018(7)	0.0068(7)
C3	0.0408(8)	0.0257(7)	0.0309(8)	-0.0027(6)	0.0024(6)	-0.0039(7)
C4	0.0343(9)	0.0362(9)	0.0416(9)	-0.0057(7)	0.0050(7)	0.0053(7)
C5	0.0314(8)	0.0356(8)	0.0398(9)	-0.0008(7)	-0.0042(7)	0.0034(7)
C6	0.0323(7)	0.0252(7)	0.0309(7)	-0.0027(6)	-0.0032(6)	-0.0029(7)
C7	0.0397(8)	0.0326(8)	0.0301(8)	-0.0011(6)	-0.0027(6)	0.0026(8)
C8	0.0388(9)	0.0335(8)	0.0295(7)	-0.0006(6)	-0.0013(6)	0.0017(7)
C9	0.0370(8)	0.0318(8)	0.0274(7)	0.0020(6)	0.0005(6)	0.0007(7)
C10	0.0315(8)	0.0368(8)	0.0293(7)	0.0003(6)	-0.0016(6)	0.0065(7)
C11	0.0302(8)	0.0443(9)	0.0274(7)	0.0011(6)	-0.0014(6)	-0.0006(7)
C12	0.0382(9)	0.0322(8)	0.0237(7)	0.0011(6)	0.0016(6)	-0.0054(7)
C13	0.0373(9)	0.0369(9)	0.0299(8)	0.0035(7)	-0.0016(7)	0.0051(7)
C14	0.0305(8)	0.0412(8)	0.0276(7)	0.0004(6)	-0.0038(6)	0.0014(7)
C15	0.0317(8)	0.0349(8)	0.0213(7)	0.0017(6)	0.0025(6)	-0.0014(7)
C16	0.0550(12)	0.0561(12)	0.0367(9)	-0.0018(9)	0.0067(9)	-0.0162(10)
Cl1	0.0776(3)	0.0446(2)	0.0298(2)	-0.00252(16)	0.0078(2)	0.0012(2)
Cl2	0.0649(3)	0.0365(2)	0.0412(2)	-0.00480(16)	-0.0075(2)	-0.0067(2)

Table 8. Hydrogen atomic coordinates and isotropic atomic displacement

parameters (Å^2) for (*S*)-17.

	x/a	y/b	z/c	U(eq)
H1	0.5751	0.1094	0.2031	0.042
H2	0.5431	0.1137	0.2752	0.043
H4	-0.0589	0.3960	0.2652	0.045
H5	-0.0208	0.3917	0.1933	0.043
H7	0.1834	0.3358	0.1326	0.041
H8	0.3303	-0.0233	0.1361	0.041
H9	0.0031	0.1476	0.0872	0.038
H10	-0.2892	-0.0307	0.0534	0.039
H11	-0.4073	-0.2917	0.0228	0.041
H13	0.1941	-0.5228	0.0623	0.042
H14	0.3068	-0.2634	0.0942	0.04
H16A	0.6657	0.2302	0.1390	0.074
H16B	0.5790	0.4281	0.1409	0.074
H16C	0.5499	0.3187	0.0992	0.074