Catalytic Asymmetric Synthesis of Cyclic Sulfamides from Conjugated Dienes

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Supporting Information

Table of Contents

General methods	S-2
Experimental procedures	S-2
Characterization data	S-3
X-ray structure of product **4f**	S-9
HPLC data for determination of enantiomeric excess	S-17
NMR spectra of compounds	S-32

General methods. All commercial reagents were used without further purification. Column chromatography was performed with silica gel (230-400 mesh). ^{1}H NMR spectra were recorded on 300 or 400 MHz spectrometers at ambient temperature. ^{13}C NMR spectra were recorded on 75 or 100 MHz spectrometers at ambient temperature. IR spectra were recorded on a FT-IR spectrometer. Melting points were uncorrected. Optical rotations were recorded on an Autopol III polarimeter. Enantioselective excess was collected using Varian 9050 and Agilent 1100 HPLC instruments.

Olefin **3a** was purchased and used directly. Olefins **3b, 3d, 3m-n** were prepared from the corresponding α,ß-unsaturated aldehydes using $MePPh_3Br$. Olefins **3c, 3e-i** were prepared using preformed diethyl allylphosphonate and the corresponding aldehydes.[1] Olefins **3j-k** were prepared using 1-chloro-2,4-pentadiene and the corresponding alcohol.[2] Olefin **3l** was prepared from the corresponding alcohol.[3]

[1] a) P. Fourgeaud, C. Midrier, J.-P. Vors, J.-N. Volle, J.-L. Pirat, D. Virieux, *Tetrahedron* 2010, **66**, 758; b) G.A. Krauss, J. Kim, *Org. Lett*. 2004, **6**, 3115.
[2] a) K. Maruyama, N. Nagai, Y. Naruta, *J. Org. Chem*. 1986, **51**, 5083; b) M. Kimura, A. Ezoe, M. Mori, Y. Tamaru, *J. Am. Chem. Soc*. 2005, **127**, 201.
[3] R.K. Haynes, K.-P. Lam, K.-Y. Wu, I.D. Williams, L.-L. Yeung, *Tetrahedron* 1999, **55**, 89.

Representative catalytic asymmetric diamination procedure (Table 1, entry 1): To a flame-dried 1.5 mL vial equipped with a magnetic stir bar was added $Pd_2(dba)_3$ (0.0046 g, 0.005 mmol) and **L7** (0.0108 g, 0.02 mmol). The sealed vial was evacuated and filled with argon three times, followed by addition of toluene (0.1 mL, distilled from sodium). The mixture was immersed in an oil bath (65 ^{o}C) and stirred for 10 min. *trans*-Penta-1,3-diene (**3a**) (0.0136 g, 0.20 mmol) was added followed by *N,N'*-di-*tert*-butylthiadiaziridine 1,1-dioxide (**2**) (0.062 g, 0.30 mmol) in one portion and the reaction mixture was stirred at 65 ^{o}C for 3 h. The crude product was purified by flash chromatography (silica gel, 25:1 hexanes: ethyl acetate). A second column was used to remove excess dba (silica gel, toluene until the yellow color elutes, then 25:1 hexanes: ethyl acetate). Cyclic sulfamide **4a** was obtained as a white solid (0.053 g, 97% yield, 90% ee).

Representative diamination on gram scale (Table 1, entry 8): To a flame-dried 15 mL vial equipped with a magnetic stir bar was added $Pd_2(dba)_3$ (0.0915 g, 0.10 mmol) and **L7** (0.2476 g, 0.46 mmol). The sealed vial was evacuated and filled with argon three times, followed by addition of toluene (3.5 mL, distilled from sodium). The mixture was immersed in an oil bath (65 ^{o}C) and stirred for 10 min. *trans*-1-Phenyl-hexa-3,5-diene (**3h**) (1.10 g, 6.96 mmol) was added followed by *N,N'*-di-*tert*-butylthiadiaziridine 1,1-dioxide (**2**) (1.86 g, 9.03 mmol) in one portion and the reaction mixture was stirred at 65 ^{o}C for 3 h. The crude product was purified by flash chromatography (silica gel, 15:1 (v/v) hexanes: ethyl acetate). A

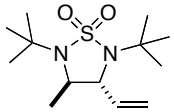
second column was used to remove excess dba (silica gel, toluene until the yellow color elutes, then 15:1 (v/v) hexanes: ethyl acetate). Cyclic sulfamide **4h** was obtained as a white solid (2.24 g, 88% yield, 92% ee).

Representative procedure for removal of *t*-Butyl groups (Scheme 3). A mixture of sulfamide **4g** (0.094 g, 0.27 mmol) in CF_3CO_2H-hexanes (1:1 (v/v), 2.4 mL) was stirred at room temperature for 7 h, concentrated, and subsequently purified by flash chromatography (silica gel, 4:1 (v/v) hexanes: ethyl acetate) to give compound **8g** as a white solid (0.058 g, 91% yield, 93% ee). R.G. Cornwall, B. Zhao, Y. Shi, *Org. Lett*. 2011, **13**, 434.

Representative procedure for deprotection to free diamine (Scheme 3). To a 25 mL round-bottom flask equipped with a magnetic stir bar and reflux condenser was added sulfamide **4g** (0.258 g, 0.736 mmol) and phenol (0.263 g, 2.79 mmol). 2N HBr (8.5 mL) was added and the mixture was vigorously refluxed for 24 h. The reaction was allowed to cool to room temperature and washed with Et_2O to remove excess phenol as monitored by TLC. The acidic aqueous layer was made basic with solid NaOH and extensively extracted with Et_2O as monitored by TLC. The combined organic layers were dried over Na_2SO_4, filtered, and concentrated to yield diamine **9g** as a pale yellow oil (0.114 g, 88% yield, 93% ee). S.V. Pansare, A.N. Rai, S.N. Kate, *Synlett* 1998, 623.

The ee of free diamine 9g was determined after derivatization to the di-*m*-toluoyl amide by the following procedure: To a 5 mL vial charged with diamine **9g** (0.008 g, 0.045 mmol) was added NaOH solution (2.0 M, 0.27 mL, 0.54 mmol) and CH_2Cl_2 (1.2 mL). Upon stirring at rt for 2 min, *m*-toluoyl chloride (0.015 g, 0.10 mmol) was added via syringe and the resulting mixture was stirred at rt for 10 min. A portion (30 μL) of the organic layer was diluted with Hex/IPA (1:1) (2 mL) and submitted to HPLC analysis (Chiralpak IC column, Hex:IPA 95:5, 1mL/min).
B. Zhao, X. Peng, S. Cui, Y. Shi, *J. Am. Chem. Soc*. 2010, **132**, 11009

Table 1, Entry 1

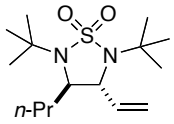


White solid; mp 91-95 ^oC; $[\alpha]^{20}_D$= +39.1 (c 1.0, $CHCl_3$) (90% ee); IR (film) 1371, 1275, 1138 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 6.07 (ddd, *J* = 17.1, 10.2, 6.9 Hz, 1H), 5.37 (d, *J* = 17.1 Hz, 1H), 5.21 (d, *J* = 10.2 Hz, 1H), 3.61 (d, *J* = 6.9 Hz, 1H), 3.34 (q, *J* = 6.6 Hz, 1H), 1.42 (d, *J* = 6.6 Hz, 3H), 1.41 (s, 9H), 1.39 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.1, 116.7, 63.3, 56.91, 56.89, 55.8, 29.0, 28.9, 22.9;

HRMS calcd. for $C_{13}H_{26}N_2NaO_2S$ $(M+Na)^+$: 297.1607. Found: 297.1603.

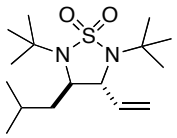
R.G. Cornwall, B. Zhao, Y. Shi, *Org. Lett*. 2011, **13**, 434.

Table 1, Entry 2



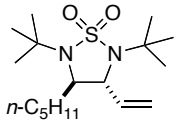
Pale yellow oil; $[\alpha]^{20}_D$= +82.4 (c 0.71, $CHCl_3$) (90% ee); IR (film) 1644, 1398, 1290 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 6.07 (ddd, *J* = 17.1, 10.2, 6.9 Hz, 1H), 5.34 (dd, *J* = 17.1, 0.6 Hz, 1H), 5.20 (dd, *J* = 10.2, 0.6 Hz, 1H), 3.76 (d, *J* = 6.9 Hz, 1H), 3.10 (dd, *J* = 11.1, 2.7 Hz, 1H), 1.98-1.81 (m, 1H), 1.66-1.51 (m, 1H), 1.44-1.36 (m, 2H), 1.40 (s, 9H), 1.39 (s, 9H), 0.97 (t, *J* = 7.8 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 139.7, 116.5, 60.1, 57.0, 56.9, 38.2, 28.9, 19.4, 14.0; Anal. calcd. for $C_{15}H_{30}N_2O_2S$: C, 59.56; H, 10.00; N, 9.26. Found: C, 59.33; H, 9.79; N, 9.41.

Table 1, Entry 3



White solid; mp 51-54 °C; $[\alpha]^{20}_D$= +29.7 (c 0.37, $CHCl_3$) (90% ee); IR (film) 1469, 1290, 1143 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 6.07 (ddd, *J* = 16.8, 10.2, 6.6 Hz, 1H), 5.34 (d, *J* = 16.8 Hz, 1H), 5.20 (d, *J* = 10.2 Hz, 1H), 3.75 (d, *J* = 6.9 Hz, 1H), 3.20 (dd, *J* = 11.7, 2.4 Hz, 1H), 2.07-1.88 (m, 1H), 1.75-1.57 (m, 2H), 1.39 (s, 9H), 1.38 (s, 9H), 0.97 (d, *J* = 6.6 Hz, 3H), 0.93 (d, *J* = 6.6 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 139.6, 116.5, 60.0, 58.7, 57.1, 56.8, 44.9, 29.0, 28.8, 25.7, 24.2, 21.3. Anal. calcd. for $C_{16}H_{32}N_2O_2S$: C, 60.72; H, 10.19; N, 8.85. Found: C, 60.90; H, 9.91; N, 8.86.

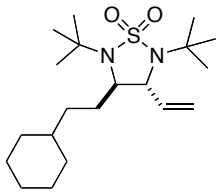
Table 1, Entry 4



Pale yellow oil; $[\alpha]^{20}_D$= +25.6 (c 0.5, $CHCl_3$) (91% ee); IR (film) 1468, 1290, 1196, 1143 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 6.07 (ddd, *J* = 17.1, 10.2, 6.6 Hz, 1H), 5.35 (d, *J* = 17.1 Hz, 1H), 5.20 (d, *J* = 10.2 Hz, 1H), 3.75 (d, *J* = 6.6 Hz, 1H), 3.07 (dd, *J* = 11.4, 2.7 Hz, 1H), 1.98-1.80 (m, 1H), 1.66-1.52 (m, 1H), 1.39 (s, 9H), 1.37 (s, 9H), 1.41-1.25 (m, 6H), 0.95-0.84 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 139.7, 116.5, 60.4, 60.1, 57.0, 56.9, 36.1, 31.7, 28.9, 25.9, 22.7, 14.1. Anal. calcd. for $C_{17}H_{34}N_2O_2S$: C, 61.77; H, 10.37; N, 8.48. Found: C, 61.92; H, 10.17; N, 8.56.

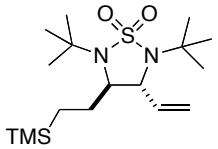
R.G. Cornwall, B. Zhao, Y. Shi, *Org. Lett*. 2011, **13**, 434.

Table 1, Entry 5



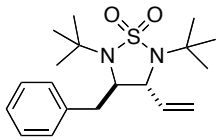
Colorless oil; $[\alpha]^{20}_D$= +24.3 (c 0.75, $CHCl_3$) (91% ee); IR (film) 1291, 1143 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 6.05 (ddd, *J* = 16.8, 10.2, 6.9 Hz, 1H), 5.36 (dd, *J* = 16.8, 0.9 Hz, 1H), 5.19 (dd, *J* = 10.2, 0.9 Hz, 1H), 3.73 (d, *J* = 6.9 Hz, 1H), 3.02 (dd, *J* = 11.1, 2.4 Hz, 1H), 1.97-1.81 (m, 1H), 1.80-1.54 (m, 6H), 1.40 (s, 9H), 1.38 (s, 9H), 1.28-1.12 (m, 6H), 0.80-0.99 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.6, 116.5, 60.9, 60.0, 56.9, 56.8, 37.8, 34.1, 33.8, 33.6, 33.3, 28.91, 28.89, 26.7, 26.5, 26.4; Anal. calcd. for $C_{20}H_{38}N_2O_2S$: C, 64.82; H, 10.34; N, 7.56. Found: C, 64.64; H, 10.49; N, 7.28.

Table 1, Entry 6



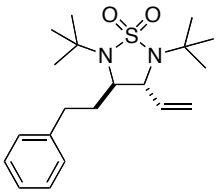
White solid; mp 129-131 °C; $[\alpha]^{20}_D$= +30.0 (c 0.83, $CHCl_3$) (91% ee); IR (film) 1279 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 6.06 (ddd, *J* = 17.1, 10.5, 6.6 Hz, 1H), 5.38 (d, *J* = 17.1 Hz, 1H), 5.21 (d, *J* = 10.5 Hz, 1H), 3.81 (d, *J* = 6.6 Hz, 1H), 2.97 (dd, *J* = 10.8, 2.7 Hz, 1H), 1.87-1.68 (m, 1H), 1.64-1.48 (m, 1H), 1.40 (s, 9H), 1.38 (s, 9H), 0.54 (td, *J* = 13.5, 4.2 Hz, 1H), 0.39 (td, *J* = 13.5, 4.2 Hz, 1H), 0.01 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.8, 116.5, 63.4, 59.4, 56.9, 30.5, 28.93, 28.90, 13.4, -1.66; HRMS calcd. for $C_{17}H_{36}N_2NaO_2SSi$ $(M+Na)^+$: 383.2159. Found: 383.2153.

Table 1, Entry 7



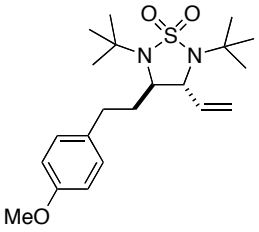
White solid; mp 86-88 °C; $[\alpha]^{20}_D$= +23.3 (c 0.90, $CHCl_3$) (93% ee); IR (film) 1398, 1292, 1143 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.39-7.33 (m, 2H), 7.31-7.25 (m, 1H), 7.24-7.19 (m, 2H), 5.95 (ddd, *J* = 17.2, 10.4, 6.8 Hz, 1H), 5.08 (d, *J* = 10.4 Hz, 1H), 5.04 (d, *J* = 17.2 Hz, 1H), 3.75 (d, *J* = 6.8 Hz, 1H), 3.48 (dd, *J* = 10.8, 4.4 Hz, 1H), 3.10 (dd, *J* = 14.0, 4.4 Hz, 1H) 3.03 (dd, *J* = 14.0, 10.8 Hz, 1H), 1.46 (s, 9H), 1.44 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.4, 137.8, 129.2, 129.1, 127.2, 116.7, 61.4, 59.3, 57.5, 57.1, 42.0, 29.2, 29.0; HRMS calcd. for $C_{19}H_{30}N_2NaO_2S$ $(M+Na)^+$: 373.1920. Found: 373.1923.

Table 1, Entry 8



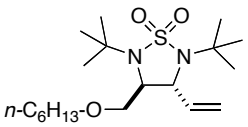
White solid; mp 50-54 ^{o}C; $[\alpha]^{20}_{D}$= +20.0 (c 0.39, $CHCl_3$) (92% ee); IR (film) 1454, 1286, 1142 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.34-7.12 (m, 5H), 6.03 (ddd, *J* = 16.8, 10.2, 6.9 Hz, 1H), 5.33 (d, *J* = 16.8 Hz, 1H), 5.21 (d, *J* = 10.2 Hz, 1H), 3.75 (d, *J* = 6.8 Hz, 1H), 3.10 (dd, *J* = 10.8, 2.4 Hz, 1H), 2.86-2.72 (m, 1H), 2.70-2.57 (m, 1H), 2.34-2.17 (m, 1H), 2.20-1.86 (m, 1H), 1.40 (s, 9H), 1.31 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.6, 139.2, 128.8, 128.6, 126.5, 116.8, 60.1, 59.5, 57.1, 56.9, 37.4, 32.6, 29.0, 28.8; HRMS calcd. for $C_{20}H_{32}N_2NaO_2S$ $(M+Na)^+$: 387.2077. Found: 387.2080.

Table 1, Entry 9



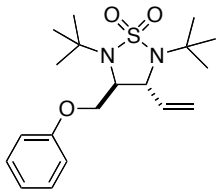
Yellow solid; mp 46-50 ^{o}C; $[\alpha]^{20}_{D}$= +11.8 (c 0.60, $CHCl_3$) (90% ee); IR (film) 1612, 1513, 1466, 1286, 1142 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.09 (d, *J* = 8.8 Hz, 2H), 6.85 (d, *J* = 8.8 Hz, 2H), 6.04 (ddd, *J* = 16.8, 10.4, 6.8 Hz, 1H), 5.34 (d, *J* = 16.8 Hz, 1H), 5.21 (d, *J* = 10.4 Hz, 1H), 3.79 (s, 3H), 3.77 (d, *J* = 6.8 Hz, 1H),), 3.09 (dd, *J* = 11.2, 2.4 Hz, 1H), 2.80-2.69 (m, 1H), 2.63-2.52 (m, 1H), 2.29-2.17 (m, 1H), 1.98-1.85 (m, 1H), 1.41 (s, 9H), 1.31 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.3, 139.2, 132.5, 129.5, 116.7, 114.2, 60.1, 59.4, 57.1, 56.9, 55.5, 37.5, 31.6, 29.0, 28.8; HRMS calcd. for $C_{21}H_{34}N_2NaO_3S$ $(M+Na)^+$: 417.2182. Found: 417.2195.

Table 1, Entry 10



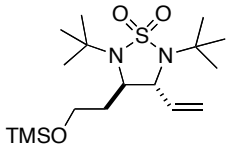
Pale yellow oil; $[\alpha]^{20}_{D}$= +21.7 (c 0.83, $CHCl_3$) (93% ee); IR (film) 1468, 1296, 1145 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.08 (ddd, *J* = 16.8, 10.4, 6.4 Hz, 1H), 5.44 (d, *J* = 16.8 Hz, 1H), 5.25 (d, *J* = 10.4 Hz, 1H), 4.15 (d, *J* = 6.4 Hz, 1H), 3.61-3.48 (m, 2H), 3.47-3.38 (m, 2H), 3.33 (dd, *J* = 10.8, 4.4 Hz, 1H), 1.65-1.53 (m, 2H), 1.43 (s, 9H), 1.40 (s, 9H), 1.40-1.22 (m, 6H), 0.90 (t, *J* = 6.4 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 139.0, 117.1, 71.7, 58.8, 58.1, 57.1, 31.8, 29.7, 28.9, 28.8, 26.1, 22.8, 14.2; HRMS calcd. for $C_{19}H_{38}N_2NaO_3S$ $(M+Na)^+$: 397.2495. Found: 397.2488.

Table 1, Entry 11



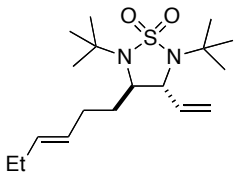
White solid; mp 80-82 °C; $[\alpha]^{20}_{D}$= +42.6 (c 0.97, $CHCl_3$) (93% ee); IR (film) 1600, 1498, 1398, 1295, 1144 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.38-7.25 (m, 2H), 7.03-6.90 (m, 3H), 6.14 (dddd, *J* = 16.8, 10.2, 6.3, 0.9 Hz, 1H), 5.50 (dd, *J* = 16.8, 0.9 Hz, 1H), 5.31 (dd, *J* = 10.2, 0.9 Hz, 1H), 4.21 (d, *J* = 6.3 Hz, 1H), 4.12 (t, *J* = 9.6 Hz, 1H), 3.95 (dd, *J* = 9.6, 3.9 Hz, 1H), 3.57 (dd, *J* = 10.2, 3.9 Hz, 1H), 1.43 (s, 9H), 1.39 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.3, 138.5, 129.9, 121.7, 117.6, 114.8, 68.6, 58.6, 58.3, 57.35, 57.30, 28.9, 28.7; Anal. calcd. for $C_{19}H_{30}N_2O_3S$: C, 62.26; H, 8.25; N, 7.64. Found: C, 62.58; H, 8.08; N, 7.67.

Table 1, Entry 12



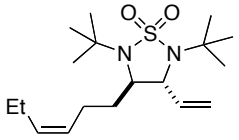
Colorless oil; $[\alpha]^{20}_{D}$= +28.9 (c 0.61, $CHCl_3$) (91% ee); IR (film) 1644, 1398, 1291 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 6.06 (ddd, *J* = 17.1, 10.5, 6.9 Hz, 1H), 5.33 (d, *J* = 17.1, Hz, 1H), 5.18 (d, *J* = 10.5 Hz, 1H), 3.99 (d, *J* = 6.9 Hz, 1H), 3.72-3.61 (m, 2H), 3.31 (dd, *J* = 10.5, 2.4 Hz, 1H), 2.15-2.00 (m, 1H), 1.85-1.72 (m, 1H), 1.37 (s, 9H), 1.35 (s, 9H), 0.08 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 139.4, 116.6, 60.7, 59.7, 58.6, 57.1, 56.9, 38.5, 29.0, 28.9, -0.47; Anal. calcd. for $C_{17}H_{36}N_2O_3SSi$: C, 54.21; H, 9.63; N, 7.44. Found: C, 54.02; H, 9.38; N, 7.55.

Table 1, Entry 13



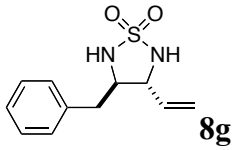
Colorless oil; $[\alpha]^{20}_{D}$= +25.0 (c 0.46, $CHCl_3$) (91% ee); IR (film) 1644, 1289, 1142 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 6.04 (ddd, *J* = 16.8, 9.9, 6.6 Hz, 1H), 5.57-5.44 (m, 1H), 5.42-5.30 (m, 1H), 5.34 (d, *J* = 16.8 Hz, 1H), 5.19 (d, *J* = 10.2 Hz, 1H), 3.78 (d, *J* = 7.2 Hz, 1H), 3.13 (dd, *J* = 11.1, 2.7 Hz, 1H), 2.22-1.88 (m, 5H), 1.74-1.56 (m, 1H), 1.39 (s, 9H), 1.37 (s, 9H), 0.95 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 139.4, 134.0, 127.3, 116.5, 59.9, 59.5, 57.1, 56.9, 35.5, 29.2, 28.9, 25.7, 14.0; Anal. calcd. for $C_{18}H_{34}N_2O_2S$: C, 63.11; H, 10.00; N, 8.18. Found: C, 63.34; H, 9.89; N, 7.93.

Table 1, Entry 14



Colorless oil; $[\alpha]^{20}_D$= +27.2 (c 1.2, $CHCl_3$) (91% ee); IR (film) 1727, 1644, 1398, 1290, 1143 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 6.06 (ddd, *J* = 17.1, 10.2, 6.9 Hz, 1H), 5.52-5.24 (m, 2H), 5.36 (d, *J* = 17.1 Hz, 1H), 5.21 (d, *J* = 10.2 Hz, 1H), 3.79 (d, *J* = 6.9 Hz, 1H), 3.11 (dd, *J* = 10.8, 2.4 Hz, 1H), 2.24-1.90 (m, 5H), 1.74-1.58 (m, 1H), 1.41 (s, 9H), 1.38 (s, 9H), 0.97 (t, *J* = 7.8 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 139.4, 133.3, 127.2, 116.7, 60.1, 59.7, 57.1, 56.9, 35.8, 29.0, 28.9, 23.7, 20.8, 14.4; Anal. calcd. for $C_{18}H_{34}N_2O_2S$: C, 63.11; H, 10.00; N, 8.18. Found: C, 62.83; H, 9.82; N, 7.96.

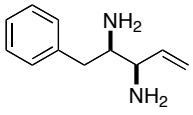
Scheme 3



8g

White solid; mp 77-79 °C; $[\alpha]^{20}_D$= +24.3 (c 0.75, $CHCl_3$) (93% ee); IR (film) 3261, 1497, 1167 cm^{-1}, ^1H NMR (300 MHz, $CDCl_3$) δ 7.40-7.19 (m, 5H), 5.76 (ddd, *J* = 17.1, 9.9, 7.2 Hz, 1H), 5.38 (d, *J* = 17.1 Hz, 1H), 5.29 (d, *J* = 9.9 Hz, 1H), 4.85 (d, *J* = 6.0 Hz, 1H), 4.67 (d, *J* = 7.2 Hz, 1H), 4.10-3.96 (m, 1H), 3.80-3.65 (m, 1H), 2.99 (dd, *J* = 14.1, 4.8 Hz, 1H), 2.88 (dd, *J* = 14.1, 8.7 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 136.6, 133.6, 129.3, 129.0, 127.3, 120.7, 64.9, 63.5, 38.4. HRMS calcd. for $C_{11}H_{15}N_2O_2S$ ($M+H^+$): 239.0849. Found: 239.0847.

Scheme 3

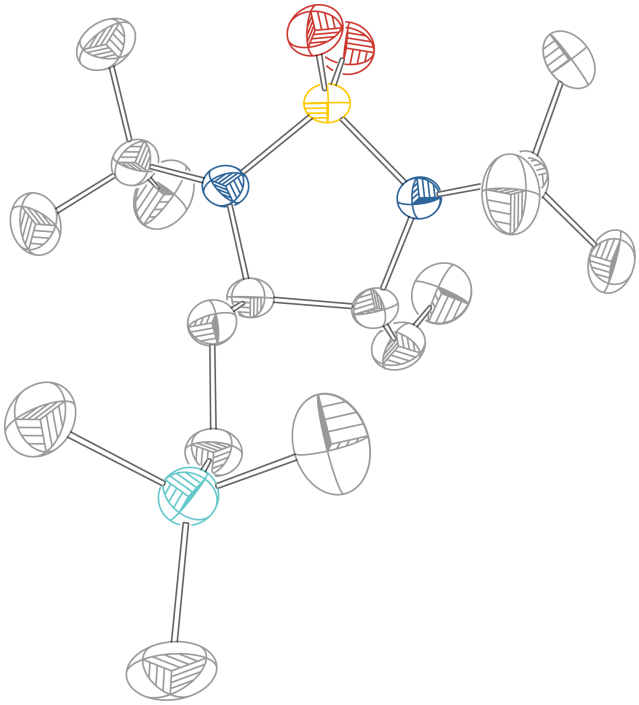


9g

Pale yellow oil; $[\alpha]^{20}_D$= +43.7 (c 0.71, $CHCl_3$) (93% ee); IR (film) 3374, 3295, 1639, 1602, 1495, 1453 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.35-7.28 (m, 2H), 7.25-7.19 (m, 3H), 5.92 (ddd, *J* = 17.2, 10.4, 6.4 Hz, 1H), 5.25 (d, *J* = 17.2 Hz, 1H), 5.18 (d, *J* = 10.4 Hz, 1H), 3.29-3.22 (m, 1H), 2.99-2.89 (m, 2H), 2.50 (dd, *J* = 14.4, 10.4 Hz, 1H), 1.18 (brs, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.5, 139.8, 129.4, 128.7, 126.5, 115.4, 58.7, 57.1, 41.2.

H. Du, W. Yuan, B. Zhao, Y. Shi, *J. Am. Chem. Soc*. 2007, **129**, 11688.

X-ray Structure of 4f.



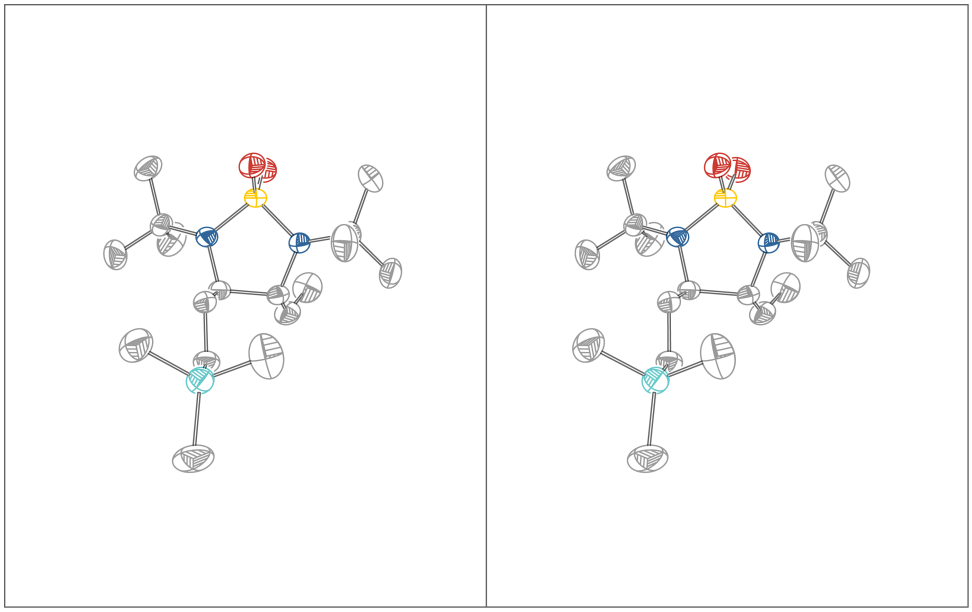


Table 1. Crystal data and structure refinement for **4f.**

Identification code	ys200	
Empirical formula	C17 H36 N2 O2 S Si	
Formula weight	360.63	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P21 21 21	
Unit cell dimensions	a = 10.963(3) Å	α= 90∞.
	b = 11.569(4) Å	β= 90∞.
	c = 17.410(5) Å	γ = 90∞.
Volume	2208.0(12) $Å^3$	
Z	4	
Density (calculated)	1.085 Mg/m^3	
Absorption coefficient	0.211 mm^{-1}	
F(000)	792	
Crystal size	0.43 x 0.39 x 0.35 mm^3	
Theta range for data collection	2.11 to 28.43°.	
Index ranges	-14<=h<=14, -14<=k<=15, -23<=l<=23	
Reflections collected	44743	
Independent reflections	5432 [R(int) = 0.0255]	
Completeness to theta = 28.43∞	99.2 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9291 and 0.9150	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5432 / 0 / 217	
Goodness-of-fit on F^2	1.034	
Final R indices [I>2sigma(I)]	R1 = 0.0312, wR2 = 0.0857	
R indices (all data)	R1 = 0.0360, wR2 = 0.0892	
Absolute structure parameter	0.00(6)	
Largest diff. peak and hole	0.171 and -0.150 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (≈^{2}x 10^3) for ys200. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	7214(2)	9233(3)	4160(1)	98(1)
C(2)	5652(3)	10531(2)	5341(1)	93(1)
C(3)	6118(3)	7950(2)	5527(1)	99(1)
C(4)	4486(2)	8816(1)	4226(1)	51(1)
C(5)	4085(1)	9800(1)	3692(1)	44(1)
C(6)	3100(1)	9448(1)	3110(1)	40(1)
C(7)	3615(1)	8701(1)	2446(1)	39(1)
C(8)	2693(2)	7827(1)	2155(1)	56(1)
C(9)	2176(2)	7823(2)	1478(1)	72(1)
C(10)	1318(1)	10926(1)	2939(1)	49(1)
C(11)	321(2)	10167(2)	2616(2)	85(1)
C(12)	1194(2)	12179(2)	2658(1)	74(1)
C(13)	1246(2)	10965(2)	3822(1)	81(1)
C(14)	5182(1)	9436(1)	1435(1)	49(1)
C(15)	6234(2)	9777(2)	1969(1)	74(1)
C(16)	5339(2)	8175(2)	1184(1)	69(1)
C(17)	5154(2)	10195(2)	717(1)	81(1)
N(1)	3981(1)	9538(1)	1851(1)	40(1)
N(2)	2553(1)	10459(1)	2725(1)	44(1)
O(1)	4087(1)	11705(1)	2007(1)	63(1)
O(2)	2375(1)	10915(1)	1309(1)	68(1)
S(1)	3239(1)	10763(1)	1920(1)	42(1)
Si(1)	5871(1)	9140(1)	4819(1)	57(1)

Table 3. Bond lengths [≈] and angles [∞] for ys200.

C(1)-Si(1)	1.870(2)
C(2)-Si(1)	1.865(2)
C(3)-Si(1)	1.866(2)
C(4)-C(5)	1.534(2)
C(4)-Si(1)	1.8744(17)
C(5)-C(6)	1.5360(19)
C(6)-N(2)	1.4760(16)
C(6)-C(7)	1.5500(19)
C(7)-N(1)	1.4733(17)
C(7)-C(8)	1.516(2)
C(8)-C(9)	1.307(3)
C(10)-N(2)	1.5042(18)
C(10)-C(11)	1.510(3)
C(10)-C(12)	1.536(2)
C(10)-C(13)	1.541(3)
C(14)-N(1)	1.5078(18)
C(14)-C(17)	1.527(2)
C(14)-C(15)	1.532(3)
C(14)-C(16)	1.533(2)
N(1)-S(1)	1.6381(12)
N(2)-S(1)	1.6287(12)
O(1)-S(1)	1.4407(12)
O(2)-S(1)	1.4349(12)

C(5)-C(4)-Si(1)	114.70(11)
C(4)-C(5)-C(6)	113.86(12)
N(2)-C(6)-C(5)	112.02(11)
N(2)-C(6)-C(7)	104.56(11)
C(5)-C(6)-C(7)	112.53(10)
N(1)-C(7)-C(8)	112.69(12)
N(1)-C(7)-C(6)	104.89(10)
C(8)-C(7)-C(6)	112.17(12)
C(9)-C(8)-C(7)	126.30(16)
N(2)-C(10)-C(11)	110.52(13)
N(2)-C(10)-C(12)	109.86(13)
C(11)-C(10)-C(12)	111.51(17)
N(2)-C(10)-C(13)	107.64(13)
C(11)-C(10)-C(13)	110.56(18)

C(12)-C(10)-C(13)	106.61(16)
N(1)-C(14)-C(17)	109.30(14)
N(1)-C(14)-C(15)	110.19(12)
C(17)-C(14)-C(15)	111.37(16)
N(1)-C(14)-C(16)	107.99(13)
C(17)-C(14)-C(16)	108.45(16)
C(15)-C(14)-C(16)	109.46(16)
C(7)-N(1)-C(14)	121.66(11)
C(7)-N(1)-S(1)	112.44(9)
C(14)-N(1)-S(1)	122.43(9)
C(6)-N(2)-C(10)	122.56(11)
C(6)-N(2)-S(1)	111.94(9)
C(10)-N(2)-S(1)	123.45(10)
O(2)-S(1)-O(1)	114.22(8)
O(2)-S(1)-N(2)	111.05(8)
O(1)-S(1)-N(2)	111.78(7)
O(2)-S(1)-N(1)	112.33(7)
O(1)-S(1)-N(1)	109.99(7)
N(2)-S(1)-N(1)	96.10(6)
C(2)-Si(1)-C(3)	109.48(11)
C(2)-Si(1)-C(1)	110.56(15)
C(3)-Si(1)-C(1)	109.48(15)
C(2)-Si(1)-C(4)	109.65(10)
C(3)-Si(1)-C(4)	109.50(10)
C(1)-Si(1)-C(4)	108.15(9)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\approx^2$x 10^3) for ys200. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

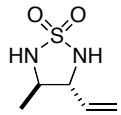
	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	64(1)	150(3)	79(1)	-24(2)	-13(1)	-11(2)
C(2)	139(2)	72(1)	68(1)	-20(1)	-30(1)	23(1)
C(3)	133(2)	79(2)	85(2)	13(1)	-48(2)	24(2)
C(4)	56(1)	50(1)	48(1)	8(1)	-6(1)	5(1)
C(5)	48(1)	41(1)	42(1)	3(1)	-1(1)	6(1)
C(6)	37(1)	38(1)	43(1)	9(1)	2(1)	2(1)
C(7)	38(1)	33(1)	45(1)	4(1)	-1(1)	0(1)
C(8)	56(1)	37(1)	76(1)	0(1)	6(1)	-11(1)
C(9)	62(1)	66(1)	88(1)	-15(1)	-10(1)	-21(1)
C(10)	41(1)	48(1)	59(1)	5(1)	5(1)	11(1)
C(11)	46(1)	84(1)	127(2)	-18(1)	5(1)	-2(1)
C(12)	67(1)	54(1)	102(2)	11(1)	14(1)	24(1)
C(13)	77(1)	100(2)	66(1)	8(1)	23(1)	37(1)
C(14)	51(1)	50(1)	47(1)	-5(1)	14(1)	-2(1)
C(15)	42(1)	94(1)	85(1)	-20(1)	11(1)	-10(1)
C(16)	74(1)	57(1)	77(1)	-16(1)	22(1)	6(1)
C(17)	108(2)	80(1)	56(1)	13(1)	35(1)	9(1)
N(1)	42(1)	35(1)	42(1)	2(1)	4(1)	0(1)
N(2)	42(1)	44(1)	46(1)	11(1)	6(1)	11(1)
O(1)	71(1)	38(1)	79(1)	-2(1)	20(1)	-10(1)
O(2)	79(1)	76(1)	49(1)	12(1)	-11(1)	22(1)
S(1)	49(1)	34(1)	41(1)	6(1)	2(1)	2(1)
Si(1)	68(1)	58(1)	45(1)	-4(1)	-17(1)	12(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (≈^{2}x 10 ^{3}) for ys200.

	x	y	z	U(eq)
H(1A)	7202	9961	3896	146
H(1B)	7177	8616	3792	146
H(1C)	7953	9169	4453	146
H(2A)	4841	10560	5550	139
H(2B)	5765	11165	4992	139
H(2C)	6235	10587	5751	139
H(3A)	6861	8087	5804	149
H(3B)	6173	7226	5260	149
H(3C)	5446	7926	5880	149
H(4A)	4648	8135	3917	62
H(4B)	3815	8632	4569	62
H(5A)	4793	10080	3414	52
H(5B)	3777	10432	4002	52
H(6)	2458	9014	3376	47
H(7)	4341	8289	2628	47
H(8)	2473	7238	2491	67
H(9A)	2367	8395	1123	87
H(9B)	1619	7247	1352	87
H(11A)	386	10144	2067	128
H(11B)	-460	10476	2758	128
H(11C)	404	9400	2819	128
H(12A)	1250	12198	2108	111
H(12B)	1836	12639	2875	111
H(12C)	419	12484	2815	111
H(13A)	455	11237	3976	122
H(13B)	1861	11479	4016	122
H(13C)	1378	10204	4025	122
H(15A)	6264	9252	2396	111
H(15B)	6107	10549	2154	111
H(15C)	6990	9742	1692	111
H(16A)	4681	7962	848	104
H(16B)	5333	7683	1628	104
H(16C)	6101	8089	918	104
H(17A)	5902	10102	437	122

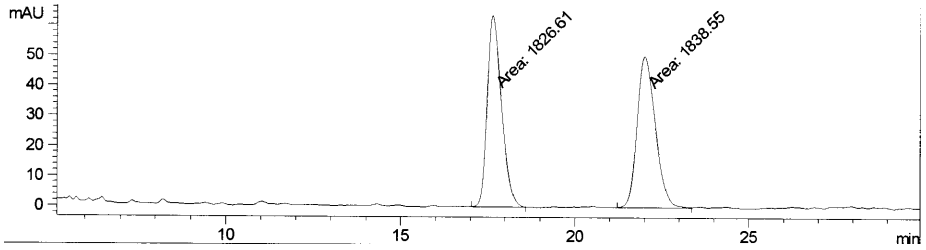
H(17B)	5062	10990	865	122
H(17C)	4480	9971	399	122

Table 1, entry 1



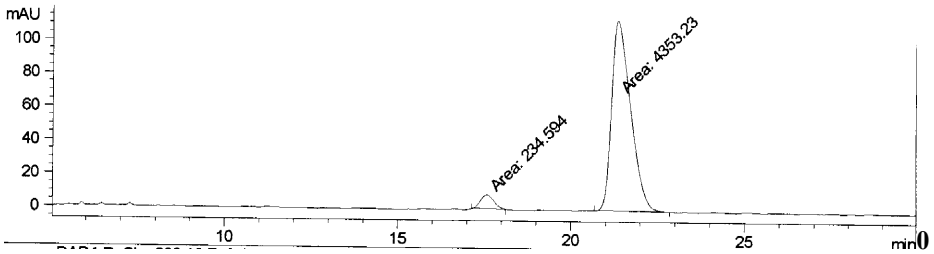
HPLC Conditions: **Column**: Chiralpak IC (Column No. IC00CE-MJ032), Daicel Chemical Industries, Ltd. **Eluent**: Hexanes/IPA (70/30); **Flow rate**: 1.0 mL/min; **Detection**: UV210

Racemic



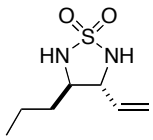
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.628	MM	0.4810	1826.61499	63.29630	49.8372
2	21.996	MM	0.6136	1838.54614	49.93508	50.1628

Chiral



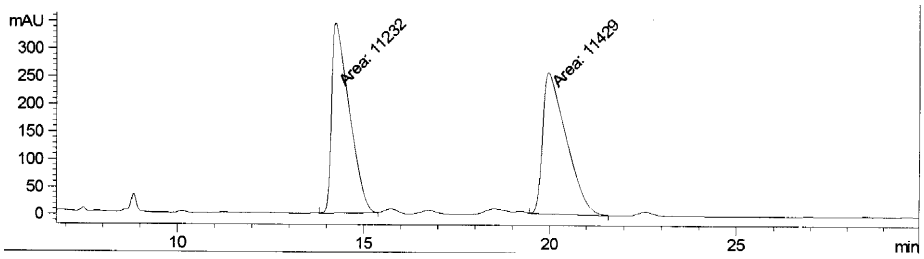
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.573	MM	0.4612	234.59427	8.47831	5.1134
2	21.313	MM	0.6390	4353.23145	113.53429	94.8866

Table 1, entry 2



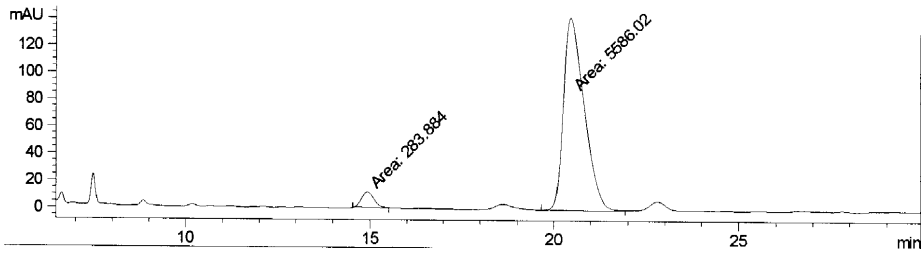
HPLC Conditions: **Column**: Chiralpak IC (Column No. IC00CE-MJ032), Daicel Chemical Industries, Ltd. **Eluent**: Hexanes/IPA (70/30); **Flow rate**: 1.0 mL/min; **Detection**: UV210

Racemic



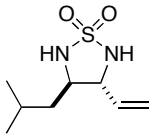
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.231	MM	0.5422	1.12320e4	345.23639	49.5652
2	19.955	MM	0.7421	1.14290e4	256.66498	50.4348

Chiral



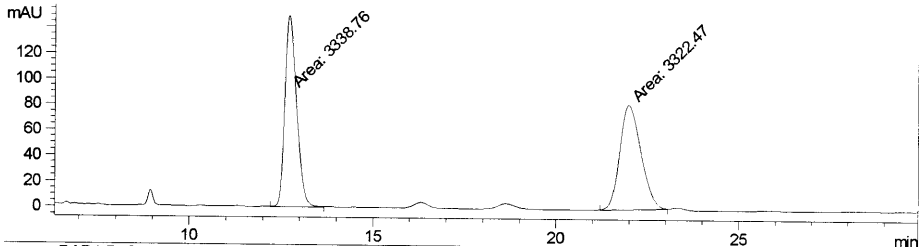
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.919	MM	0.4087	283.88440	11.57713	4.8363
2	20.430	MV	0.6551	5586.01807	142.11674	95.1637

Table 1, entry 3



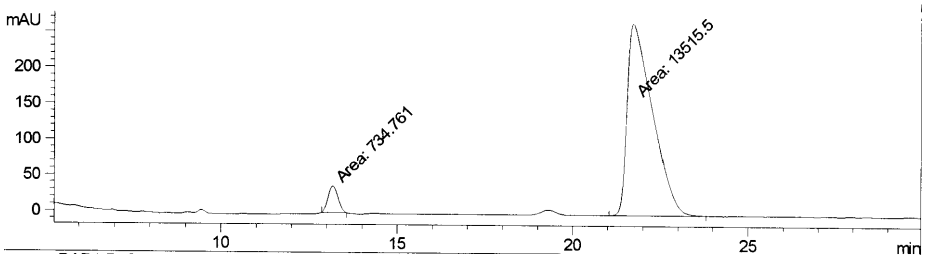
HPLC Conditions: **Column**: Chiralpak IC (Column No. IC00CE-MJ032), Daicel Chemical Industries, Ltd. **Eluent**: Hexanes/IPA (70/30); **Flow rate**: 1.0 mL/min; **Detection**: UV210

Racemic



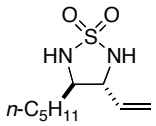
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.695	MV	0.3721	3338.75513	149.54039	50.1223
2	21.972	MM	0.6782	3322.46582	81.65154	49.8777

Chiral



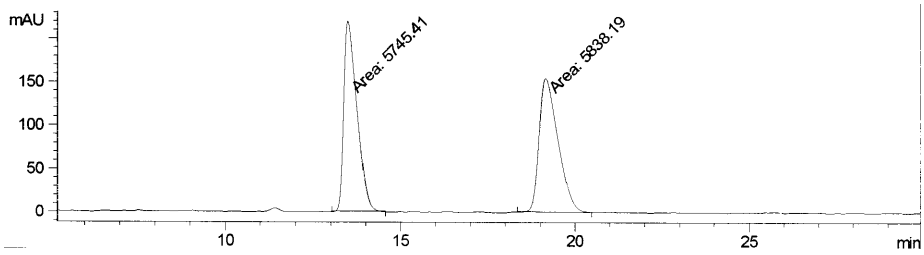
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.159	MM	0.3310	734.76068	36.99473	5.1561
2	21.711	MM	0.8444	1.35155e4	266.78217	94.8439

Table 1, entry 4



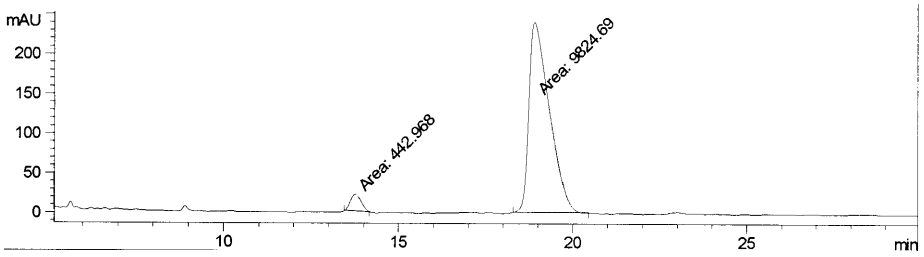
HPLC Conditions: **Column**: Chiralpak IC (Column No. IC00CE-MJ032), Daicel Chemical Industries, Ltd. **Eluent**: Hexanes/IPA (70/30); **Flow rate**: 1.0 mL/min; **Detection**: UV210

Racemic



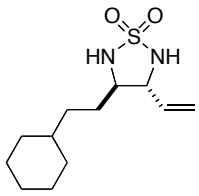
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.483	VM	0.4363	5745.41406	219.47356	49.5995
2	19.151	MM	0.6325	5838.19385	153.83551	50.4005

Chiral



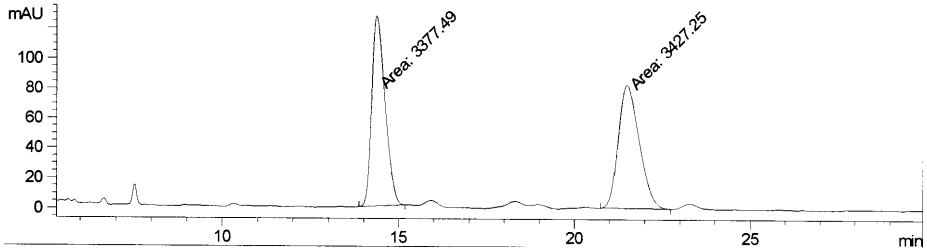
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.760	MM	0.3485	442.96762	21.18212	4.3142
2	18.903	MM	0.6847	9824.68945	239.13670	95.6858

Table 1, entry 5



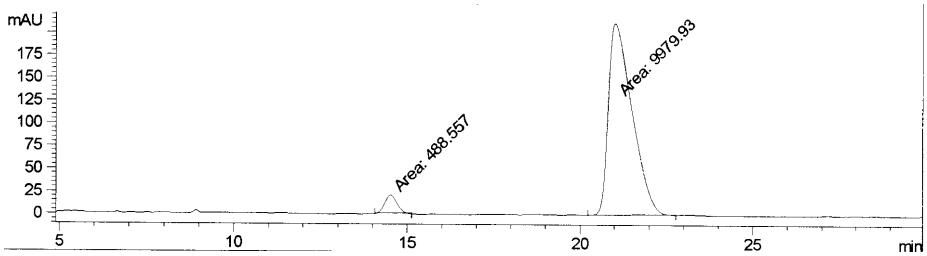
HPLC Conditions: **Column**: Chiralpak IC (Column No. IC00CE-MJ032), Daicel Chemical Industries, Ltd. **Eluent**: Hexanes/IPA (70/30); **Flow rate**: 1.0 mL/min; **Detection**: UV210

Racemic



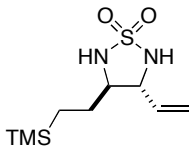
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.360	MM	0.4438	3377.49414	126.82595	49.6344
2	21.479	MM	0.6945	3427.24829	82.24599	50.3656

Chiral



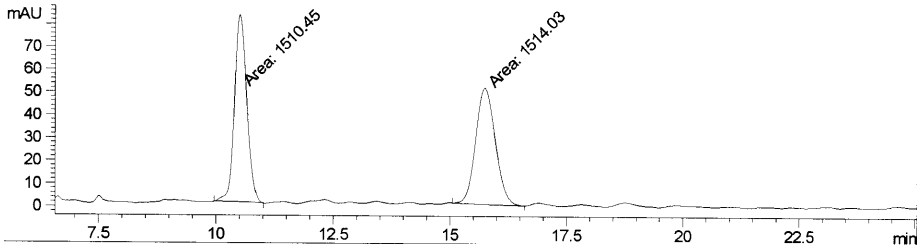
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.508	MM	0.4061	488.55731	20.04921	4.6669
2	21.005	MV	0.7876	9979.92969	211.19110	95.3331

Table 1, entry 6



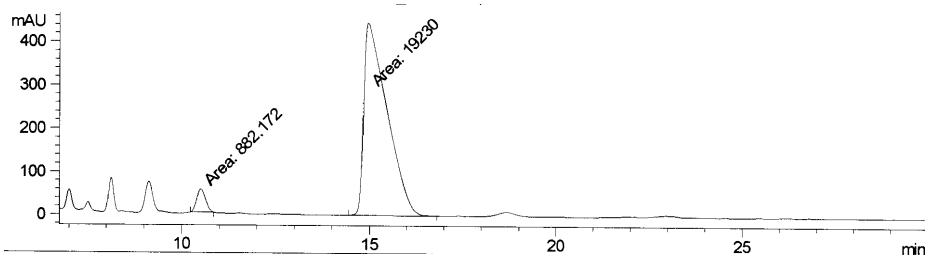
HPLC Conditions: **Column**: Chiralpak IC (Column No. IC00CE-MJ032), Daicel Chemical Industries, Ltd. **Eluent**: Hexanes/IPA (70/30); **Flow rate**: 1.0 mL/min; **Detection**: UV210

Racemic



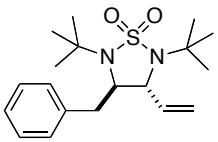
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.502	MM	0.3063	1510.44727	82.18114	49.9408
2	15.742	MM	0.4963	1514.02917	50.84820	50.0592

Chiral



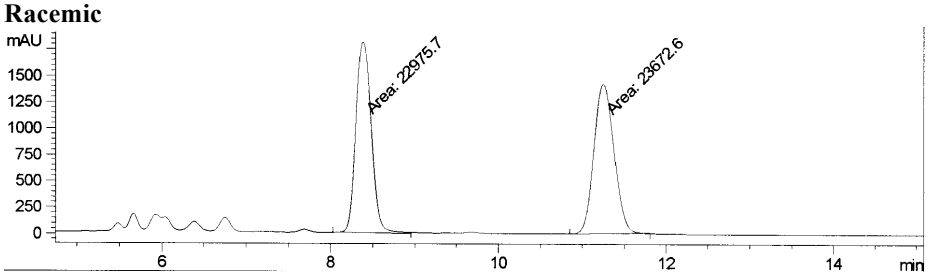
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.501	MM	0.2748	882.17200	53.49947	4.3863
2	14.952	VM	0.7204	1.92300e4	444.92090	95.6137

Table 1, entry 7



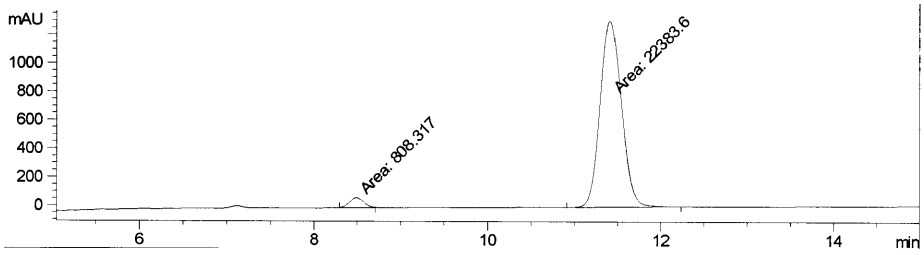
HPLC Conditions: **Column**: Chiralpak IC (Column No. IC00CE-MJ032), Daicel Chemical Industries, Ltd. **Eluent**: Hexanes/IPA (80/20); **Flow rate**: 1.0 mL/min; **Detection**: UV210

Racemic



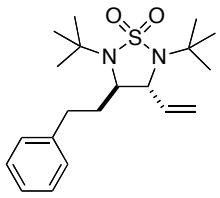
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.379	MM	0.2112	2.29757e4	1812.76794	49.2531
2	11.248	MM	0.2778	2.36726e4	1420.46838	50.7469

Chiral



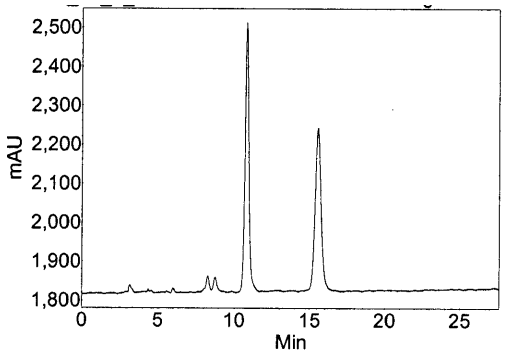
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.486	MM	0.1910	808.31714	70.55151	3.4853
2	11.410	MM	0.2849	2.23836e4	1309.51123	96.5147

Table 1, entry 8



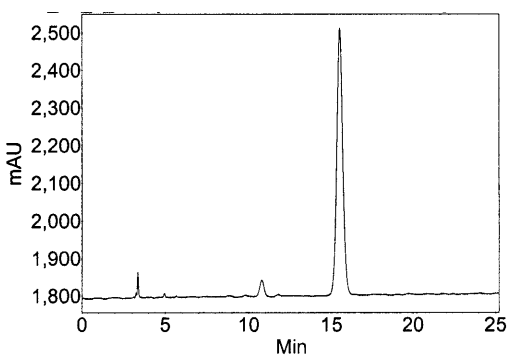
HPLC Conditions: **Column**: Chiralpak IC (Column No. IC00CE-NJ016), Daicel Chemical Industries, Ltd. **Eluent**: Hexanes/IPA (80/20); **Flow rate**: 1.0 mL/min; **Detection**: UV200

Racemic



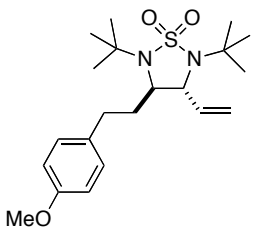
Index	Time [Min]	Area % [%]
2	10.81	50.909
1	15.55	49.091
Total		100.000

Chiral



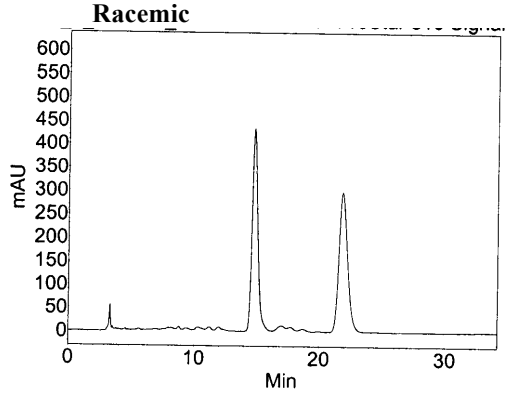
Index	Time [Min]	Area % [%]
2	10.78	3.771
1	15.51	96.229
Total		100.000

Table 1, entry 9



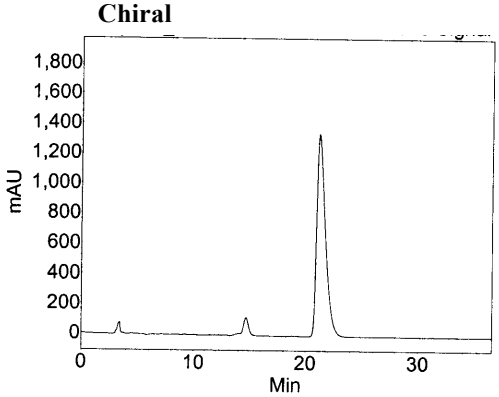
HPLC Conditions: **Column**: Chiralpak IC (Column No. IC00CE-NJ016), Daicel Chemical Industries, Ltd. **Eluent**: Hexanes/IPA (80/20); **Flow rate**: 1.0 mL/min; **Detection**: UV230

Racemic



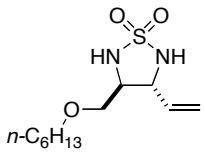
Index	Time [Min]	Area % [%]
1	14.77	49.691
2	21.84	50.309
Total		100.000

Chiral



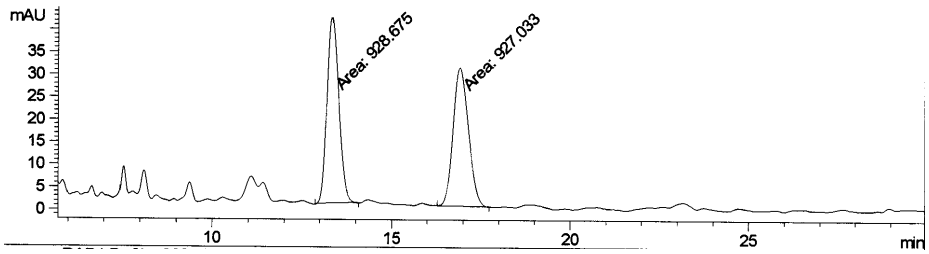
Index	Time [Min]	Area % [%]
2	14.68	4.931
1	21.18	95.069
Total		100.000

Table 1, entry 10



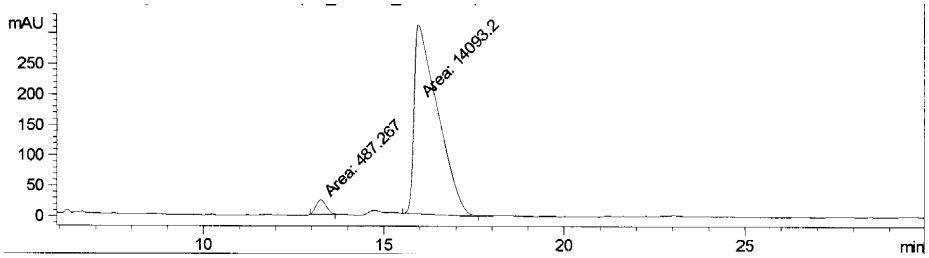
HPLC Conditions: **Column**: Chiralpak IC (Column No. IC00CE-MJ032), Daicel Chemical Industries, Ltd. **Eluent**: Hexanes/IPA (70/30); **Flow rate**: 1.0 mL/min; **Detection**: UV210

Racemic



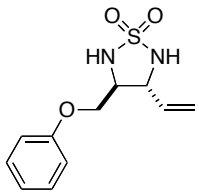
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.321	MM	0.3759	928.67542	41.18030	50.0442
2	16.891	MM	0.5038	927.03345	30.66966	49.9558

Chiral



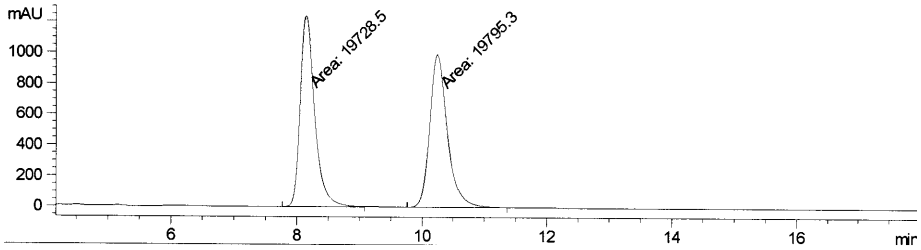
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.247	MM	0.3392	487.26651	23.94153	3.3419
2	15.952	MM	0.7573	1.40932e4	310.18173	96.6581

Table 1, entry 11



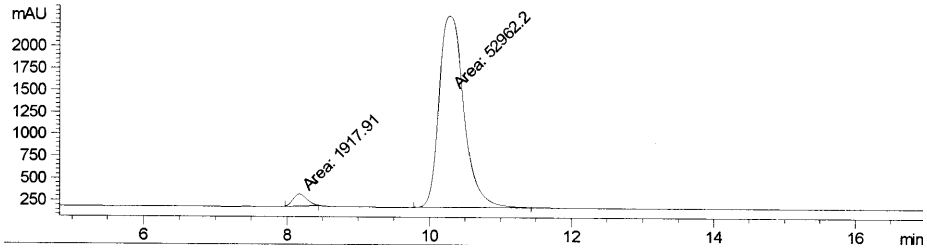
HPLC Conditions: **Column**: Chiralpak IA (Column No. IA00CE-ML034), Daicel Chemical Industries, Ltd. **Eluent**: Hexanes/IPA (80/20); **Flow rate**: 1.0 mL/min; **Detection**: UV210

Racemic



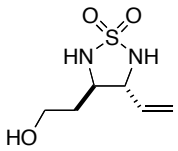
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.144	MM	0.2649	1.97285e4	1241.44409	49.9154
2	10.241	MM	0.3328	1.97953e4	991.23071	50.0846

Chiral



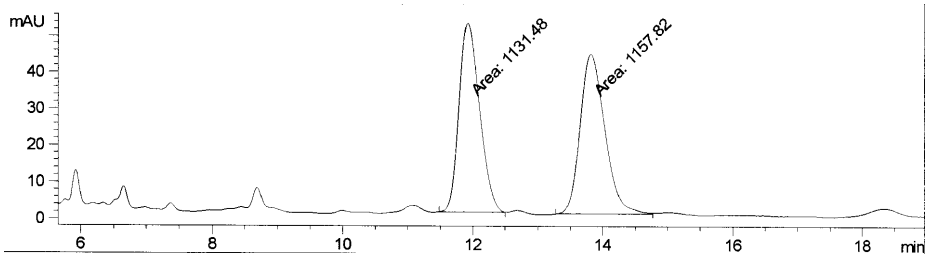
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.173	MM	0.2313	1917.90613	138.20412	3.4947
2	10.282	MM	0.4083	5.29622e4	2161.80103	96.5053

Table 1, entry 12



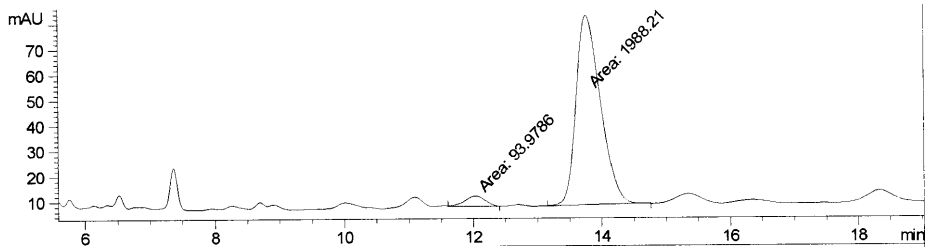
HPLC Conditions: **Column**: Chiralpak IC (Column No. IC00CE-MJ032), Daicel Chemical Industries, Ltd. **Eluent**: Hexanes/IPA (70/30); **Flow rate**: 1.0 mL/min; **Detection**: UV210

Racemic



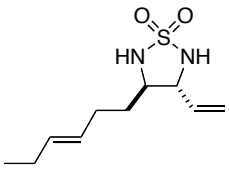
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.916	MM	0.3648	1131.47742	51.68816	49.4246
2	13.811	MV	0.4436	1157.82422	43.50105	50.5754

Chiral



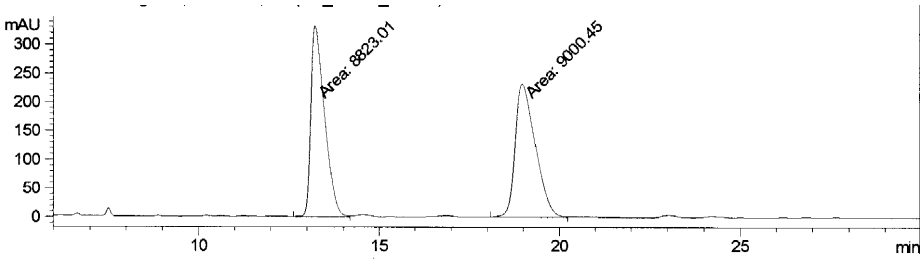
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.027	MM	0.3753	93.97863	4.17313	4.5135
2	13.763	MM	0.4439	1988.20605	74.64914	95.4865

Table 1, entry 13



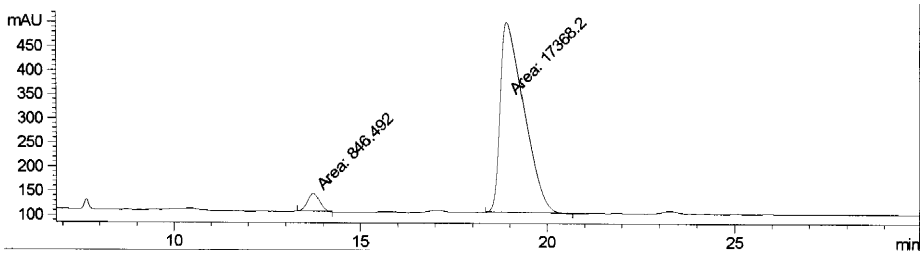
HPLC Conditions: **Column**: Chiralpak IC (Column No. IC00CE-MJ032), Daicel Chemical Industries, Ltd. **Eluent**: Hexanes/IPA (70/30); **Flow rate**: 1.0 mL/min; **Detection**: UV210

Racemic



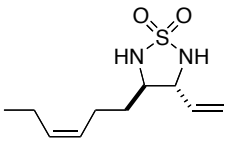
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.224	MV	0.4430	8823.01270	331.94012	49.5022
2	18.969	MV	0.6483	9000.44922	231.39961	50.4978

Chiral



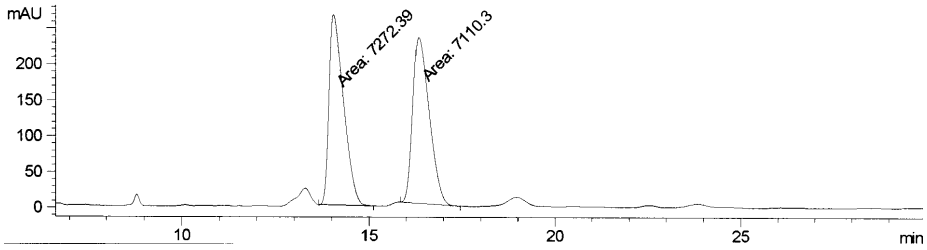
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.731	MM	0.3802	846.49249	37.10445	4.6473
2	18.903	MM	0.7354	1.73682e4	393.63812	95.3527

Table 1, entry 14



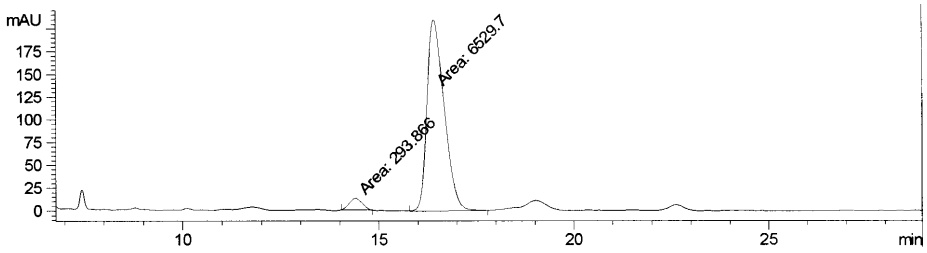
HPLC Conditions: **Column**: Chiralpak IC (Column No. IC00CE-MJ032), Daicel Chemical Industries, Ltd. **Eluent**: Hexanes/IPA (70/30); **Flow rate**: 1.0 mL/min; **Detection**: UV210

Racemic



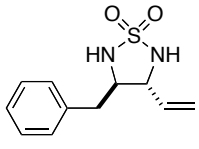
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.043	VM	0.4565	7272.38525	265.50165	50.5635
2	16.332	MM	0.5127	7110.29785	231.15279	49.4365

Chiral



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.401	MM	0.3835	293.86566	12.77134	4.3066
2	16.406	MM	0.5180	6529.70215	210.09157	95.6934

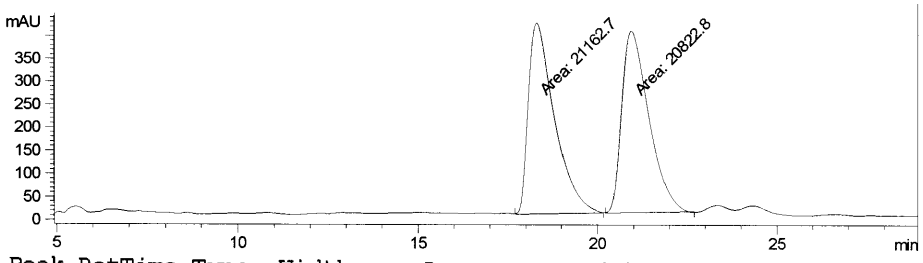
Scheme 3



8g

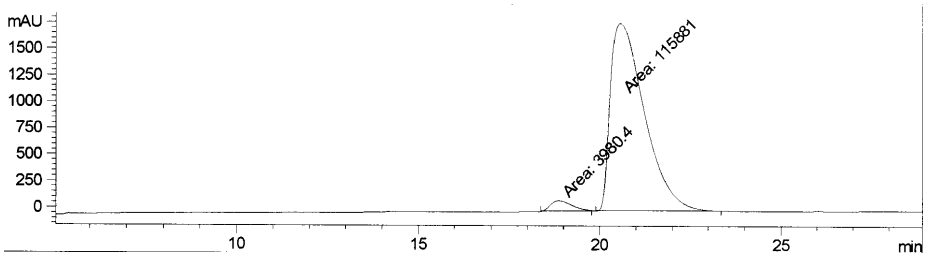
HPLC Conditions: **Column**: Chiralpak OD-H (Column No. ODHOCE-FB013), Daicel Chemical Industries, Ltd. **Eluent**: Hexanes/IPA (90/10); **Flow rate**: 1.0 mL/min; **Detection**: UV210

Racemic



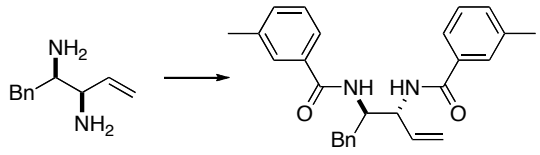
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.295	MM	0.8512	2.11627e4	414.35797	50.4048
2	20.932	MM	0.8811	2.08228e4	393.89938	49.5952

Chiral



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.863	MM	0.6797	3980.39795	97.59868	3.3208
2	20.550	VM	1.0912	1.15881e5	1769.96094	96.6792

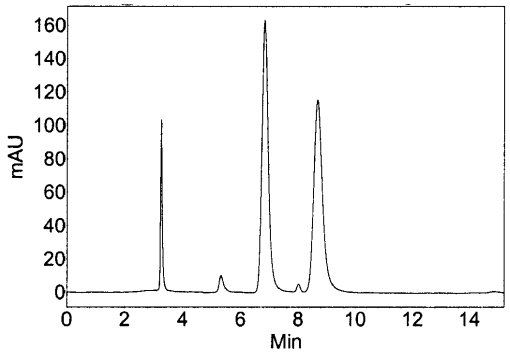
Scheme 3



9g

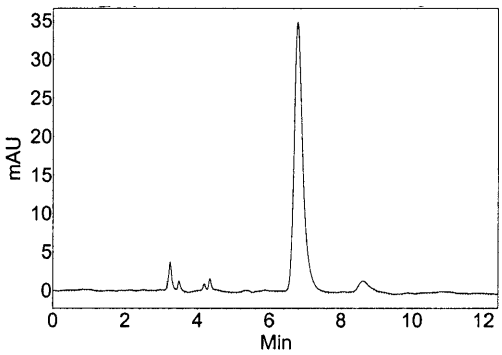
HPLC Conditions: **Column**: Chiralpak IC (Column No. IC00CE-NJ016), Daicel Chemical Industries, Ltd. **Eluent**: Hexanes/IPA (95/5); **Flow rate**: 1.0 mL/min; **Detection**: UV220

Racemic

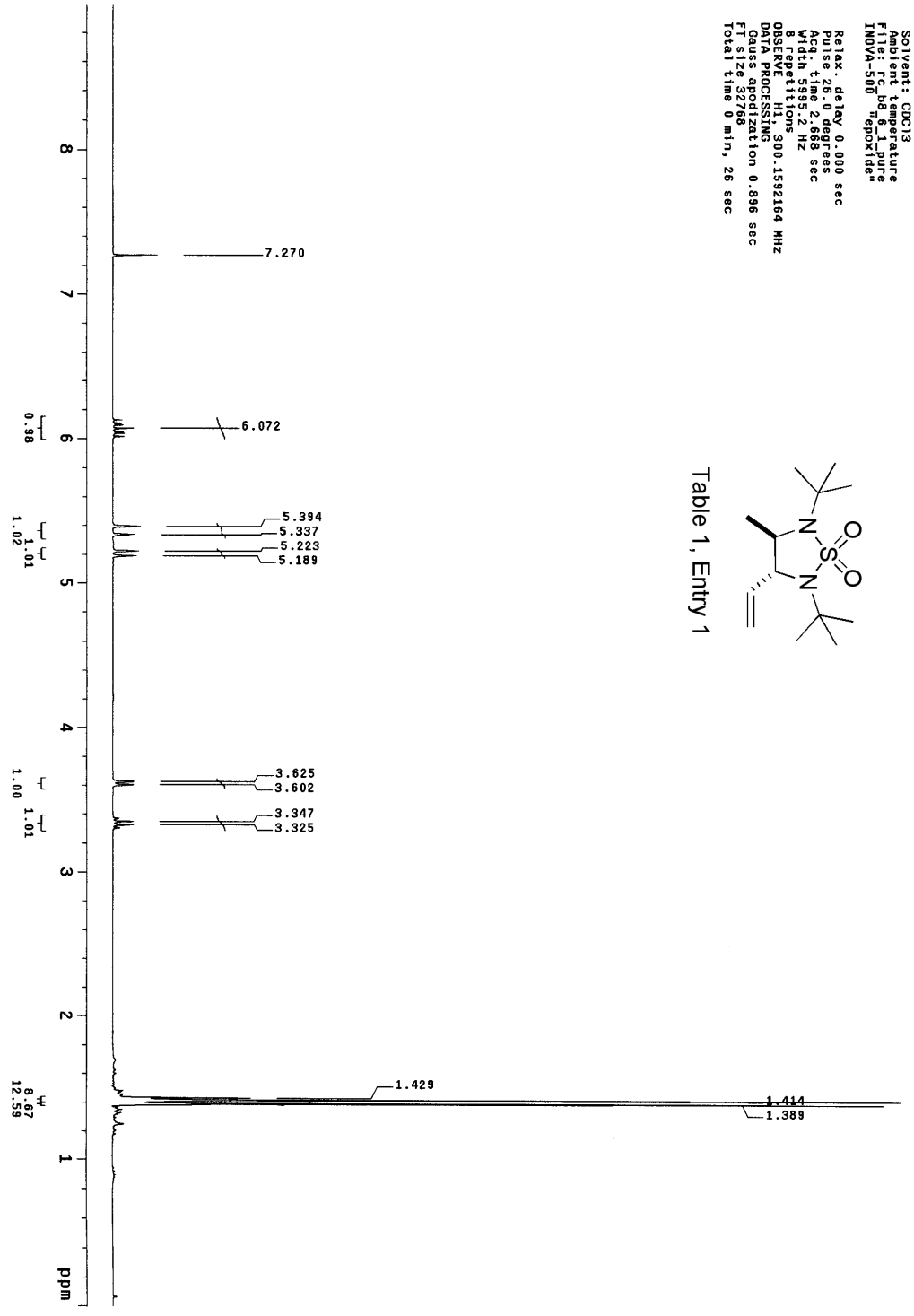


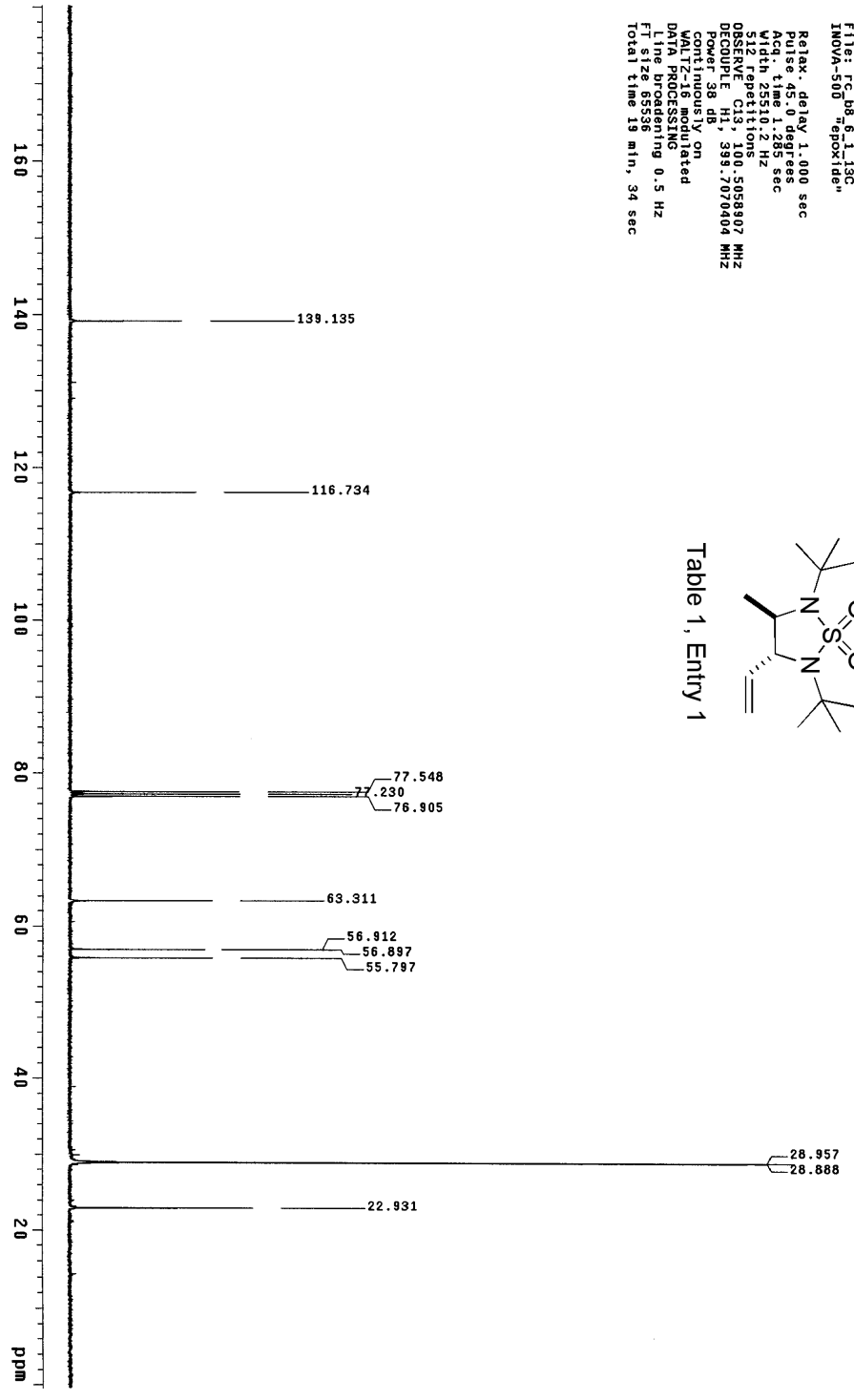
Index	Time [Min]	Area % [%]
1	6.83	49.933
2	8.68	50.067
Total		100.000

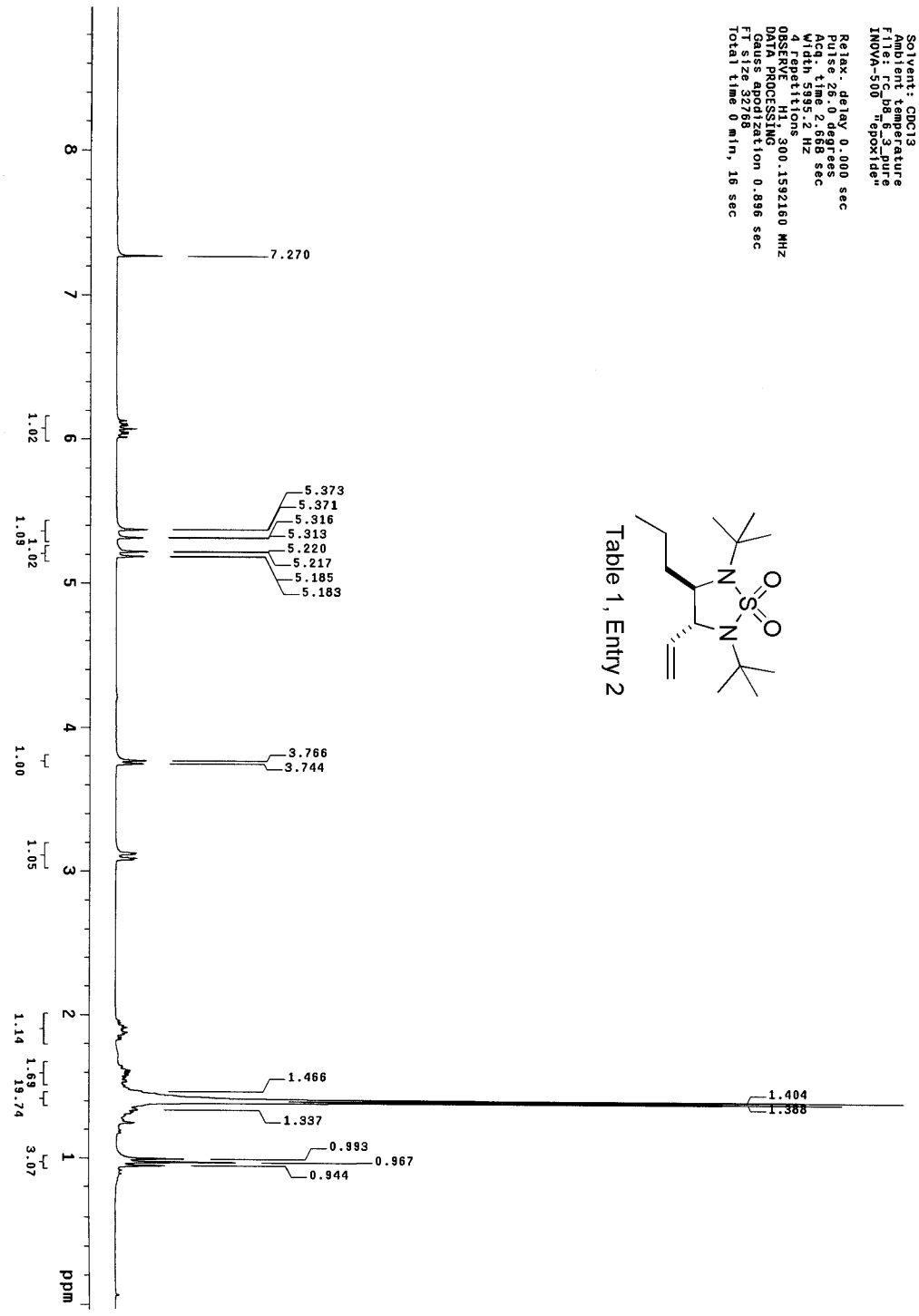
Chiral

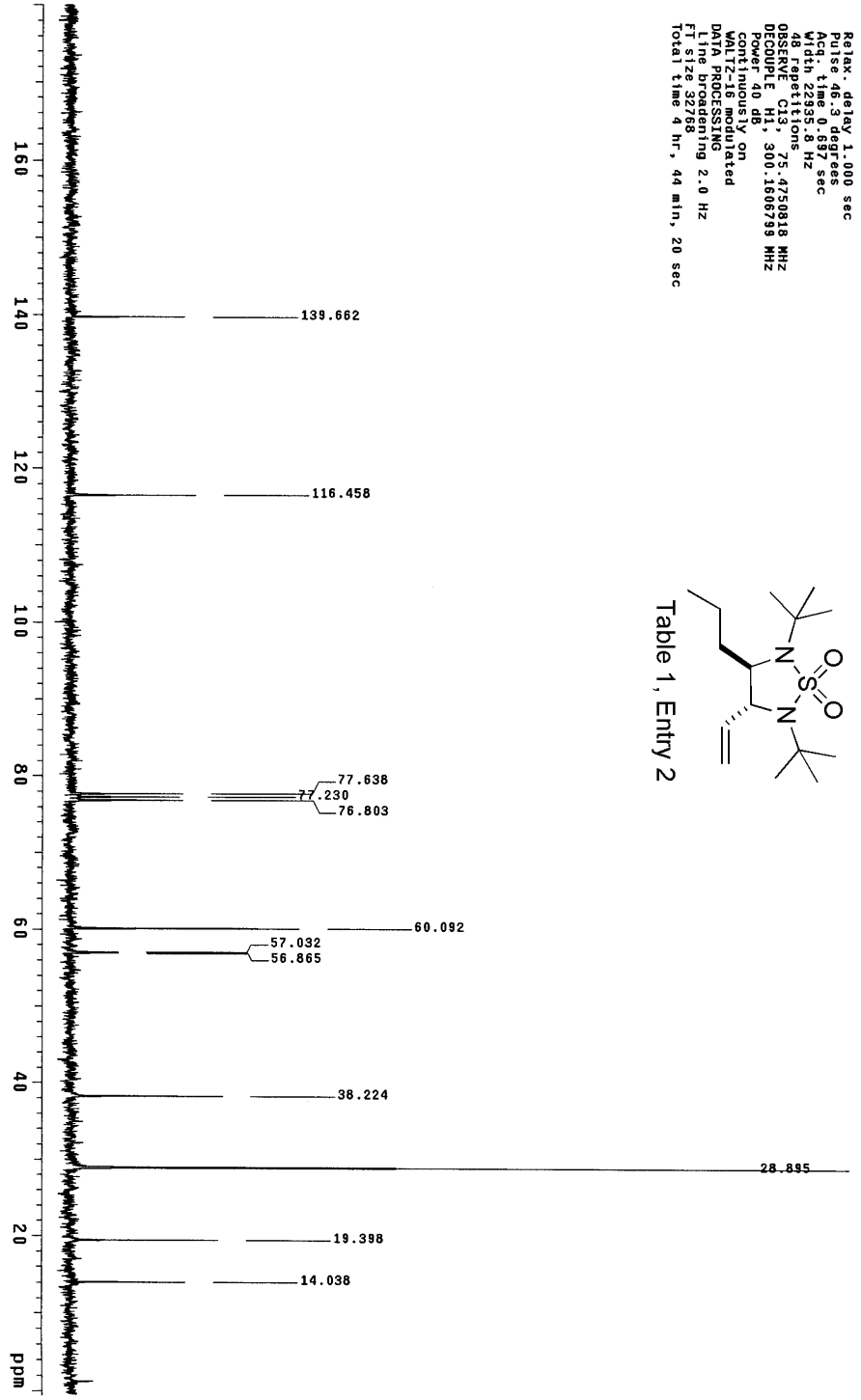


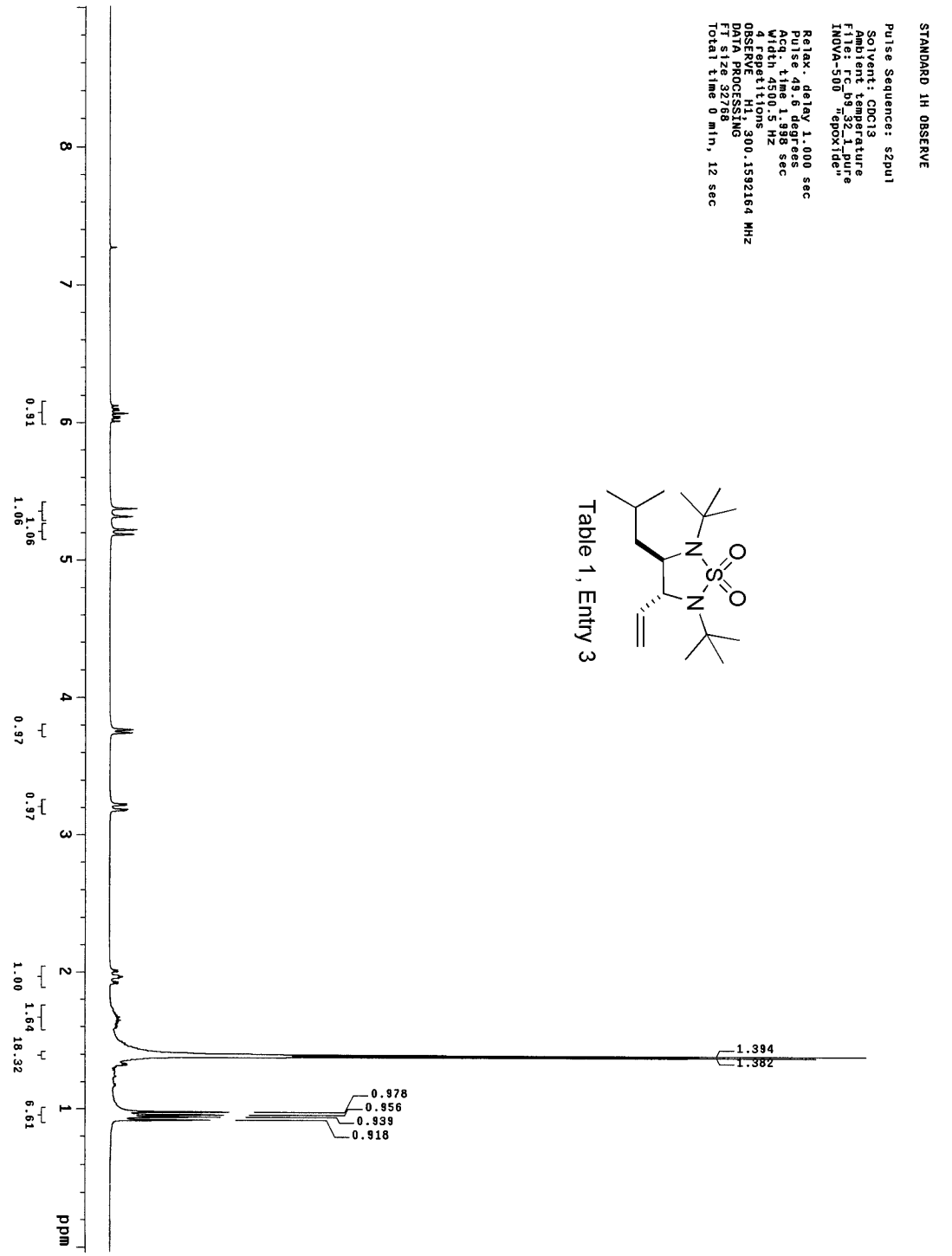
Index	Time [Min]	Area % [%]
1	6.78	96.604
2	8.63	3.396
Total		100.000

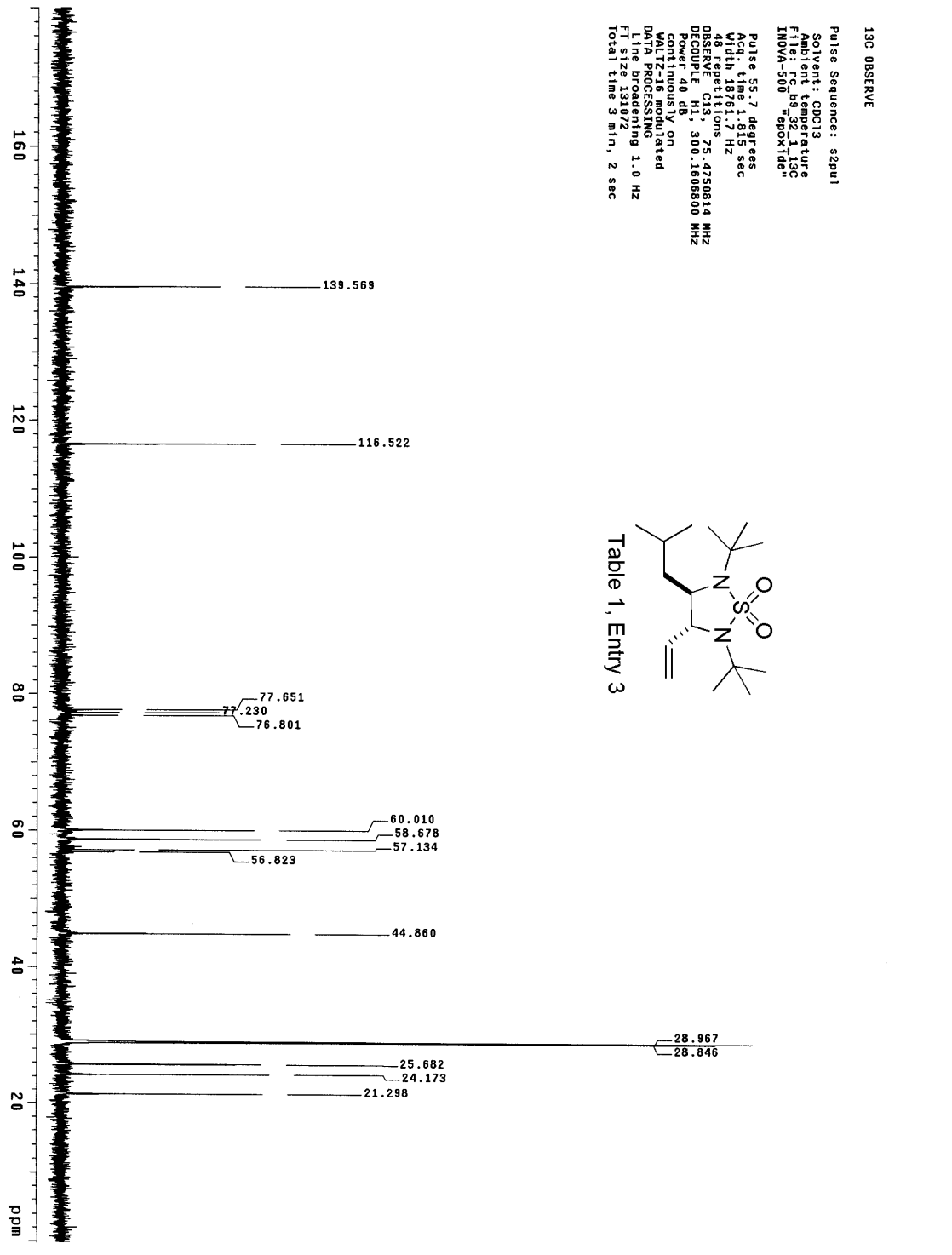

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: rc_b8_6_1_pure
INOVA-500 "epoxide"
Relax. delay 0.000 sec
Pulse 26.0 degrees
Acq. time 2.668 sec
Width 5995.2 Hz
8 repetitions
OBSERVE H1, 300.1592164 MHz
DATA PROCESSING
Gauss apodization 0.896 sec
FT size 32768
Total time 0 min, 26 sec
Table 1, Entry 1
7.270
6.072
5.394
5.337
5.223
5.189
3.625
3.602
3.347
3.325
1.429
1.414
1.389
0.98
1.02
1.01
1.00
1.01
8.67
12.59
ppm


b8-6-1
Archive directory: /home/DATA/walkup/cornwall
Sample directory: b8-6-1_20120316_01
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Sample #46, Operator: cornwall
File: rc_b8_6_1_13C
INOVA-500 "epoxide"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.285 sec
Width 25510.2 Hz
512 repetitions
OBSERVE C13, 100.5058907 MHz
DECOUPLE H1, 399.7070404 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 19 min, 34 sec
Table 1, Entry 1
139.135
116.734
77.548
77.230
76.905
63.311
56.912
56.897
55.797
28.957
28.888
22.931
160
140
120
100
80
60
40
20
ppm


STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: r_bo_3_pure
INOVA-500 "r_bo_3_pure"
Relax. delay 0.000 sec
Pulse 26.0 degrees
Acq. time 2.668 sec
Width 5995.2 Hz
4 repetitions
OBSERVE H1, 300.1592160 MHz
DATA PROCESSING
Gauss apodization 0.896 sec
FT size 32768
Total time 0 min, 16 sec
Table 1, Entry 2
7.270
5.373
5.371
5.316
5.313
5.220
5.217
5.185
5.183
3.766
3.744
1.466
1.404
1.388
1.337
0.993
0.967
0.944
1.02
1.09
1.02
1.00
1.05
1.14
1.69
19.74
3.07
ppm


13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: rc_b8_6_3_carbon
INOVA-500 "epoxide"
Relax. delay 1.000 sec
Pulse 45.3 degrees
Acq. time 0.697 sec
Width 22935.8 Hz
48 repetitions
OBSERVE C13, 75.4750818 MHz
DECOUPLE H1, 300.1606799 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 32768
Total time 4 hr, 44 min, 20 sec
Table 1, Entry 2
139.662
116.458
77.638
77.230
76.803
60.092
57.032
56.865
38.224
28.895
19.398
14.038
ppm


STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: rc-bg-32-1-pure
INOVA-500 "epoxide"
Relax. delay 1.000 sec
Pulse 49.6 degrees
Acq. time 1.998 sec
Width 4500.5 Hz
4 repetitions
OBSERVE H1, 300.1592164 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 12 sec
Table 1, Entry 3
1.394
1.382
0.978
0.956
0.939
0.918
0.91
1.06
0.97
0.97
1.00
1.64
18.32
6.61
ppm


13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: rc_b9_32_1_13C
INOVA-500 "epoxide"
Pulse 55.7 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
48 repetitions
OBSERVE C13, 75.4750814 MHz
DECOUPLE H1, 300.1606800 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 3 min, 2 sec
Table 1, Entry 3
139.569
116.522
77.651
77.230
76.801
60.010
58.678
57.134
56.823
44.860
28.967
28.846
25.682
24.173
21.298
160
140
120
100
80
60
40
20
ppm

STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: rc_b8_6_10-1H
INOVA-500 "epoxide"

Relax. delay 1.000 sec
Pulse 49.6 degrees
Acq. time 1.998 sec
Width 4500.5 Hz
4 repetitions
OBSERVE H1, 300.1592161 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 12 sec

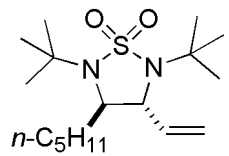
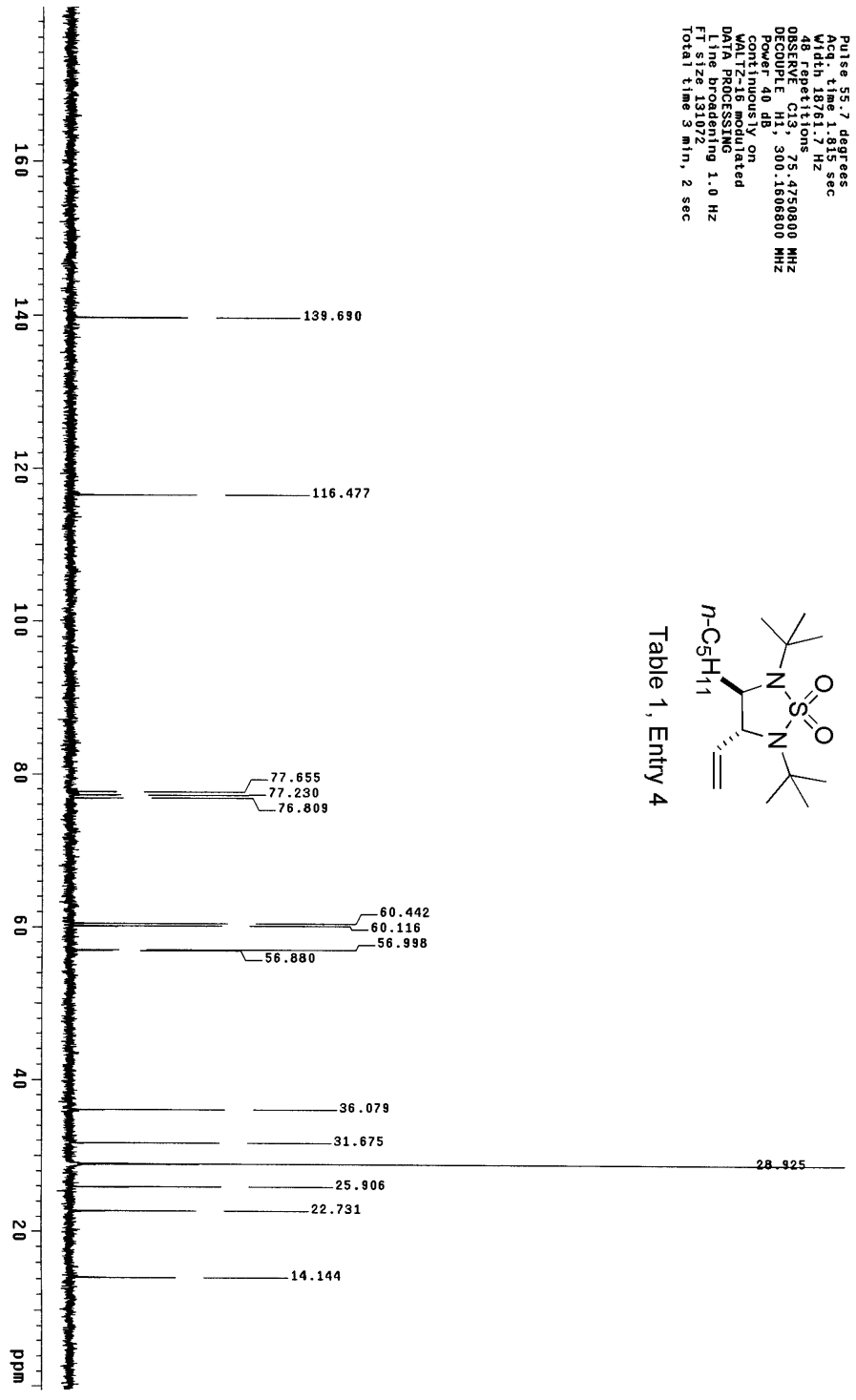


Table 1, Entry 4


13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: rc_b8_6_10-13C
INOVA-500 "epoxide"
Pulse 55.7 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
48 repetitions
OBSERVE C13, 75.4750800 MHz
DECOUPLE H1, 300.1606800 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 3 min, 2 sec
n-C5H11
Table 1, Entry 4
139.690
116.477
77.655
77.230
76.809
60.442
60.116
56.998
56.880
36.079
31.675
28.925
25.906
22.731
14.144
160
140
120
100
80
60
40
20
ppm

STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: rc_b9_32_2_pure
INOVA-500 "epoxide"

Relax. delay 1.000 sec
Pulse 49.6 degrees
Acq. time 1.998 sec
Width 4500.5 Hz
4 repetitions
OBSERVE H1, 300.1592159 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 12 sec

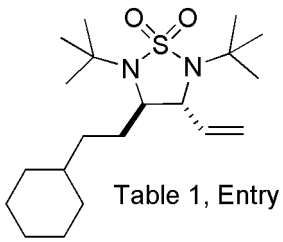
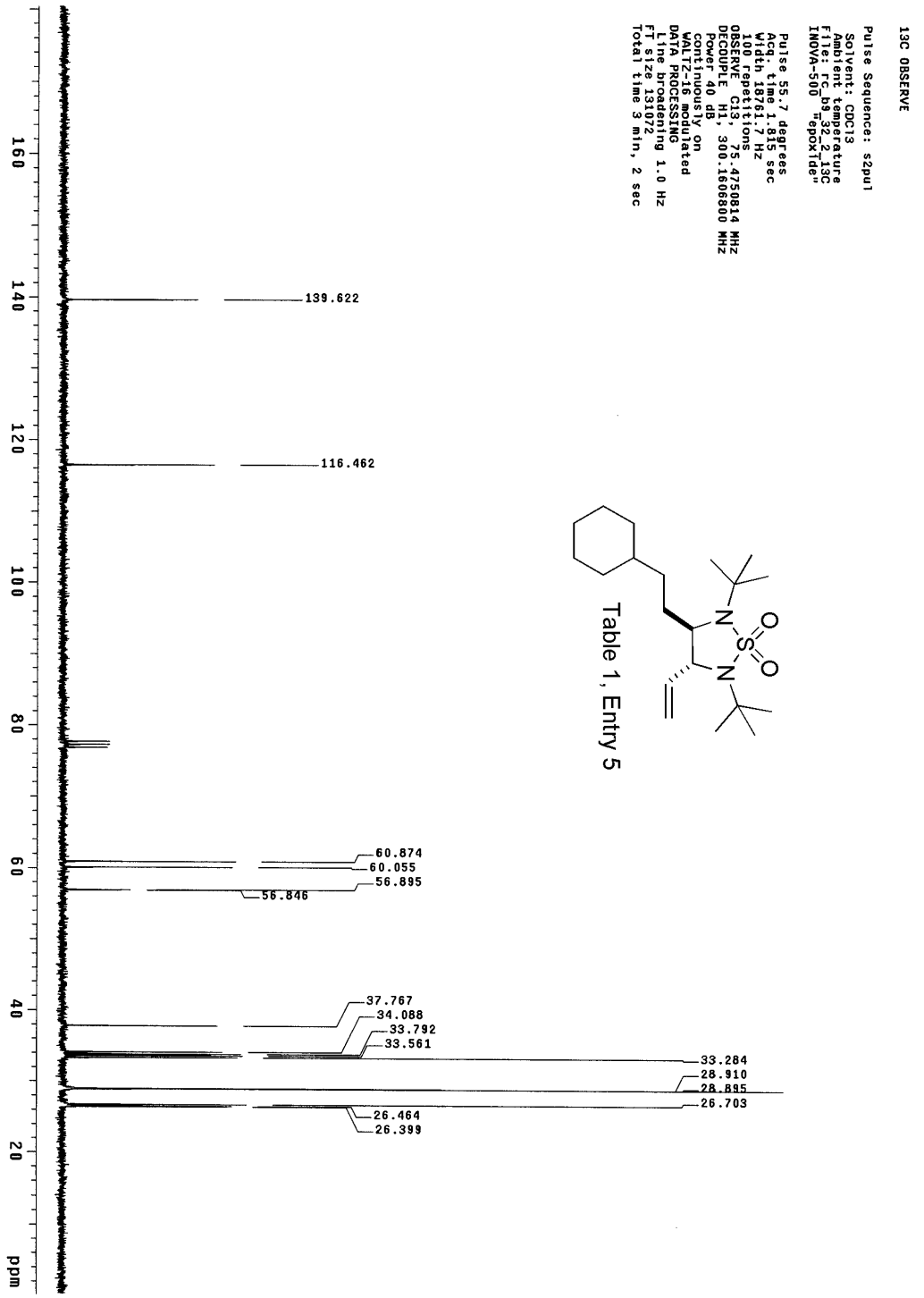
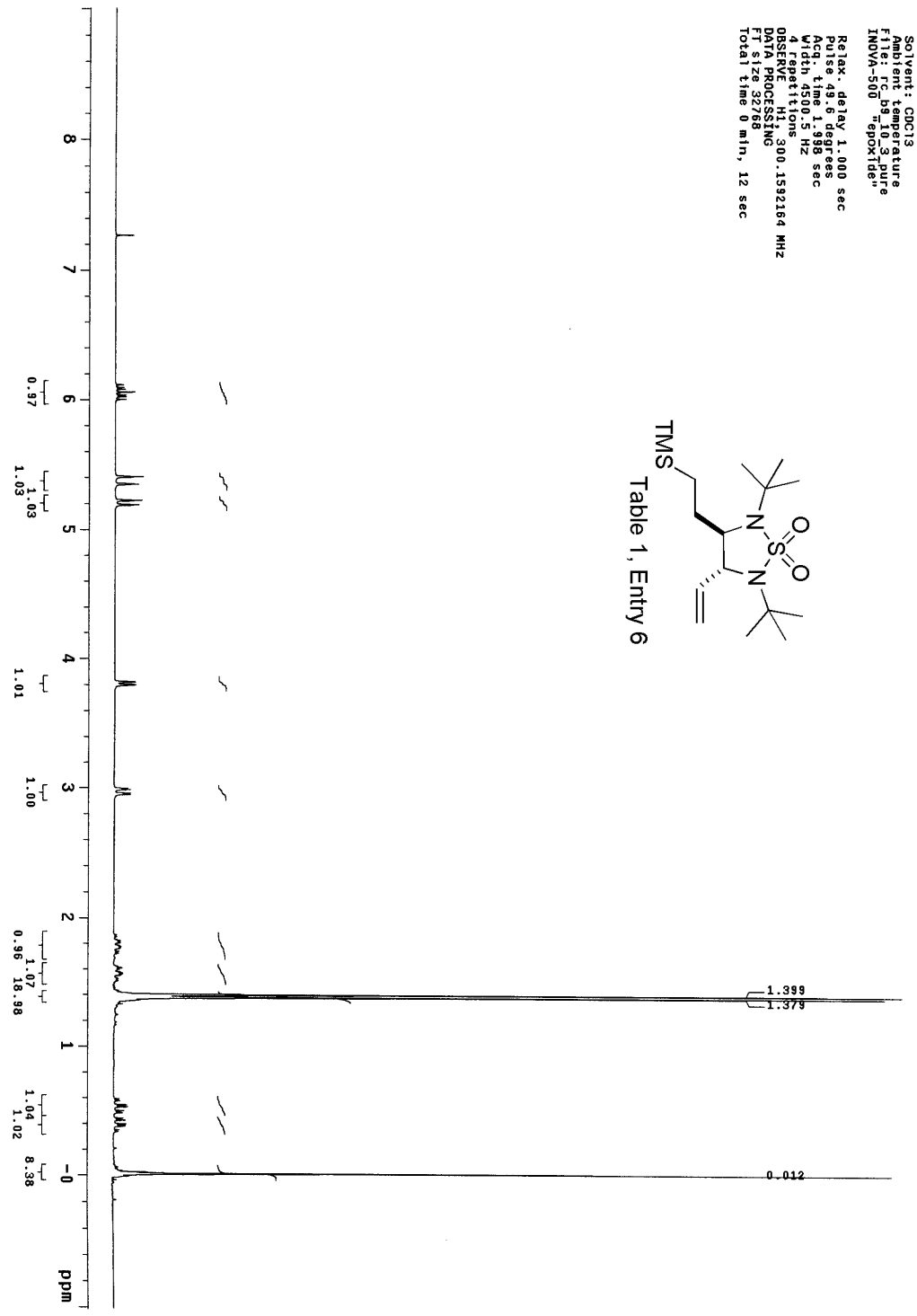
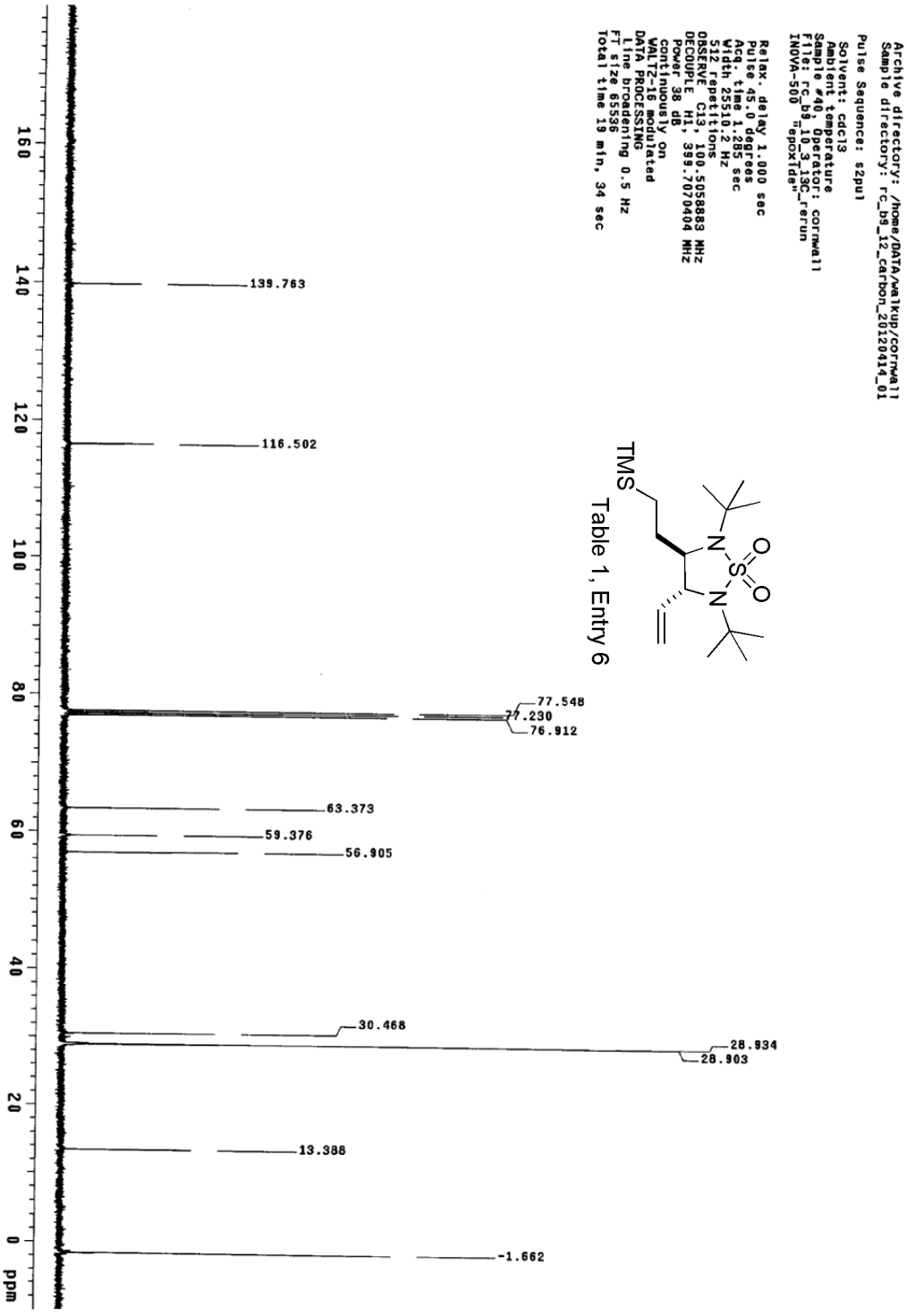


Table 1, Entry 5


13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: rc_b9_32_2_13C
INOVA-500 "epoxide"
Pulse 55.7 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
1000 repetitions
OBSERVE C13, 75.4750814 MHz
DECOUPLE H1, 300.1600800 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 3 min, 2 sec
Table 1, Entry 5
139.622
116.462
60.874
60.055
56.895
56.846
37.767
34.088
33.792
33.561
33.284
28.910
28.895
26.703
26.464
26.399
160
140
120
100
80
60
40
20
ppm


STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: rc_b9_10_3_pure
INOVA-500 "epoxide"
Relax. delay 1.000 sec
Pulse 49.6 degrees
Acq. time 1.998 sec
Width 4500.5 Hz
4 repetitions
OBSERVE H1, 300.1592164 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 12 sec
TMS
Table 1, Entry 6
1.399
1.379
0.012
0.97
1.03
1.03
1.01
1.00
0.96
1.07
18.98
1.04
1.02
8.38
8
7
6
5
4
3
2
1
-0
ppm


Archive directory: /home/DATA/walkup/cornwall
Sample directory: rc_b9_12_carbon_20120414_01
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Sample #40, Operator: cornwall
File: rc_b9_10_3_13C_rerun
INOVA-500 "epoxide"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.280 sec
Width 25510.2 Hz
512 repetitions
OBSERVE C13, 100.5058883 MHz
DECOUPLE H1, 399.7070404 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 19 min, 34 sec
TMS
Table 1, Entry 6
139.763
116.502
77.548
77.230
76.912
63.373
59.376
56.905
30.468
28.934
28.903
13.388
-1.662
160
140
120
100
80
60
40
20
0
ppm

b9_10_1

Archive directory: /home/DATA/walkup/cornwall
Sample directory: b9_10_1_20120305_01

Pulse Sequence: s2pul

Solvent: cdcl3
Ambient temperature
Sample #14, Operator: cornwall
File: b9_10_1_pure
INOVA-500 "epoxide"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.556 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.7050307 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 28 sec

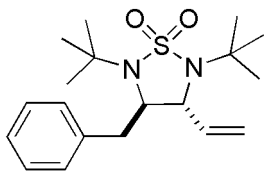
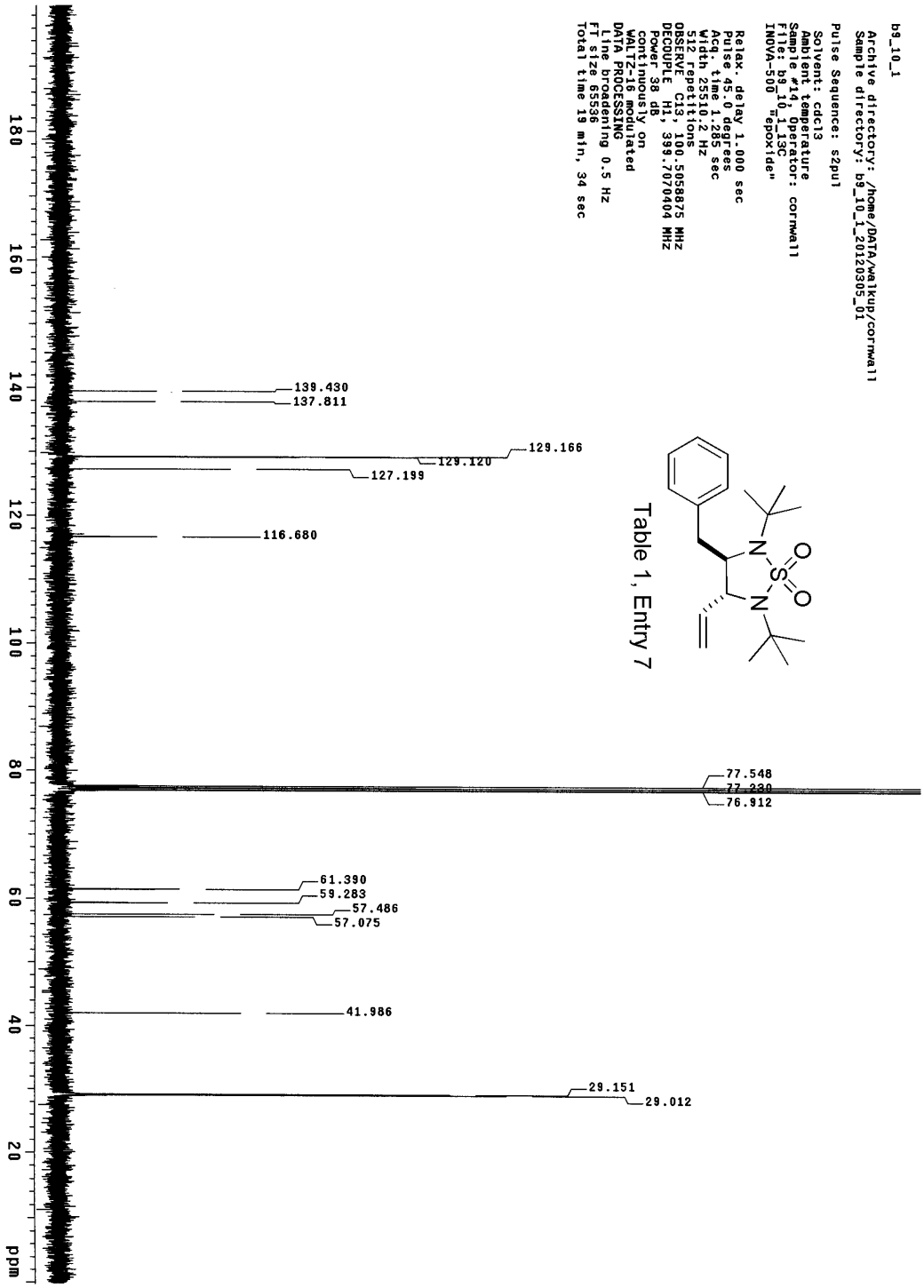
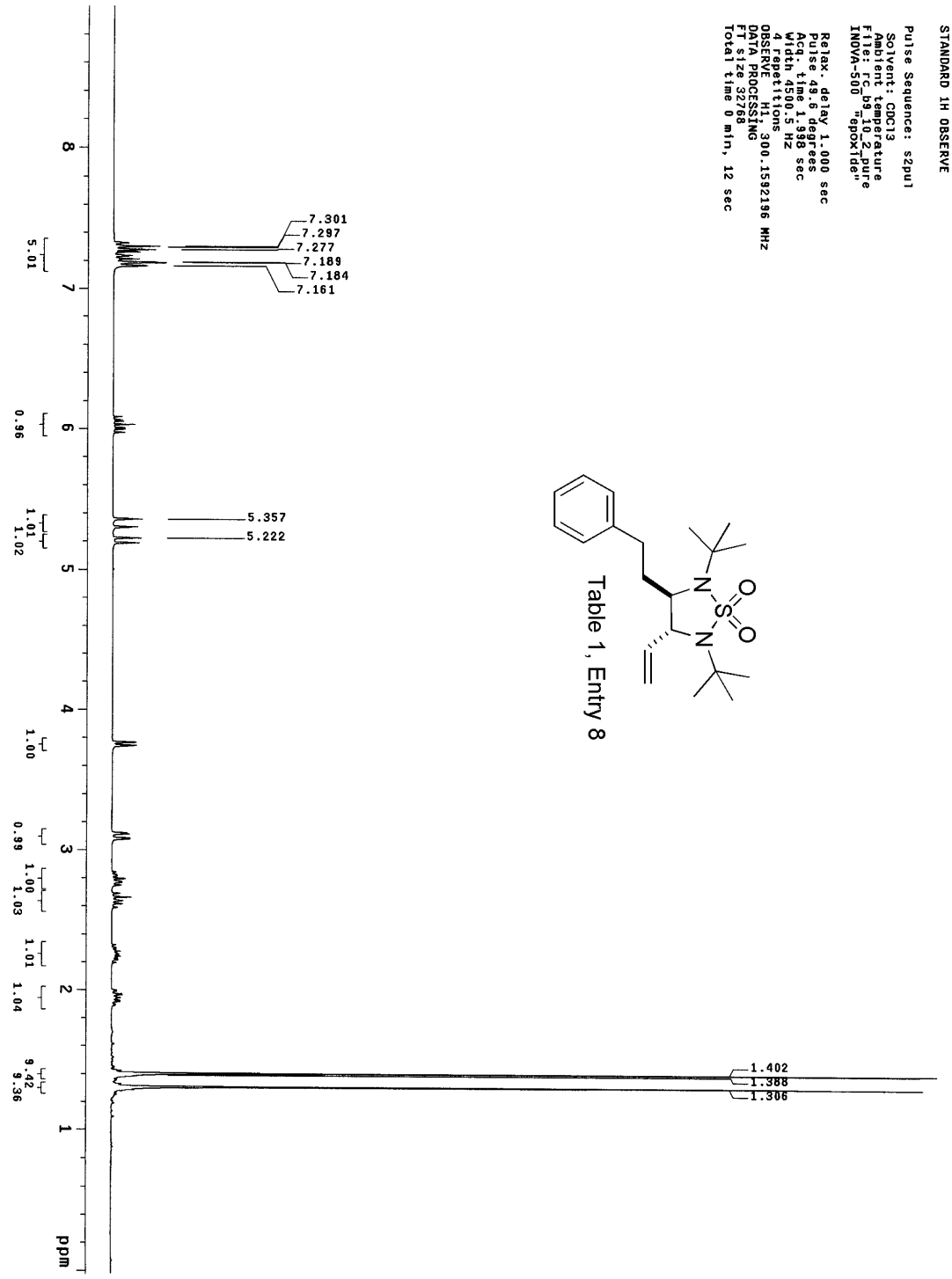
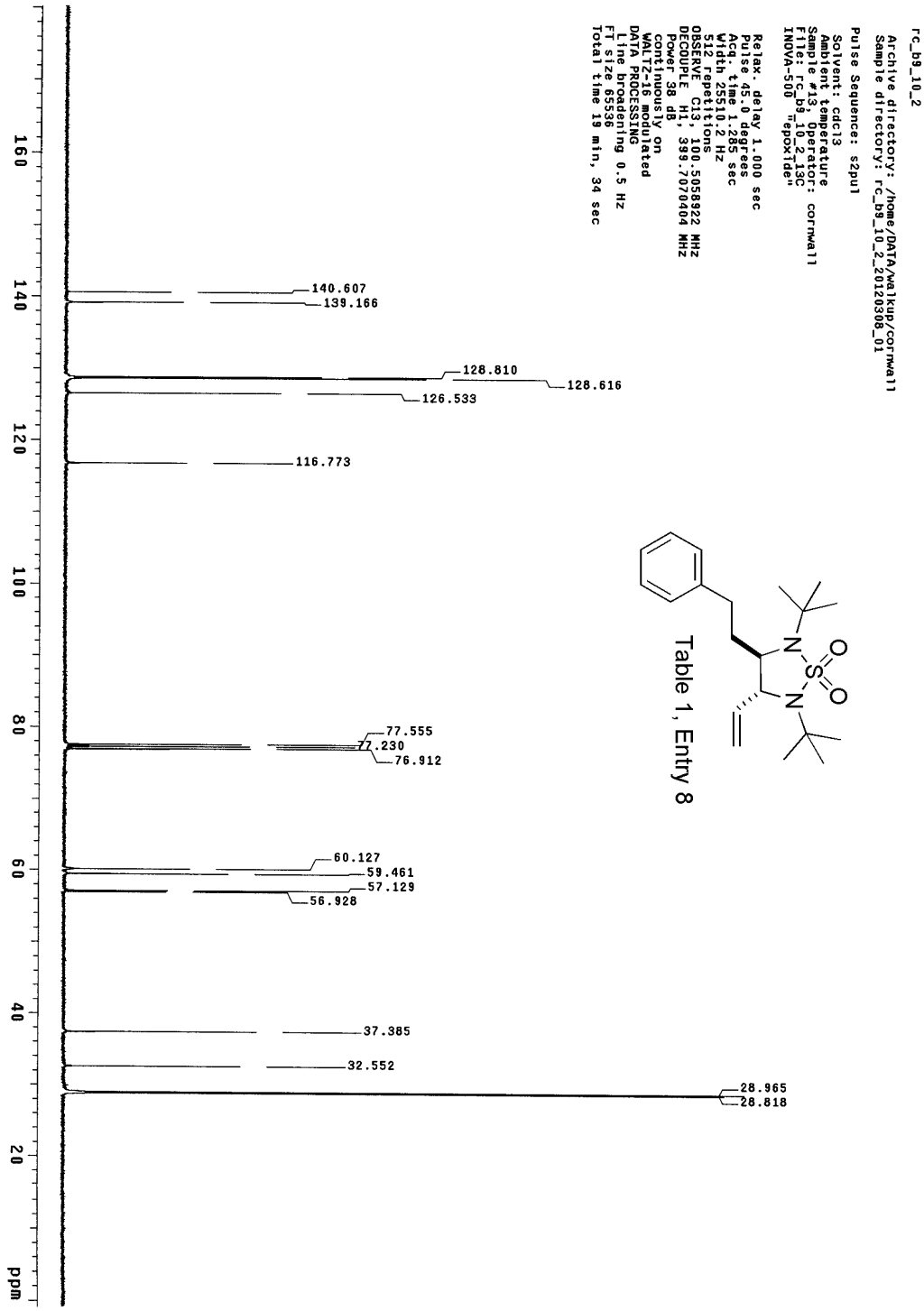
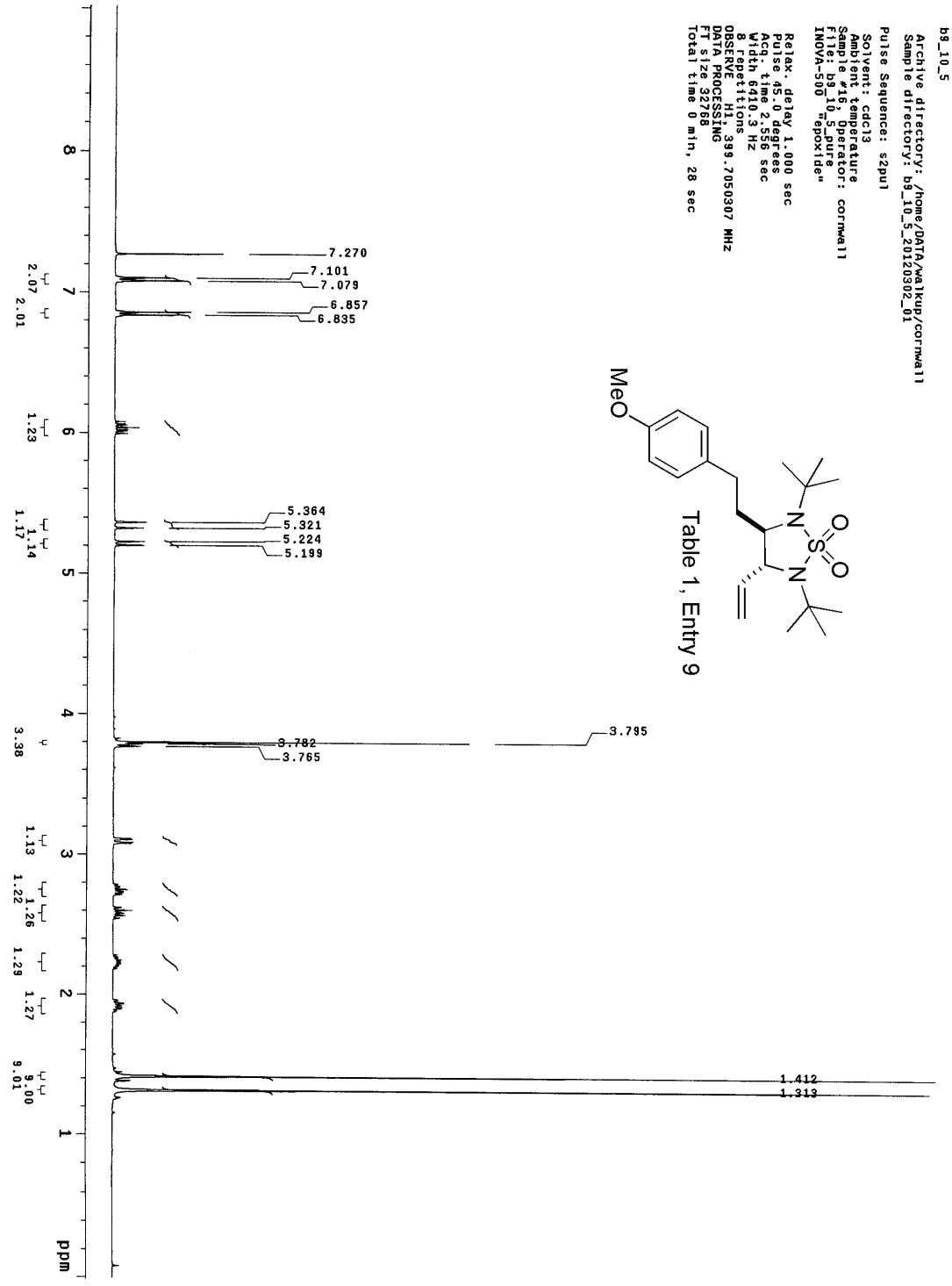


Table 1, Entry 7


b9_10_1
Archive directory: /home/DATA/walkup/cornwall
Sample directory: b9_10_1_20120305_01
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Sample_id: operator: cornwall
File: b9_10_1_13C
INOVA-500 "epoxide"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.285 sec
Width 25510.2 Hz
512 repetitions
OBSERVE C13, 100.5058875 MHz
DECOUPLE H1, 399.7070404 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
FT size 65536
Line broadening 0.5 Hz
Total time 19 min, 34 sec
Table 1, Entry 7
139.430
137.811
129.166
129.120
127.199
116.680
77.548
77.230
76.912
61.390
59.283
57.486
57.075
41.986
29.151
29.012
180
160
140
120
100
80
60
40
20
ppm


STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: rc_b9_10_2_pure
INOVA-500 "epox1de"
Relax. delay 1.000 sec
Pulse 45.6 degrees
Acq. time 1.998 sec
Width 4500.5 Hz
4 repetitions
OBSERVE H1, 300.1592196 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 12 sec
7.301
7.297
7.277
7.189
7.184
7.161
5.357
5.222
1.402
1.388
1.306
5.01
0.96
1.01
1.02
1.00
0.99
1.00
1.03
1.01
1.04
9.42
9.36
ppm
Table 1, Entry 8


rc_b9_10_2
Archive directory: /home/DATA/walkup/cornwall
Sample directory: rc_b9_10_2_20120308_01
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Sample #13, Operator: cornwall
File: rc_b9_10_2_13C
INOVA-500 "epoxide"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.285 sec
Width 25510.2 Hz
512 repetitions
OBSERVE C13, 100.5058922 MHz
DECOUPLE H1, 399.7070404 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 19 min, 34 sec
140.607
139.166
128.810
128.616
126.533
116.773
77.555
77.230
76.912
60.127
59.461
57.129
56.928
37.385
32.552
28.965
28.818
160
140
120
100
80
60
40
20
ppm
Table 1, Entry 8


b9_10_5
Archive directory: /home/DATA/walkup/cornwall
Sample directory: b9_10_5_20120302_01
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Sample #16, Operator: cornwall
File: b9_10_5_pure
INOVA-500 "epoxide"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.556 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.7050307 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 28 sec
MeO
Table 1, Entry 9
7.270
7.101
7.079
6.857
6.835
5.364
5.321
5.224
5.199
3.795
3.782
3.765
1.412
1.313
2.07
2.01
1.23
1.17
1.14
3.38
1.13
1.22
1.26
1.29
1.27
9.00
9.01
ppm

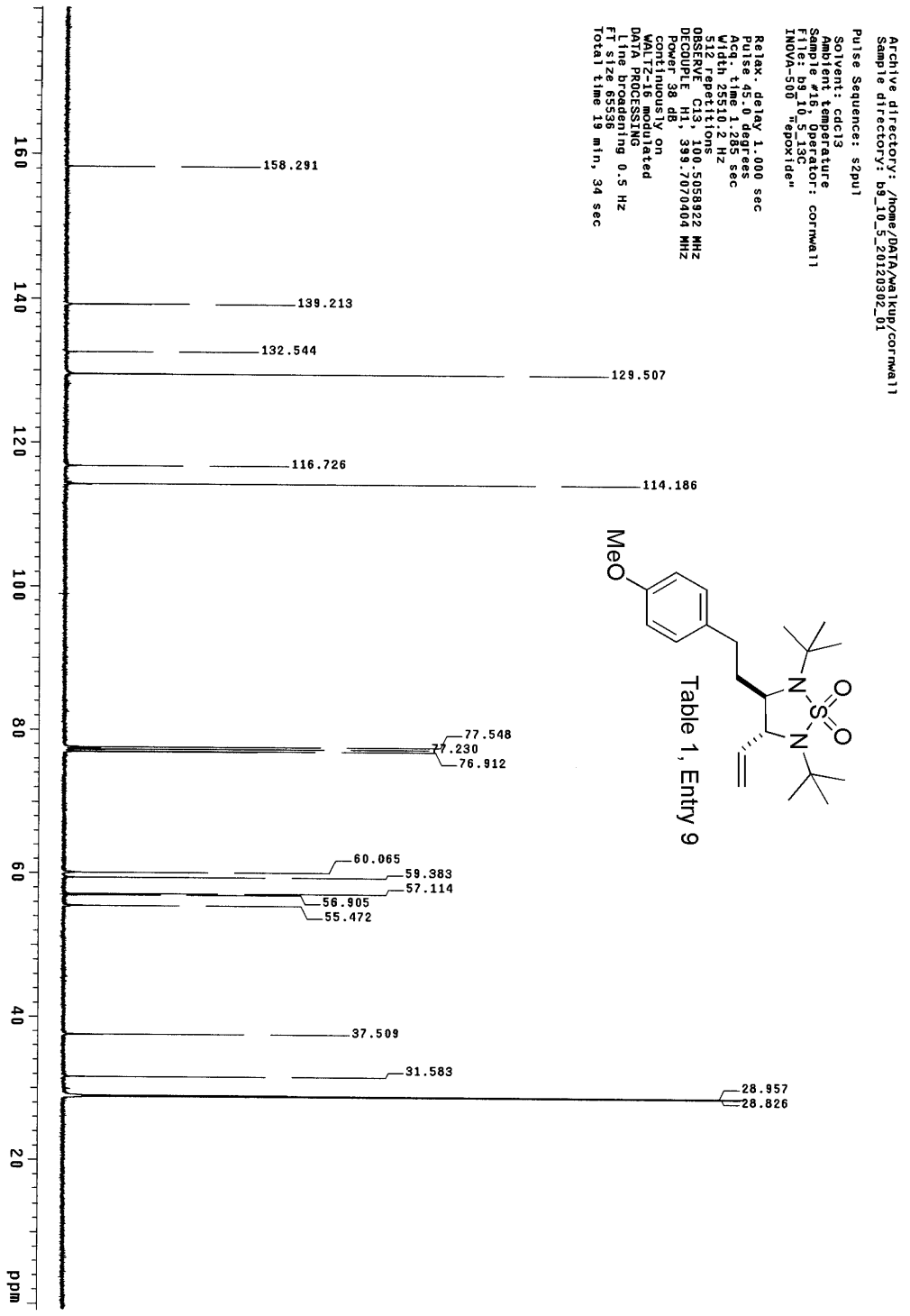
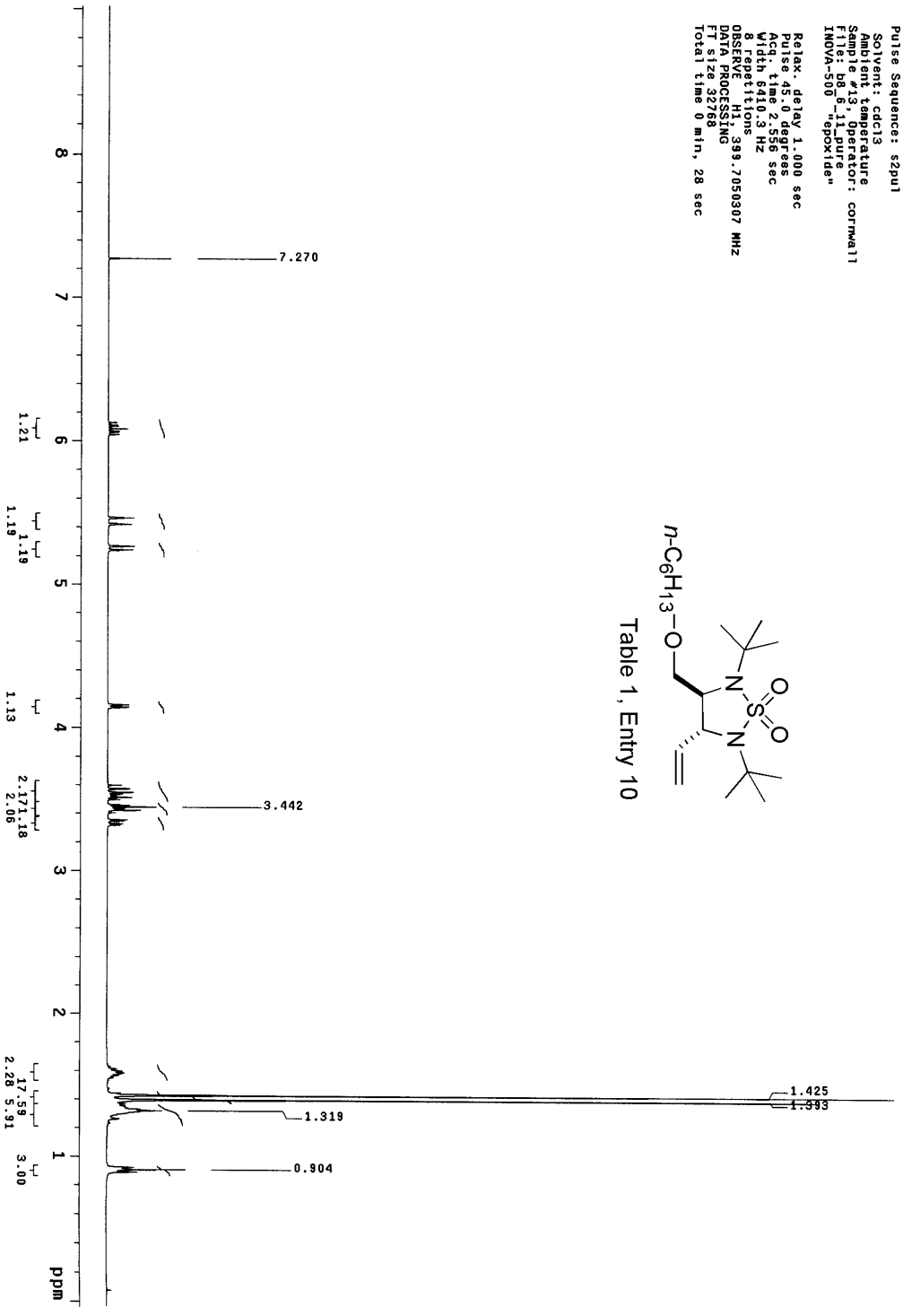
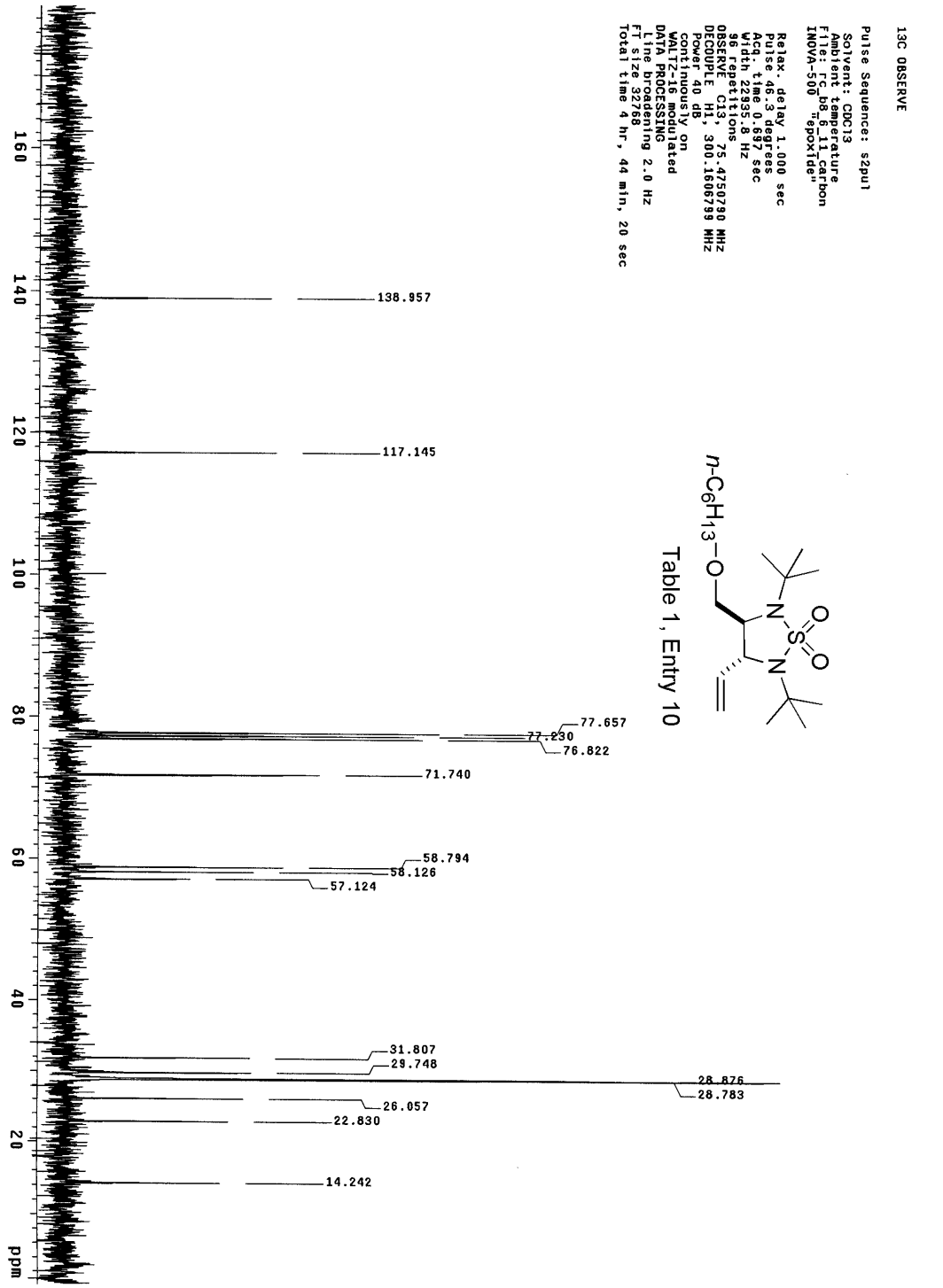


Table 1, Entry 9


b8_6_11
Archive directory: /home/DATA/walkup/cornwall
Sample directory: b8_6_11_20120305_01
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Sample #13, Operator: cornwall
File: b8_6_11_pure
INOVA-500 "epoxide"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.556 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.7050307 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 28 sec
n-C6H13−O
Table 1, Entry 10
7.270
3.442
1.425
1.393
1.319
0.904
1.21
1.19
1.19
1.13
2.171.18
2.06
17.59
2.28 5.91
3.00
ppm


13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: rc_b8_6_11_carbon
INOVA-500 "epoxide"
Relax. delay 1.000 sec
Pulse 46.3 degrees
Acq. time 0.697 sec
Width 22935.8 Hz
96 repetitions
OBSERVE C13, 75.4750790 MHz
DECOUPLE H1, 300.1606799 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 32768
Total time 4 hr, 44 min, 20 sec
n-C6H13-O
Table 1, Entry 10
138.957
117.145
77.657
77.230
76.822
71.740
58.794
58.126
57.124
31.807
29.748
28.876
28.783
26.057
22.830
14.242
160
140
120
100
80
60
40
20
ppm

STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: rc_b8_28_8_11162012
INOVA-500 "epoxide"

Relax. delay 1.000 sec
Pulse 49.6 degrees
Acq. time 1.998 sec
Width 4500.5 Hz
8 repetitions
OBSERVE H1, 300.1592196 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 24 sec

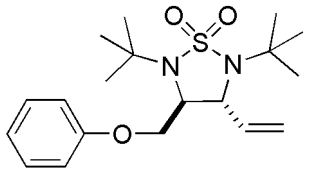
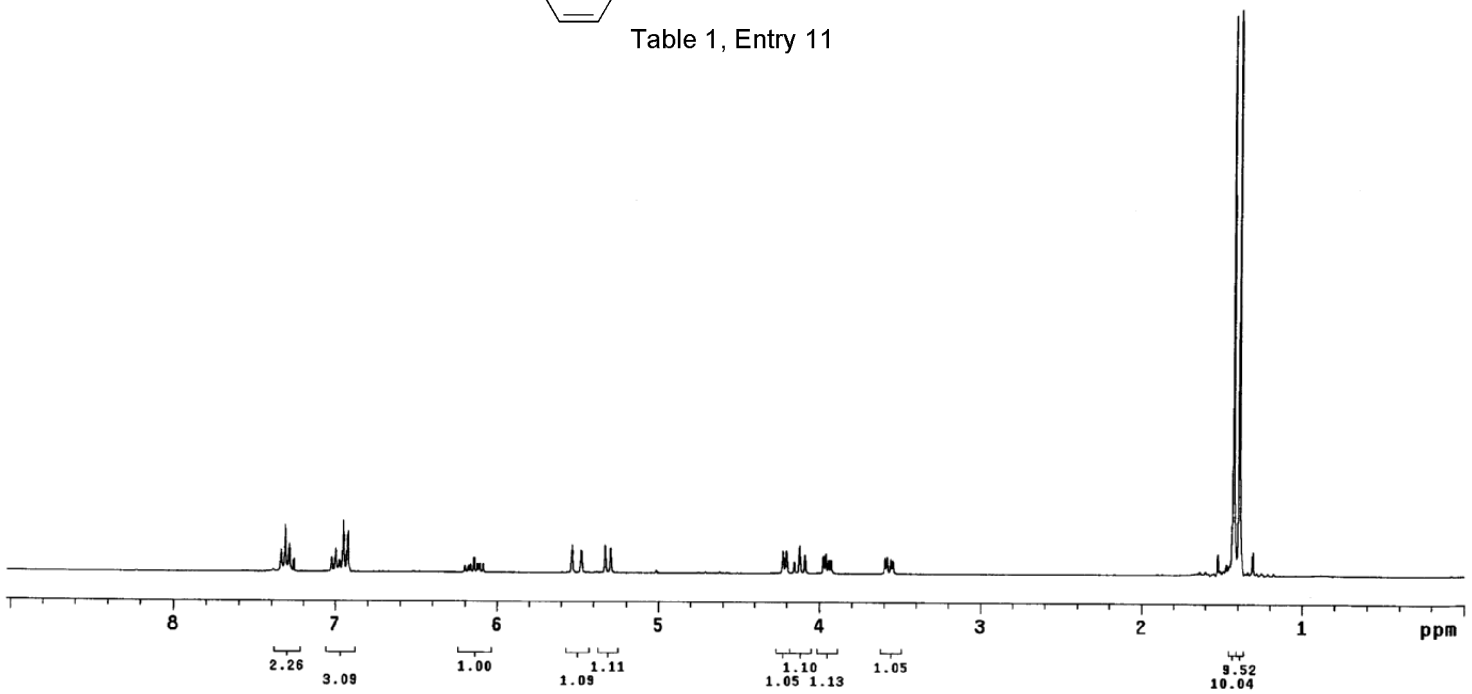
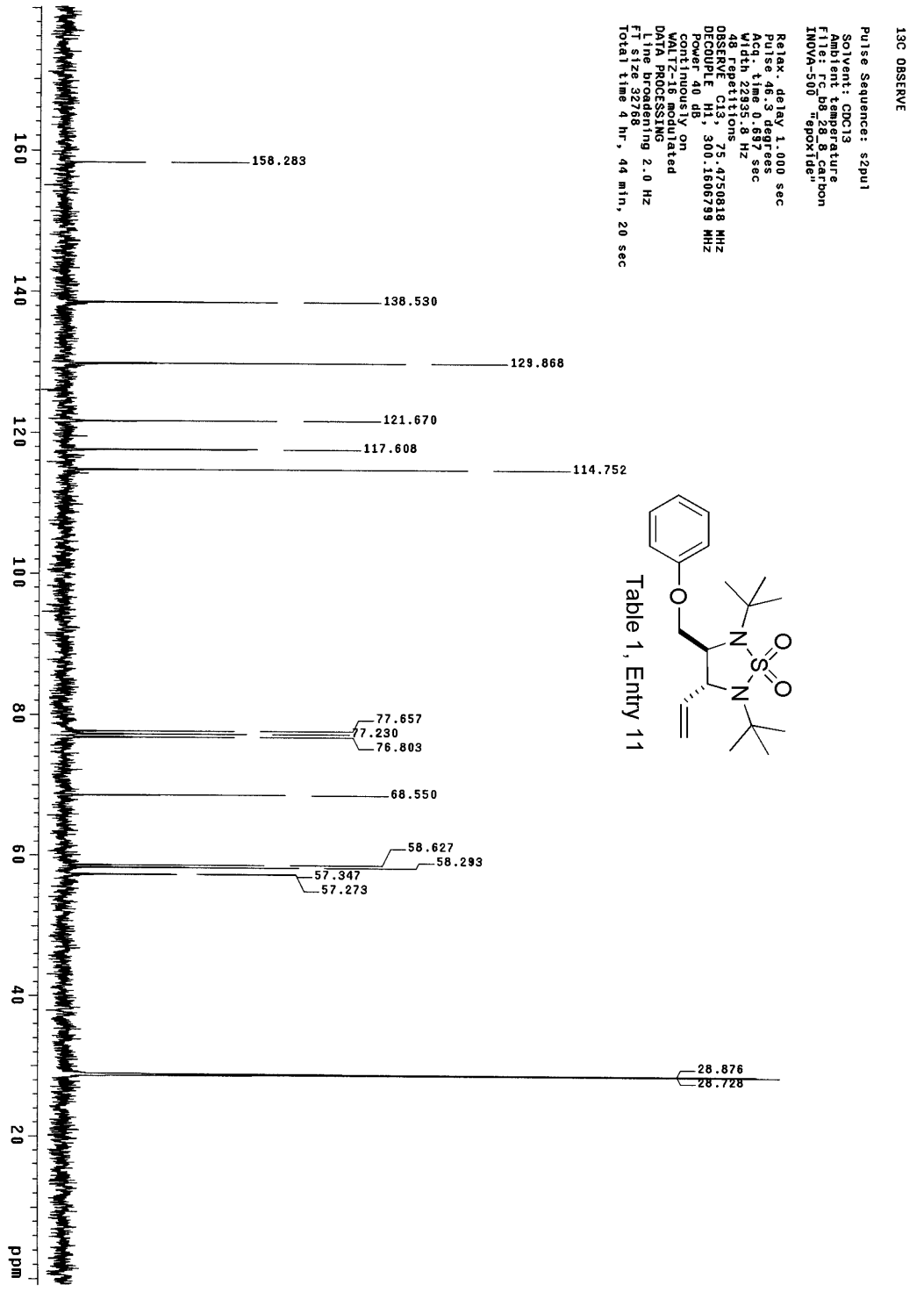
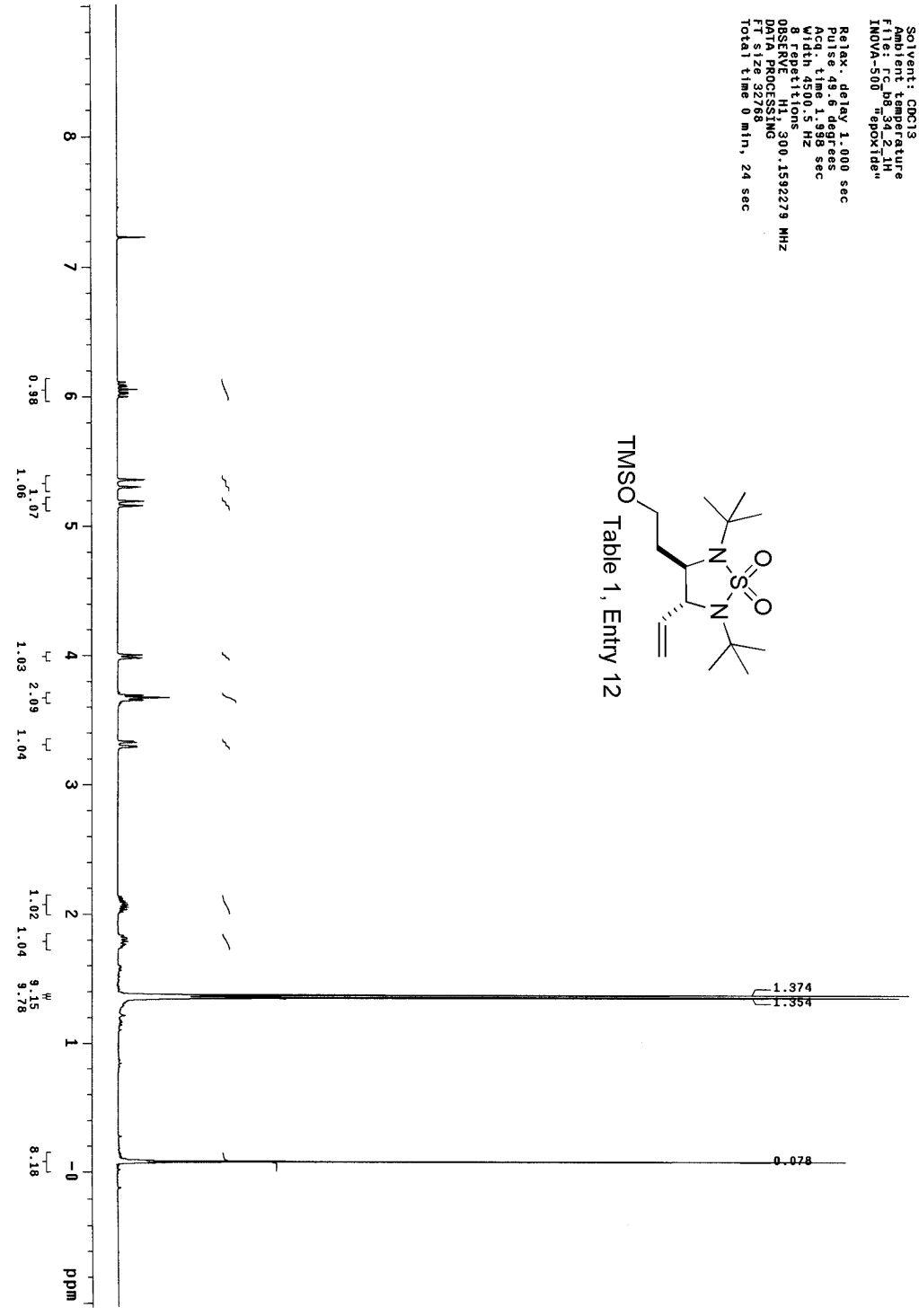
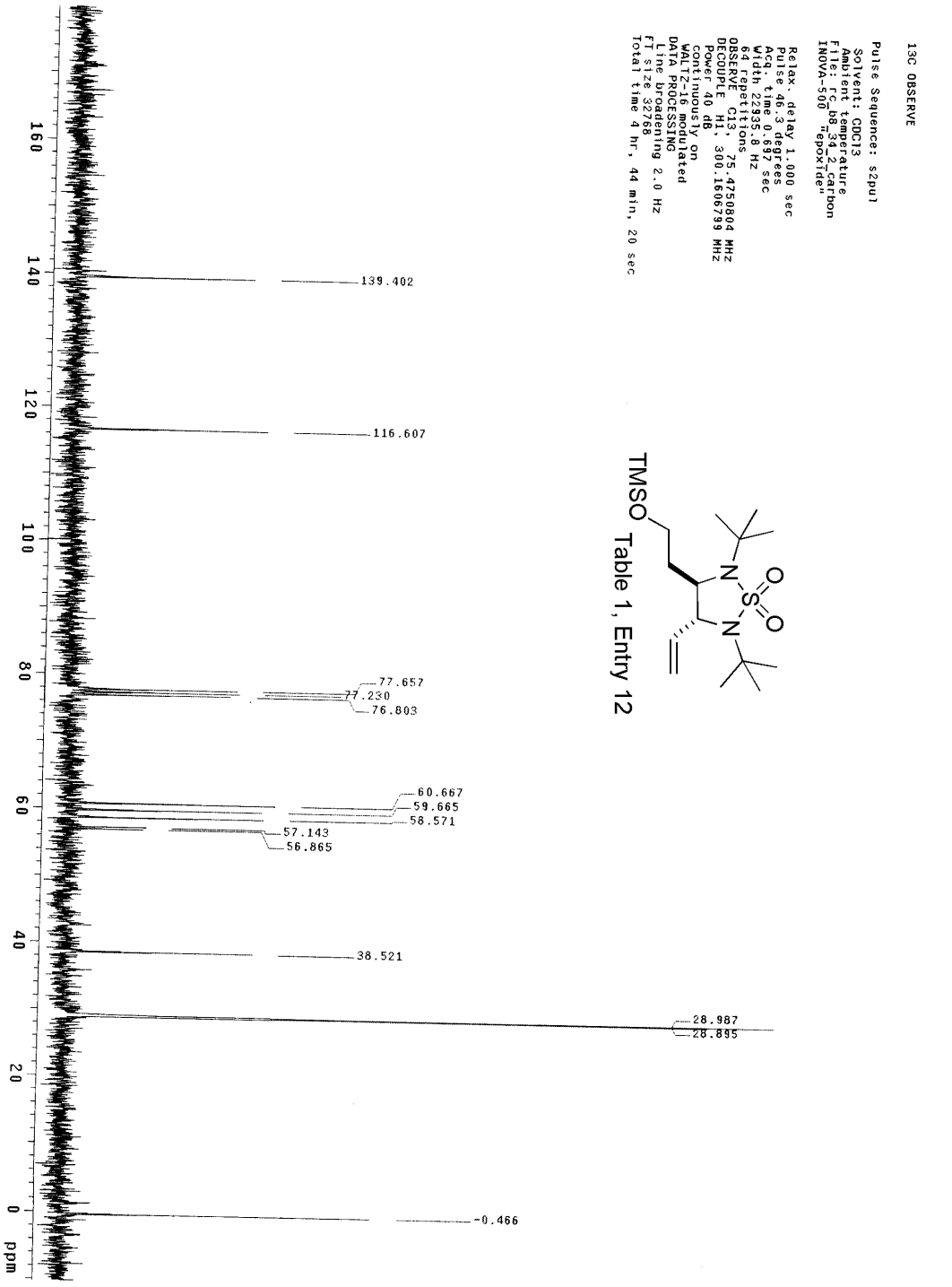


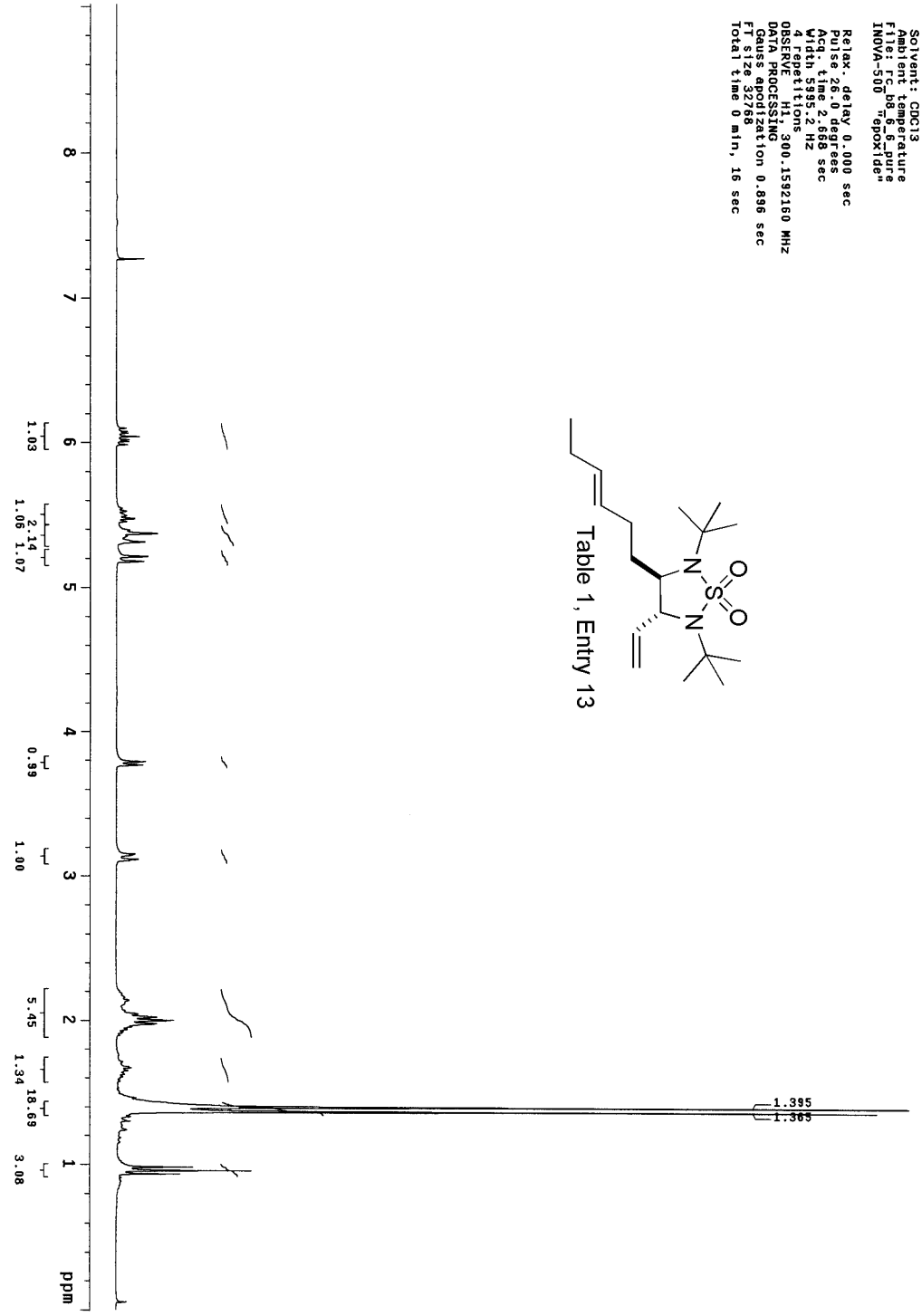
Table 1, Entry 11

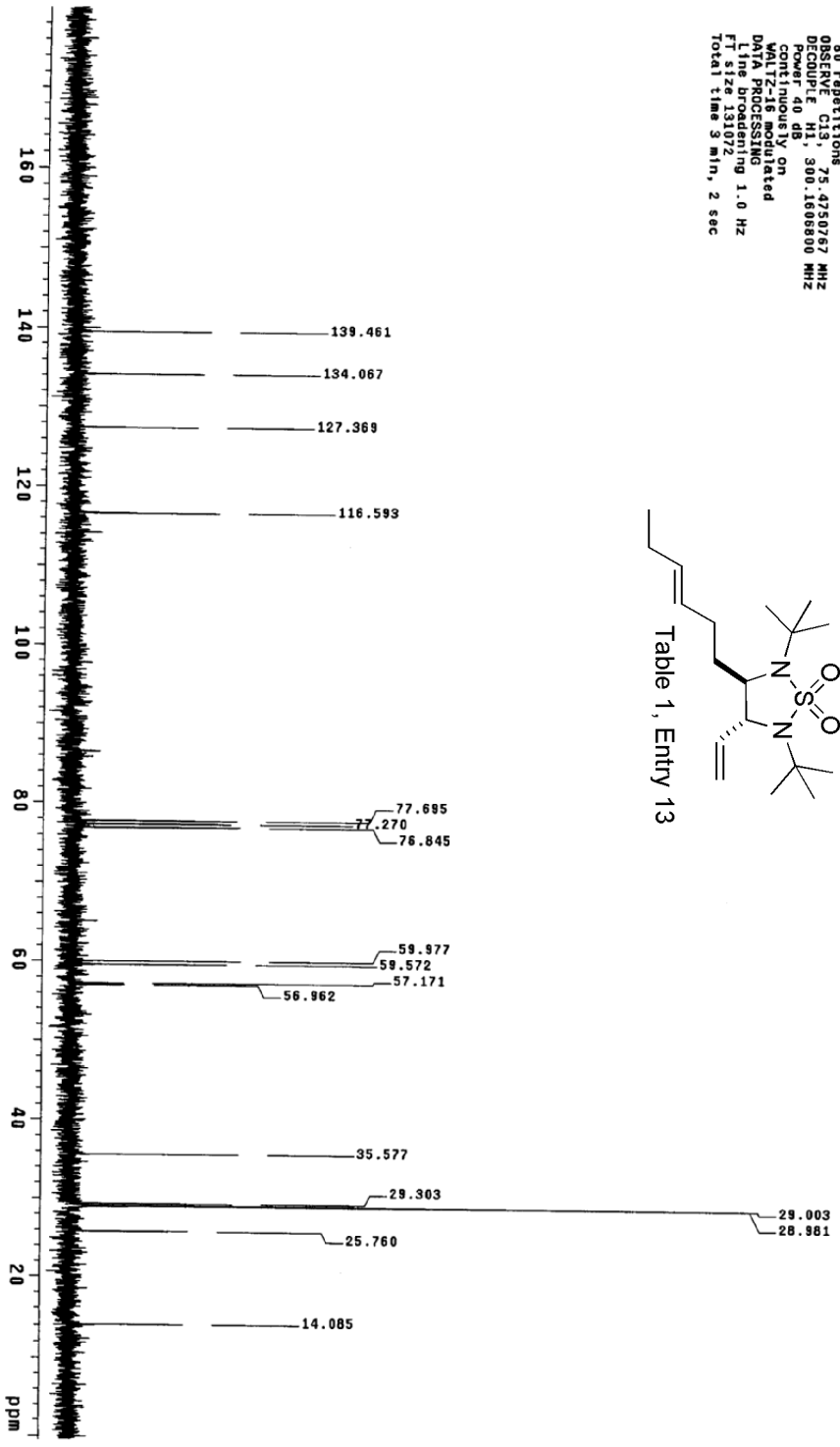



13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: rc_b8_28_8_carbon
INOVA-500 "epoxide"
Relax. delay 1.000 sec
Pulse 46.3 degrees
Acq. time 0.897 sec
Width 22935.8 Hz
48 repetitions
OBSERVE C13, 75.4750818 MHz
DECOUPLE H1, 300.1606799 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
FT line broadening 2.0 Hz
FT size 32768
Total time 4 hr, 44 min, 20 sec
158.283
138.530
129.868
121.670
117.608
114.752
77.657
77.230
76.803
68.550
58.627
58.293
57.347
57.273
28.876
28.728
160
140
120
100
80
60
40
20
ppm
Table 1, Entry 11


STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: rc_b8_34_2_1H
INOVA-500 "epoxide"
Relax. delay 1.000 sec
Pulse 49.6 degrees
Acq. time 1.998 sec
Width 4500.5 Hz
8 repetitions
OBSERVE H1, 300.1592279 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 24 sec
TMSO
Table 1, Entry 12
1.374
1.354
0.078
0.98
1.06
1.07
1.03
2.09
1.04
1.02
1.04
9.15
9.78
8.18
8
7
6
5
4
3
2
1
-0
ppm


13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: rc_bs_94_2_carbon
INOVA-500 "epoxide"
Relax. delay 1.000 sec
Pulse 46.3 degrees
Acq. time 0.697 sec
Width 29850.8 Hz
64 repetitions
OBSERVE C13, 75.4750804 MHz
DECOUPLE H1, 300.1606799 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 32768
Total time 4 hr, 44 min, 20 sec
TMSO
Table 1, Entry 12
139.402
116.607
77.657
77.230
76.803
60.667
59.665
58.571
57.143
56.865
38.521
28.987
28.895
-0.466
ppm


STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: rc_b8_6_6_pure
INOVA-500 "epoxide"
Relax. delay 0.000 sec
Pulse 26.0 degrees
Acq. time 2.668 sec
Width 5995.2 Hz
4 repetitions
OBSERVE H1, 300.1592160 MHz
DATA PROCESSING
Gauss apodization 0.896 sec
FT size 32768
Total time 0 min, 16 sec
Table 1, Entry 13
1.395
1.383
1.03
2.14
1.06 1.07
0.99
1.00
5.45
1.34
18.69
3.08
ppm


13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: rc_bR_6_6_pure_13C_11142012
INOVA-500 "epoxide"
Pulse 55.7 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
80 repetitions
OBSERVE C13, 75.4750767 MHz
DECOUPLE H1, 300.1606800 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
FT size 131072
Total time 3 min, 2 sec
Table 1, Entry 13
139.461
134.067
127.369
116.593
77.695
77.270
76.845
59.977
59.572
57.171
56.962
35.577
29.303
29.003
28.981
25.760
14.085
ppm

STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: rc_b8_6_5_pure
INOVA-500 "epoxide"

Relax. delay 0.000 sec
Pulse 26.0 degrees
Acq. time 2.668 sec
Width 5995.2 Hz
4 repetitions
OBSERVE H1, 300.1592164 MHz
DATA PROCESSING
Gauss apodization 0.896 sec
FT size 32768
Total time 0 min, 16 sec

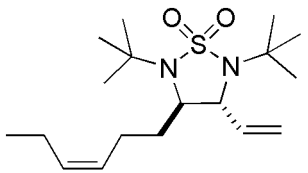


Table 1, Entry 14

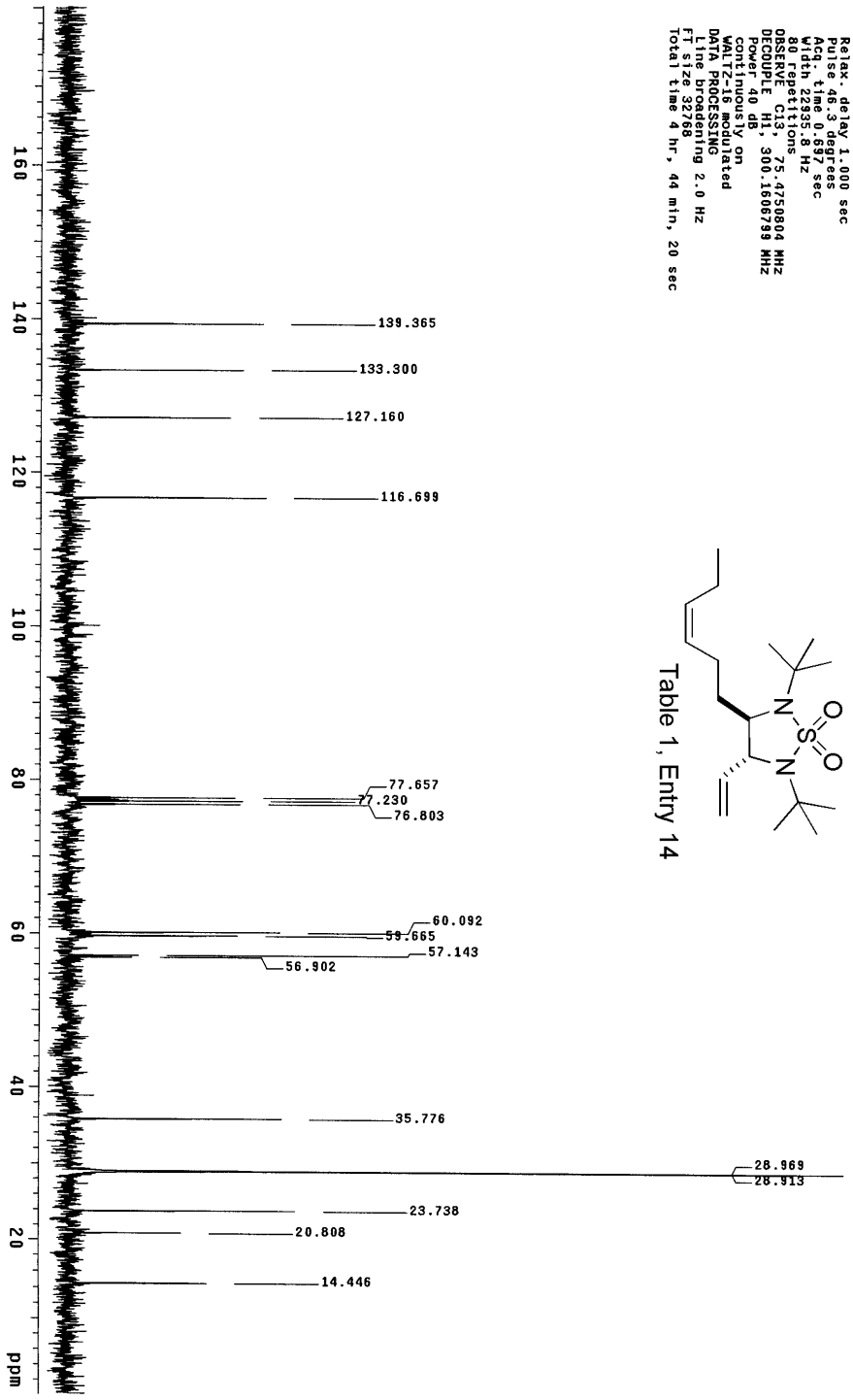
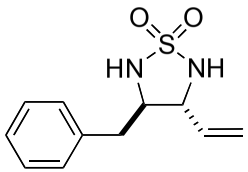


Table 1, Entry 14

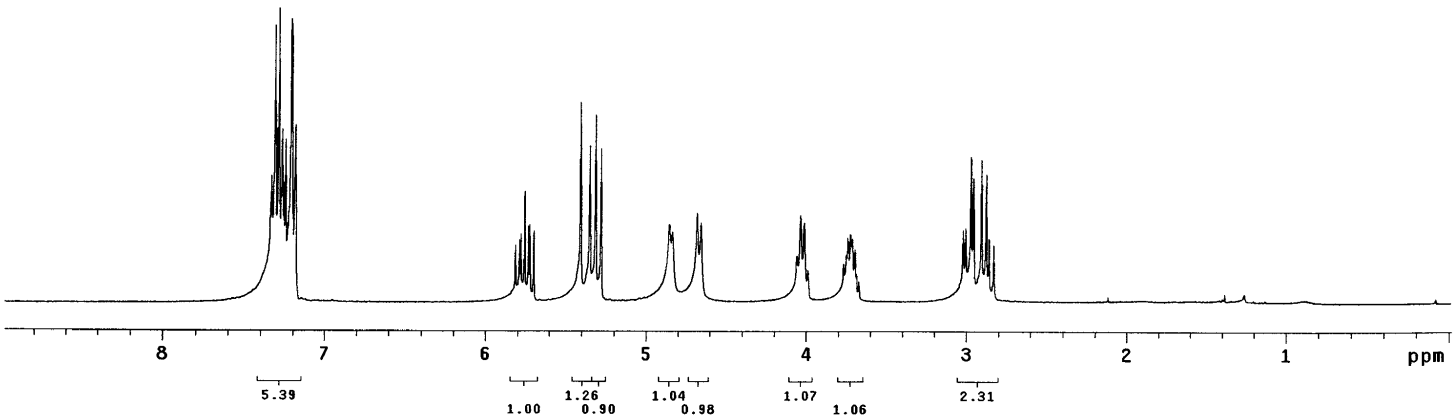
STANDARD 1H OBSERVE

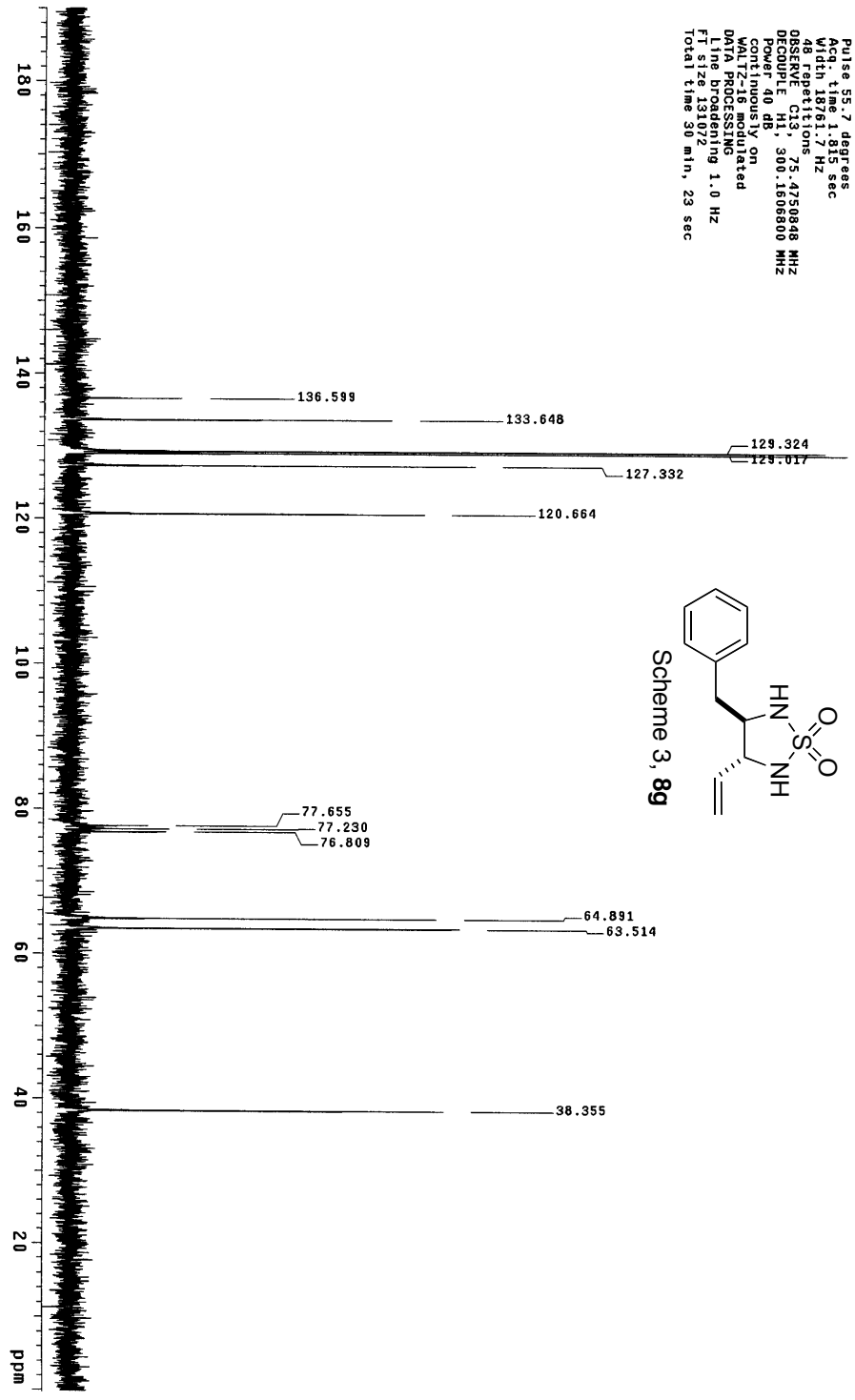
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: rc_b9_38_2_1H
INOVA-500 "epoxide"

Relax. delay 1.000 sec
Pulse 49.6 degrees
Acq. time 1.998 sec
Width 4500.5 Hz
4 repetitions
OBSERVE H1, 300.1592196 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 12 sec



Scheme 3, **8g**

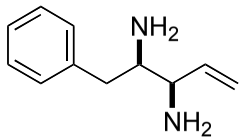



13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: c:\vnmr\data\...
INOVA-500 "epoxide"
Pulse 55.7 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
48 repetitions
OBSERVE C13, 75.4750848 MHz
DECOUPLE H1, 300.1606800 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 30 min, 23 sec
136.599
133.648
129.324
129.017
127.332
120.664
77.655
77.230
76.809
64.891
63.514
38.355
ppm
Scheme 3, 8g

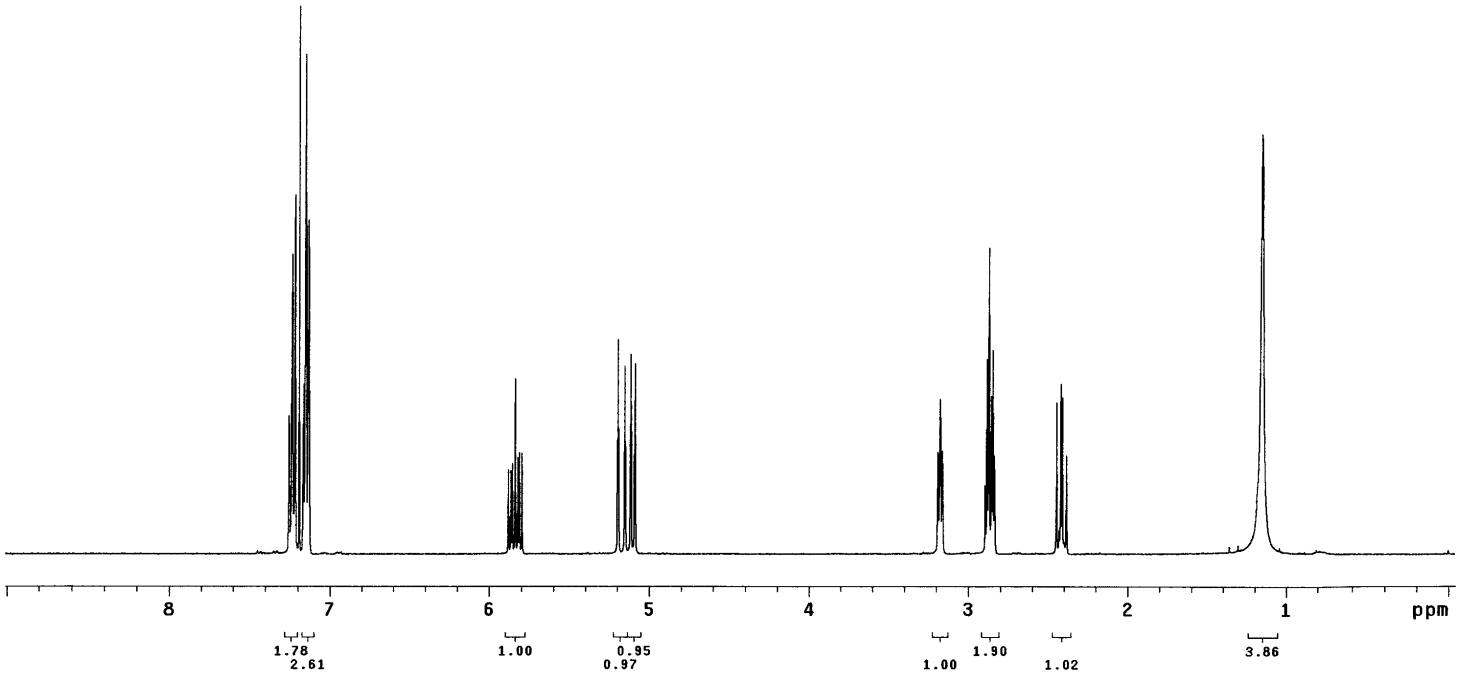
Archive directory: /home/DATA/walkup/cornwall
Sample directory: rc_b9_38_20120428_01

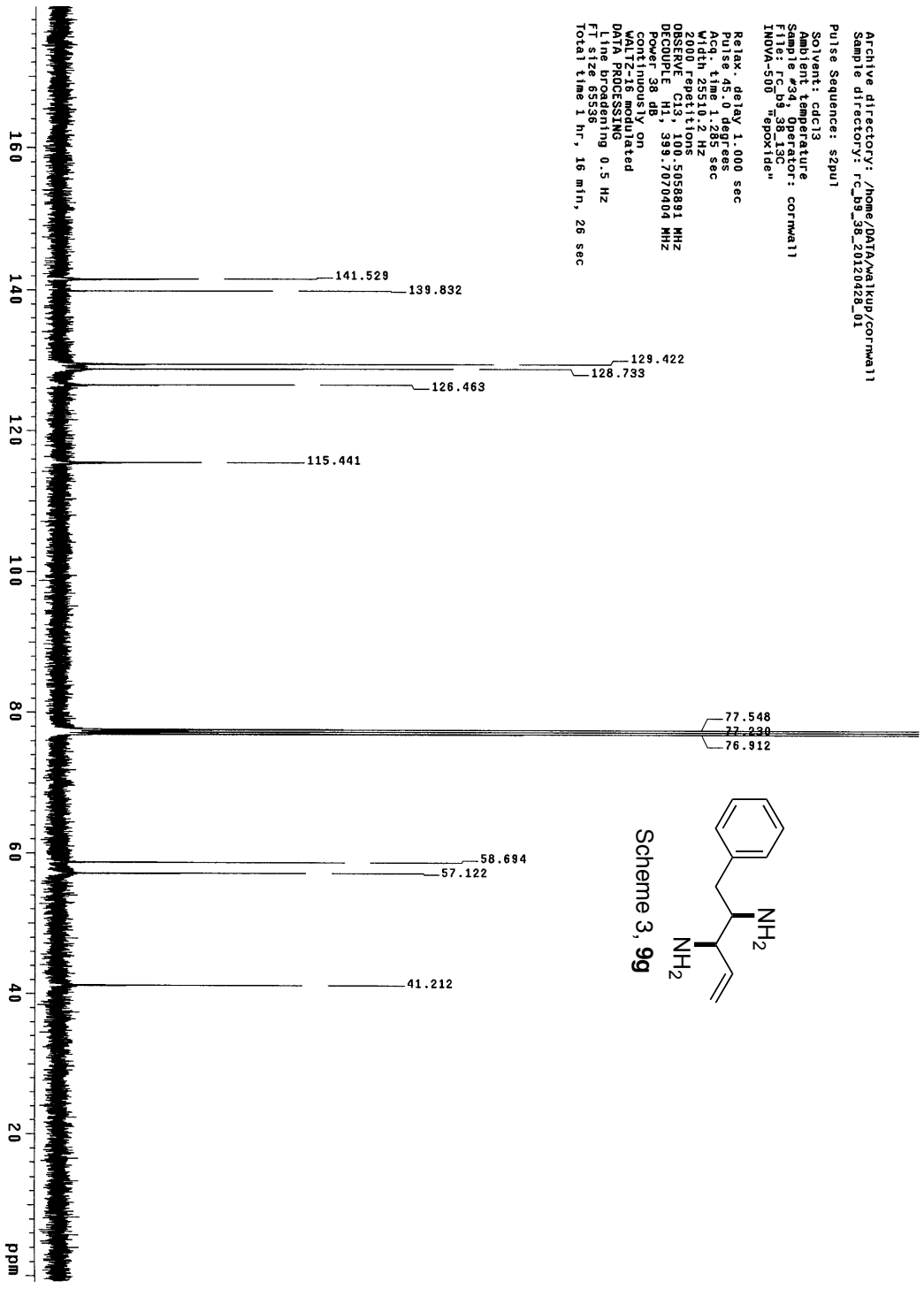
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Sample #34, Operator: cornwall
File: rc_b9_38_1H
INOVA-500 "epoxide"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.556 sec
Width 6410.3 Hz
16 repetitions
OBSERVE H1, 399.7050625 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 57 sec



Scheme 3, **9g**




Archive directory: /home/DATA/walkup/cornwall
Sample directory: rc_b9_38_20120428_01
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Sample #34 Operator: cornwall
File: rc_b9_38_13C
INOVA-500 "epoxide"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.285 sec
Width 25510.2 HZ
2000 repetitions
OBSERVE C13, 100.5058891 MHz
DECOUPLE H1, 399.7070404 MHz
Power 58 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 16 min, 26 sec
141.529
139.832
129.422
128.733
126.463
115.441
77.548
77.230
76.912
58.694
57.122
41.212
160
140
120
100
80
60
40
20
ppm
NH2
NH2

Scheme 3, **9g**