Benzhydryldimethylsilyl Allylic Silanes: Syntheses and Applications to [3+2] Annulation Reactions

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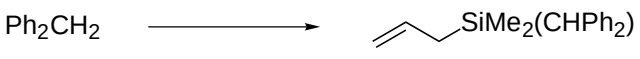
Supporting Information

Contents:

General. ^{1}H NMR and ^{13}C NMR spectra were recorded at ambient temperature at 400 and 100 MHz, and 500 MHz and 125 MHz, respectively, using a Bruker DRX400 or 500 spectrometer. The data are reported as follows: chemical shift in ppm from internal tetramethylsilane on the δ scale, multiplicity (br = broad, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constants (Hz) and integration. Multiplets were reported either as single chemical shifts or as ranges. For cases where the multiplets spanned less than 0.1 ppm at the indicated field, chemical shifts were reported as single values. For cases where multiplets spanned more than 0.1 ppm, chemical shifts were reported as ranges. High resolution mass spectra were acquired on a VG Analytical 7070E or Fisons Autospec spectrometer, and were obtained by peak matching. Microanalysis were performed by Atlantic Microlab, Atlanta, GA. Analytical gas-liquid chromatography (GLC) was performed on a Hewlett Packard 5890 Level 4 chromatograph, equipped with a split mode injection system and a flame ionization detector. Fused silica capillary column (30 m × 0.32 mm) wall-coated with DB-1 (J & W Scientific) was used with helium as the carrier gas. Melting points are reported uncorrected. Analytical thin layer chromatography was performed using EM Reagents 0.25 mm silica gel 60–F plates. Liquid chromatography was performed using forced flow (flash chromatography) of the indicated solvent system on EM Reagents silica gel (SiO_2) 60 (230–400) mesh. All reactions were carried out under an atmosphere of nitrogen in glassware which had been flame-dried under a stream of nitrogen. Unless otherwise noted, all reagents were commercially obtained and, where appropriate, purified prior to use. THF was distilled from sodium and benzophenone ketyl. Toluene was dried by filtration through alumina. Methylene chloride was distilled over CaH_2 prior to use. LiCl was dried at 150 °C at 0.05 mmHg for 8 h, then stored in an Innovative Technologies nitrogen atmosphere drybox. $BF_3{\cdot}OEt_2$ was distilled from Et_2O and stored in a sealed tube. $TiCl_4$ was distilled and stored in a sealed tube. Hydrocinnamaldehyde was distilled prior to use. Chlorosulfonyl isocyanate was purchased from Aldrich and distilled over K_2CO_3 prior to use.

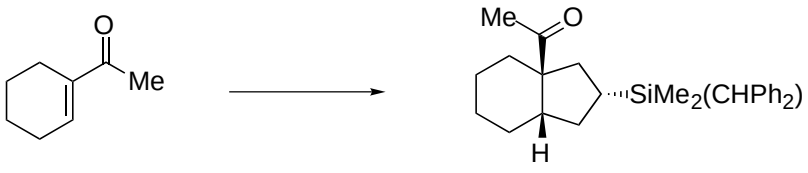
I. Synthesis and Annulations of Allylbenzhydryldimethylsilylsilane



1

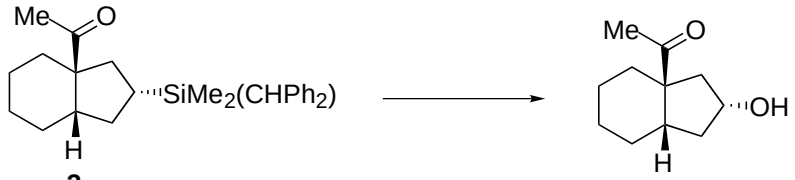
Allyldimethylbenzhydrylsilane (1). Diphenylmethane (5.04 g, 30.0 mmol) and *n*-BuLi (1.45 M solution in hexanes, 20.7 mL, 30.0 mmol) in 20 mL of Et_2O were heated under reflux for 20 h under nitrogen. The resultant red solution was transferred by cannula to a solution of allyldimethylchlorosilane (2.93 mL, 20 mmol) in 20 mL of Et_2O at 22 °C. After 12 h, 20 mL of saturated aqueous NH_4Cl and 20 mL of hexanes were added to the reaction mixture. The layers were separated and the aqueous layer was extracted with 3 × 50 mL of hexanes. The combined organic layers were washed with 20 mL of brine, dried ($MgSO_4$), filtered, and concentrated *in vacuo*. Purification by flash chromatography and bulb-to-bulb distillation (0.05 mmHg, 125 °C) afforded **1** as a colorless

oil (4.38 g, 82%): ^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.27 (m, 8H), 7.14 (m, 2H), δ 5.67 (m, 1H), 4.82 (m, 2H), 3.60 (s, 1H), 1.53 (dd, *J* = 8.5, 1.0 Hz, 2H), 0.07 (s, 6H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 143.0, 135.0, 129.3, 128.9, 125.7, 114.1, 45.2, 23.1, –3.3; IR (thin film) 1629, 1493 cm^{-1}; HRMS (CI/isobutane) *m* / *z* calcd for $C_{18}H_{22}Si$ (M^+) 266.1491, found 266.1494. Anal. Calcd for $C_{18}H_{22}Si$: C, 81.14; H, 8.32. Found: C, 81.32; H, 8.47.



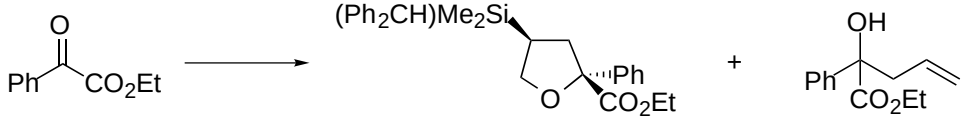
2 **3**

***trans*-1-Acetyl-8-benzhydryldimethylsilylbicyclo[4.3.0]nonane (3).** To $TiCl_4$ (0.065 mL, 0.60 mmol) in 1 mL of CH_2Cl_2 was added 2,6-di-*tert*-butylpyridine (0.013 mL, 0.050 mmol) at –20 °C. After 5 min, 1-acetylcyclohexene (0.064 mL, 0.50 mmol) was added to the solution. After 20 min at –20 °C, the resultant yellow suspension was cooled to –78 °C and a solution of **1** (0.20 g, 0.75 mmol) in 1 mL of CH_2Cl_2 was added. The reaction mixture was allowed to warm to 0 °C. After 4 h, the reaction was quenched with 5 mL of saturated aqueous NH_4Cl, the layers were separated, and the aqueous layer was extracted with 3 × 20 mL of CH_2Cl_2. The combined organic layers were washed with 10 mL of brine, dried (Na_2SO_4), filtered and concentrated *in vacuo*. Purification by flash chromatography (hexanes to 40:60 CH_2Cl_2/hexanes) afforded **3** as a colorless oil (0.18 g, 92%): ^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.24 (m, 8H), 7.14 (m, 2H), 3.52 (s, 1H), 2.33 (m, 1H), 2.01 (s, 3H), 1.77–1.25 (m, 11H), 1.17–1.01 (m, 2H), 0.05 (s, 3H), 0.04 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 213.4, 143.1, 129.1, 128.8, 125.7, 58.7, 45.4, 41.6, 38.0, 32.5, 31.7, 27.0, 25.8, 23.9, 22.5, 22.3, –4.3, –4.7; IR (thin film) 2926, 1703, 1494 cm^{-1}; HRMS (CI/isobutane) *m* / *z* calcd for $C_{26}H_{34}OSi$ (M^+) 390.2379, found 390.2376. Anal. Calcd for $C_{26}H_{34}OSi$: C, 79.95; H, 8.78. Found: C, 79.72; H, 8.87.



3 **4**

***trans*-1-Acetyl-8-hydroxybicyclo[4.3.0]nonane (4).** To a solution of **3** (0.22 g, 0.56 mmol) in 5 mL of THF was added *n*-Bu_4NF (1M solution in THF, 0.90 mL, 0.90 mmol) dropwise by syringe at 0 °C. The ice bath was removed and the reaction mixture was allowed to warm to 22 °C. After 30 min, MeOH (1.3 mL) was added to the reaction mixture followed by $KHCO_3$ (0.084 mg, 0.84 mmol) and H_2O_2 (30%, 0.63 mL, 5.6 mmol). After 12 h, the reaction mixture was diluted with 10 mL of CH_2Cl_2 and 10 mL of H_2O, the layers were separated, and the aqueous layer was extracted with 3 × 20 mL of CH_2Cl_2. The combined organic layers were dried ($MgSO_4$), filtered, and concentrated *in vacuo*. Purification by flash chromatography (20:80 to 40:60 EtOAc/hexanes) afforded **4** as a colorless oil (0.083 g, 81%). Spectral data was identical to that reported in the literatures:[1] ^{1}H NMR ($CDCl_3$, 500 MHz) δ 4.38 (m, 1H), 2.40–2.35 (m, 1H), 2.26 (br, 1H), 2.21–2.01 (m, 2H and s, 3H), 1.91–1.87 (m, 1H), 1.91–1.37 (m, 8 H), 1.25–1.18 (m, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 213.4, 71.6, 58.1, 44.9, 39.7, 38.7, 30.5, 26.5, 25.9, 23.4, 22.1; IR (thin film) 3418, 2932, 2860, 1698 cm^{-1}.



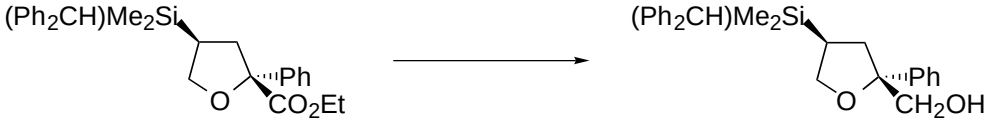
5 **6** **7**

[1] (a) Knölker, H.-J.; Wanzl, G. *Synlett* **1995**, 378-382. (b) Groaning, M. D.; Brengel, G. P.; Meyers, A. I. *J. Org. Chem.* **1998**, *63*, 5517-5522.

(2*R,4*S**)-4-Benzhydryldimethylsilyl-2-phenyl-tetrahydrofuran-2-carboxylic acid ethyl ester (6).** To a solution of ethyl benzoylformate (0.16 mL, 1.0 mmol) and allylbenzhydryldimethylsilane **1** (0.319 g, 1.20 mmol) in 5 mL of dry CH_2Cl_2 was added dropwise $SnCl_4$ (0.2 M solution in CH_2Cl_2, 6.0 mL, 1.2 mmol) by syringe at 22 °C. After 10 min, 1 mL of Et_3N was added, followed by 10 mL of water. The layers were separated and the aqueous layer was extracted with 3 × 20 mL of EtOAc. The combined organic layers were washed with 10 mL of brine, dried (Na_2SO_4), filtered, and concentrated *in vacuo*. Purification by flash chromatography (benzene to 2:98 EtOAc/benzene) afforded colorless oils **7** (0.012 g, 5%) and **6** (0.361 g, 81%) as a single stereoisomer as determined by ^{1}H NMR spectroscopy: ^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.41 (m, 2H), 7.30–7.18 (m, 11H), 7.10 (m, 2H), 4.10 (q, *J* = 7.1 Hz, 2H), 3.88 (t, *J* = 7.4 Hz, 1H), 3.63 (dd, *J* = 12.0, 8.4 Hz, 1 H), 3.49 (s, 1H), 2.29 (t, *J* = 12.8 Hz, 1 H), 2.21 (dd, *J* = 12.7, 7.4 Hz, 1H), 1.31 (m, 1H), 1.16 (t, *J* = 7.1 Hz, 3H), 0.07 (s, 6H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 173.7, 142.4, 142.0, 129.1, 129.0, 128.9, 128.5, 126.0, 87.7, 71.8, 61.9, 45.5, 40.5, 25.4, 14.5, –4.1, –4.6; IR (thin film) 1724, 1252 cm^{-1}; HRMS (CI/isobutane) *m* / *z* calcd for $C_{28}H_{31}SiO_3$ $(M - H)^+$ 443.2043, found 443.2027. Anal. Calcd for $C_{28}H_{32}SiO_3$: C, 75.64; H, 7.26. Found: C, 75.56; H, 7.23.

6 **8**

(2*R,4*S**)-4-Hydroxy-2-phenyl-tetrahydrofuran-2-carboxylic acid ethyl ester (8).** Using the same procedure given for **4** with **6** (0.234 g, 0.530 mmol), *n*-Bu_4NF (1 M solution in THF, 0.84 mL, 0.84 mmol), MeOH (1.0 mL), $KHCO_3$ (0.080 g, 0.80 mmol) and H_2O_2 (30%, 0.60 mL, 5.3 mmol) afforded **8**, after purification by flash chromatography (10:90 to 50:50 EtOAc/hexanes), as a colorless oil (0.098 g, 78%): ^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.51 (d, *J* = 7.5 Hz, 2H), 7.34–7.26 (m, 3H), 4.54 (m, 1H), 4.17 (q, *J* = 7.1 Hz, 2H), 4.12 (m, 2H), 3.03 (d, *J* = 13.6 Hz, 1H), 2.95 (d, *J* = 4.4 Hz, 1H), 2.27 (dd, *J* = 13.6, 5.2 Hz, 1H), 1.21 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 174.3, 141.5, 128.7, 128.2, 125.8, 87.4, 77.1, 72.5, 62.3, 47.0, 14.4; IR (thin film) 3478, 1731 cm^{-1}; HRMS (CI/isobutane) *m* / *z* calcd for $C_{13}H_{17}O_4$ $(M + H)^+$ 237.1127, found 237.1126. Anal. Calcd for $C_{13}H_{16}O_4$: C, 66.07; H, 6.83. Found: C, 65.97; H, 6.99.



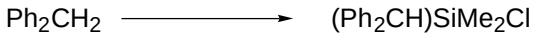
6 **9**

(2*R,4*S**)-4-Benzhydryldimethylsilyl-2-hydroxymethyl-2-phenyltetrahydrofuran (9).** $LiAlH_4$ (0.040 g, 1.1 mmol) was suspended in 3 mL of dry Et_2O under nitrogen and a solution of **6** (0.39 g, 0.88 mmol) in 2 mL of dry Et_2O was added at 0 °C. After 10 min, the excess hydride was carefully quenched by the dropwise and sequential addition of 0.03 mL of water, 0.03 mL of a 10% aqueous NaOH, and an additional 0.1 mL of water. The reaction mixture was filtered and the solid was washed with 3 × 10 mL of Et_2O. The combined filtrate and washes were dried ($MgSO_4$), filtered, and concentrated *in vacuo*. Purification by flash chromatography (15:85 EtOAc/hexane) afforded **9** as a colorless oil (0.330 g, 93%): ^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.34–7.09 (m, 15H), 3.82 (t, *J* = 8.4 Hz, 1H), 3.61–3.52 (m, 3H), 3.48 (s, 1H), 2.25 (dd, *J* = 8.6, 4.2 Hz, 1H), 2.01 (t, *J* = 12.6 Hz, 1H), 1.83 (dd, *J* = 12.1, 6.6 Hz, 1H), 1.32 (m, 1H), 0.06 (s, 3H), 0.05 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 144.4, 142.6, 142.56, 129.1, 129.02, 129.00, 128.6, 127.4, 126.0, 125.9, 125.8, 88.2, 71.2, 68.5, 45.5, 36.5, 25.5, –4.1, –4.4; IR (thin film) 3440, 1596, 1493, 1252 cm^{-1}; HRMS (CI/isobutane) *m* / *z* calcd for $C_{26}H_{29}OSi$ $(M - OH)^+$ 385.1988, found 385.1983. Anal. Calcd for $C_{26}H_{30}O_2Si$: C, 77.57; H, 7.52. Found: C, 77.49; H, 7.68.

9 **10**

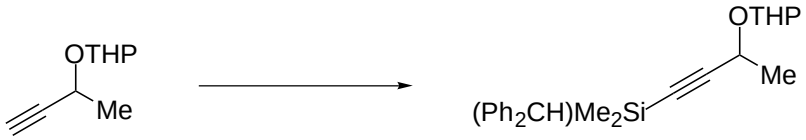
(2*R,4*S**)-4-Hydroxy-2-hydroxymethyl-2-phenyltetrahydrofuran (10).** Using the same procedure given for **4** with **9** (0.21 g, 0.52 mmol), *n*-Bu_4NF (1 M solution in THF, 0.83 mL, 0.83 mmol), MeOH (1.0 mL), $KHCO_3$ (0.078 g, 0.78 mmol) and H_2O_2 (30%, 0.59 mL, 5.2 mmol) afforded **10**, after purification by flash chromatography (10:90 to 50:50 EtOAc/hexanes), as a white solid (0.081 g, 80%): mp 104–105 °C; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.39–7.29 (m, 4H), 7.25 (t, *J* = 7.0 Hz, 1H), 4.85 (br, 1H), 4.61 (br, 1H), 4.36 (m, 1H), 4.06 (dd, *J* = 9.6, 1.1 Hz, 1H), 3.73 (dd, *J* = 9.6, 2.8 Hz, 1H), 3.64 (d, *J* = 11.7 Hz, 1H), δ 3.63 (d, *J* = 11.7 Hz, 1H), δ 2.54 (d, *J* = 14.0 Hz, 1H), δ 2.40 (dd, *J* = 14.0, 5.5 Hz, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 144.6, 128.9, 127.7, 125.5, 87.8, 76.5, 73.2, 69.8, 44.9; IR (KBr) 3276, 1055 cm^{-1}; HRMS (CI/isobutane) *m* / *z* calcd for $C_{11}H_{15}O_3$ $(M + H)^+$ 195.1021, found 195.1025. Anal. Calcd for $C_{11}H_{14}O_3$: C, 68.01; H, 7.27. Found: C, 67.92; H, 7.26.

II. Syntheses of (*E*)-Benzhydryldimethylcrotylsilanes



11

Chlorobenzhydryldimethylsilane (11). Diphenylmethane (16.8 g, 100 mmol) and *n*-BuLi (2.0 M solution in hexanes, 75.0 mL, 150 mmol) in 100 mL of Et_2O were heated under reflux for 48 h under nitrogen. The resultant red solution was transferred by cannula to a solution of dimethyldichlorosilane (72.8 mL, 600 mmol) in 200 mL of hexanes at –78 °C. After 1 h, the reaction mixture was allowed to warm to 0°C and stirred for an additional 3 h. The reaction mixture was filtered and the filtrate was concentrated *in vacuo*. Purification by distillation (0.10 mmHg, 130–132 °C) afforded **11** as a colorless oil (18.3 g, 70%): ^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.30 (m, 8H), 7.14 (m, 2H), 3.75 (s, 1H), 0.40 (s, 6H); ^{13}C NMR ($CDCl_3$, 500 MHz) δ 140.9, 129.5, 129.1, 126.5, 47.6, 2.0; IR (thin film) 1597, 1493, 1254 cm^{-1}.

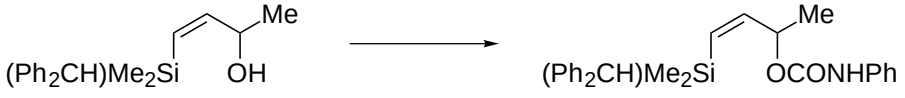


12 **13**

(±)-2-[3-(Benzhydryldimethylsilyl)-1-methyl-2-propynyloxy]tetrahydropyran (13). To a cooled (–78 °C) solution of **12** (3.93 g, 25.5 mmol) in 25 mL of THF was added *n*-BuLi (2.2 M solution in hexane, 11.6 mL, 25.5 mmol) dropwise by syringe. After 5 min, the mixture was allowed to warm to 0 °C. A solution of chlorodimethylbenzhydrylsilane (6.46 g, 24.7 mmol) in 25 mL of THF was added dropwise. The ice/H_2O bath was removed, and, after 30 min, 50 mL of 10% aqueous NaCl and 50 mL of hexanes were added to the reaction mixture. The layers were separated, and the aqueous layer was extracted with 3 × 50 mL of hexanes. The combined organic layers were dried (Na_2SO_4), filtered, and concentrated *in vacuo*. Purification by flash chromatography (1:99 to 5:95 EtOAC/hexanes) afforded **13** as a colorless oil (8.3 g, 88%). ^{1}H NMR spectroscopic analysis indicated that the acetal was an approximately 60:40 mixture of diastereoisomers: ^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.35 (m, 8H, major+minor), 7.26 (m, 8H, major+minor), 7.14 (m, 4H, major+minor), 4.83 (dd, *J* = 4.2, 2.7 Hz, 1H, minor), 4.76 (t, *J* = 3.3 Hz, 1H, major), 4.53 (q, *J* = 6.8 Hz, 1H, minor), 4.44 (q, *J* = 6.7 Hz, 1H, major), 3.93 (ddd, *J* = 12.2, 10.0, 2.9 Hz, 1 H, major), 3.81 (ddd, *J* = 11.4, 8.2, 3.2 Hz, 1H, minor), 3.58 (s, 1 H, major), 3.57 (s, 1H, minor), 3.47 (m, 2H, major+minor), 1.88–1.50 (m, 12H, major+minor), 1.44 (d, *J* = 6.8 Hz, 3 H, minor), 1.41 (d, *J* = 6.7 Hz, 3H, major), 0.14 (s, 6H, minor), 0.13 (s, 6H, major). ^{13}C NMR ($CDCl_3$, 125 MHz) major isomer: δ 142.4, 129.4, 128.7, 125.9, 108.4, 96.4, 87.8, 63.0, 62.4, 45.2, 31.0, 25.9, 22.3, 20.0, –1.4. Characteristic signals of the minor isomer: 142.3, 129.4, 128.7, 109.3, 97.4, 87.0, 63.4, 61.6, 19.4. IR (thin film) 2943, 2171, 1597, 1494 cm^{-1}; HRMS (CI/isobutane) *m* / *z* calcd for $C_{24}H_{31}O_2Si$ $(M + H)^+$ 379.2093, found 379.2098. Anal. Calcd for $C_{24}H_{30}O_2Si$: C, 76.14; H, 7.99. Found: C, 75.85; H, 8.04.

13 **23**

(±)-(*Z*)-1-Benzhydryldimethylsilyl-1-buten-3-ol (23). To a cooled (0 °C) solution of $BH_3 \cdot SMe_2$ (0.28 mL, 3.0 mmol) in 4 mL of THF was added cyclohexene (0.60 mL, 6.0 mmol). The reaction mixture was allowed to warm to 22 °C and gradually became milky white. After 2.5 h, the reaction mixture was cooled to 0 °C and **13** (0.757 g, 2.00 mmol) was added dropwise neat. The reaction mixture was allowed to warm to 22 °C and gradually became clear and colorless. After 2.5 h, the reaction mixture was cooled to 0 °C and AcOH (glacial, 0.28 mL, 5.0 mmol) was added dropwise. The reaction mixture was allowed to warm to 22 °C. After 12 h, the reaction mixture was poured into 10 mL of saturated aqueous $NaHCO_3$ and diluted with 10 mL of Et_2O. The layers were separated, and the aqueous layer was extracted with 3 × 10 mL of Et_2O. The combined organic layers were washed with 10 mL of brine, dried (Na_2SO_4), filtered, and concentrated *in vacuo*. The resultant pale yellow slurry was diluted with 10 mL of MeOH, and *p*-TsOH (0.008 g, 0.04 mmol) was added. The reaction mixture was stirred at 22 °C for 12 h, and then concentrated *in vacuo*. Purification by flash chromatography (5:95 to 20:80 EtOAC/hexanes) afforded **23** as a colorless oil (0.51 g, 86%) with an *Z*/*E* ratio > 99:1 as indicated by capillary GC analysis of the unpurified reaction mixture: GC t_R 4.9 min (DB-1, 1 min at 200 °C then ramped to 250 °C at 5 °C / min, 16 psi); ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.27 (m, 8H), 7.15 (m, 2H), 6.24 (dd, *J* = 14.2, 8.9 Hz, 1H), 5.60 (d, *J* = 14.2 Hz, 1H), 4.01 (m, 1H), 3.62 (s, 1H), 1.10 (d, *J* = 6.2 Hz, 3H), 1.01 (d, *J* = 3.1 Hz, 1H), 0.23 (s, 3H), 0.13 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 153.2, 142.82, 142.79, 129.4, 129.3, 128.8, 127.7, 125.9, 125.8, 69.0, 46.4, 23.1, –0.5, –0.7; IR (thin film) 3359, 2969, 1596, 1494, 1249 cm^{-1}; HRMS (CI/isobutane) *m* / *z* calcd for $C_{19}H_{23}Si$ $(M - OH)^+$ 279.1569, found 279.1568. Anal. Calcd for $C_{19}H_{24}OSi$: C, 76.97; H, 8.16. Found: C, 76.78; H, 8.08.



23 **14**

(±)-(*Z*)-1-Benzhydryldimethylsilyl-1-buten-3-ol *N*-phenylcarbamate (14). Phenyl isocyanate (0.330 mL, 3.05 mmol) was added to alcohol **23** (0.820 g, 2.77 mmol) at 22 °C. After stirring for 15 h, the resultant slurry was diluted with hexanes and then filtered. The filtrate was concentrated *in vacuo*. Purification by flash chromatography (5:95 EtOAc/hexanes) afforded **14** as a pale yellow oil (1.1 g, 96%): ^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.27–7.03 (m, 15H), 6.53 (s, 1H), 6.25 (dd, *J* = 14.5, 9.0 Hz, 1H), 5.67 (d, *J* = 14.5 Hz, 1H), 5.37 (m, 1H), 3.70 (s, 1H), 1.21 (d, *J* = 6.0 Hz, 3H), 0.24 (s, 3H), 0.22 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 153.1, 147.8, 142.8, 138.4, 130.3, 129.5, 129.48, 129.34, 128.85, 128.79, 125.78, 125.7, 123.8, 119.1, 72.5, 45.9, 21.5, –0.9, –1.0; IR (thin film) 3396, 3328, 1732, 1602, 1218 cm^{-1}; HRMS (CI/isobutane) *m* / *z* calcd for $C_{26}H_{30}NO_2Si$ $(M + H)^+$ 416.2046, found 416.2056. Anal. Calcd for $C_{26}H_{29}NO_2Si$: C, 75.40; H, 7.03; N, 3.37. Found: C, 75.07; H, 7.09; N, 3.32.

14 **16**

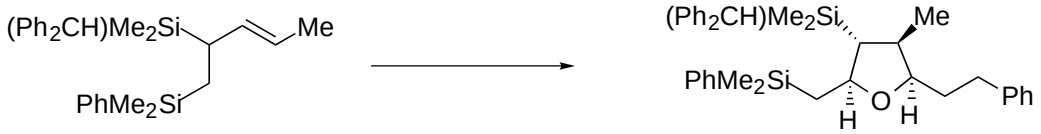
(±)-(*E*)-2-Benzhydryldimethylsilyl-3-pentene (16). A solution of CuI·2LiCl in THF was first prepared by stirring CuI (0.19 g, 1.0 mmol) and LiCl (0.085 g, 2.0 mmol) in 5 mL of THF at 22 °C for 10 min and then cooled to –78 °C. In a separate flask, to a cooled (–78 °C) solution of carbamate **14** (0.415 g, 1.00 mmol) in 4 mL of THF was added dropwise by syringe n-BuLi (2.7 M solution in hexanes, 0.37 mL, 1.0 mmol), and stirring for 5 min at –78 °C. The reaction mixture was then added to the CuI·2LiCl solution dropwise by cannula. After 30 min, MeLi (1.05 M solution in Et_2O, 0.95 mL, 1.0 mmol) was added dropwise by syringe, and the reaction mixture was allowed to warm to 22 °C without removing the cold bath. After 15 h, 30 mL of saturated aqueous NH_4Cl and 30 mL of Et_2O were added, and the mixture was stirred until the aqueous layer became transparent blue. The layers were separated, and the aqueous layer was extracted with 3 × 30 mL of Et_2O. The combined

organic layers were washed with 10 mL of brine, dried (Na_2SO_4), filtered, and concentrated in vacuo. Purification by flash chromatography (hexanes) afforded **16** as a colorless oil (0.246 g, 84 %) with an *E*/*Z* ratio > 99:1 and γ:α ratio of > 99:1 as indicated by capillary GC analysis of the unpurified reaction mixture: GC t_R 4.1 min (DB-1, 1 min at 200 °C then ramped to 250 °C at 5 °C / min, 16 psi); ^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.35 (m, 8H), 7.23 (m, 2H), 5.41 (ddq, *J* = 15.3, 8.1, 1.5 Hz, 1H), 5.20 (dqd, *J* = 15.2, 6.3, 1.2 Hz, 1H), 3.72 (s, 1H), 1.72 (dt, *J* = 6.4, 1.2 Hz, 3H), 1.66 (quintet, *J* = 7.3 Hz, 1H), 1.04 (d, *J* = 7.3 Hz, 3H), 0.12 (s, 3H), 0.11 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 143.24, 143.22, 133.8, 129.4, 129.3, 128.79, 128.77, 125.63, 125.62 122.0, 44.2, 24.8, 18.7, 14.5, –4.7, –4.8; IR (thin film) 2956, 1596, 1494, 1248 cm^{-1}; HRMS (CI/isobutane) *m* / *z* calcd for $C_{20}H_{26}Si$ (M^+) 294.1804, found 294.1803. Anal. Calcd for $C_{20}H_{26}Si$: C, 81.58; H, 8.91. Found: C, 81.34; H, 8.92.

14 **15**

(±)-(*E*)-2-Benzhydryldimethylsilyl-1-dimethylphenylsilyl-3-pentene (15). Using the procedure given for **16** with carbamate **14** (1.38 mg, 3.33 mmol), *n*-BuLi (1.9 M solution in hexanes, 1.75 mL, 3.33 mmol), CuI (0.634 g, 3.33 mmol), LiCl (0.282 g, 6.66 mmol), and (dimethylphenylsilyl)methylmagnesium chloride (1.25 M solution in THF, 2.66 mL, 3.33 mmol) afforded **15**, after purification by flash chromatography (hexanes), as a colorless oil (1.23 g, 86%) with an *E*/*Z* ratio of 98:2 and γ:α ratio of > 99:1 as indicated by capillary GC analysis of the unpurified reaction mixture: GC t_R 8.9 min (DB-1, 1 min at 200 °C then ramped to 275 °C at 10 °C / min, 16 psi); ^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.37–7.08 (m, 15H), 5.00 (m, 2H), 3.60 (s, 1H), 1.59 (ddd, *J* = 11.8, 8.9, 2.9 Hz, 1H), 1.52 (d, *J* = 5.3 Hz, 3H), 0.73 (dd, *J* = 14.8, 3.2 Hz, 1H), 0.68 (dd, *J* = 14.8, 11.6 Hz, 1H), 0.14 (s, 3H), 0.07 (s, 3H), –0.02 (s, 3H), –0.05 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 143.2, 140.6, 134.1, 133.5, 129.4, 129.3, 129.1, 128.8, 128.7, 128.0, 125.6, 125.5, 123.2, 43.9, 26.5, 18.5, 15.0, –1.4, –2.4, –4.5, –5.1; IR (thin film) 2956, 1596, 1494, 1246 cm^{-1}; HRMS (CI/isobutane) *m* / *z* calcd for $C_{28}H_{36}Si_2$ (M^+) 428.2355, found 428.2362. Anal. Calcd for $C_{28}H_{36}Si_2$: C, 78.44; H, 8.46. Found: C, 78.60; H, 8.46.

III. Annulations of (*E*)-Benzhydryldimethylcrotylsilanes



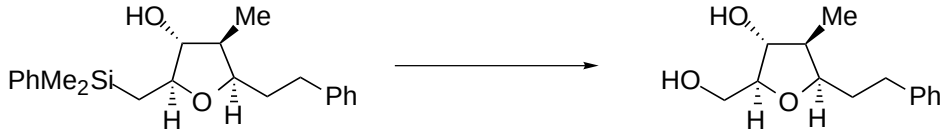
15 **17**

(2*S,3*R**,4*S**,5*R**)-3-Benzhydryldimethylsilyl-2-dimethylphenylsilylmethyl-4-methyl-5-(2-phenylethyl)tetrahydrofuran (17).** To a cooled (–78 °C) solution of (*E*)-2-(dimethylbenzhydrylsilyl)-1-(dimethylphenylsilyl)-3-pentene **15** (0.429 g, 1.00 mmol) in 4 mL of CH_2Cl_2 was added hydrocinnamaldehyde (0.40 mL, 3.0 mmol), and then $BF_3{\cdot}OEt_2$ (0.37 mL, 3.0 mmol) was added dropwise by syringe. The reaction mixture was allowed to warm to –45 °C. After 4 days at –45 °C, the reaction mixture was diluted with 30 mL of CH_2Cl_2 and 10 mL of saturated aqueous $NaHCO_3$ was added. The layers were separated, and the aqueous layer was extracted with 3 × 30 mL of CH_2Cl_2. The combined organic layers were washed with 20 mL of brine, dried (Na_2SO_4), filtered, and concentrated *in vacuo*. Purification by flash chromatography (20:80 to 30:70 CH_2Cl_2/hexanes) afforded **17** as a colorless oil (0.497 g, 88%): ^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.52 (m, 2H), 7.34 (m, 3H), 7.27–7.08 (m, 15H), 3.67 (td, *J* = 9.7, 2.6 Hz, 1H), 3.49 (s, 1H), 3.32 (ddd, *J* = 9.3, 5.9, 3.8 Hz, 1H), 2.74 (ddd, *J* = 14.2, 9.9, 4.5, 1H), 2.54 (ddd, *J* = 13.8, 9.8, 6.8 Hz, 1H), 1.93 (m 1H), 1.70 (m, 1H), 1.55 (m, 1H), 0.92 (dd, *J* = 14.7, 2.8 Hz, 1H), 0.87 (dd, *J* = 14.7, 10.2 Hz, 1H), 0.72 (dd, *J* = 9.0, 5.5 Hz, 1 H), 0.67 (d, *J* = 6.8 Hz, 3H), 0.33 (s, 3H), 0.30 (s, 3H), 0.02 (s, 3H), –0.02 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 143.12, 143.06, 140.6, 134.2, 129.4, 129.1, 129.0, 128.9, 128.85, 128.76, 128.1, 126.1, 125.82, 125.80, 81.0, 78.3, 45.1, 44.5, 39.8, 33.9, 33.1, 25.6, 18.3, –1.1, –1.9, –3.3, –3.4; IR (thin film)

2955, 1596, 1494, 1249 cm^{-1}; HRMS (FAB) *m* / *z* calcd for $C_{37}H_{46}OSi_2Na$ $(M + Na)^+$ 585.2985, found 585.2994. Anal. Calcd for $C_{37}H_{46}OSi_2$: C, 78.96; H, 8.24. Found: C, 78.99; H, 8.15.

17 **18**

(2*S,3*R**,4*S**,5*R**)-2-Dimethylphenylsilylmethyl-3-hydroxy-4-methyl-5-(2-phenylethyl)tetrahydrofuran (18).** The same procedure given for **4** was followed. The reagents used were: **17** (0.265 g, 0.470 mmol), *n*-Bu_4NF (1.0 M solution in THF, 0.75 mL, 0.75 mmol), MeOH (1 mL), $KHCO_3$ (0.070 g, 0.70 mmol), H_2O_2 (30%, 0.53 mL, 4.7 mmol). Purification by flash chromatography (20:80 to 30:70 CH_2Cl_2/hexanes) afforded **18** as a colorless oil (0.141 g, 85%): ^1H NMR ($CDCl_3$, 500 MHz) δ 7.57 (m, 2H), 7.35 (m, 3H), 7.26 (m, 2H), 7.18 (m, 3H), 3.90 (ddd, *J* = 10.3, 5.9, 4.6 Hz, 1H), 3.49 (td, *J* = 7.3, 4.8 Hz, 1H), 3.37 (m, 1H), 2.79 (ddd, *J* = 14.6, 9.3, 5.2 Hz, 1H), 2.54 (ddd, *J* = 13.8, 9.8, 6.6 Hz 1H), 1.95 (m 1H), 1.76 (m, 1H), 1.66 (m, 1H), 1.47 (br, 1H), 1.20 (d, *J* = 7.4 Hz, 2H), 0.90 (d, *J* = 7.3 Hz, 3H), 0.39 (s, 3H), 0.36 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 142.7, 139.6, 134.1, 129.5, 128.9, 128.8, 128.3, 126.2, 86.2, 83.5, 79.1, 45.4, 33.4, 33.3, 22.8, 13.3, –1.4, –2.1; IR (thin film) 3385, 1112 cm^{-1}; HRMS (FAB+) *m* / *z* calcd for $C_{22}H_{30}O_2SiNa$ $(M + Na)^+$ 377.1913, found 377.1917. Anal. Calcd for $C_{22}H_{30}O_2Si$: C, 74.53; H, 8.53. Found: C, 74.47; H, 8.49.



18 **19**

(2*S,3*R**,4*S**,5*R**)-3-Hydroxy-2-hydroxymethyl-4-methyl-5-(2-phenylethyl)tetrahydrofuran (19).** Potassium bromide (0.043 g, 0.36 mmol) and anhydrous NaOAc (0.074 g, 0.90 mmol) were added to a stirred solution of **18** (0.106 g, 0.300 mmol) in 1.0 mL of AcOH (glacial). The reaction mixture was cooled to 0 °C and AcOOH (32%, 0.39 mL, 1.8 mmol) was added dropwise, during which time Br_2 was generated and the reaction mixture became orange. After the addition, more anhydrous NaOAc (0.23 g, 2.8 mmol) and AcOOH (32%, 1.27 mL, 5.40 mmol) were added. After 14 h at 22 °C, the reaction mixture was diluted with 30 mL of Et_2O, and 3 g of $Na_2S_2O_3$ was added. The reaction mixture was stirred vigorously for 30 min, filtered through Celite, washed with Et_2O, and concentrated *in vacuo*. The residue was dissolved in 30 mL of EtOAc, washed with 5 mL of saturated aqueous $NaHCO_3$ and 5 mL of brine, dried (Na_2SO_4), filtered, and concentrated *in vacuo*. Purification by flash chromatography (10:90 to 60:40 EtOAc/hexanes) afforded **19** as a colorless oil (0.053 g, 75%): ^1H NMR ($CDCl_3$, 500 MHz) δ 7.22 (t, *J* = 7.5 Hz, 2H), 7.20 (m, 3H), 4.08 (ddd, *J* = 8.9, 6.3, 4.9 Hz, 1H), 3.78 (m, 2H), 3.69 (m, 2H), 2.79 (ddd, *J* = 14.0, 9.8, 5.7 Hz, 1H), 2.63 (ddd, *J* = 13.9, 9.6, 6.7 Hz, 1H), 2.22 (br, 2H), 2.14 (m, 1H), 1.76 (m, 2H), 0.95 (d, *J* = 7.3 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 142.4, 128.9, 126.4, 85.7, 80.3, 79.9, 63.5, 45.2, 33.3, 33.1, 12.6; IR (thin film) 3382, 1455, 1056 cm^{-1}; HRMS (CI/isobutane) *m* / *z* calcd for $C_{14}H_{21}O_3$ $(M + H)^+$ 237.1490, found 237.1487.

16 **20**

(3*S,4*R**,5*S**)-4-Benzhydryldimethylsilyl-3,5-dimethyl-2-pyrrolidinone (20).** To a cooled (0 °C) solution of **16** (0.176 g, 0.600 mmol) in 5 mL of toluene was added chlorosulfonyl isocyanate (0.063 mL, 0.72 mmol). The reaction mixture was allowed to warm to 22 °C. After 2 h, the reaction mixture was concentrated *in vacuo*. The residue was dissolved in 5 mL of CH_2Cl_2, and 5 mL of 25% aqueous Na_2SO_3 was added. The reaction mixture was then stirred vigorously at 22 °C for 20 h. The layers were separated and the

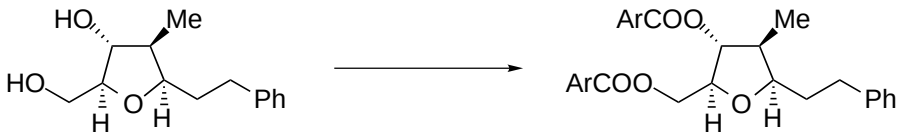
aqueous layer was extracted with 3 × 20 mL of CH_2Cl_2. The combined organic layers were washed with 10 mL of brine, dried (Na_2SO_4), filtered, and concentrated *in vacuo*. Purification by flash chromatography (10:90 to 80:20 EtOAc/hexanes) afforded **20** as a white solid (0.178 g, 88%): mp 190–192 °C; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.25 (m, 8H), 7.15 (m, 2H), 6.48 (s, 1H), 3.60 (s, 1H), 3.46 (quintet, J = 6.2 Hz, 1H), 2.27 (m, 1H), 1.07 (d, J = 7.1 Hz, 3H), 1.01 (d, J = 6.1 Hz, 3H), 0.88 (t, J = 8.2 Hz, 1H), 0.11 (s, 6H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 180.8, 142.4, 129.29, 129.26, 129.0, 126.1, 50.5, 44.6, 39.7, 37.1, 24.7, 19.2, –3.8, –3.9; IR (KBr) 3198, 1693 cm^{-1}; HRMS (CI/isobutane) m / z calcd for $C_{21}H_{28}NOSi$ $(M + H)^+$ 338.1941, found 338.1948.

20 **21**

(3*R,4*R**,5*S**)-3,5-Dimethyl-4-hydroxy-2-pyrrolidinone (21).** To a solution of **20** (0.068 g, 0.20 mmol) in 8 mL of THF was added *n*-Bu_4NF (1 M solution in CH_2Cl_2, 0.32 mL, 0.32 mmol) dropwise by syringe at 0 °C. The reaction mixture was then stirred at 22 °C for 0.5 h, then 0.5 mL of MeOH was added, followed by $KHCO_3$ (30 mg, 0.30 mmol) and H_2O_2 (30%, 0.23 mL, 2.0 mmol). After 12 h, the reaction mixture was diluted with 30 mL of Et_2O, and 1 g of $Na_2S_2O_3$ was added. The mixture was stirred vigorously for 0.5 h, filtered through Celite, washed with Et_2O, dried ($MgSO_4$), and concentrated *in vacuo*. Purification by flash chromatography (5:95 to 10:90 MeOH/CH_2Cl_2) afforded **21** as a white solid (0.021 g, 81%): mp 99–101 °C; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 6.40 (s, 1H), 3.54 (m, 2H), 3.45 (quintet, J = 6.3 Hz, 1H), 2.39 (quintet, J = 7.3 Hz, 1H), 1.30 (d, J = 6.3 Hz, 3H), 1.24 (d, J = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 177.6, 82.5, 56.2, 45.8, 19.5, 13.6; IR (KBr) 3382, 3224, 1694 cm^{-1}; HRMS (CI/isobutane) m / z calcd for $C_6H_{11}NO_2$ (M^+) 129.0789, found 129.0791. Anal. Calcd for $C_6H_{11}NO_2$: C, 55.80; H, 8.58; N, 10.84. Found: C, 55.51; H, 8.56; N, 10.59.

18 **22**

(3*S,4*S**,5*R**)-4-Methyl-7-phenyl-1-hepten-3,5-diol (22).** To a solution of **18** (0.080 g, 0.22 mmol) in 2 mL of DMSO was added *t*-BuOK (35 mg, 0.31 mmol) at 22 °C. After 1 h, the reaction mixture became dark orange, and 5 mL of saturated aqueous NH_4Cl was added. The reaction mixture was extracted with 3 × 10 mL of Et_2O, the combined organic layers were washed with 10 mL of brine, dried (Na_2SO_4), filtered, and concentrated *in vacuo*. Purification by flash chromatography (10:90 to 20:80 EtOAc/hexanes) afforded **22** as a colorless oil (0.039 g, 81%): ^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.27 (t, J = 7.5 Hz, 2H), 7.19 (m, 3H), 5.87 (ddd, J = 17.2, 10.6, 5.1 Hz, 1H), 5.25 (d, J = 17.2 Hz, 1H), 5.15 (d, J = 10.6 Hz, 1H), 4.38 (m, 1H), 3.94 (m, 1H), 2.86 (br, 1H), 2.85–2.75 (m, 2H), 2.64 (m, 1H), 1.87 (m, 1H), 1.69 (m, 1H), 1.61 (m, 1 H), 0.92 (d, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 142.4, 140.1, 128.9, 126.3, 115.1, 77.9, 76.0, 42.0, 37.5, 32.9, 5.4; IR (thin film) 3360, 1645, 1455 cm^{-1}; HRMS (CI/isobutane) m / z calcd for $C_{14}H_{21}O_2$ $(M + H)^+$ 221.1541, found 221.1548. Anal. Calcd for $C_{14}H_{20}O_2$: C, 76.31; H, 9.16. Found: C, 76.04; H, 9.37.



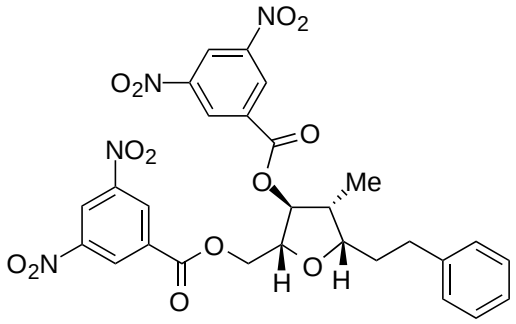
19 **24**

Ar = 3,5-NO_2-C_6H_3-

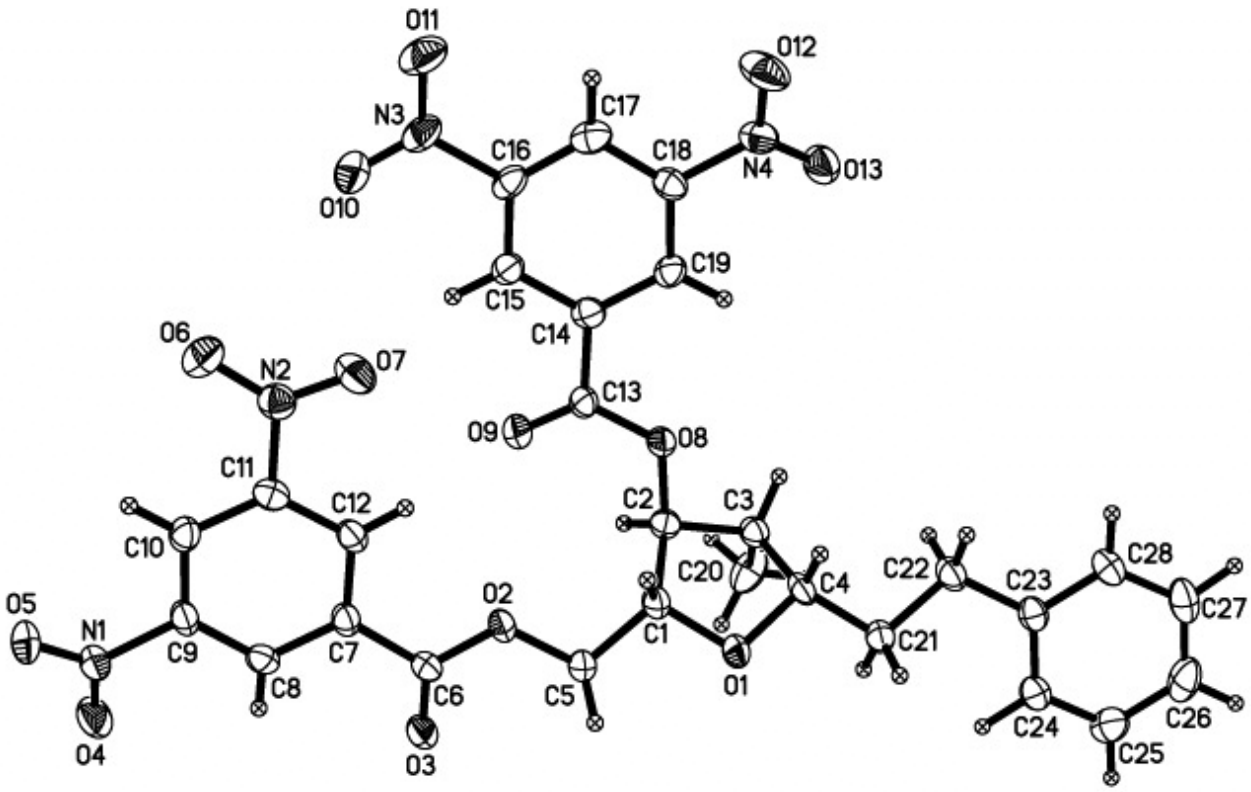
(2*S,3*R**,4*S**,5*R**)-3-(3,5-Dinitrophenylcarboxy)-2-(3,5-dinitrophenylcarboxymethyl)-4-methyl-5-(2-phenylethyl)tetrahydrofuran (24).** To a solution of **19** (0.033 g, 0.14 mmol) in 3 mL of CH_2Cl_2 was added 3,5-dinitro-benzoyl chloride (0.097 g, 0.42 mmol), 4-(*N*,*N*-dimethylamino)pyridine (0.005 g,

0.03 mmol), and Et_3N (0.058 mL, 0.42 mmol). After stirring at 22 °C for 12 h, 10 mL of saturated aqueous Na_2HPO_4 and 10 mL of CH_2Cl_2 were added. The layers were separated, and the aqueous layer was washed with 3 × 10 mL of CH_2Cl_2. The combined organic layers were washed with 10 mL of brine, dried ($MgSO_4$), filtered, and concentrated *in vacuo*. Purification by flash chromatography (10:90 EtOAc/hexanes) afforded **24** as a pale yellow solid (0.070 g, 80%). Recrystallization from CH_2Cl_2/EtOAc/hexanes (10:10:80) provided a single crystal suitable for X-ray analysis. ^{1}H NMR ($CDCl_3$, 500 MHz) δ 9.24 (t, *J* = 2.1 Hz, 1H), 9.22 (t, *J* = 2.1 Hz, 1H), 9.20 (d, *J* = 2.1 Hz, 2H), 9.13 (d, *J* = 2.1 Hz, 2H), 7.29 (t, *J* = 7.5 Hz, 2H), 7.20 (m, 3H), 5.17 (dd, *J* = 3.5, 1.3 Hz, 1H), 4.80 (dd, *J* = 11.7, 4.5 Hz, 1H), 4.70 (dd, *J* = 11.7, 5.7 Hz, 1H), 4.29 (dt, *J* = 5.5, 4.5 Hz, 1H), 4.21 (dt, *J* = 8.5, 5.0 Hz, 1H), 2.85 (ddd, *J* = 15.4, 10.0, 5.6 Hz, 1H), 2.71 (ddd, *J* = 16.0, 9.7, 6.4 Hz, 1H), 2.55 (m, 1H), 2.00 (m, 1H), 1.86 (m, 1H), 1.19 (d, *J* = 7.4 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 162.81, 162.80, 149.2, 149.1, 141.8, 133.9, 133.6, 130.0, 129.9, 128.9, 128.8, 126.6, 123.3, 123.0, 85.3, 81.7, 81.1, 66.7, 42.7, 33.1, 32.1, 12.6.

IV. X-ray Analysis of Compound 24



24



X-ray Data Collection, Structure Solution and Refinement for **24**:

A colorless crystal of approximate dimensions 0.05 x 0.15 x 0.25 mm was mounted on a glass fiber and transferred to a Bruker CCD platform diffractometer. The SMART[1] program package was used to determine the unit-cell parameters and for data collection (30 sec/frame scan time for a sphere of diffraction data). The raw frame data was processed using SAINT[2] and SADABS[3] to yield the reflection data file. Subsequent calculations were carried out using the SHELXTL[4] program. There were no systematic absences nor any diffraction symmetry other than the Friedel condition. The centrosymmetric triclinic space group $P\bar{1}$ was assigned and later determined to be correct.

The structure was solved by direct methods and refined on F^2 by full-matrix least-squares techniques. The analytical scattering factors[5] for neutral atoms were used throughout the analysis. Hydrogen atoms were located from a difference-Fourier map and refined (x,y,z and U_{iso}). At convergence, wR2 = 0.1365 and GOF = 1.004

for 503 variables refined against 6607 data (As a comparison for refinement on F, R1 = 0.0544 for those 3838 data with $I > 2.0\sigma(I)$).

References.

1. SMART Software Users Guide, Version 5.0, Bruker Analytical X-Ray Systems, Inc.; Madison, WI 1999.
2. SAINT Software Users Guide, Version 6.0, Bruker Analytical X-Ray Systems, Inc.; Madison, WI 1999.
3. Sheldrick, G. M. SADABS, Bruker Analytical X-Ray Systems, Inc.; Madison, WI 1999.
4. Sheldrick, G. M. SHELXTL Version 5.10, Bruker Analytical X-Ray Systems, Inc.; Madison, WI 1999.
5. International Tables for X-Ray Crystallography 1992, Vol. C., Dordrecht: Kluwer Academic Publishers.

Definitions:

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

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

$Goof = S = [\Sigma[w(F_o^2-F_c^2)^2] / (n-p)]^{1/2}$ where n is the number of reflections and p is the total number of parameters refined.

The thermal ellipsoid plot is shown at the 50% probability level.

Table 1. Crystal data and structure refinement for **24**.

Empirical formula	$C_{28}\ H_{24}\ N_4\ O_{13}$	
Formula weight	624.51	
Temperature	163(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	$P\bar{1}$	
Unit cell dimensions	a = 11.5924(8) Å	α= 72.9710(10)°.
	b = 11.7511(8) Å	β= 66.0370(10)°.

	c = 12.3782(9) Å γ = 65.7610(10)°.
Volume	1388.84(17) Å^3
Z	2
Density (calculated)	1.493 Mg/m^3
Absorption coefficient	0.121 mm^{-1}
F(000)	648
Crystal size	0.25 x 0.15 x 0.05 mm^3
Theta range for data collection	1.82 to 28.28°.
Index ranges	$-15 \le h \le 15$, $-14 \le k \le 15$, $-16 \le l \le 16$
Reflections collected	15122
Independent reflections	6607 [R(int) = 0.0471]
Completeness to theta = 28.28°	95.6 %
Absorption correction	None
Max. and min. transmission	0.9940 and 0.9705
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	6607 / 0 / 503
Goodness-of-fit on F^2	1.004
Final R indices [I > 2sigma(I)]	R1 = 0.0544, wR2 = 0.1092
R indices (all data)	R1 = 0.1124, wR2 = 0.1365
Extinction coefficient	0.0024(8)
Largest diff. peak and hole	0.350 and -0.264 e.Å^{-3}

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for **24**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	10820(2)	3133(2)	5168(1)	27(1)
O(2)	7316(2)	4316(2)	6787(1)	28(1)
O(3)	6622(2)	3295(2)	8647(1)	32(1)
O(4)	1893(2)	5131(2)	11061(2)	39(1)
O(5)	882(2)	7128(2)	10568(2)	38(1)
O(6)	2161(2)	8619(2)	6234(2)	42(1)
O(7)	4290(2)	8038(2)	5242(2)	36(1)
O(8)	8762(2)	4375(2)	3502(1)	25(1)
O(9)	6601(2)	4570(2)	4568(1)	34(1)
O(10)	3225(2)	8234(2)	3178(2)	41(1)
O(11)	3811(2)	9427(2)	1486(2)	43(1)
O(12)	8530(2)	8376(2)	-971(2)	45(1)
O(13)	9760(2)	6423(2)	-672(2)	35(1)
N(1)	1787(2)	6125(2)	10358(2)	31(1)
N(2)	3320(2)	7952(2)	6132(2)	29(1)
N(3)	4063(2)	8523(2)	2248(2)	32(1)
N(4)	8746(2)	7325(2)	-369(2)	29(1)
C(1)	9431(2)	3734(2)	5292(2)	24(1)
C(2)	9215(2)	3291(2)	4359(2)	24(1)

C(3)	10603(2)	2468(2)	3687(2)	24(1)
C(4)	11514(2)	2920(2)	3945(2)	24(1)
C(5)	8657(2)	3378(3)	6593(2)	29(1)
C(6)	6410(2)	4145(2)	7852(2)	25(1)
C(7)	5074(2)	5158(2)	7956(2)	23(1)
C(8)	4068(2)	5171(2)	9049(2)	24(1)
C(9)	2846(2)	6105(2)	9174(2)	25(1)
C(10)	2554(3)	7041(2)	8244(2)	26(1)
C(11)	3577(2)	6986(2)	7158(2)	25(1)
C(12)	4832(2)	6070(2)	6987(2)	24(1)
C(13)	7447(2)	4902(2)	3708(2)	23(1)
C(14)	7120(2)	5951(2)	2734(2)	24(1)
C(15)	5786(2)	6719(2)	2929(2)	25(1)
C(16)	5470(2)	7702(2)	2040(2)	26(1)
C(17)	6417(3)	7949(2)	959(2)	28(1)
C(18)	7715(2)	7125(2)	785(2)	25(1)
C(19)	8103(3)	6147(2)	1649(2)	24(1)
C(20)	10783(3)	1078(3)	4151(3)	39(1)
C(21)	12929(2)	2038(3)	3813(2)	26(1)
C(22)	13727(3)	1860(3)	2507(2)	29(1)
C(23)	15195(2)	1057(2)	2244(2)	26(1)
C(24)	15694(3)	138(2)	3097(2)	30(1)
C(25)	17044(3)	-579(3)	2809(3)	36(1)
C(26)	17922(3)	-379(3)	1654(3)	41(1)
C(27)	17430(3)	519(3)	803(3)	38(1)
C(28)	16079(3)	1230(3)	1095(2)	30(1)

Table 3. Bond lengths [Å] and angles [°] for **24**.

O(1)-C(1)	1.430(3)
O(1)-C(4)	1.438(3)
O(2)-C(6)	1.332(3)
O(2)-C(5)	1.461(3)
O(3)-C(6)	1.203(3)
O(4)-N(1)	1.229(3)
O(5)-N(1)	1.225(3)
O(6)-N(2)	1.221(3)
O(7)-N(2)	1.229(2)
O(8)-C(13)	1.332(3)
O(8)-C(2)	1.461(3)
O(9)-C(13)	1.212(3)
O(10)-N(3)	1.232(3)
O(11)-N(3)	1.219(3)
O(12)-N(4)	1.225(2)
O(13)-N(4)	1.224(3)
N(1)-C(9)	1.480(3)
N(2)-C(11)	1.475(3)
N(3)-C(16)	1.473(3)
N(4)-C(18)	1.473(3)
C(1)-C(5)	1.521(3)
C(1)-C(2)	1.537(3)
C(2)-C(3)	1.525(3)

C(3)-C(20)	1.519(4)
C(3)-C(4)	1.527(3)
C(4)-C(21)	1.509(3)
C(6)-C(7)	1.499(3)
C(7)-C(8)	1.380(3)
C(7)-C(12)	1.393(3)
C(8)-C(9)	1.371(3)
C(9)-C(10)	1.385(3)
C(10)-C(11)	1.382(3)
C(11)-C(12)	1.384(3)
C(13)-C(14)	1.495(3)
C(14)-C(15)	1.394(3)
C(14)-C(19)	1.396(3)
C(15)-C(16)	1.385(3)
C(16)-C(17)	1.386(3)
C(17)-C(18)	1.382(3)
C(18)-C(19)	1.383(3)
C(21)-C(22)	1.529(3)
C(22)-C(23)	1.518(3)
C(23)-C(24)	1.389(3)
C(23)-C(28)	1.390(3)
C(24)-C(25)	1.387(4)
C(25)-C(26)	1.398(4)
C(26)-C(27)	1.375(4)
C(27)-C(28)	1.387(4)
C(1)-O(1)-C(4)	106.71(16)
C(6)-O(2)-C(5)	116.12(18)
C(13)-O(8)-C(2)	117.28(18)
O(5)-N(1)-O(4)	125.0(2)
O(5)-N(1)-C(9)	117.9(2)
O(4)-N(1)-C(9)	117.2(2)
O(6)-N(2)-O(7)	124.7(2)
O(6)-N(2)-C(11)	117.7(2)
O(7)-N(2)-C(11)	117.6(2)
O(11)-N(3)-O(10)	124.9(2)
O(11)-N(3)-C(16)	117.8(2)
O(10)-N(3)-C(16)	117.3(2)
O(13)-N(4)-O(12)	124.1(2)
O(13)-N(4)-C(18)	117.68(19)
O(12)-N(4)-C(18)	118.2(2)
O(1)-C(1)-C(5)	106.53(18)
O(1)-C(1)-C(2)	106.03(18)
C(5)-C(1)-C(2)	116.1(2)
O(8)-C(2)-C(3)	107.51(18)
O(8)-C(2)-C(1)	109.76(19)
C(3)-C(2)-C(1)	104.84(19)
C(20)-C(3)-C(2)	110.6(2)
C(20)-C(3)-C(4)	113.2(2)
C(2)-C(3)-C(4)	102.00(19)
O(1)-C(4)-C(21)	109.54(18)
O(1)-C(4)-C(3)	103.28(18)
C(21)-C(4)-C(3)	117.4(2)
O(2)-C(5)-C(1)	104.79(19)
O(3)-C(6)-O(2)	125.0(2)

O(3)-C(6)-C(7)	123.2(2)
O(2)-C(6)-C(7)	111.75(19)
C(8)-C(7)-C(12)	120.0(2)
C(8)-C(7)-C(6)	118.3(2)
C(12)-C(7)-C(6)	121.8(2)
C(9)-C(8)-C(7)	119.3(2)
C(8)-C(9)-C(10)	123.1(2)
C(8)-C(9)-N(1)	118.8(2)
C(10)-C(9)-N(1)	118.1(2)
C(11)-C(10)-C(9)	116.0(2)
C(10)-C(11)-C(12)	123.2(2)
C(10)-C(11)-N(2)	118.4(2)
C(12)-C(11)-N(2)	118.4(2)
C(11)-C(12)-C(7)	118.4(2)
O(9)-C(13)-O(8)	124.9(2)
O(9)-C(13)-C(14)	123.1(2)
O(8)-C(13)-C(14)	111.99(19)
C(15)-C(14)-C(19)	120.5(2)
C(15)-C(14)-C(13)	117.8(2)
C(19)-C(14)-C(13)	121.7(2)
C(16)-C(15)-C(14)	118.5(2)
C(15)-C(16)-C(17)	123.0(2)
C(15)-C(16)-N(3)	118.6(2)
C(17)-C(16)-N(3)	118.4(2)
C(18)-C(17)-C(16)	116.4(2)
C(17)-C(18)-C(19)	123.4(2)
C(17)-C(18)-N(4)	118.2(2)
C(19)-C(18)-N(4)	118.4(2)
C(18)-C(19)-C(14)	118.2(2)
C(4)-C(21)-C(22)	111.0(2)
C(23)-C(22)-C(21)	116.0(2)
C(24)-C(23)-C(28)	118.3(2)
C(24)-C(23)-C(22)	122.9(2)
C(28)-C(23)-C(22)	118.7(2)
C(25)-C(24)-C(23)	120.7(2)
C(24)-C(25)-C(26)	120.2(3)
C(27)-C(26)-C(25)	119.3(3)
C(26)-C(27)-C(28)	120.2(3)
C(27)-C(28)-C(23)	121.3(3)

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **24**. 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}
O(1)	20(1)	36(1)	22(1)	-12(1)	-2(1)	-6(1)
O(2)	21(1)	34(1)	18(1)	-4(1)	-1(1)	-4(1)
O(3)	27(1)	36(1)	21(1)	-1(1)	-4(1)	-6(1)
O(4)	36(1)	42(1)	25(1)	-1(1)	-1(1)	-13(1)

O(5)	25(1)	45(1)	33(1)	-19(1)	-2(1)	1(1)
O(6)	32(1)	42(1)	44(1)	-1(1)	-17(1)	-3(1)
O(7)	41(1)	36(1)	25(1)	-3(1)	-6(1)	-14(1)
O(8)	21(1)	25(1)	20(1)	1(1)	-5(1)	-3(1)
O(9)	23(1)	45(1)	25(1)	1(1)	-4(1)	-9(1)
O(10)	28(1)	43(1)	41(1)	-13(1)	-10(1)	1(1)
O(11)	43(1)	31(1)	51(1)	-3(1)	-27(1)	2(1)
O(12)	61(1)	29(1)	27(1)	3(1)	-11(1)	-7(1)
O(13)	33(1)	34(1)	28(1)	-6(1)	-5(1)	-6(1)
N(1)	24(1)	43(1)	23(1)	-10(1)	-4(1)	-9(1)
N(2)	32(1)	28(1)	28(1)	-6(1)	-11(1)	-10(1)
N(3)	29(1)	26(1)	41(1)	-12(1)	-19(1)	3(1)
N(4)	35(1)	26(1)	24(1)	-4(1)	-12(1)	-6(1)
C(1)	20(1)	26(1)	21(1)	-6(1)	-3(1)	-4(1)
C(2)	23(1)	23(1)	22(1)	1(1)	-8(1)	-7(1)
C(3)	24(1)	23(1)	21(1)	-4(1)	-5(1)	-5(1)
C(4)	24(1)	27(1)	15(1)	-5(1)	0(1)	-8(1)
C(5)	20(1)	37(2)	21(1)	-5(1)	-5(1)	-3(1)
C(6)	25(1)	33(1)	19(1)	-7(1)	-6(1)	-9(1)
C(7)	21(1)	27(1)	21(1)	-8(1)	-3(1)	-8(1)
C(8)	28(1)	28(1)	20(1)	-5(1)	-9(1)	-10(1)
C(9)	21(1)	31(1)	21(1)	-10(1)	-1(1)	-9(1)
C(10)	22(1)	28(1)	27(1)	-11(1)	-7(1)	-5(1)
C(11)	30(1)	26(1)	22(1)	-4(1)	-10(1)	-11(1)
C(12)	24(1)	29(1)	18(1)	-6(1)	-4(1)	-11(1)
C(13)	20(1)	27(1)	22(1)	-10(1)	-5(1)	-4(1)
C(14)	27(1)	23(1)	23(1)	-9(1)	-10(1)	-3(1)
C(15)	24(1)	28(1)	25(1)	-11(1)	-10(1)	-2(1)
C(16)	23(1)	25(1)	32(1)	-13(1)	-14(1)	1(1)
C(17)	35(2)	19(1)	32(1)	-6(1)	-17(1)	-3(1)
C(18)	29(1)	25(1)	20(1)	-6(1)	-7(1)	-8(1)
C(19)	22(1)	23(1)	26(1)	-8(1)	-9(1)	-1(1)
C(20)	30(2)	22(2)	64(2)	-7(1)	-19(2)	-4(1)
C(21)	22(1)	33(2)	23(1)	-7(1)	-4(1)	-10(1)
C(22)	27(2)	30(2)	21(1)	-5(1)	-2(1)	-6(1)
C(23)	25(1)	25(1)	24(1)	-9(1)	-3(1)	-8(1)
C(24)	27(2)	33(2)	25(1)	-9(1)	-4(1)	-8(1)
C(25)	32(2)	36(2)	38(2)	-15(1)	-15(1)	0(1)
C(26)	23(2)	48(2)	51(2)	-29(2)	-5(1)	-3(1)
C(27)	31(2)	44(2)	34(2)	-17(1)	5(1)	-16(1)
C(28)	33(2)	30(2)	23(1)	-6(1)	-2(1)	-11(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **24**.

	x	y	z	U(eq)
H(1A)	9180(20)	4690(20)	5117(19)	19(6)
H(2A)	8600(20)	2840(20)	4693(18)	12(5)
H(3A)	10710(20)	2660(20)	2850(20)	27(6)
H(4A)	11560(20)	3720(20)	3440(20)	29(7)

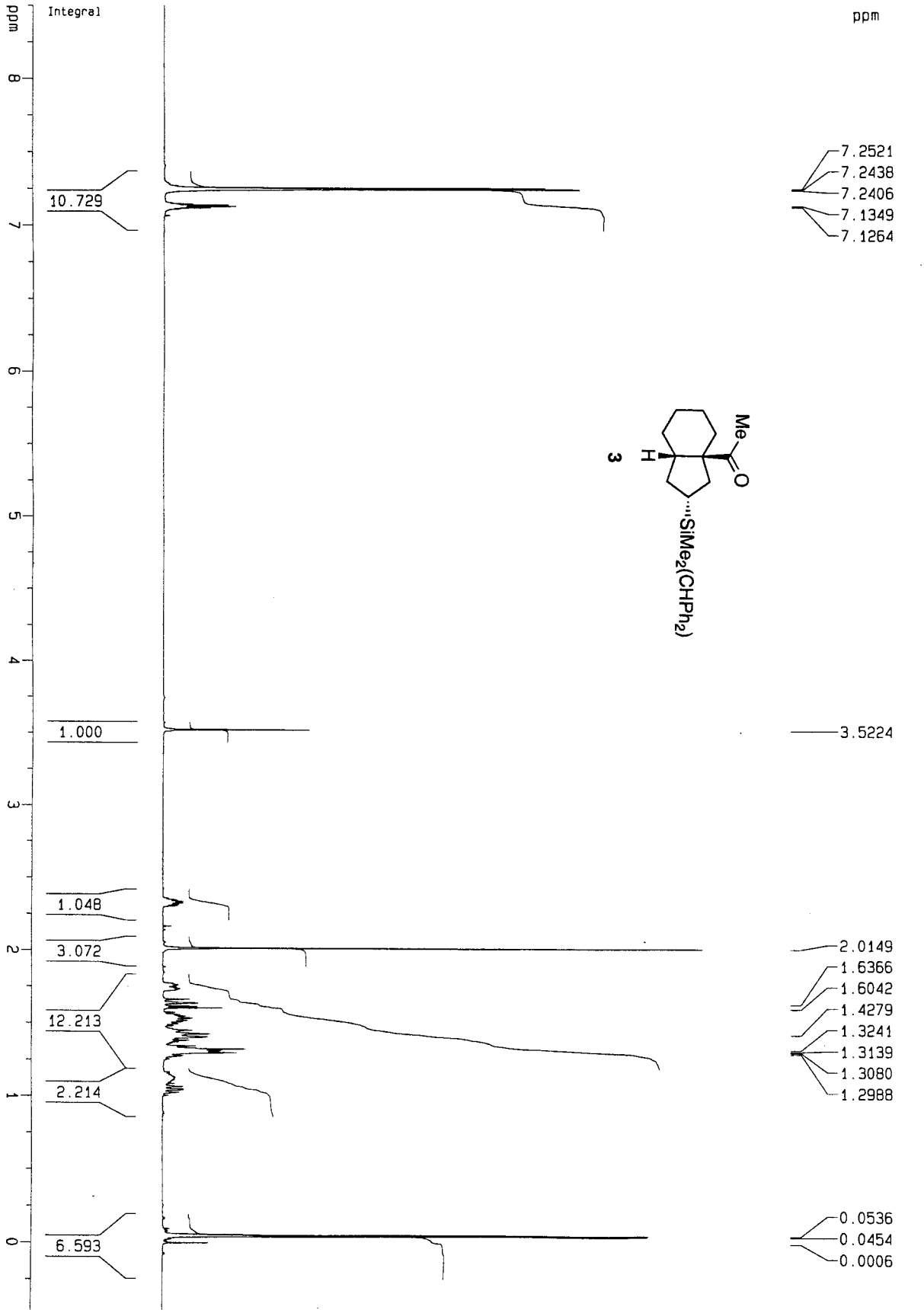
H(5A)	8620(20)	2510(30)	6730(20)	34(7)
H(5B)	9100(20)	3430(20)	7080(20)	24(6)
H(8A)	4200(20)	4640(20)	9640(20)	27(7)
H(10A)	1730(30)	7700(20)	8350(20)	34(7)
H(12A)	5450(20)	6040(20)	6270(20)	29(7)
H(15A)	5120(30)	6590(30)	3660(20)	45(8)
H(17A)	6190(20)	8610(20)	390(20)	29(7)
H(19A)	8990(20)	5640(20)	1496(18)	18(6)
H(20A)	11690(30)	490(30)	3750(20)	49(8)
H(20B)	10060(30)	840(30)	4090(20)	54(9)
H(20C)	10620(30)	930(20)	5010(20)	36(8)
H(21A)	12920(20)	1230(20)	4300(20)	29(7)
H(21B)	13320(20)	2390(20)	4090(20)	28(7)
H(22A)	13290(30)	1470(30)	2220(20)	41(8)
H(22B)	13680(30)	2650(30)	1990(20)	38(7)
H(24A)	15070(30)	-10(20)	3930(20)	41(8)
H(25A)	17390(30)	-1180(20)	3390(20)	34(7)
H(26A)	18850(30)	-820(30)	1470(20)	43(8)
H(27A)	18000(30)	660(20)	-10(20)	44(8)
H(28A)	15810(20)	1840(20)	500(20)	19(6)

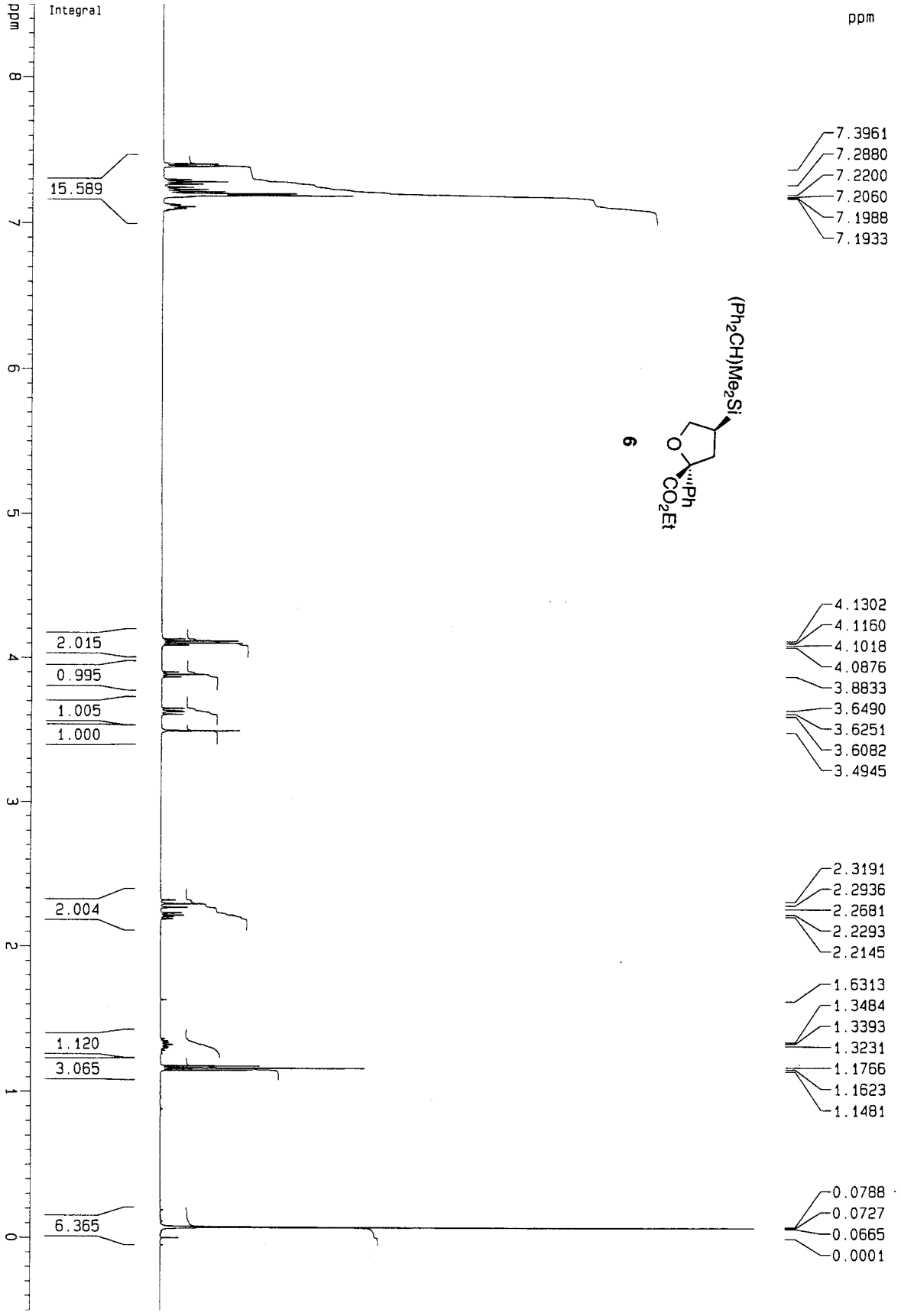
Table 6. Torsion angles [°] for **24**.

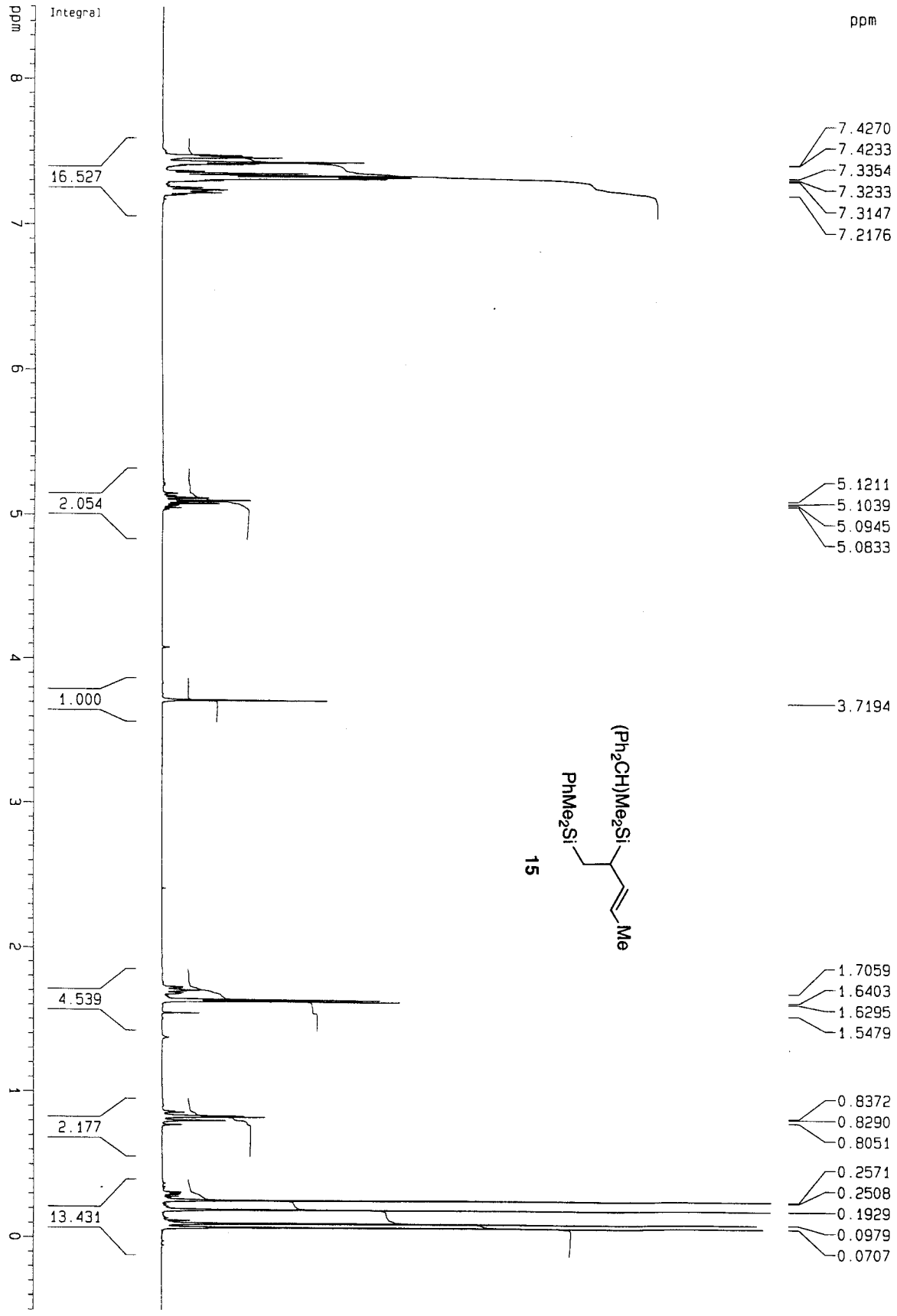
C(4)-O(1)-C(1)-C(5)	152.2(2)
C(4)-O(1)-C(1)-C(2)	27.9(2)
C(13)-O(8)-C(2)-C(3)	151.44(19)
C(13)-O(8)-C(2)-C(1)	-95.1(2)
O(1)-C(1)-C(2)-O(8)	-118.1(2)
C(5)-C(1)-C(2)-O(8)	123.8(2)
O(1)-C(1)-C(2)-C(3)	-2.9(2)
C(5)-C(1)-C(2)-C(3)	-121.0(2)
O(8)-C(2)-C(3)-C(20)	-143.6(2)
C(1)-C(2)-C(3)-C(20)	99.7(2)
O(8)-C(2)-C(3)-C(4)	95.7(2)
C(1)-C(2)-C(3)-C(4)	-21.0(2)
C(1)-O(1)-C(4)-C(21)	-167.6(2)
C(1)-O(1)-C(4)-C(3)	-41.8(2)
C(20)-C(3)-C(4)-O(1)	-80.8(2)
C(2)-C(3)-C(4)-O(1)	38.0(2)
C(20)-C(3)-C(4)-C(21)	39.8(3)
C(2)-C(3)-C(4)-C(21)	158.60(19)
C(6)-O(2)-C(5)-C(1)	175.1(2)
O(1)-C(1)-C(5)-O(2)	161.21(19)
C(2)-C(1)-C(5)-O(2)	-81.0(3)
C(5)-O(2)-C(6)-O(3)	0.5(3)
C(5)-O(2)-C(6)-C(7)	179.9(2)
O(3)-C(6)-C(7)-C(8)	5.2(4)
O(2)-C(6)-C(7)-C(8)	-174.2(2)
O(3)-C(6)-C(7)-C(12)	-174.6(2)
O(2)-C(6)-C(7)-C(12)	6.0(3)
C(12)-C(7)-C(8)-C(9)	-1.7(3)

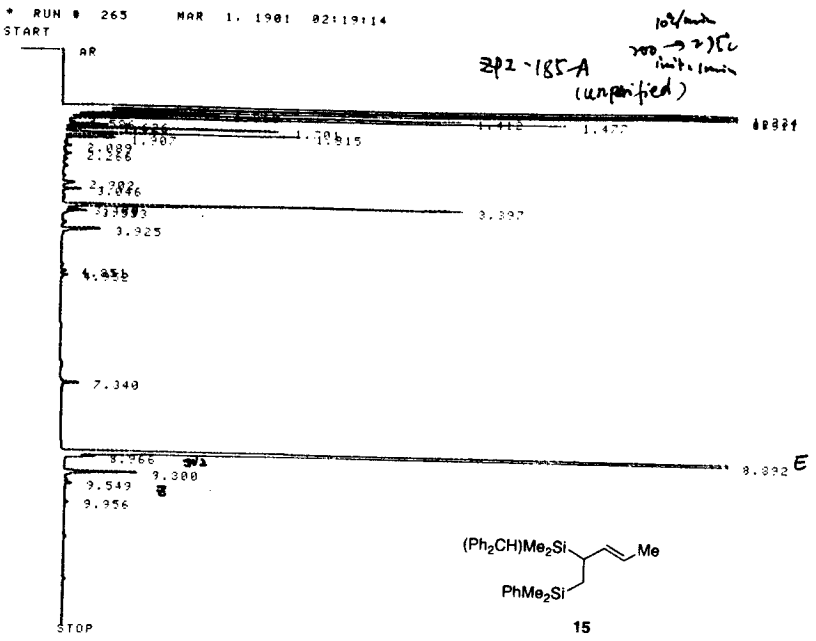
C(6)-C(7)-C(8)-C(9)	178.5(2)
C(7)-C(8)-C(9)-C(10)	1.2(4)
C(7)-C(8)-C(9)-N(1)	-178.6(2)
O(5)-N(1)-C(9)-C(8)	158.0(2)
O(4)-N(1)-C(9)-C(8)	-21.3(3)
O(5)-N(1)-C(9)-C(10)	-21.8(3)
O(4)-N(1)-C(9)-C(10)	158.9(2)
C(8)-C(9)-C(10)-C(11)	0.0(4)
N(1)-C(9)-C(10)-C(11)	179.7(2)
C(9)-C(10)-C(11)-C(12)	-0.6(4)
C(9)-C(10)-C(11)-N(2)	179.5(2)
O(6)-N(2)-C(11)-C(10)	-12.2(3)
O(7)-N(2)-C(11)-C(10)	166.9(2)
O(6)-N(2)-C(11)-C(12)	167.9(2)
O(7)-N(2)-C(11)-C(12)	-13.1(3)
C(10)-C(11)-C(12)-C(7)	0.1(4)
N(2)-C(11)-C(12)-C(7)	180.0(2)
C(8)-C(7)-C(12)-C(11)	1.1(3)
C(6)-C(7)-C(12)-C(11)	-179.1(2)
C(2)-O(8)-C(13)-O(9)	2.1(3)
C(2)-O(8)-C(13)-C(14)	-176.79(18)
O(9)-C(13)-C(14)-C(15)	11.5(3)
O(8)-C(13)-C(14)-C(15)	-169.5(2)
O(9)-C(13)-C(14)-C(19)	-166.9(2)
O(8)-C(13)-C(14)-C(19)	12.1(3)
C(19)-C(14)-C(15)-C(16)	-2.2(3)
C(13)-C(14)-C(15)-C(16)	179.4(2)
C(14)-C(15)-C(16)-C(17)	0.8(4)
C(14)-C(15)-C(16)-N(3)	-179.4(2)
O(11)-N(3)-C(16)-C(15)	175.9(2)
O(10)-N(3)-C(16)-C(15)	-4.8(3)
O(11)-N(3)-C(16)-C(17)	-4.3(3)
O(10)-N(3)-C(16)-C(17)	175.0(2)
C(15)-C(16)-C(17)-C(18)	2.2(4)
N(3)-C(16)-C(17)-C(18)	-177.6(2)
C(16)-C(17)-C(18)-C(19)	-4.0(4)
C(16)-C(17)-C(18)-N(4)	177.9(2)
O(13)-N(4)-C(18)-C(17)	-158.0(2)
O(12)-N(4)-C(18)-C(17)	21.2(3)
O(13)-N(4)-C(18)-C(19)	23.8(3)
O(12)-N(4)-C(18)-C(19)	-157.0(2)
C(17)-C(18)-C(19)-C(14)	2.8(4)
N(4)-C(18)-C(19)-C(14)	-179.2(2)
C(15)-C(14)-C(19)-C(18)	0.5(3)
C(13)-C(14)-C(19)-C(18)	178.9(2)
O(1)-C(4)-C(21)-C(22)	-176.2(2)
C(3)-C(4)-C(21)-C(22)	66.6(3)
C(4)-C(21)-C(22)-C(23)	176.3(2)
C(21)-C(22)-C(23)-C(24)	25.2(4)
C(21)-C(22)-C(23)-C(28)	-156.0(2)
C(28)-C(23)-C(24)-C(25)	0.6(4)
C(22)-C(23)-C(24)-C(25)	179.5(2)
C(23)-C(24)-C(25)-C(26)	0.4(4)
C(24)-C(25)-C(26)-C(27)	-1.2(4)

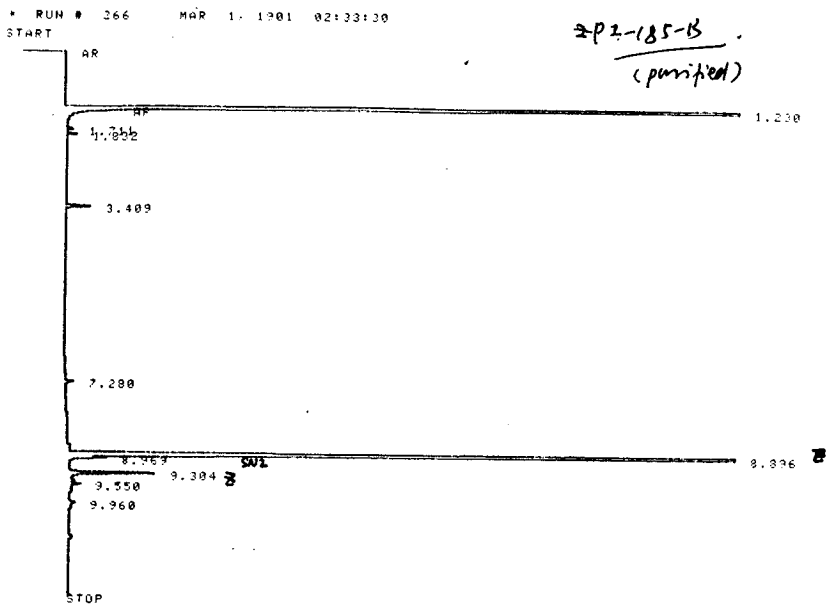
C(25)-C(26)-C(27)-C(28)	0.9(4)
C(26)-C(27)-C(28)-C(23)	0.1(4)
C(24)-C(23)-C(28)-C(27)	-0.9(4)
C(22)-C(23)-C(28)-C(27)	-179.7(2)


Integral
ppm
10.729
1.000
1.048
3.072
12.213
2.214
6.593
7.2521
7.2438
7.2406
7.1349
7.1264
3.5224
2.0149
1.6366
1.6042
1.4279
1.3241
1.3139
1.3080
1.2988
0.0536
0.0454
0.0006
Me
O
H
SiMe2(CHPh2)
3


Integral
ppm
15.589
2.015
0.995
1.005
1.000
2.004
1.120
3.065
6.365
7.3961
7.2880
7.2200
7.2060
7.1988
7.1933
4.1302
4.1160
4.1018
4.0876
3.8833
3.6490
3.6251
3.6082
3.4945
2.3191
2.2936
2.2681
2.2293
2.2145
1.6313
1.3484
1.3393
1.3231
1.1766
1.1623
1.1481
0.0788
0.0727
0.0665
0.0001
(Ph2CH)Me2Si
O
Ph
CO2Et
6


Integral
ppm
16.527
2.054
1.000
4.539
2.177
13.431
7.4270
7.4233
7.3354
7.3233
7.3147
7.2176
5.1211
5.1039
5.0945
5.0833
3.7194
1.7059
1.6403
1.6295
1.5479
0.8372
0.8290
0.8051
0.2571
0.2508
0.1929
0.0979
0.0707
PhMe2Si
(Ph2CH)Me2Si
Me
15





RUN# 265 MAR 1. 1901 02:19:14

AREA%

RT	AREA	TYPE	WIDTH	AREA%	
1.311	121535	TBV	.019	30.57948	
1.360	3671	TVP	.020	.92321	
1.412	10253	TPB	.019	2.57849	
1.477	12520	BB	.017	3.14861	
1.586	290	BV	.027	.07293	
1.626	1288	VP	.021	.32391	
1.701	7247	PB	.024	1.82252	
1.815	7727	PV	.023	1.94323	
1.907	1694	VV	.023	.42602	
2.089	437	VP	.041	.10990	
2.266	380	VP	.034	.09556	
2.902	677	PB	.040	.17026	
3.046	811	BB	.031	.20396	
3.397	18821	PV	.033	4.73322	
3.480	707	VV	.032	.17780	
3.533	1322	VB	.037	.33246	
3.925	2572	PB	.047	.64682	
4.851	322	VV	.039	.08098	
4.952	458	VB	.044	.11518	
7.340	993	VB	.039	.24973	
8.892	196902	PV	.036	49.51814	E
8.966	1594	VB	.035	.40087	α
9.300	4461	BB	.042	1.12188	Z
9.549	512	VV	.043	.12876	
9.956	382	PV	.049	.09607	

TOTAL AREA= 397636
MUL FACTOR=1.0000E+00

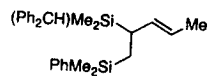
E/Z = 98/2
γ/α > 99/1

RUN# 266 MAR 1. 1901 02:33:30

AREA%

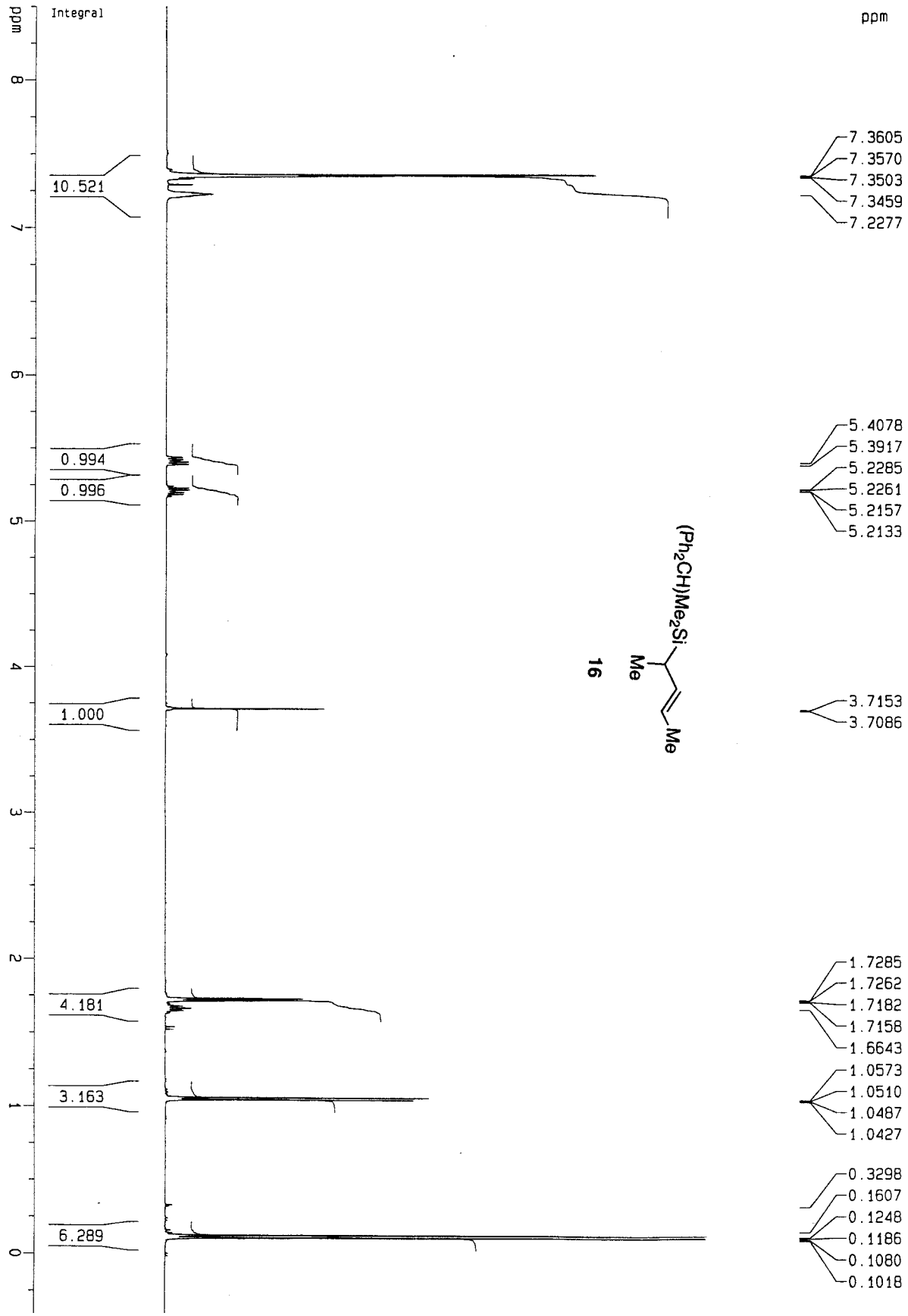
RT	AREA	TYPE	WIDTH	AREA%	
1.711	347	BB	.033	.14612	
1.832	499	BB	.031	.21012	
3.409	1455	BB	.040	.61268	
7.280	374	PB	.035	.15749	
8.896	226241	PV	.038	95.26739	E
8.969	1949	VB	.036	.82070	α
9.304	5419	PV	.044	2.28188	Z
9.550	721	VV	.046	.30360	
9.960	475	VV	.059	.20002	

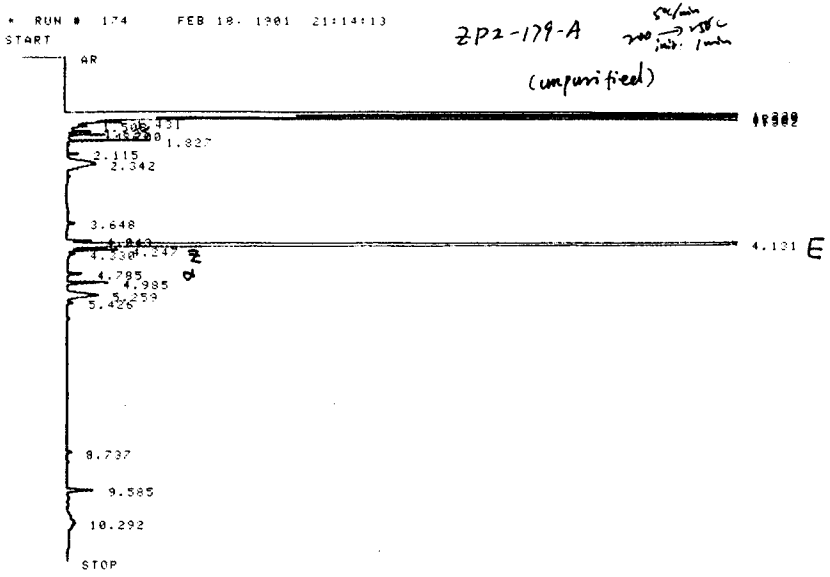
TOTAL AREA= 237480
MUL FACTOR=1.0000E+00



15

E/Z = 98/2
SN2'/SN2 > 99/1


Integral
ppm
10.521
0.994
0.996
1.000
4.181
3.163
6.289
7.3605
7.3570
7.3503
7.3459
7.2277
5.4078
5.3917
5.2285
5.2261
5.2157
5.2133
3.7153
3.7086
1.7285
1.7262
1.7182
1.7158
1.6643
1.0573
1.0510
1.0487
1.0427
0.3298
0.1607
0.1248
0.1186
0.1080
0.1018
(Ph2CH)Me2Si
Me
Me
16



ZP2-179-A (unpurified)

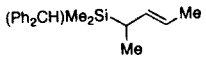
5%/min 200 → 250°C init. 1 min

RUN# 174 FEB 18, 1901 21:14:13

AREA%

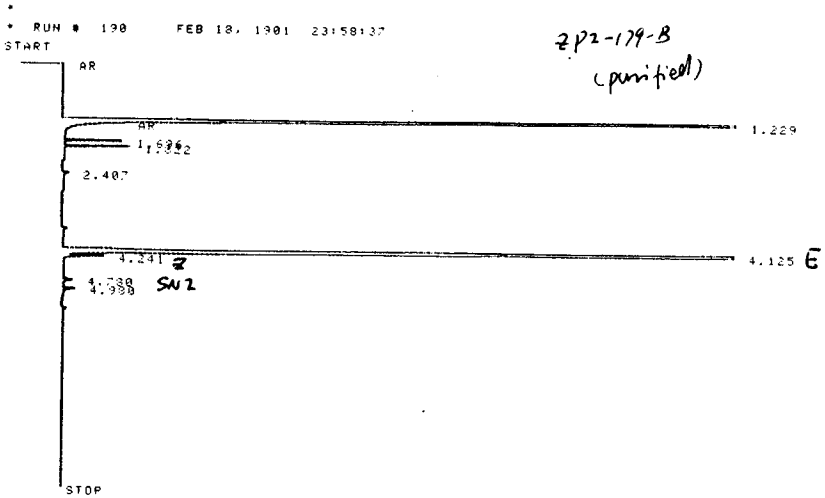
RT	AREA	TYPE	WIDTH	AREA%	
1.309	68249	TBP	.013	17.89593	
1.362	23929	TPP	.013	6.27455	
1.431	562	TPV	.013	.14736	
1.508	364	BP	.025	.09545	
1.625	908	PP	.020	.23733	
1.700	1065	PV	.020	.27926	
1.827	2587	VB	.021	.67835	
2.115	495	BP	.023	.12980	
2.342	5911	VV	.140	1.54996	
3.648	633	PB	.050	.16598	
4.043	748	PP	.025	.19614	
4.131	260558	PB	.034	68.32230	E
4.247	1981	BP	.020	.51945	Z
4.330	383	PP	.038	.10043	
4.785	899	PV	.041	.23573	X
4.985	2292	VB	.038	.60100	
5.259	4755	PV	.105	1.24683	
5.426	410	VB	.043	.10751	
8.737	391	BB	.046	.10253	
9.585	2022	PV	.053	.53020	
10.292	2532	BP	.343	.66393	

TOTAL AREA= 381366
MUL FACTOR=1.0000E+00



16

E/Z >99/1
SN2'/SN2 >99/1



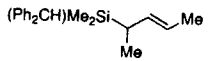
ZP2-179-B (purified)

RUN# 190 FEB 18, 1901 23:58:37

AREA%

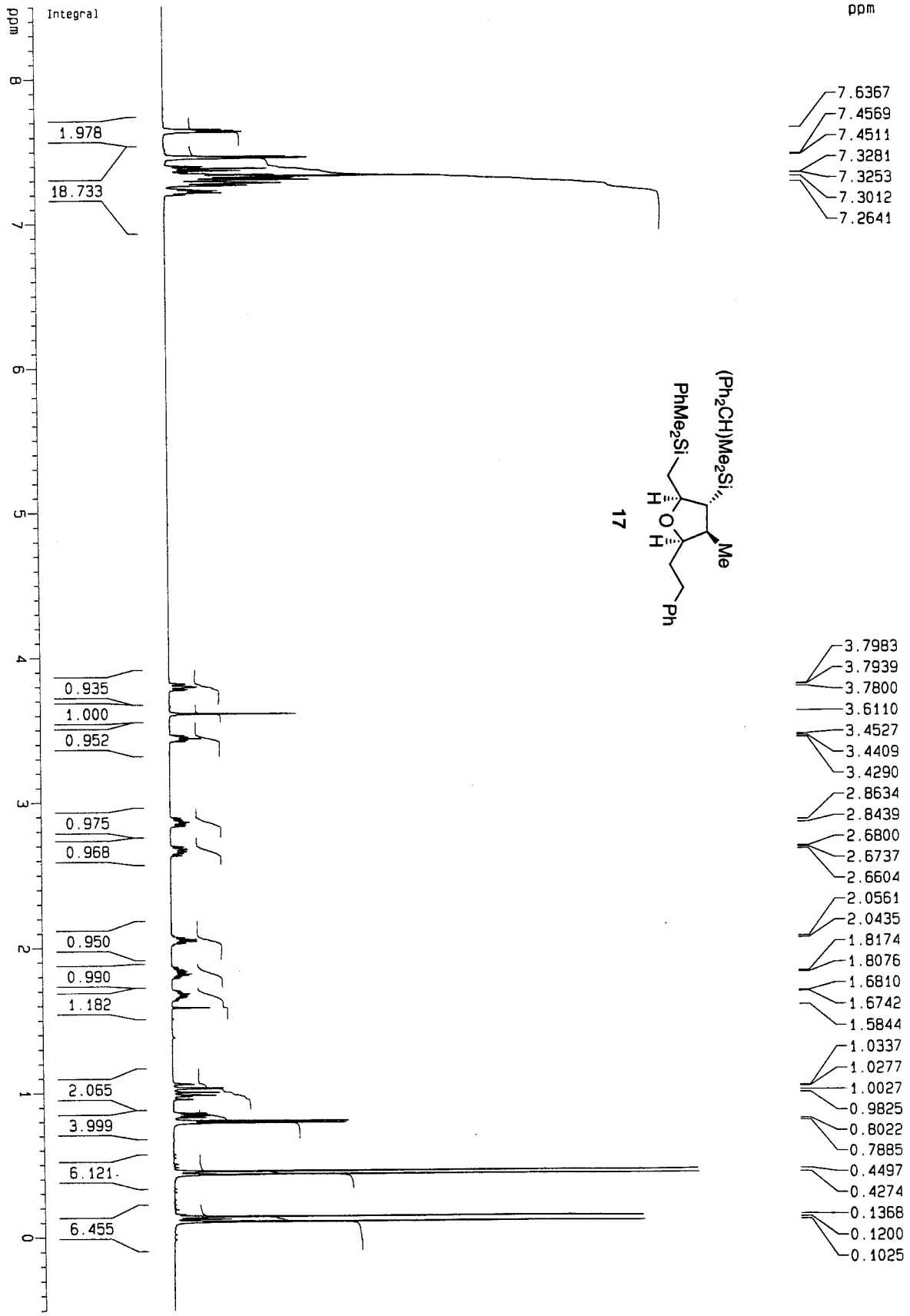
RT	AREA	TYPE	WIDTH	AREA%	
1.696	1505	PB	.018	.54209	
1.822	1831	BB	.013	.65951	
2.407	249	PB	.028	.08969	
4.125	271123	PB	.037	97.65587	E
4.241	1419	BB	.028	.51111	Z
4.730	694	BP	.047	.24997	SN2
4.980	810	PB	.043	.29175	

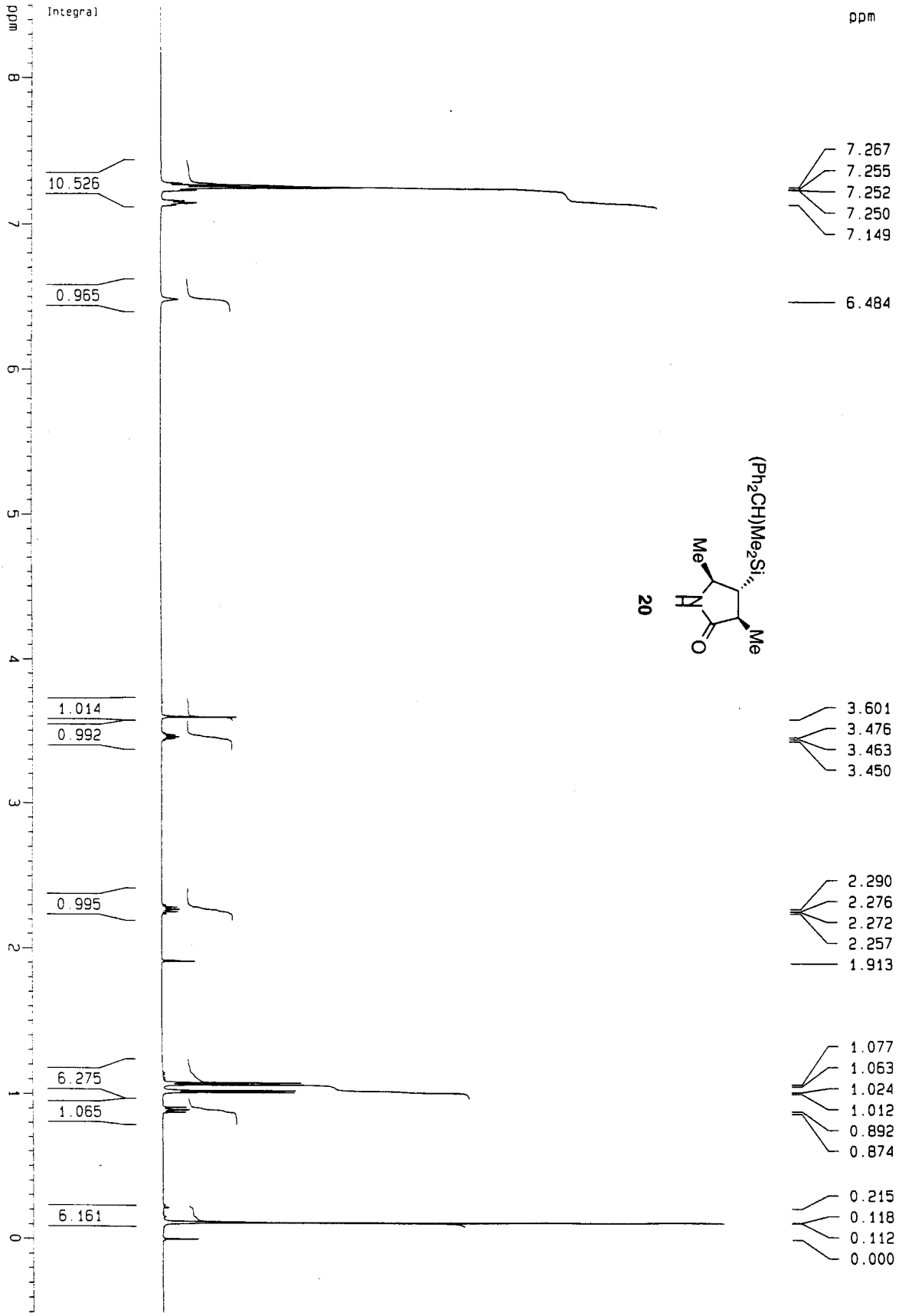
TOTAL AREA= 277631
MUL FACTOR=1.0000E+00



16

E/Z >99/1
SN2'/SN2 >99/1


ppm
Integral
1.978
18.733
0.935
1.000
0.952
0.975
0.968
0.950
0.990
1.182
2.065
3.999
6.121
6.455
7.6367
7.4569
7.4511
7.3281
7.3253
7.3012
7.2641
3.7983
3.7939
3.7800
3.6110
3.4527
3.4409
3.4290
2.8634
2.8439
2.6800
2.6737
2.6604
2.0561
2.0435
1.8174
1.8076
1.6810
1.6742
1.5844
1.0337
1.0277
1.0027
0.9825
0.8022
0.7885
0.4497
0.4274
0.1368
0.1200
0.1025
(Ph2CH)Me2Si
PhMe2Si
Me
Ph
H
O
H
17


Integral
ppm
10.526
0.965
1.014
0.992
0.995
6.275
1.065
6.161
7.267
7.255
7.252
7.250
7.149
6.484
3.601
3.476
3.463
3.450
2.290
2.276
2.272
2.257
1.913
1.077
1.063
1.024
1.012
0.892
0.874
0.215
0.118
0.112
0.000
(Ph2CH)Me2Si
Me
Me
N
H
O
20