Synthesis of ($\pm$)-5-*epi*-Citreo viral and ($\pm$)-Citreo viral and the Kinetic Resolution of an Allylic Silane by a [3 + 2] Annulation Reaction

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Supporting Information

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General. ^1H NMR and ^{13}C NMR spectra were recorded at ambient temperature at 500 MHz and 125 MHz, respectively, using a Bruker DRX 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. High resolution mass spectra were acquired on a VG Analytical 7070E or Fisons Autospec spectrometer, and were obtained by peak matching. Microanalyses were performed by Atlantic Microlab, Atlanta, GA. 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). Enantiomeric excesses were determined by HPLC analysis on a Hewlett Packard series 1100 using a Chiralcel OJ column, and the enantiomerically enriched material was compared with racemic material. 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, Et_2O , and CH_2Cl_2 were dried by filtration through alumina according to the procedure of Grubbs.¹

I. Synthesis of ($\pm$)-5-*epi*-citreo viral **16**

Acetic acid 2-methyl-3-(dimethylphenylsilyl)-(E)-but-1-enyl ester (6). To a solution of PhMe_2SiCl (8.40 mL, 50.0 mmol) in 50 mL of dry THF was added lithium wire (2.10 g, 300 mmol). After 16 h at 23 °C, the resultant dark red solution was added to a cooled (–30 °C) suspension of CuI (4.50 g, 25.0 mmol) in 10 mL of THF by cannula. After 4 h at –30 °C, the slurry was cooled to –78 °C, followed by the addition of aldehyde **8** (2.40 mL, 25.0 mmol) dropwise by syringe. The reaction mixture was stirred at –78 °C for 1 h and then warmed to –30 °C for an additional 2 h. Ac_2O (9.40 mL, 100 mmol) was then added and the reaction mixture was allowed to warm to 23 °C, and after 12 h, 100 mL of Et_2O was added. The mixture was washed sequentially with 60 mL of 1 N HCl, 30 mL of saturated NaHCO_3 , and 30 mL of brine. The organic layer was dried

¹ Pangborn, A. B.; Giardello, M. A.; Grubbs, R. H.; Rosen, R. K.; Timmers, F. J. *Organometallics* **1996**, 15, 1518–1520.

(MgSO₄), filtered, and concentrated *in vacuo*. Purification by flash chromatography (benzene) afforded **6** as a colorless oil (5.80 g, 88%): ¹H NMR (CDCl₃, 500 MHz) δ 7.57 (m, 2H), 7.42 (m, 3H), 6.87 (d, *J* = 1.3 Hz, 1H), 2.19 (s, 3H), 1.82 (q, *J* = 7.5 Hz, 1H), 1.60 (d, *J* = 1.4 Hz, 3H), 1.18 (d, *J* = 7.5 Hz, 3H), 0.40 (s, 3H), 0.37 (s, 3H); ¹³C NMR (CDCl₃, 125 MHz) δ 168.2, 138.0, 133.9, 129.3, 129.0, 127.7, 125.2, 26.8, 20.8, 14.3, 14.1, -4.0, -4.7; IR (thin film) 1745, 1227 cm⁻¹; HRMS (CI/NH₃) *m/z* calcd for C₁₅H₂₂O₂Si (M⁺) 262.1389, found 262.1387. Anal. Calcd for C₁₅H₂₂O₂Si: C, 68.67; H, 8.46. Found: C, 68.78; H, 8.42.

(2R*,3S*,4S*,5S*)-3-Acetoxy-4-dimethylphenylsilyl-2,4,5-trimethyltetrahydrofuran-2-carboxylic acid ethyl ester (9). To a cooled (-78 °C) solution of ethyl pyruvate (0.400 mL, 3.60 mmol) and allylsilane **6** (0.870 g, 3.30 mmol) in 12 mL of dry CH₂Cl₂ was added dropwise SnCl₄ (0.200 M solution in CH₂Cl₂, 18.0 mL, 3.60 mmol) by syringe. After 1 h, 0.5 mL of Et₃N was added followed by 10 mL of water. The mixture was warmed to 23 °C and the layers were separated. The aqueous layer was extracted with 3 × 20 mL of EtOAc. The combined organic layers were washed with 10 mL of brine, dried (MgSO₄), filtered, and concentrated *in vacuo*. Purification by flash chromatography (5:95 to 10:90 EtOAc/hexanes) afforded a colorless oil **9** (1.06 g, 85%) as a single stereoisomer as determined by ¹H NMR spectroscopy: ¹H NMR (CDCl₃, 500 MHz) δ 7.51 (m, 2H), 7.38 (m, 3H), 5.66 (s, 1H), 4.23 (q, *J* = 6.4 Hz, 1H), 4.10 (m, 2H), 1.86 (s, 3H), 1.27 (s, 3H), 1.24 (t, *J* = 7.1 Hz, 3H), 1.14 (d, *J* = 6.3 Hz, 3H), 1.08 (s, 3H), 0.39 (s, 3H), 0.38 (s, 3H); ¹³C NMR (CDCl₃, 125 MHz) δ 173.5, 169.6, 135.6, 134.2, 129.3, 127.7, 82.0, 79.7, 77.7, 60.9, 35.5, 20.2, 19.7, 15.4, 13.9, 9.5, -5.6, -5.8; IR (thin film) 1742 cm⁻¹; HRMS (CI/NH₃) *m/z* calcd for C₂₀H₂₉O₅Si, (M - H)⁺ 377.1784, found 378.1787. Anal. Calcd for C₂₀H₃₀O₅Si: C, 63.46; H, 7.99. Found: C, 63.17; H, 7.96.

(2S*,3S*,4S*,5S*)-4-Dimethylphenylsilyl-3-hydroxy-2-hydroxymethyl-2,4,5-trimethyltetrahydrofuran (10). To a cooled (0 °C) suspension of LiAlH₄ (0.433 g, 11.4 mmol) in 30 mL of Et₂O was added a solution of **9** (2.15 g, 5.69 mmol) in 40 mL of Et₂O. After 10 h at 23 °C, the excess hydride was carefully quenched by 2 g of Na₂SO₄·10H₂O. The mixture was filtered and the solid was washed with 3 × 20 mL of EtOAc. The combined filtrate was concentrated *in vacuo*. Purification by flash chromatography (30:70 to 50:50 EtOAc/hexanes) afforded **10** as a white solid (1.52 g, 91%): mp 98–99 °C; ¹H NMR (CDCl₃, 500 MHz) δ 7.52 (m, 2H), 7.38 (m, 3H), 4.17 (d, *J* = 6.2 Hz, 1H), 3.88 (q, *J* = 6.3 Hz, 1H), 3.31 (m, 2H), 2.15 (t, *J* = 6.3 Hz, 1H), 1.36 (d, *J* = 6.2 Hz, 1H), 1.13 (s, 3H), 1.08 (d, *J* = 6.3 Hz, 3H), 1.02 (s, 3H), 0.38 (s, 3H), 0.37 (s, 3H); ¹³C NMR (CDCl₃, 125 MHz) δ 136.7, 134.6, 130.0, 128.6, 82.4, 78.6, 76.4, 68.8, 37.6, 20.1, 16.5, 9.1, -4.9, -5.1; IR (KBr) 3350 cm⁻¹; HRMS (CI/NH₃) *m/z* calcd for C₁₆H₂₅O₃Si, (M - H)⁺ 293.1673, found 293.1673. Anal. Calcd for C₁₆H₂₆O₃Si: C, 65.27; H, 8.91. Found: C, 65.41; H, 8.92.

(2S*,3S*,4S*,5S*)-3-Benzyloxy-2-benzyloxymethyl-4-dimethylphenylsilyl-2,4,5-trimethyltetrahydrofuran (11). To a suspension of NaH (1.00 g, 60% oil dispersion, 25.0 mmol, washed with 3 × 10 mL of hexanes) in 30 mL of dry DMF was added a solution of diol **10** (1.47 g, 5.00 mmol) in 30 mL of DMF. After 0.5 h at 23 °C, BnBr (3.00 mL, 25.0 mmol) and *n*-Bu₄NI (0.0900 g, 0.250 mmol) were added, and the reaction mixture was stirred for 12 h at 70 °C. The mixture was cooled to 23 °C, and 60 mL of saturated NH₄Cl was added. The mixture was extracted with 3 × 60 mL of Et₂O, and the combined organic layers were washed with brine (30 mL), dried (MgSO₄), and concentrated *in vacuo*. Purification by flash chromatography (5:95 to 10:90 EtOAc/hexanes) afforded **11** as a colorless oil (2.06 g, 87%): ¹H NMR (CDCl₃, 500 MHz) δ 7.48-7.18 (m, 15H), 4.54 (d, *J* = 11.6 Hz, 1H), 4.46 (d, *J* = 12.0 Hz, 1H), 4.42 (d, *J* = 12.0 Hz, 1H), 4.18 (d, *J* = 11.6 Hz, 1H), 4.02 (s, 1H), 3.87 (q, *J* = 6.3 Hz, 1H), 3.21 (d, *J* = 9.1 Hz, 1H), 3.12 (d, *J* = 9.1 Hz, 1H), 1.22 (s, 3H), 1.09 (s, 3H), 1.00 (d, *J* = 6.3 Hz, 3H), 0.31 (s, 3H), 0.30 (s, 3H); ¹³C NMR (CDCl₃, 125 MHz) δ 138.8, 138.3, 136.8, 134.4, 129.1, 128.3, 128.1, 127.7, 127.6, 127.5, 127.1, 86.3, 81.3, 76.2, 75.5, 73.4, 72.0, 37.5, 20.7, 15.7, 9.3,

−4.9, −5.2; IR (thin film) 2858, 1454 cm^{−1}; HRMS (CI/NH₃) *m/z* calcd for C₂₃H₃₁O₂Si (M - BnO) 367.1729, found 367.1722. Anal. Calcd for C₃₀H₃₈O₃Si: C, 75.91; H, 8.08. Found: C, 75.73; H, 8.06.

(2S*,3S*,4S*,5S*)-3-Benzyloxy-2-benzyloxymethyl-4-hydroxy-2,4,5-trimethyltetrahydrofuran (12) and (2S*,3S*,4R*,5S*)-3-Benzyloxy-2-benzyloxymethyl-2,4,5-trimethyltetrahydrofuran (13). To a suspension of KH (0.144 g, 3.60 mmol) in 5 mL of NMP was added cumene hydroperoxide (0.490 mL, 88.0% solution, 2.90 mmol). After 20 min, silane **11** (0.340 g, 0.720 mmol) in 5 mL of NMP was added dropwise followed by TBAF (1.44 mL, 1 M solution in THF, 1.44 mmol). After 24 h at 23 °C, 10 mL of saturated Na₂S₂O₃ was added, and the mixture was stirred for 30 min at 23 °C. The mixture was then extracted with 5 × 40 mL of *t*-BuOMe. The combined organic layers were washed with brine (30 mL), dried (MgSO₄), and concentrated *in vacuo*. Purification by flash chromatography (5:95 to 30:70 EtOAc/hexanes) afforded **12** (0.216 g, 85%) and **13** (0.015 g, 6%) as colorless oils. **12**: ¹H NMR (CDCl₃, 500 MHz) δ 7.31 (m, 10H), 4.63 (d, *J* = 12.0 Hz, 1H), 4.59 (m, 2H), 4.52 (d, *J* = 12.0 Hz, 1H), 3.89 (s, 1H), 3.86 (q, *J* = 6.7 Hz, 1H), 3.41 (d, *J* = 9.6 Hz, 1H), 3.35 (d, *J* = 9.6 Hz, 1H), 2.58 (s, 1H), 1.25 (s, 3H), 1.19 (s, 3H), 1.14 (d, *J* = 6.5 Hz, 3H); ¹³C NMR (CDCl₃, 125 MHz) δ 138.6, 137.9, 128.5, 128.4, 127.8, 127.7, 127.6, 127.5, 88.9, 82.1, 81.1, 79.7, 76.9, 73.9, 73.5, 20.8, 17.2, 15.7; IR (thin film) 3440, 1454 cm^{−1}; HRMS (CI/NH₃) *m/z* calcd for C₂₂H₂₉O₄ (M + H)⁺ 357.2067, found 357.2062. Anal. Calcd for C₂₂H₂₈O₄: C, 74.12; H, 7.92. Found: C, 73.88; H, 7.99. **13**: ¹H NMR (CDCl₃, 500 MHz) δ 7.31 (m, 10H), 4.52 (m, 4H), 4.05 (m, 1H), 4.01 (d, *J* = 6.5 Hz, 1H), 3.32 (d, *J* = 9.5 Hz, 1H), 3.30 (d, *J* = 9.5 Hz, 1H), 2.32 (m, 1H), 1.28 (s, 3H), 1.17 (d, *J* = 6.4 Hz, 3H), 0.96 (d, *J* = 7.2 Hz, 3H); ¹³C NMR (CDCl₃, 125 MHz) δ 138.7, 138.4, 128.22, 128.17, 127.43, 127.39, 127.3, 127.2, 83.5, 82.7, 76.5, 74.8, 73.4, 72.4, 40.1, 20.7, 16.6, 8.1; IR (thin film) 2977, 1454, 1087 cm^{−1}; HRMS (EI) *m/z* calcd for C₂₂H₂₈O₃ (M⁺) 340.2038, found 340.2026. Anal. Calcd for C₂₂H₂₈O₃: C, 77.60; H, 8.29. Found: C, 77.78; H, 8.36.

(2S*,3S*,4S*,5S*)-3-Benzyloxy-4-hydroxy-2-hydroxymethyl-2,4,5-trimethyltetrahydrofuran (28). Dibenzy ether **12** (0.235 g, 0.660 mmol) was dissolved in 5 mL of EtOH, 10% Pd-C (0.130 g) was added, and

the mixture was hydrogenated with H_2 supplied by a balloon (1 atm). The progress of the reaction was monitored by TLC. After 2 h, the reaction mixture was filtered, and the filtrate was concentrated *in vacuo*. Purification by flash chromatography (50:50 to 80:20 EtOAc/hexanes) afforded **28** as a colorless oil (0.163 g, 93%): 1H NMR ($CDCl_3$, 500 MHz) δ 7.34 (m, 4H), 7.30 (m, 1H), 4.71 (d, J = 12.0 Hz, 1H), 4.61 (d, J = 12.0 Hz, 1H), 3.82 (q, J = 6.5 Hz, 1H and s, 1H), 3.48 (d, J = 11.5 Hz, 1H), 3.45 (d, J = 11.5 Hz, 1H), 2.96 (br, 2H), 1.26 (s, 3H), 1.17 (s, 3H), 1.15 (d, J = 6.5 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 138.0, 128.2, 127.5, 127.2,

88.0, 82.4, 80.7, 79.5, 73.3, 69.0, 19.7, 16.9, 15.2; IR (thin film) 3406, 1454 cm^{-1} ; HRMS (CI/ NH_3) m/z calcd for $C_{15}H_{22}O_4$ ($M + H$)⁺ 267.1596, found 267.1603. Anal. Calcd for $C_{15}H_{22}O_4$: C, 67.63; H, 8.33. Found: C, 67.40; H, 8.25.

(1S*,4R*,6S*,7R*)-7-Benzyloxy-1,4,6-trimethyl-2,5-dioxabicyclo[2.2.1]heptan-3-one (14). To a solution of diol **28** (0.332 g, 1.25 mmol) in 10 mL of CH_2Cl_2 was added powered 4Å molecular sieves (0.600 g), NMO (0.293 g, 2.50 mmol), and TPAP (0.0660 g, 0.190 mmol) at 0 °C. The reaction mixture was allowed to warm to 23 °C over the course of 1.5 h, and was then diluted with 20 mL of CH_2Cl_2 . The reaction mixture was filtered through a plug of silica gel, which was washed with 40 mL of CH_2Cl_2 . The filtrate was concentrated *in vacuo*. Purification by flash chromatography (5:95 to 10:90 EtOAc/hexanes) afforded **14** as a colorless oil (0.288 g, 88%): 1H NMR ($CDCl_3$, 500 MHz) δ 7.35 (m, 5H), 4.72 (d, J = 11.9 Hz, 1H), 4.64 (d, J = 11.9 Hz, 1H), 3.86 (q, J = 6.8 Hz, 1H), 3.44 (s, 1H), 1.49 (s, 3H), 1.48 (s, 3H), 1.38 (d, J = 6.8 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 171.7, 138.8, 128.6, 128.3, 127.8, 89.7, 85.9, 82.7, 80.6, 73.8, 15.2, 12.7, 11.2; IR (thin film) 1790, 1455, 1116 cm^{-1} ; HRMS (CI/ NH_3) m/z calcd for $C_{15}H_{18}O_4$ (M^+) 262.1205, found 262.1200. Anal. Calcd for $C_{15}H_{18}O_4$: C, 68.67; H, 6.92. Found: C, 68.96; H, 6.94.

(2S*,3S*,4S*,5S*)-3-Benzyloxy-2-(2-carboethoxy-(*E*)-propenyl)-4-hydroxy-2,4,5-trimethyltetrahydrofuran (15). *i*-Bu₂AlH (0.19 mL, 1.5 M solution in toluene, 0.29 mmol) was added dropwise to a solution of lactone **14** (0.156 g, 0.600 mmol) in 5 mL of CH_2Cl_2 at -78 °C. After 1.5 h at -78 °C, 3 mL of HCl solution (1 N in MeOH) was added, and the mixture was allowed to warm to 23 °C. After diluting with 30 mL of CH_2Cl_2 , the solution was washed with 30 mL of 1 N HCl and the aqueous layer was extracted with 3 × 30 mL of CH_2Cl_2 . The combined organic layers were washed with saturated $NaHCO_3$ (30 mL), brine (20 mL), dried ($MgSO_4$), and concentrated *in vacuo*. The crude lactol was dissolved in 5 mL of dry benzene, and (carboethoxyethylidene)triphenylphosphorane (0.325 g, 0.900 mmol) was added. The solution was heated at reflux for 10 h. Following dilution with 80 mL of Et_2O , the reaction mixture was washed with 20 mL of saturated $NaHCO_3$ and 20 mL of brine. The organic layer was dried ($MgSO_4$) and concentrated *in vacuo*. Purification by flash chromatography (20:80 EtOAc/hexanes) afforded **15** as a colorless oil (0.186 g, 89%): 1H NMR ($CDCl_3$, 500 MHz) δ 7.37 (m, 4H), 7.30 (m, 1H), 6.87 (d, J = 1.4 Hz, 1H), 4.82 (d, J = 11.8 Hz, 1H), 4.62 (d, J = 11.8 Hz, 1H), 4.20 (m, 2H), 3.89 (s, 1H), 3.68 (q, J = 6.4 Hz, 1H), 1.94 (d, J = 1.4 Hz, 3H), 1.29 (t, J = 7.1 Hz, 3H), 1.24 (s, 3H), 1.17 (d, J = 6.4 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 168.5, 148.4, 138.0, 128.4, 127.7, 127.5, 127.4, 92.2, 81.9, 80.3, 77.1, 72.7, 60.7, 21.7, 16.4, 14.2, 13.5, 12.4; IR (thin film) 3478, 1700,

1644 cm^{-1} ; HRMS (CI/ NH_3) m/z calcd for $\text{C}_{20}\text{H}_{28}\text{O}_5$ (M^+) 348.1937, found 348.1930. Anal. Calcd for $\text{C}_{20}\text{H}_{28}\text{O}_5$: C, 68.93; H, 8.10. Found: C, 69.12; H, 8.09.

(2S*,3S*,4S*,5S*)-2-(2-Carboethoxy-(E)-propenyl)-3,4-dihydroxy-2,4,5-trimethyltetrahydrofuran (29).

To a solution of benzyl ether **15** (0.220 g, 0.630 mmol) in 10 mL of 1,2-dichloroethane was added 1 mL of aqueous pH 7 buffer and DDQ (0.861 g, 3.80 mmol). The reaction mixture was allowed to stir for 12 h at 50 °C. After cooling to 23 °C, 20 mL of saturated NaHCO_3 was slowly added. The mixture was poured into 100 mL of EtOAc, and the layers were separated. The organic layer was washed with 30 mL of brine and the combined aqueous layers were extracted with 3×40 mL of EtOAc. The combined organic layers were dried (MgSO_4) and concentrated *in vacuo*. Purification by flash chromatography (30:70 to 50:50 EtOAc/hexanes) afforded **28** as a colorless oil (0.150 g, 92%): ^1H NMR (CDCl_3 , 500 MHz) δ 6.89 (d, $J = 1.4$ Hz, 1H), 4.19 (q, $J = 7.2$ Hz, 2H), 4.08 (d, $J = 5.8$ Hz, 1H), 3.69 (q, $J = 6.3$ Hz, 1H), 3.52 (d, $J = 5.9$ Hz, 1H), 3.13 (s, 1H), 1.98 (d, $J = 1.3$ Hz, 3H), 1.30 (t, $J = 7.2$ Hz, 3H), 1.28 (s, 3H), 1.17 (s, 3H), 1.16 (d, $J = 6.0$ Hz, 3H); ^{13}C NMR (CDCl_3 , 125 MHz) δ 169.0, 148.4, 127.3, 85.2, 82.1, 80.1, 77.3, 61.0, 21.1, 16.0, 14.1, 14.0, 12.6; IR (thin film) 3443, 1694, 1651 cm^{-1} ; HRMS (CI/ NH_3) m/z calcd for $\text{C}_{13}\text{H}_{23}\text{O}_5$ ($\text{M} + \text{H}^+$) 259.1545, found 259.1548. Anal. Calcd for $\text{C}_{13}\text{H}_{22}\text{O}_5$: C, 60.43; H, 8.59. Found: C, 60.14; H, 8.67.

(2S*,3S*,4S*,5S*)-3,4-Dihydroxy-2-(2-hydroxymethyl-(E)-propenyl)-2,4,5-trimethyltetrahydrofuran (30).

To a cooled (-78 °C) solution of **29** (0.120 g, 0.460 mmol) in CH_2Cl_2 (6 mL) was added a solution of *i*- Bu_2AlH (1.50 M in toluene, 1.38 mL, 2.10 mmol). The resulting solution was maintained at -78 °C for 1 h and then allowed to warm to 0 °C. After 0.5 h at 0 °C, a saturated aqueous solution of Rochelle's salt (10 mL) was added, and the mixture was stirred vigorously for 1 h. The resulting solution was extracted with 3×30 mL of EtOAc. The combined organic layers were washed with brine (10 mL), dried (MgSO_4), and concentrated *in vacuo*. Purification by flash chromatography (70:30 EtOAc/hexanes to EtOAc) afforded **30** as a colorless oil (0.078 g, 84%): ^1H NMR (CDCl_3 , 500 MHz) δ 5.70 (d, $J = 1.1$ Hz, 1H), 4.08 (d, $J = 6.3$ Hz, 1H), 4.01 (d, $J = 4.2$ Hz, 2H), 3.80 (q, $J = 6.4$ Hz, 1H), 2.80-2.30 (br, 3H), 1.86 (s, 3H), 1.33 (s, 3H), 1.24 (s, 3H), 1.22 (d, $J = 6.4$ Hz, 3H); ^{13}C NMR (CDCl_3 , 125 MHz) δ 135.4, 132.6, 86.5, 82.3, 81.0, 78.2, 68.7, 22.1, 16.4, 14.8, 14.0; IR (thin film) 3443, 2983 cm^{-1} ; HRMS (CI/ NH_3) m/z calcd for $\text{C}_{11}\text{H}_{19}\text{O}_3$ ($\text{M} - \text{OH}^+$) 199.1334, found 199.1344.

(2S*,3S*,4S*,5S*)-3,4-Dihydroxy-2-(2-formyl-(E)-propenyl)-2,4,5-trimethyltetrahydrofuran [(±)-5-epi-citreoviral] (16).

A mixture of **30** (0.040 g, 0.180 mmol), MnO_2 (0.300 g, 3.45 mmol), and CH_2Cl_2 (6 mL) was

stirred for 2 h at 23 °C. The reaction mixture was then filtered through Celite, and the Celite was washed with CH_2Cl_2 (3×10 mL) and EtOAc (3×10 mL). The combined filtrate was concentrated *in vacuo*. Purification by flash chromatography (40:60 to 60:40 EtOAc/hexanes) afforded **16** as a white solid (0.021 g, 55%): ^1H NMR (CDCl_3 , 500 MHz) δ 9.36 (s, 1H), 6.65 (d, $J = 1.2$ Hz, 1H), 4.11 (d, $J = 6.0$ Hz, 1H), 3.75 (q, $J = 6.4$ Hz, 1H), 2.78 (d, $J = 6.1$ Hz, 1H), 2.28 (s, 1H), 1.89 (d, $J = 1.2$ Hz, 3H), 1.34 (s, 3H), 1.23 (s, 3H), 1.20 (d, $J = 6.4$ Hz, 3H); ^{13}C NMR (CDCl_3 , 125 MHz) δ 195.9, 160.9, 137.8, 85.0, 82.5, 80.3, 78.1, 21.0, 16.5, 14.4, 9.4; IR (KBr) 3330, 1667, 1628 cm^{-1} ; HRMS (CI/ NH_3) m/z calcd for $\text{C}_{10}\text{H}_{17}\text{O}_3$ ($\text{M} - \text{CHO}$) $^+$ 185.1181, found 185.1177. Anal. Calcd for $\text{C}_{11}\text{H}_{18}\text{O}_4$: C, 61.65; H, 8.47. Found: C, 61.45; H, 8.49.

II. Syntheses of ($\pm$)-citreoiviral 1

2-Hydroxy-3-(triisopropylsilyloxy)-propionic acid ethyl ester (32). To diol **31**² (1.06 g, 7.90 mmol) in 20 mL of dry DMF was added imidazole (0.537 g, 7.90 mmol) and TIPSCl (1.70 mL, 7.90 mmol). After 12 h at 23 °C, 30 mL of saturated aqueous NH_4Cl was added and the solution was extracted with 3×50 mL of Et_2O . The combined organic layers were washed with brine (20 mL), dried (MgSO_4), and concentrated *in vacuo*. Purification by flash chromatography (4:96 EtOAc/hexanes) afforded **32** as a colorless oil (1.60 g, 70%): ^1H NMR (CDCl_3 , 500 MHz) δ 4.20 (m, 3H), 4.02 (m, 1H), 3.92 (m, 1H), 3.11 (d, $J = 8.4$ Hz, 1H), 1.26 (m, 3H), 1.02 (m, 21H); ^{13}C NMR (CDCl_3 , 125 MHz) δ 172.7, 72.0, 65.5, 61.4, 17.7, 14.1, 11.8; IR (thin film) 3541, 1746 cm^{-1} ; HRMS (FAB) m/z calcd for $\text{C}_{14}\text{H}_{31}\text{O}_4\text{Si}$ ($\text{M} + \text{H}$) $^+$ 291.1991, found 291.1989. Anal. Calcd for $\text{C}_{14}\text{H}_{30}\text{O}_4\text{Si}$: C, 57.89; H, 10.42. Found: C, 57.85; H, 10.40.

2-Oxo-3-(triisopropylsilyloxy)propionic acid ethyl ester (17). To a solution of **32** (1.87 g, 6.50 mmol) in 50 mL of CH_2Cl_2 was added Dess-Martin periodinane (5.50 g, 13.0 mmol). After stirring for 3 h at 23 °C, $\text{Na}_2\text{S}_2\text{O}_3$ (12 g) and saturated aqueous NaHCO_3 (100 mL) were added and the mixture was stirred for 0.5 h. The mixture was extracted with 3×60 mL of CH_2Cl_2 . The combined organic layers were washed with brine (20 mL), dried (MgSO_4), and concentrated *in vacuo*. Purification by bulb-to-bulb distillation afforded **17** as a colorless oil (1.70 g, 91%), which was unstable upon standing and was used in the next step immediately. **17**: ^1H NMR (CDCl_3 , 500 MHz) δ 4.76 (s, 2H), 4.30 (q, $J = 7.1$ Hz, 2H), 1.35 (t, $J = 7.1$ Hz, 3H), 1.13 (m, 3H), 1.06 (m, 18H); ^{13}C NMR (CDCl_3 , 125 MHz) δ 192.5, 161.2, 67.7, 62.2, 17.7, 13.9, 11.9; IR (thin film) 1757, 1731 cm^{-1} .

² Choi, D.; Stables, J. P.; Kohn, H. *Bioorg. Med. Chem.* **1996**, *4*, 2105-2114.

(2*S,3*R**,4*R**,5*R**)-3-Acetoxy-4,5-dimethyl-4-dimethylphenylsilyl-2-triisopropylsilyloxymethyl-tetrahydrofuran-2-carboxylic acid ethyl ester (18).** To a cooled (−78 °C) solution of allylsilane **6** (1.10 g, 4.20 mmol) and α -keto ester **17** (1.00 g, 3.50 mmol) in 10 mL of CH₂Cl₂ was added dropwise a solution of SnCl₄ (0.200 M in CH₂Cl₂, 19.0 mL, 3.80 mmol). After 2 h at −78 °C, 1 mL of Et₃N was added followed by 20 mL of water. The mixture was extracted with 3 × 50 mL of EtOAc. The combined organic layers were washed with brine (20 mL), dried (MgSO₄), and concentrated *in vacuo*. Purification by flash chromatography (2:98 to 5:95 EtOAc/hexanes) afforded **18** as a colorless oil (1.59 g, 83%): ¹H NMR (CDCl₃, 500 MHz) δ 7.48 (m, 2H), 7.35 (m, 3H), 5.85 (s, 1H), 4.08 (m, 3H), 3.91 (d, *J* = 10.2 Hz, 1H), 3.80 (d, *J* = 10.2 Hz, 1H), 1.89 (s, 3H), 1.19 (t, *J* = 7.1 Hz, 3H), 1.03 (m, 27H), 0.32 (s, 3H), 0.31 (s, 3H); ¹³C NMR (CDCl₃, 125 MHz) δ 171.9, 168.9, 135.4, 134.4, 129.3, 127.7, 85.7, 79.0, 78.0, 65.6, 60.9, 35.8, 20.5, 17.8, 15.4, 14.0, 11.9, 9.9, −5.5, −5.6; IR (thin film) 1755, 1464 cm^{−1}; HRMS (FAB) *m/z* calcd for C₂₉H₅₀O₆Si₂Na (M + Na)⁺ 573.3044, found 573.3046. Anal. Calcd for C₂₉H₅₀O₆Si₂: C, 63.24; H, 9.16. Found: C, 63.10; H, 9.20.

(2*S,3*R**,4*R**,5*R**)-4,5-Dimethyl-4-dimethylphenylsilyl-3-hydroxy-2-hydroxymethyl-2-triisopropylsilyloxymethyltetrahydrofuran (33).** To a suspension of LiBH₄ (1.50 g, 69.0 mmol) in 30 mL of Et₂O was added a solution of **18** (3.80 g, 6.90 mmol) in 30 mL of Et₂O dropwise by syringe. After 12 h at 23 °C, 30 mL of saturated aqueous NH₄Cl was carefully added to the reaction mixture followed by 30 mL of 1 N HCl. The layers were separated and the aqueous layer was extracted with 3 × 50 mL of Et₂O. The combined organic layers were washed with brine (30 mL), dried (MgSO₄), and concentrated *in vacuo*. Purification by flash chromatography (5:95 to 15:85 EtOAc/hexanes) afforded **33** as a white solid (2.80 g, 87%): mp 113–115 °C; ¹H NMR (CDCl₃, 500 MHz) δ 7.49 (m, 2H), 7.36 (m, 3H), 4.08 (d, *J* = 5.7 Hz, 1H), 3.94 (d, *J* = 10.1 Hz, 1H), 3.88 (d, *J* = 10.1 Hz, 1H), 3.78 (q, *J* = 6.3 Hz, 1H), 3.61 (d, *J* = 5.7 Hz, 1H), 3.38 (dd, *J* = 11.3, 9.9 Hz, 1H), 3.16 (dd, *J* = 11.5, 2.8 Hz, 1H), 2.07 (dd, *J* = 9.7, 2.8 Hz, 1H), 1.07 (m, 27H), 0.39 (s, 3H), 0.35 (s, 3H); ¹³C NMR (CDCl₃, 125 MHz) δ 136.3, 134.4, 129.3, 127.7, 82.8, 80.3, 75.9, 65.1, 63.8, 37.1, 17.8, 15.6, 11.7, 8.5, −5.1, −5.7; IR (KBr) 3358, 1463 cm^{−1}; Anal. Calcd for C₂₅H₄₆O₄Si₂: C, 64.34; H, 9.94. Found: C, 64.07; H, 9.94.

(2*S,3*R**,4*R**,5*R**)-4-Dimethylphenylsilyl-3-hydroxy-2-hydroxymethyl-2,4,5-trimethyltetrahydrofuran (19).** To diol **33** (2.79 g, 6.00 mmol) in 60 mL of CH₂Cl₂ was added Et₃N (1.25 mL, 9.00 mmol) and MsCl (0.510 mL, 6.60 mmol). After 1 h at 23 °C, the mixture was diluted with 400 mL of Et₂O and was washed with saturated aqueous NaHCO₃ (50 mL), water (2 × 30 mL), and brine (40 mL). The organic layer was dried (MgSO₄) and concentrated *in vacuo*. The unpurified mesylate was dissolved in 45 mL of Et₂O and was added dropwise to a suspension of LiAlH₄ (1.37 g, 36.0 mmol) in 45 mL of Et₂O. After stirring for 6 h at 23 °C, the excessive LiAlH₄ was quenched with 5 g of Na₂SO₄·10H₂O. The mixture was filtered and the solid was washed with 3 × 50 mL of EtOAc. The filtrate was concentrated *in vacuo* and was purified by flash chromatography (40:60 EtOAc/hexanes) to afford **19** as a colorless oil (1.56 g, 88%): ¹H NMR (CDCl₃, 500 MHz) δ 7.53 (m, 2H), 7.39 (m, 3H), 4.12 (d, *J* = 9.6 Hz, 1H), 3.90 (q, *J* = 6.3 Hz, 1H), 3.58 (m, 2H), 2.78 (d, *J* = 9.6 Hz, 1H), 2.71 (dd, *J* = 8.8, 4.4 Hz, 1H), 1.08 (d, *J* = 6.3 Hz, 3H), 0.96 (s, 3H), 0.91 (s, 3H), 0.40 (s, 3H), 0.38 (s, 3H); ¹³C

NMR (CDCl₃, 125 MHz) δ 136.1, 134.2, 129.4, 127.9, 84.5, 81.8, 74.7, 66.5, 36.9, 22.0, 15.7, 8.2, -5.5, -5.8; IR (thin film) 3312, 1040 cm⁻¹; HRMS (CI/NH₃) m/z calcd for C₁₆H₂₆O₃Si (M⁺) 294.1651, found 294.1648. Anal. Calcd for C₁₆H₂₆O₃Si: C, 65.27; H, 8.91. Found: C, 65.27; H, 8.89.

(2S*,3R*,4R*,5R*)-3-Benzyloxy-2-benzyloxymethyl-4-dimethylphenylsilyl-2,4,5-trimethyltetrahydrofuran (34).

NaH (0.660 g, 16.5 mmol, 60.0% dispersion in mineral oil, washed with 3 $\times$ 15 mL of hexanes) was suspended in 20 mL of dry DMF, and then a solution of **19** (0.970 g, 3.30 mmol) in 20 mL of dry DMF was added dropwise. After 0.5 h at 23 $^{\circ}$ C, BnBr (1.96 mL, 16.5 mmol) and *n*-Bu₄NI (0.060 g, 0.160 mmol) were added. The reaction mixture was heated at 70 $^{\circ}$ C for 15 h. The mixture was cooled to 23 $^{\circ}$ C and saturated aqueous NH₄Cl (40 mL) was added. The solution was extracted with 3 $\times$ 60 mL of Et₂O. The combined organic layers were washed with brine (30 mL), dried (MgSO₄), and concentrated *in vacuo*. Purification by flash chromatography (3:97 to 5:95 EtOAc/hexanes) afforded **34** as a colorless oil (1.42 g, 81%): ¹H NMR (CDCl₃, 500 MHz) δ 7.61-7.21 (m, 15H), 4.66 (d, *J* = 10.9 Hz, 1H), 4.62 (d, *J* = 12.2 Hz, 1H), 4.51 (d, *J* = 12.2 Hz, 1H), 4.36 (d, *J* = 10.9 Hz, 1H), 3.96 (q, *J* = 6.3 Hz, 1H), 3.91 (s, 1H), 3.64 (d, *J* = 9.7 Hz, 1H), 3.54 (d, *J* = 9.7 Hz, 1H), 1.31 (s, 3H), 1.15 (s, 3H), 1.14 (d, *J* = 6.3 Hz, 3H), 0.45 (s, 3H), 0.44 (s, 3H); ¹³C NMR (CDCl₃, 125 MHz) δ 138.7, 138.4, 136.7, 134.4, 129.2, 128.1, 127.7, 127.6, 127.24, 127.19, 91.5, 81.0, 74.6, 74.3, 73.4, 73.3, 37.8, 24.3, 15.7, 9.2, -4.8, -5.1; IR (thin film) 2963, 1427, 1108 cm⁻¹; HRMS (CI/NH₃) m/z calcd for C₂₂H₂₉O₂Si (M - BnO)⁺ 353.1937, found 353.1933. Anal. Calcd for C₃₀H₃₈O₃Si: C, 75.91; H, 8.08. Found: C, 76.10; H, 8.02.

(2S*,3R*,4R*,5R*)-3-Benzyloxy-2-benzyloxymethyl-4-hydroxy-2,4,5-trimethyltetrahydrofuran (20).

To a cooled (0 $^{\circ}$ C) solution of KH (0.654 g, 16.4 mmol) in 15 mL of NMP was added dropwise cumene hydroperoxide (2.20 mL, 88.0% solution, 13.1 mmol). After the solution was warmed to 23 $^{\circ}$ C, a solution of **34** (1.55 g, 3.27 mmol) in 15 mL of NMP was added dropwise, followed by the addition of *n*-Bu₄NF (1.00 M solution in THF, 6.60 mL, 6.60 mmol). After stirring for 40 h at 23 $^{\circ}$ C, 50 mL of saturated Na₂S₂O₃ was added and the mixture was stirred vigorously for 0.5 h. The solution was extracted with 5 $\times$ 60 mL of *t*-BuOMe. The combined organic layers were washed with brine (50 mL), dried (MgSO₄), and concentrated *in vacuo*. Purification by flash chromatography (5:95 to 30:70 EtOAc/hexanes) afforded **20** as a colorless oil (0.943 g, 81%): ¹H NMR (CDCl₃, 500 MHz) δ 7.29 (m, 10H), 4.66 (d, *J* = 12.0 Hz, 1H), 4.62 (d, *J* = 12.0 Hz, 1H), 4.58 (d, *J* = 12.3 Hz, 1H), 4.51 (d, *J* = 12.3 Hz, 1H), 3.80 (q, *J* = 6.4 Hz, 1H), 3.74 (s, 1H), 3.52 (d, *J* = 9.9 Hz, 1H), 3.42 (d, *J* = 9.9 Hz, 1H), 1.47 (br, 1H), 1.40 (s, 3H), 1.21 (s, 3H), 1.15 (d, *J* = 6.4 Hz, 3H); ¹³C NMR (CDCl₃, 125 MHz) δ 138.5, 138.4, 128.3, 128.1, 127.54, 127.52, 127.3, 127.2, 93.2, 80.8, 80.4, 76.9, 73.5, 73.4, 73.3, 24.4, 16.4, 14.0; IR (thin film) 3440, 1454, 1086 cm⁻¹; HRMS (CI/ NH₃) m/z calcd for C₁₅H₂₁O₄ (M - Bn)⁺ 265.1440, found 265.1442. Anal. Calcd for C₂₂H₂₈O₄: C, 74.12; H, 7.92. Found: C, 73.82; H, 7.98.

(2*S,3*R**,4*R**,5*R**)-4-*tert*-Butyldimethylsilyloxy-3-benzyloxy-2-benzyloxymethyl-2,4,5-**

trimethyltetrahydrofuran (35). To alcohol **20** (1.03 g, 2.88 mmol) in 30 mL of CH₂Cl₂ was added 2,6-lutidine (0.840 mL, 7.20 mmol) and TBSOTf (1.65 mL, 7.20 mmol). After stirring for 10 h at 23 °C, the reaction mixture was diluted with 200 mL of Et₂O. The solution was washed with saturated aqueous NaHCO₃ (30 mL), water (3 × 30 mL), and brine (30 mL). The organic layer was dried (MgSO₄) and concentrated *in vacuo*. Purification by flash chromatography (2:98 EtOAc/hexanes) afforded **35** as a colorless oil (1.30 g, 96%): ¹H NMR (CDCl₃, 500 MHz) δ 7.35 (m, 10H), 4.72 (d, *J* = 11.4 Hz, 1H), 4.67 (d, *J* = 11.4 Hz, 1H), 4.62 (d, *J* = 12.2 Hz, 1H), 4.54 (d, *J* = 12.2 Hz, 1H), 3.90 (q, *J* = 6.4 Hz, 1H), 3.82 (s, 1H), 3.62 (d, *J* = 9.7 Hz, 1H), 3.47 (d, *J* = 9.7 Hz, 1H), 1.51 (s, 3H), 1.30 (s, 3H), 1.22 (d, *J* = 6.3 Hz, 3H), 0.96 (s, 9H), 0.19 (s, 3H), 0.18 (s, 3H); ¹³C NMR (CDCl₃, 125 MHz) δ 138.7, 138.6, 128.2, 128.1, 127.6, 127.4, 127.3, 94.7, 83.4, 80.5, 77.5, 73.9, 73.6, 73.4, 25.7, 24.7, 18.0, 16.7, 14.2, -2.1, -2.3; IR (thin film) 2956, 1455, 1094 cm⁻¹; HRMS (CI/ NH₃) *m/z* calcd for C₂₀H₃₃O₃Si (M - BnOCH₂)⁺ 349.2199, found 349.2201. Anal. Calcd for C₂₈H₄₂O₄Si: C, 71.44; H, 8.99. Found: C, 71.70; H, 9.05.

(2*S,3*R**,4*R**,5*R**)-4-(*tert*-Butyldimethylsilyloxy)-3-benzyloxy-2-hydroxymethyl-2,4,5-**

trimethyltetrahydrofuran (21). Dibenzyl ether **35** (1.30 g, 2.76 mmol) was hydrogenated (1 atm) with 10% Pd-C (0.350 g) in 35 mL of EtOH for 40 min at 23 °C. The mixture was then filtered and the filtrate was concentrated *in vacuo*. Purification by flash chromatography (15:85 to 40:60 EtOAc/hexanes) afforded **21** as a colorless oil (0.891 g, 85%): ¹H NMR (CDCl₃, 500 MHz) δ 7.35 (m, 5H), 4.81 (d, *J* = 11.4 Hz, 1H), 4.55 (d, *J* = 11.4 Hz, 1H), 3.89 (s, 1H), 3.86 (q, *J* = 6.3 Hz, 1H), 3.58 (dd, *J* = 11.8, 7.7 Hz, 1H), 3.53 (dd, *J* = 11.8, 6.2 Hz, 1H), 2.38 (dd, *J* = 7.5, 6.3 Hz, 1H), 1.33 (s, 3H), 1.30 (s, 3H), 1.17 (d, *J* = 6.3 Hz, 3H), 0.91 (s, 9H), 0.16 (s, 3H), 0.15 (s, 3H); ¹³C NMR (CDCl₃, 125 MHz) δ 137.8, 128.6, 128.0, 127.4, 95.8, 83.6, 79.9, 77.1, 73.8, 67.2, 25.7, 24.3, 18.1, 16.3, 13.9, -1.8, -2.1; IR (thin film) 3456 cm⁻¹; HRMS (CI/ NH₃) *m/z* calcd for C₂₀H₃₃O₃Si (M - CH₂OH)⁺ 349.2199, found 349.2190. Anal. Calcd for C₂₁H₃₆O₄Si: C, 66.27; H, 9.53. Found: C, 66.17; H, 9.56.

(2*S,3*R**,4*R**,5*R**)-4-(*tert*-Butyldimethylsilyloxy)-3-benzyloxy-2-(2-carboethoxy-(*E*)-propenyl)-2,4,5-**

trimethyltetrahydrofuran (22). TPAP (0.041 g, 0.120 mmol) was added to a mixture of **21** (0.890 g, 2.33 mmol), powdered 4 Å molecular sieves (1.16 g), and NMO (0.409 g, 3.50 mmol) in 20 mL of CH₂Cl₂. The reaction mixture was allowed to stir at 23 °C for 1 h and was diluted with 20 mL of CH₂Cl₂. The mixture was filtered through a plug of silica gel and was washed with 3 × 40 mL of CH₂Cl₂. The filtrate was concentrated *in vacuo*. The unpurified aldehyde was dissolved in 20 mL of dry benzene and

(carboethoxyethylidene)triphenylphosphorane (2.54 g, 7.00 mmol) was added. The solution was heated at reflux for 10 h. Following cooling and dilution with 200 mL of Et₂O, the reaction mixture was washed with 40 mL of saturated NaHCO₃ and 40 mL of brine. The organic layer was dried (MgSO₄) and concentrated *in vacuo*. Purification by flash chromatography (20:80 EtOAc/hexanes) afforded **22** as a colorless oil (1.02 g, 95%): ¹H NMR (CDCl₃, 500 MHz) δ 7.35 (m, 5H), 7.08 (d, *J* = 1.4 Hz, 1H), 4.78 (d, *J* = 11.5 Hz, 1H), 4.67 (d, *J* = 11.5 Hz, 1H), 4.20 (q, *J* = 7.2 Hz, 2H), 3.90 (q, *J* = 6.4 Hz, 1H), 3.82 (s, 1H), 2.08 (d, *J* = 1.4 Hz, 3H), 1.46 (s, 3H), 1.31 (t, *J* = 7.1 Hz, 3H), 1.16 (s, 3H), 1.15 (d, *J* = 6.2 Hz, 3H), 0.92 (s, 9H), 0.14 (s, 3H), 0.13 (s, 3H); ¹³C NMR (CDCl₃, 125 MHz) δ 169.0, 141.5, 138.1, 128.3, 128.2, 127.6, 127.4, 94.7, 82.1, 79.9, 77.8, 73.3, 60.5, 27.7, 25.7, 18.0, 15.9, 14.17, 14.16, 13.4, -2.0, -2.3; IR (thin film) 1713, 1651 cm⁻¹; HRMS (CI/ NH₃) *m/z* calcd for C₂₂H₃₃O₅Si (M - C₄H₉)⁺ 405.2097, found 405.2096. Anal. Calcd for C₂₆H₄₂O₅Si: C, 67.49; H, 9.16. Found: C, 67.71; H, 9.36.

(2*S,3*R**,4*R**,5*R**)-4-(*tert*-Butyldimethylsilyloxy)-2-(2-carboethoxy-(*E*)-propenyl)-3-hydroxy-2,4,5-trimethyltetrahydrofuran (**36**).** To benzyl ether **22** (1.02 g, 2.20 mmol) in 40 mL of 1,2-dichloroethane was added DDQ (3.00 g, 13.2 mmol) and 4 mL of pH 7 buffer solution. The reaction mixture was stirred at 50 °C for 40 h. After cooling to 23 °C, saturated aqueous NaHCO₃ (50 mL) was slowly added. The mixture was poured into 200 mL of Et₂O, and the layers were separated. The organic layer was washed with 50 mL of brine and the combined aqueous layers were extracted with 3 × 60 mL of Et₂O. The combined organic layers were dried (MgSO₄) and concentrated *in vacuo*. Purification by flash chromatography (2:98 to 10:90 EtOAc/hexanes) afforded **36** as a colorless oil (0.690 g, 84%): ¹H NMR (CDCl₃, 500 MHz) δ 6.86 (s, 1H), 4.18 (q, *J* = 7.2 Hz, 2H), 3.94 (d, *J* = 7.5 Hz, 1H), 3.86 (q, *J* = 6.4 Hz, 1H), 2.03 (d, *J* = 0.9 Hz, 3H), 1.95 (d, *J* = 7.5 Hz, 1H), 1.48 (s, 3H), 1.29 (t, *J* = 7.1 Hz, 3H), 1.13 (d, *J* = 6.4 Hz, 3H), 1.08 (s, 3H), 0.85 (s, 9H), 0.12 (s, 3H), 0.11 (s, 3H); ¹³C NMR (CDCl₃, 125 MHz) δ 168.6, 141.8, 129.0, 87.2, 82.1, 82.0, 78.8, 60.8, 26.2, 25.7, 17.9, 16.1, 14.5, 14.2, 13.7, -2.3, -2.5; IR (thin film) 3484, 1693, 1650 cm⁻¹; HRMS (CI/ NH₃) *m/z* calcd for C₁₉H₃₄O₄Si (M - H₂O)⁺ 354.2226, found 354.2225. Anal. Calcd for C₁₉H₃₆O₅Si: C, 61.25; H, 9.75. Found: C, 61.28; H, 9.85.

Methanesulfonic acid (2*S,3*R**,4*R**,5*R**)-4-(*tert*-butyldimethylsilyloxy)-2-(2-carboethoxy-(*E*)-propenyl)-2,4,5-trimethyltetrahydrofuran-3-yl ester (**37**).** To a solution of alcohol **36** (0.690 g, 1.85 mmol) in 20 mL of CH₂Cl₂ were added pyridine (1.49 mL, 18.5 mmol), DMAP (0.023g, 0.190 mmol), and MsCl (1.43 mL, 18.5 mmol). The reaction mixture was stirred for 2 d at 23 °C and was then diluted with 150 mL of CH₂Cl₂. The mixture was washed with 50 mL of 1 N HCl, the layers were separated, and the aqueous layer was extracted with 50 mL of CH₂Cl₂. The combined organic layers were washed with saturated aqueous 30 mL of NaHCO₃, 30 mL of brine, dried (MgSO₄), filtered, and concentrated *in vacuo*. Purification by flash chromatography (4:96 EtOAc/hexanes) afforded **37** as a colorless oil (0.797 g, 96%): ¹H NMR (CDCl₃, 500 MHz) δ 6.78 (d, *J* = 1.4 Hz, 1H), 4.81 (s, 1H), 4.17 (m, 2H), 4.01 (q, *J* = 6.5 Hz, 1H), 3.08 (s, 3H), 2.05 (d, *J* = 1.3 Hz, 3H), 1.54 (s, 3H), 1.28 (t, *J* = 7.1 Hz, 3H), 1.23 (s, 3H), 1.16 (d, *J* = 6.5 Hz, 3H), 0.87 (s, 9H), 0.14 (s, 3H), 0.13 (s, 3H); ¹³C NMR (CDCl₃, 125 MHz) δ 168.6, 139.5, 129.1, 93.1, 82.3, 81.3, 80.1, 60.7, 38.7, 25.6, 25.3, 17.9, 17.8, 15.1,

14.2, 13.2, -2.1, -2.3; IR (thin film) 1713, 1658 cm^{-1} ; HRMS (CI/ NH_3) m/z calcd for $\text{C}_{18}\text{H}_{33}\text{O}_6\text{SiS}$ ($\text{M} - \text{OEt}$)⁺ 405.1767, found 405.1762. Anal. Calcd for $\text{C}_{20}\text{H}_{38}\text{O}_7\text{SiS}$: C, 53.31; H, 8.51. Found: C, 53.60; H, 8.63.

3-((1*R,2*S**,4*R**,5*R**)-3,6-dioxa-2,4,5-trimethyl-bicyclo[3.1.0]hex-2-yl)-2-methylacrylic acid ethyl ester (4).** To a solution of **37** (0.0690 g, 0.150 mmol) in 3 mL of dry THF was added $n\text{-Bu}_4\text{NF}$ (1.00 M solution in THF, 0.230 mL, 0.230 mmol, pre-treated with 4 Å molecular sieves for 4 h). After stirring for 12 h at 23 °C, saturated aqueous NH_4Cl solution (5 mL) was added. The mixture was extracted with 3×20 mL of Et_2O . The combined organic layers were washed with 10 mL of brine, dried (MgSO_4), filtered, and concentrated *in vacuo*. Purification by flash chromatography (5:95 to 10:90 EtOAc /hexanes) afforded **4** as a colorless oil (0.033 g, 92%). Spectral data were identical to those reported in the literature:³ ^1H NMR (CDCl_3 , 500 MHz) δ 6.82 (d, J = 1.4 Hz, 1H), 4.20 (q, J = 7.1 Hz, 2H), 4.12 (q, J = 6.8 Hz, 1H), 3.46 (s, 1H), 2.03 (d, J = 1.4 Hz, 3H), 1.45 (s, 3H), 1.39 (s, 3H), 1.29 (t, J = 7.2 Hz, 3H), 1.14 (d, J = 6.8 Hz, 3H); ^{13}C NMR (CDCl_3 , 125 MHz) δ 168.1, 142.3, 130.2, 79.6, 76.8, 67.3, 66.7, 60.8, 20.8, 18.6, 14.2, 13.8, 13.6; IR (thin film) 1713, 1651 cm^{-1} ; HRMS (CI/ NH_3) m/z calcd for $\text{C}_{13}\text{H}_{21}\text{O}_4$ ($\text{M} + \text{H}$)⁺ 241.1440, found 241.1430.

(2*S,3*R**,4*R**,5*R**)-2-(2-Carboethoxy-(*E*)-propenyl)-3,4-dihydroxy-2,4,5-trimethyltetrahydrofuran (38).** To epoxide **4** (0.120 g, 0.500 mmol) in 15 mL of THF and 5 mL of H_2O was added HClO_4 (70.0% solution, 5.00 mL) at 0 °C. The reaction mixture was stirred for 10 h at 23 °C, and saturated aqueous NaHCO_3 (30 mL) was added slowly. The solution was extracted with 3×40 mL of CH_2Cl_2 , and the combined organic layers were washed with brine (20 mL), dried (MgSO_4), and concentrated *in vacuo*. Purification by flash chromatography (10:90 to 40: 60 EtOAc /hexanes) afforded **38** as a colorless oil (0.056 g, 44%). Spectral data were identical to those reported in the literature:⁴ ^1H NMR (CDCl_3 , 500 MHz) δ 6.97 (d, J = 1.3 Hz, 1H), 4.18 (m, 2H), 3.96 (d, J = 4.7 Hz, 1H), 3.82 (q, J = 6.3 Hz, 1H), 2.09 (d, J = 4.9 Hz, 1H), 1.97 (d, J = 1.4 Hz, 3H), 1.74 (s, 1H), 1.36 (s, 3H), 1.30 (t, J = 7.1 Hz, 3H), 1.22 (s, 3H), 1.19 (d, J = 6.3 Hz, 3H); ^{13}C NMR (CDCl_3 , 125 MHz) δ 168.2, 146.8, 127.6, 85.3, 83.9, 80.4, 77.6, 60.9, 20.2, 17.3, 14.2, 13.5, 12.1.

³ Hatakeyama, S.; Sakurai, K.; Numata, H.; Ochi, N.; Takano, S. *J. Am. Chem. Soc.* **1988**, *110*, 5201-5203.

⁴ Whang, K.; Venkataraman, H.; Kim, Y. G.; Cha, J. K. *J. Org. Chem.* **1991**, *56*, 7174-7177.

(2S*,3R*,4R*,5R*)-3,4-Dihydroxy-2-(2-hydroxymethyl-(E)-propenyl)-2,4,5-trimethyltetrahydrofuran (39). A solution of *i*-Bu₂AlH (1.50 M in toluene, 0.470 mL, 0.700 mmol) was added to a cooled (−78 °C) solution of **38** (0.036 g, 0.140 mmol) dropwise by syringe. After 1 h at −78 °C, the reaction mixture was allowed to warm to 0 °C and was maintained at 0 °C for 0.5 h. The excessive *i*-Bu₂AlH was then quenched with 0.5 mL of MeOH, followed by a 5 mL of saturated aqueous solution of Rochelle's salt. The mixture was stirred vigorously for 1 h. The resulting solution was extracted with 3 × 20 mL of EtOAc. The combined organic layers were washed with brine (10 mL), dried (MgSO₄), and concentrated *in vacuo*. Purification by flash chromatography (70:30 EtOAc/hexanes to EtOAc) afforded **39** as a colorless oil (0.024 g, 80%). Spectral data were identical to those reported in the literature:⁴ ¹H NMR (CDCl₃, 500 MHz) δ 5.74 (s, 1H), 3.95 (m, 3H), 3.81 (q, *J* = 6.3 Hz, 1H), 1.97 (s, br, 3H), 1.86 (s, 3H), 1.34 (s, 3H), 1.21 (s, 3H), 1.18 (d, *J* = 6.3 Hz, 3H); ¹³C NMR (CDCl₃, 125 MHz) δ 135.3, 132.2, 85.5, 83.9, 81.1, 77.6, 68.2, 21.0, 17.8, 14.7, 12.3.

(2S*,3R*,4R*,5R*)-3,4-Dihydroxy-2-(2-formyl-(E)-propenyl)-2,4,5-trimethyltetrahydrofuran [(±)-citroviral] (1). Triol **39** (0.021 g, 0.100 mmol) and MnO₂ (0.174 g, 2.00 mmol) in 3 mL of Et₂O were stirred at 23 °C for 4 h. The mixture was filtered through Celite and the Celite was washed with EtOAc (3 × 10 mL). The filtrate was concentrated *in vacuo*. Purification by flash chromatography (50:55 EtOAc/hexanes) afforded **1** as a colorless oil (0.013 g, 61%). Spectral data were identical to those reported in the literature:⁴ ¹H NMR (CDCl₃, 500 MHz) δ 9.37 (s, 1H), 6.68 (d, *J* = 1.1 Hz, 1H), 3.99 (d, *J* = 4.9 Hz, 1H), 3.89 (q, *J* = 6.4 Hz, 1H), 2.03 (br, 1H), 1.88 (d, *J* = 1.1 Hz, 3H), 1.56 (s, 1H), 1.42 (s, 3H), 1.26 (s, 3H), 1.21 (d, *J* = 6.3 Hz, 3H); ¹³C NMR (CDCl₃, 125 MHz) δ 195.2, 159.2, 137.4, 85.3, 84.3, 80.5, 78.2, 20.0, 18.0, 12.2, 10.1.

III. Kinetic resolution of allylsilane 6

(R)-2-Oxopropionic acid 4,4-dimethyl-2-oxo-tetrahydrofuran-3-yl ester (23). A solution of (*R*)-pantolactone (13.1 g, 100 mmol), pyruvic acid (34.7 mL, 500 mmol), and *p*-toluenesulfonic acid (0.950 g, 5.00 mmol) in 300 mL of dry benzene was heated at reflux with azeotropic removal of water for 40 h. The reaction mixture was cooled and then washed with 300 mL of aqueous saturated NaHCO₃, and the aqueous layer was extracted with 3 × 150 mL of EtOAc. The combined organic layers were washed with brine (50 mL), dried (MgSO₄), and concentrated *in vacuo*. Purification by flash chromatography (30:70 EtOAc/hexanes) followed by recrystallization from EtOAc/hexanes afforded **23** as colorless needles (11.0 g, 55%); mp 77–79 °C; ¹H NMR (CDCl₃, 500 MHz) δ 5.38 (s, 1H), 4.09 (d, *J* = 9.1 Hz, 1H), 4.06 (d, *J* = 9.1 Hz, 1H), 2.50 (d, *J* = 1.0 Hz, 3H), 1.22 (s, 3H), 1.16 (s, 3H); ¹³C NMR (CDCl₃, 125 MHz) δ 189.9, 171.0, 159.2, 76.6, 76.2, 40.2, 26.8, 22.7, 19.7; IR (thin film) cm^{−1}; [α]_D²³ +31.0° (*c* 1.62, CHCl₃); HRMS (CI/NH₃) *m/z* calcd for C₉H₁₃O₅ (*M* + H)⁺ 201.0765, found 201.0763. Anal. Calcd for C₉H₁₂O₅: C, 54.00; H, 6.04. Found: C, 54.15; H, 6.09.

(2R,3S,4S,5S)-3-Acetoxy-4-dimethylphenylsilyl-2,4,5-trimethyltetrahydrofuran-2-carboxylic acid (R)-4,4-dimethyl-2-oxo-tetrahydrofuran-3-yl ester (24). To a solution of **23** (0.100 g, 0.500 mmol) and ($\pm$)-allylsilane **6** (0.262 g, 1.00 mmol) in 5 mL of dry CH_2Cl_2 was added dropwise SnCl_4 (0.200 M solution in CH_2Cl_2 , 2.75 mL, 0.550 mmol) by syringe at -78°C . After 24 h at -78°C , 0.2 mL of Et_3N was added followed by 5 mL of water. The layers were separated and the aqueous layer was extracted with 3×10 mL of EtOAc . The combined organic layers were washed with 5 mL of brine, dried (MgSO_4), filtered, and concentrated *in vacuo*. Purification by flash chromatography (5:95 to 30:70 EtOAc /hexanes) afforded **(-)-6** (0.126 g, 48%) as a colorless oil and **23** (0.208 g, 90%) as a viscous colorless oil. The spectra data of the resolved **(-)-6** were identical to the racemic one (*vide supra*): $[\alpha]_D^{23} -44.9^\circ$ (c 1.49, CHCl_3). Compound **24** was determined to be a single stereoisomer by ^1H NMR spectroscopy: ^1H NMR (CDCl_3 , 500 MHz) δ 7.47 (m, 2H), 7.36 (m, 3H), 5.69 (s, 1H), 5.33 (s, 1H), 4.35 (q, $J = 6.3$ Hz, 1H), 4.04 (d, $J = 9.0$ Hz, 1H), 4.02 (d, $J = 9.0$ Hz, 1H), 1.79 (s, 3H), 1.28 (s, 3H), 1.25 (s, 3H), 1.14 (d, $J = 6.3$ Hz, 3H), 1.12 (s, 3H), 1.09 (s, 3H), 0.37 (s, 3H), 0.36 (s, 3H); ^{13}C NMR (CDCl_3 , 125 MHz) δ 172.2, 171.7, 170.1, 135.7, 134.2, 129.3, 127.8, 82.1, 79.6, 78.0, 76.0, 75.8, 40.6, 35.6, 23.0, 20.1, 19.8, 19.7, 15.3, 9.6, -5.5, -5.7; IR (thin film) 2967, 1794, 1752 cm^{-1} ; $[\alpha]_D^{23} -70.0^\circ$ (c 1.22, CHCl_3); HRMS (CI/NH_3) m/z calcd for $\text{C}_{24}\text{H}_{35}\text{O}_7\text{Si}$ ($\text{M} + \text{H}^+$) 463.2152, found 463.2138. Anal. Calcd for $\text{C}_{24}\text{H}_{34}\text{O}_7\text{Si}$: C, 62.31; H, 7.41. Found: C, 62.55; H, 7.44.

(2S,3S,4S,5S)-4-Dimethylphenylsilyl-3-hydroxy-2-hydroxymethyl-2,4,5-trimethyltetrahydrofuran ((-)-10). To a cooled (0°C) suspension of LiAlH_4 (0.042 g, 1.10 mmol) in 2 mL of Et_2O was added a solution of **23** (0.100 g, 0.220 mmol) in 2 mL of dry Et_2O . After 10 h, the excess hydride was carefully quenched by addition of 0.5 g of $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$. The mixture was filtered and the solid was washed with 3×10 mL of EtOAc . The combined filtrate was concentrated *in vacuo*. Purification by flash chromatography (30:70 to 50:50 EtOAc /hexanes) afforded **(-)-10** as a white solid (0.058 g, 90%) with an ee of $> 99\%$ as determined by chiral HPLC analysis: $[\alpha]_D^{23} -37.9^\circ$ (c 1.24, CHCl_3). The spectra data of **(-)-10** were identical to the racemic compound (*vide supra*).

(2*R*,3*R*,4*R*,5*R*)-4-Dimethylphenylsilyl-3-hydroxy-2-hydroxymethyl-2,4,5-trimethyltetrahydrofuran ((+)-10). The same procedures developed for the racemic compounds were employed. Diol (+)-**10** was obtained as a white solid with an ee of > 99% as determined by chiral HPLC analysis. The spectra data of (+)-**9** and (+)-**10** were identical to the racemic ones (*vide supra*): (+)-**9**: $[\alpha]_D^{23} +31.5^\circ$ (*c* 1.20, CHCl₃); (+)-**10**: $[\alpha]_D^{23} +39.0^\circ$ (*c* 1.45, CHCl₃).

IV. X-ray Analysis of Compound (±)-10:

X-ray Data Collection, Structure Solution and Refinement for (±)-10. A colorless crystal of approximate dimensions 0.16 x 0.26 x 0.36 mm was mounted on a glass fiber and transferred to a Bruker CCD platform diffractometer. The SMART¹ program package was used to determine the unit-cell parameters and for data collection (25 sec/frame scan time for a sphere of diffraction data). The raw frame data was processed using SAINT² and SADABS³ to yield the reflection data file. Subsequent calculations were carried out using the SHELXTL⁴ program. The diffraction symmetry was 2/*m* and the systematic absences were consistent with the monoclinic space groups *Pn* and *P2*/*n*. It was later determined that the noncentrosymmetric space group *Pn* was correct.

The structure was solved by direct methods and refined on F^2 by full-matrix least-squares techniques. The analytical scattering factors⁵ for neutral atoms were used throughout the analysis. Hydrogen atoms were included using a riding model. There were four molecules of the formula unit present and two water molecules. The hydrogen atoms associated with the water molecules were not located or included in the refinement. The structure was refined utilizing the SHELXTL⁴ TWIN command. At convergence, $wR2 = 0.1801$ and $GOF = 1.020$ for 420 variables refined against 15959 data. As a comparison for refinement on F , $R1 = 0.0660$ for those 11782 data with $I > 2.0\sigma(I)$. The absolute structure was assigned by refinement of the Flack parameter⁶ although the value (0.20) is somewhat higher than expected.

References.

1. SMART Software Users Guide, Version 5.1, 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.
6. Flack, H. D. Acta. Cryst., A39, 876-881, 1983.

Definitions:

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

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

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 (±)-10.

Identification code	kaw23 (Zhikai Peng)	
Empirical formula	C ₁₆ H ₂₆ O ₃ Si • 1/2(H ₂ O)	
Formula weight	303.47	
Temperature	163(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	Pn	
Unit cell dimensions	a = 6.5888(3) Å	a = 90°.
	b = 13.7070(7) Å	b = 90.1340(10)°.
	c = 37.641(2) Å	g = 90°.
Volume	3399.5(3) Å ³	
Z	8	
Density (calculated)	1.186 Mg/m ³	
Absorption coefficient	0.147 mm ⁻¹	
F(000)	1320	
Crystal size	0.36 x 0.26 x 0.16 mm ³	
Theta range for data collection	1.49 to 28.29°.	
Index ranges	-8 ≤ h ≤ 8, -18 ≤ k ≤ 18, -50 ≤ l ≤ 50	
Reflections collected	36749	
Independent reflections	15959 [R(int) = 0.0530]	
Completeness to theta = 28.29°	97.5 %	
Absorption correction	None	
Max. and min. transmission	0.9769 and 0.9490	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	15959 / 2 / 420	
Goodness-of-fit on F ²	1.020	
Final R indices [I > 2sigma(I)]	R1 = 0.0660, wR2 = 0.1555	
R indices (all data)	R1 = 0.1000, wR2 = 0.1801	
Absolute structure parameter	0.2(2)	
Largest diff. peak and hole	0.886 and -0.431 e.Å ⁻³	

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

	x	y	z	U(eq)
Si(1)	15520(2)	9965(2)	11729(1)	19(1)
O(1)	15461(5)	10980(2)	10701(1)	22(1)
O(2)	15851(6)	9603(2)	10088(1)	26(1)
O(3)	11757(5)	9517(3)	11111(1)	28(1)
C(1)	14207(7)	10119(3)	10653(1)	19(1)
C(2)	13818(8)	9723(4)	11041(2)	19(1)
C(3)	14658(8)	10531(5)	11292(2)	19(1)
C(4)	16384(7)	10919(4)	11054(1)	20(1)
C(5)	15444(7)	9364(3)	10453(1)	20(1)
C(6)	12308(8)	10420(4)	10456(1)	25(1)
C(7)	13024(8)	11316(4)	11356(1)	27(1)
C(8)	17278(8)	11899(4)	11144(1)	28(1)
C(9)	13367(10)	9316(5)	11940(2)	30(2)
C(10)	17659(9)	9095(5)	11655(2)	23(1)
C(11)	16404(8)	10942(4)	12054(2)	19(1)
C(12)	18407(9)	11260(5)	12072(2)	25(1)
C(13)	19033(9)	11979(4)	12311(2)	26(1)
C(14)	17648(8)	12404(4)	12547(1)	20(1)
C(15)	15613(9)	12083(4)	12534(2)	27(1)
C(16)	15027(10)	11381(5)	12292(2)	29(1)
Si(2)	5577(2)	7588(2)	8718(1)	17(1)
O(4)	9208(5)	6516(2)	9510(1)	20(1)
O(5)	11672(5)	7717(3)	9995(1)	29(1)
O(6)	5057(5)	8158(3)	9613(1)	22(1)
C(17)	8447(7)	7357(3)	9698(1)	18(1)
C(18)	6856(8)	7818(4)	9444(2)	17(1)
C(19)	6386(8)	7023(5)	9164(2)	17(1)
C(20)	8504(8)	6549(4)	9144(1)	20(1)
C(21)	10167(7)	8093(4)	9756(1)	22(1)
C(22)	7592(8)	7003(4)	10050(1)	23(1)
C(23)	4748(7)	6310(4)	9289(1)	22(1)
C(24)	8647(8)	5527(4)	8996(1)	26(1)
C(25)	3326(10)	8391(5)	8783(2)	29(2)
C(26)	7679(9)	8325(5)	8520(2)	26(1)
C(27)	4900(8)	6599(4)	8397(2)	21(1)
C(28)	2921(10)	6193(5)	8396(2)	27(1)
C(29)	2434(11)	5431(5)	8159(2)	37(2)
C(30)	3949(12)	5117(6)	7926(2)	54(2)
C(31)	5713(12)	5465(6)	7916(2)	43(2)
C(32)	6263(10)	6220(5)	8153(2)	29(1)
Si(3)	5410(2)	2505(2)	8778(1)	19(1)

O(7)	5487(5)	1513(2)	9815(1)	21(1)
O(8)	5175(6)	2891(2)	10426(1)	27(1)
O(9)	9201(5)	2959(3)	9395(1)	29(1)
C(33)	6762(7)	2367(3)	9859(1)	18(1)
C(34)	7122(8)	2755(4)	9471(2)	18(1)
C(35)	6265(8)	1944(4)	9223(2)	16(1)
C(36)	4539(7)	1584(4)	9464(1)	20(1)
C(37)	5560(7)	3138(3)	10063(1)	21(1)
C(38)	8666(8)	2058(4)	10054(1)	27(1)
C(39)	7848(8)	1136(4)	9156(1)	26(1)
C(40)	3579(8)	607(4)	9379(1)	29(1)
C(41)	7599(9)	3156(5)	8565(2)	26(1)
C(42)	3307(10)	3391(5)	8854(2)	31(2)
C(43)	4544(9)	1530(4)	8459(2)	22(1)
C(44)	2511(9)	1204(5)	8445(2)	26(1)
C(45)	1854(10)	497(5)	8206(2)	35(2)
C(46)	3186(11)	131(6)	7972(2)	45(2)
C(47)	5161(10)	401(5)	7967(2)	37(2)
C(48)	5857(9)	1110(5)	8212(2)	28(1)
Si(4)	15566(2)	4931(2)	11780(1)	19(1)
O(10)	11899(5)	5996(2)	10995(1)	22(1)
O(11)	9440(5)	4787(3)	10510(1)	28(1)
O(12)	16047(5)	4351(2)	10888(1)	22(1)
C(49)	12666(7)	5154(3)	10804(1)	17(1)
C(50)	14261(8)	4686(4)	11062(2)	17(1)
C(51)	14750(8)	5486(4)	11341(2)	16(1)
C(52)	12634(7)	5980(4)	11357(1)	17(1)
C(53)	10950(7)	4426(4)	10748(1)	22(1)
C(54)	13510(8)	5500(4)	10455(1)	23(1)
C(55)	16385(7)	6210(4)	11203(1)	23(1)
C(56)	12504(8)	7002(4)	11505(1)	26(1)
C(57)	13506(10)	4168(5)	11977(2)	30(2)
C(58)	17858(9)	4151(5)	11709(2)	23(1)
C(59)	16273(8)	5931(4)	12105(2)	20(1)
C(60)	14887(10)	6279(5)	12363(2)	31(1)
C(61)	15440(10)	7019(5)	12592(2)	32(1)
C(62)	17429(8)	7433(4)	12582(1)	22(1)
C(63)	18762(9)	7080(4)	12332(2)	27(1)
C(64)	18193(9)	6355(5)	12100(2)	24(1)
O(13)	10951(7)	7876(3)	10720(1)	38(1)
O(14)	10050(7)	4606(3)	9783(1)	38(1)

Table 3. Bond lengths [Å] and angles [°] for 10.

Si(1)-C(9)	1.854(7)
Si(1)-C(10)	1.868(6)
Si(1)-C(11)	1.904(6)
Si(1)-C(3)	1.905(6)
O(1)-C(1)	1.452(6)
O(1)-C(4)	1.462(6)
O(2)-C(5)	1.436(5)
O(3)-C(2)	1.413(7)
C(1)-C(6)	1.510(7)
C(1)-C(5)	1.519(6)
C(1)-C(2)	1.578(7)
C(2)-C(3)	1.558(8)
C(3)-C(7)	1.541(8)
C(3)-C(4)	1.544(7)
C(4)-C(8)	1.505(7)
C(11)-C(12)	1.392(9)
C(11)-C(16)	1.411(8)
C(12)-C(13)	1.394(9)
C(13)-C(14)	1.403(8)
C(14)-C(15)	1.412(8)
C(15)-C(16)	1.379(9)
Si(2)-C(25)	1.863(7)
Si(2)-C(27)	1.868(6)
Si(2)-C(26)	1.870(7)
Si(2)-C(19)	1.923(6)
O(4)-C(17)	1.444(5)
O(4)-C(20)	1.453(5)
O(5)-C(21)	1.434(6)
O(6)-C(18)	1.425(6)
C(17)-C(22)	1.519(6)
C(17)-C(21)	1.532(7)
C(17)-C(18)	1.552(7)
C(18)-C(19)	1.547(8)
C(19)-C(23)	1.531(7)
C(19)-C(20)	1.541(7)
C(20)-C(24)	1.512(7)
C(27)-C(32)	1.388(8)
C(27)-C(28)	1.418(9)
C(28)-C(29)	1.410(10)
C(29)-C(30)	1.398(10)
C(30)-C(31)	1.257(11)
C(31)-C(32)	1.412(10)
Si(3)-C(42)	1.865(7)
Si(3)-C(41)	1.877(7)

Si(3)-C(43)	1.883(7)
Si(3)-C(35)	1.926(6)
O(7)-C(33)	1.450(5)
O(7)-C(36)	1.465(6)
O(8)-C(37)	1.434(5)
O(9)-C(34)	1.428(7)
C(33)-C(38)	1.511(7)
C(33)-C(37)	1.528(6)
C(33)-C(34)	1.574(7)
C(34)-C(35)	1.557(8)
C(35)-C(36)	1.537(7)
C(35)-C(39)	1.543(7)
C(36)-C(40)	1.515(7)
C(43)-C(48)	1.397(8)
C(43)-C(44)	1.413(9)
C(44)-C(45)	1.391(9)
C(45)-C(46)	1.342(10)
C(46)-C(47)	1.353(10)
C(47)-C(48)	1.417(9)
Si(4)-C(57)	1.867(7)
Si(4)-C(58)	1.870(6)
Si(4)-C(59)	1.896(6)
Si(4)-C(51)	1.896(6)
O(10)-C(52)	1.446(5)
O(10)-C(49)	1.450(5)
O(11)-C(53)	1.425(6)
O(12)-C(50)	1.423(6)
C(49)-C(54)	1.504(6)
C(49)-C(53)	1.523(6)
C(49)-C(50)	1.565(7)
C(50)-C(51)	1.553(8)
C(51)-C(52)	1.551(7)
C(51)-C(55)	1.556(7)
C(52)-C(56)	1.511(7)
C(59)-C(64)	1.392(8)
C(59)-C(60)	1.417(8)
C(60)-C(61)	1.379(9)
C(61)-C(62)	1.428(8)
C(62)-C(63)	1.377(8)
C(63)-C(64)	1.374(9)
C(9)-Si(1)-C(10)	109.7(3)
C(9)-Si(1)-C(11)	107.2(3)
C(10)-Si(1)-C(11)	108.4(3)
C(9)-Si(1)-C(3)	109.7(3)
C(10)-Si(1)-C(3)	110.7(3)
C(11)-Si(1)-C(3)	111.0(3)

C(1)-O(1)-C(4)	107.6(3)
O(1)-C(1)-C(6)	108.1(4)
O(1)-C(1)-C(5)	108.1(4)
C(6)-C(1)-C(5)	112.8(4)
O(1)-C(1)-C(2)	104.9(4)
C(6)-C(1)-C(2)	114.3(4)
C(5)-C(1)-C(2)	108.3(4)
O(3)-C(2)-C(3)	111.6(4)
O(3)-C(2)-C(1)	113.6(4)
C(3)-C(2)-C(1)	105.1(4)
C(7)-C(3)-C(4)	111.5(5)
C(7)-C(3)-C(2)	110.1(5)
C(4)-C(3)-C(2)	98.8(4)
C(7)-C(3)-Si(1)	110.9(4)
C(4)-C(3)-Si(1)	115.0(4)
C(2)-C(3)-Si(1)	109.9(4)
O(1)-C(4)-C(8)	108.4(4)
O(1)-C(4)-C(3)	104.0(4)
C(8)-C(4)-C(3)	117.7(4)
O(2)-C(5)-C(1)	114.9(4)
C(12)-C(11)-C(16)	116.5(6)
C(12)-C(11)-Si(1)	122.7(4)
C(16)-C(11)-Si(1)	120.7(4)
C(11)-C(12)-C(13)	122.2(6)
C(12)-C(13)-C(14)	120.7(5)
C(13)-C(14)-C(15)	117.8(5)
C(16)-C(15)-C(14)	120.4(5)
C(15)-C(16)-C(11)	122.4(6)
C(25)-Si(2)-C(27)	109.0(3)
C(25)-Si(2)-C(26)	108.9(3)
C(27)-Si(2)-C(26)	108.1(3)
C(25)-Si(2)-C(19)	110.0(3)
C(27)-Si(2)-C(19)	109.7(3)
C(26)-Si(2)-C(19)	111.1(3)
C(17)-O(4)-C(20)	109.2(3)
O(4)-C(17)-C(22)	107.6(4)
O(4)-C(17)-C(21)	109.7(4)
C(22)-C(17)-C(21)	111.3(4)
O(4)-C(17)-C(18)	104.9(4)
C(22)-C(17)-C(18)	114.6(4)
C(21)-C(17)-C(18)	108.5(4)
O(6)-C(18)-C(19)	111.7(4)
O(6)-C(18)-C(17)	114.8(4)
C(19)-C(18)-C(17)	105.5(4)
C(23)-C(19)-C(20)	112.6(5)
C(23)-C(19)-C(18)	112.3(4)

C(20)-C(19)-C(18)	98.6(4)
C(23)-C(19)-Si(2)	109.4(4)
C(20)-C(19)-Si(2)	112.1(4)
C(18)-C(19)-Si(2)	111.5(4)
O(4)-C(20)-C(24)	107.5(4)
O(4)-C(20)-C(19)	104.8(4)
C(24)-C(20)-C(19)	117.8(5)
O(5)-C(21)-C(17)	111.3(4)
C(32)-C(27)-C(28)	116.5(6)
C(32)-C(27)-Si(2)	123.1(5)
C(28)-C(27)-Si(2)	120.4(4)
C(29)-C(28)-C(27)	120.1(6)
C(30)-C(29)-C(28)	117.6(7)
C(31)-C(30)-C(29)	124.2(8)
C(30)-C(31)-C(32)	119.7(7)
C(27)-C(32)-C(31)	121.8(6)
C(42)-Si(3)-C(41)	109.1(3)
C(42)-Si(3)-C(43)	109.6(3)
C(41)-Si(3)-C(43)	107.3(3)
C(42)-Si(3)-C(35)	110.0(3)
C(41)-Si(3)-C(35)	109.7(3)
C(43)-Si(3)-C(35)	111.0(3)
C(33)-O(7)-C(36)	107.2(3)
O(7)-C(33)-C(38)	108.0(4)
O(7)-C(33)-C(37)	108.3(3)
C(38)-C(33)-C(37)	112.5(4)
O(7)-C(33)-C(34)	104.8(4)
C(38)-C(33)-C(34)	114.7(4)
C(37)-C(33)-C(34)	108.1(4)
O(9)-C(34)-C(35)	111.5(4)
O(9)-C(34)-C(33)	113.4(4)
C(35)-C(34)-C(33)	105.1(4)
C(36)-C(35)-C(39)	111.5(5)
C(36)-C(35)-C(34)	98.2(4)
C(39)-C(35)-C(34)	111.4(4)
C(36)-C(35)-Si(3)	115.2(4)
C(39)-C(35)-Si(3)	110.0(4)
C(34)-C(35)-Si(3)	110.0(4)
O(7)-C(36)-C(40)	108.0(4)
O(7)-C(36)-C(35)	103.9(4)
C(40)-C(36)-C(35)	118.0(4)
O(8)-C(37)-C(33)	114.1(4)
C(48)-C(43)-C(44)	115.6(6)
C(48)-C(43)-Si(3)	122.0(5)
C(44)-C(43)-Si(3)	122.3(5)
C(45)-C(44)-C(43)	122.6(6)

C(46)-C(45)-C(44)	118.8(6)
C(45)-C(46)-C(47)	122.6(7)
C(46)-C(47)-C(48)	119.2(6)
C(43)-C(48)-C(47)	121.2(6)
C(57)-Si(4)-C(58)	109.0(3)
C(57)-Si(4)-C(59)	109.1(3)
C(58)-Si(4)-C(59)	108.0(3)
C(57)-Si(4)-C(51)	111.4(3)
C(58)-Si(4)-C(51)	109.4(3)
C(59)-Si(4)-C(51)	110.0(3)
C(52)-O(10)-C(49)	109.7(3)
O(10)-C(49)-C(54)	108.1(4)
O(10)-C(49)-C(53)	109.3(4)
C(54)-C(49)-C(53)	111.2(4)
O(10)-C(49)-C(50)	104.8(4)
C(54)-C(49)-C(50)	114.9(4)
C(53)-C(49)-C(50)	108.4(4)
O(12)-C(50)-C(51)	111.6(4)
O(12)-C(50)-C(49)	113.8(4)
C(51)-C(50)-C(49)	105.5(4)
C(52)-C(51)-C(50)	98.6(4)
C(52)-C(51)-C(55)	110.9(4)
C(50)-C(51)-C(55)	111.5(4)
C(52)-C(51)-Si(4)	113.2(4)
C(50)-C(51)-Si(4)	111.4(4)
C(55)-C(51)-Si(4)	110.7(4)
O(10)-C(52)-C(56)	108.4(4)
O(10)-C(52)-C(51)	105.7(4)
C(56)-C(52)-C(51)	118.1(4)
O(11)-C(53)-C(49)	112.1(4)
C(64)-C(59)-C(60)	117.2(6)
C(64)-C(59)-Si(4)	120.9(4)
C(60)-C(59)-Si(4)	121.9(4)
C(61)-C(60)-C(59)	120.4(6)
C(60)-C(61)-C(62)	121.1(6)
C(63)-C(62)-C(61)	117.8(6)
C(64)-C(63)-C(62)	121.0(5)
C(63)-C(64)-C(59)	122.6(5)

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

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Si(1)	27(1)	16(1)	13(1)	-1(1)	-3(1)	2(1)
O(1)	35(2)	20(2)	10(2)	2(1)	-3(1)	-3(1)
O(2)	47(2)	19(2)	13(2)	-2(1)	4(2)	3(2)
O(3)	31(2)	33(2)	20(2)	-5(2)	2(1)	-7(2)
Si(2)	21(1)	18(1)	12(1)	0(1)	-2(1)	1(1)
O(4)	30(2)	20(2)	10(1)	-1(1)	-7(1)	7(1)
O(5)	22(2)	44(2)	20(2)	4(2)	-8(1)	-3(2)
O(6)	25(2)	26(2)	16(2)	-7(1)	-1(1)	1(1)
Si(3)	26(1)	17(1)	14(1)	1(1)	-1(1)	3(1)
O(7)	32(2)	16(2)	14(2)	-1(1)	-4(1)	-2(1)
O(8)	49(2)	17(2)	14(2)	-2(1)	4(2)	1(2)
O(9)	33(2)	35(2)	19(2)	-2(2)	1(1)	-6(2)
Si(4)	24(1)	15(1)	18(1)	-1(1)	-5(1)	1(1)
O(10)	31(2)	20(2)	15(2)	-4(1)	-2(1)	5(1)
O(11)	23(2)	44(2)	18(2)	4(2)	-4(1)	-2(2)
O(12)	24(2)	24(2)	19(2)	-6(1)	2(1)	5(1)
O(13)	66(3)	24(2)	23(2)	-1(2)	-11(2)	-2(2)
O(14)	64(3)	30(2)	21(2)	1(2)	-10(2)	-5(2)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 10.

	x	y	z	U(eq)
H(2)	15259	10126	10035	39
H(3A)	11406	9015	10999	42
H(2A)	14631	9114	11076	22
H(4A)	17500	10425	11048	24
H(5A)	16754	9273	10577	24
H(5B)	14711	8734	10460	24
H(6A)	12669	10648	10218	38
H(6B)	11631	10947	10586	38
H(6C)	11390	9859	10437	38
H(7A)	12607	11598	11128	40
H(7B)	13579	11830	11509	40
H(7C)	11846	11018	11472	40
H(8A)	18342	12061	10973	42
H(8B)	17861	11878	11384	42
H(8C)	16211	12397	11135	42

H(9A)	12879	8801	11780	44
H(9B)	12265	9778	11986	44
H(9C)	13818	9024	12164	44
H(10A)	17228	8586	11488	34
H(10B)	18049	8795	11881	34
H(10C)	18822	9448	11556	34
H(12A)	19381	10978	11918	30
H(13A)	20410	12184	12313	31
H(14A)	18066	12892	12711	24
H(15A)	14642	12353	12692	32
H(16A)	13644	11185	12286	35
H(5)	12832	7806	9909	43
H(6)	5320	8665	9729	34
H(18A)	7507	8382	9320	20
H(20A)	9422	6986	9005	24
H(21A)	10809	8245	9525	27
H(21B)	9601	8706	9853	27
H(22A)	8683	6714	10192	35
H(22B)	6541	6512	10005	35
H(22C)	7000	7555	10179	35
H(23A)	5158	6022	9517	32
H(23B)	4575	5793	9112	32
H(23C)	3463	6660	9319	32
H(24A)	10071	5322	8991	39
H(24B)	8092	5517	8754	39
H(24C)	7869	5080	9146	39
H(25A)	2919	8672	8554	44
H(25B)	3674	8916	8949	44
H(25C)	2203	8006	8880	44
H(26A)	7229	8602	8293	39
H(26B)	8860	7904	8480	39
H(26C)	8051	8853	8683	39
H(28A)	1922	6434	8554	32
H(29A)	1123	5142	8157	44
H(30A)	3624	4608	7764	65
H(31A)	6672	5222	7750	52
H(32A)	7603	6475	8145	35
H(8)	5423	2296	10459	40
H(9)	9651	3363	9543	43
H(34A)	6310	3365	9436	22
H(36A)	3452	2093	9471	24
H(37A)	6318	3762	10055	25
H(37B)	4246	3242	9941	25
H(38A)	8308	1816	10290	41
H(38B)	9582	2618	10077	41
H(38C)	9343	1538	9920	41

H(39A)	8291	862	9384	38
H(39B)	9019	1413	9032	38
H(39C)	7240	620	9010	38
H(40A)	2495	472	9550	43
H(40B)	4611	94	9394	43
H(40C)	3009	623	9138	43
H(41A)	7157	3444	8340	40
H(41B)	8701	2692	8521	40
H(41C)	8083	3674	8724	40
H(42A)	2870	3666	8625	46
H(42B)	3783	3917	9009	46
H(42C)	2162	3056	8966	46
H(44A)	1555	1479	8604	31
H(45A)	488	276	8208	42
H(46A)	2724	-335	7803	54
H(47A)	6070	117	7800	44
H(48A)	7242	1303	8208	33
H(11)	8285	4696	10598	43
H(12)	15743	3904	10745	33
H(50A)	13612	4121	11185	20
H(52A)	11713	5552	11499	21
H(53A)	11515	3812	10652	26
H(53B)	10313	4277	10980	26
H(54A)	12410	5769	10310	34
H(54B)	14138	4951	10331	34
H(54C)	14531	6006	10499	34
H(55A)	15939	6487	10975	35
H(55B)	17671	5864	11169	35
H(55C)	16573	6736	11376	35
H(56A)	11093	7228	11495	39
H(56B)	13360	7439	11364	39
H(56C)	12973	7003	11752	39
H(57A)	13137	3648	11810	45
H(57B)	12316	4577	12023	45
H(57C)	13984	3880	12200	45
H(58A)	17527	3625	11542	34
H(58B)	18290	3870	11936	34
H(58C)	18958	4550	11611	34
H(60A)	13569	6002	12379	37
H(61A)	14483	7258	12759	38
H(62A)	17818	7935	12742	27
H(63A)	20094	7343	12319	32
H(64A)	19148	6133	11930	29

Table 6. Torsion angles [°] for 10.

C(4)-O(1)-C(1)-C(6)	141.6(4)
C(4)-O(1)-C(1)-C(5)	-96.1(4)
C(4)-O(1)-C(1)-C(2)	19.3(4)
O(1)-C(1)-C(2)-O(3)	131.2(4)
C(6)-C(1)-C(2)-O(3)	13.0(6)
C(5)-C(1)-C(2)-O(3)	-113.6(5)
O(1)-C(1)-C(2)-C(3)	9.0(5)
C(6)-C(1)-C(2)-C(3)	-109.3(5)
C(5)-C(1)-C(2)-C(3)	124.1(4)
O(3)-C(2)-C(3)-C(7)	-37.9(6)
C(1)-C(2)-C(3)-C(7)	85.6(5)
O(3)-C(2)-C(3)-C(4)	-154.7(4)
C(1)-C(2)-C(3)-C(4)	-31.2(5)
O(3)-C(2)-C(3)-Si(1)	84.5(5)
C(1)-C(2)-C(3)-Si(1)	-152.0(3)
C(9)-Si(1)-C(3)-C(7)	63.8(5)
C(10)-Si(1)-C(3)-C(7)	-175.0(4)
C(11)-Si(1)-C(3)-C(7)	-54.5(5)
C(9)-Si(1)-C(3)-C(4)	-168.5(4)
C(10)-Si(1)-C(3)-C(4)	-47.4(5)
C(11)-Si(1)-C(3)-C(4)	73.1(5)
C(9)-Si(1)-C(3)-C(2)	-58.1(5)
C(10)-Si(1)-C(3)-C(2)	63.0(4)
C(11)-Si(1)-C(3)-C(2)	-176.5(4)
C(1)-O(1)-C(4)-C(8)	-166.8(4)
C(1)-O(1)-C(4)-C(3)	-40.8(5)
C(7)-C(3)-C(4)-O(1)	-72.2(5)
C(2)-C(3)-C(4)-O(1)	43.5(5)
Si(1)-C(3)-C(4)-O(1)	160.5(3)
C(7)-C(3)-C(4)-C(8)	47.6(6)
C(2)-C(3)-C(4)-C(8)	163.4(5)
Si(1)-C(3)-C(4)-C(8)	-79.7(5)
O(1)-C(1)-C(5)-O(2)	-69.4(5)
C(6)-C(1)-C(5)-O(2)	50.0(6)
C(2)-C(1)-C(5)-O(2)	177.5(4)
C(9)-Si(1)-C(11)-C(12)	150.0(5)
C(10)-Si(1)-C(11)-C(12)	31.6(6)
C(3)-Si(1)-C(11)-C(12)	-90.1(6)
C(9)-Si(1)-C(11)-C(16)	-29.8(6)
C(10)-Si(1)-C(11)-C(16)	-148.2(5)
C(3)-Si(1)-C(11)-C(16)	90.0(5)
C(16)-C(11)-C(12)-C(13)	-0.5(9)
Si(1)-C(11)-C(12)-C(13)	179.7(5)
C(11)-C(12)-C(13)-C(14)	0.9(10)

C(12)-C(13)-C(14)-C(15)	-0.4(9)
C(13)-C(14)-C(15)-C(16)	-0.5(9)
C(14)-C(15)-C(16)-C(11)	1.0(10)
C(12)-C(11)-C(16)-C(15)	-0.5(9)
Si(1)-C(11)-C(16)-C(15)	179.3(5)
C(20)-O(4)-C(17)-C(22)	133.3(4)
C(20)-O(4)-C(17)-C(21)	-105.4(4)
C(20)-O(4)-C(17)-C(18)	10.9(5)
O(4)-C(17)-C(18)-O(6)	139.1(4)
C(22)-C(17)-C(18)-O(6)	21.3(6)
C(21)-C(17)-C(18)-O(6)	-103.7(5)
O(4)-C(17)-C(18)-C(19)	15.7(5)
C(22)-C(17)-C(18)-C(19)	-102.1(5)
C(21)-C(17)-C(18)-C(19)	132.9(4)
O(6)-C(18)-C(19)-C(23)	-40.3(6)
C(17)-C(18)-C(19)-C(23)	85.1(5)
O(6)-C(18)-C(19)-C(20)	-159.2(4)
C(17)-C(18)-C(19)-C(20)	-33.8(5)
O(6)-C(18)-C(19)-Si(2)	82.9(5)
C(17)-C(18)-C(19)-Si(2)	-151.7(3)
C(25)-Si(2)-C(19)-C(23)	67.5(5)
C(27)-Si(2)-C(19)-C(23)	-52.3(4)
C(26)-Si(2)-C(19)-C(23)	-171.8(4)
C(25)-Si(2)-C(19)-C(20)	-166.8(4)
C(27)-Si(2)-C(19)-C(20)	73.4(5)
C(26)-Si(2)-C(19)-C(20)	-46.1(5)
C(25)-Si(2)-C(19)-C(18)	-57.3(5)
C(27)-Si(2)-C(19)-C(18)	-177.1(4)
C(26)-Si(2)-C(19)-C(18)	63.4(4)
C(17)-O(4)-C(20)-C(24)	-159.7(4)
C(17)-O(4)-C(20)-C(19)	-33.7(5)
C(23)-C(19)-C(20)-O(4)	-77.9(5)
C(18)-C(19)-C(20)-O(4)	40.7(5)
Si(2)-C(19)-C(20)-O(4)	158.2(3)
C(23)-C(19)-C(20)-C(24)	41.4(6)
C(18)-C(19)-C(20)-C(24)	160.1(4)
Si(2)-C(19)-C(20)-C(24)	-82.4(5)
O(4)-C(17)-C(21)-O(5)	-67.2(5)
C(22)-C(17)-C(21)-O(5)	51.8(5)
C(18)-C(17)-C(21)-O(5)	178.8(4)
C(25)-Si(2)-C(27)-C(32)	144.8(5)
C(26)-Si(2)-C(27)-C(32)	26.6(6)
C(19)-Si(2)-C(27)-C(32)	-94.7(6)
C(25)-Si(2)-C(27)-C(28)	-35.5(6)
C(26)-Si(2)-C(27)-C(28)	-153.7(5)
C(19)-Si(2)-C(27)-C(28)	84.9(6)

C(32)-C(27)-C(28)-C(29)	1.2(9)
Si(2)-C(27)-C(28)-C(29)	-178.5(5)
C(27)-C(28)-C(29)-C(30)	-1.3(10)
C(28)-C(29)-C(30)-C(31)	0.5(12)
C(29)-C(30)-C(31)-C(32)	0.4(13)
C(28)-C(27)-C(32)-C(31)	-0.3(10)
Si(2)-C(27)-C(32)-C(31)	179.4(5)
C(30)-C(31)-C(32)-C(27)	-0.5(11)
C(36)-O(7)-C(33)-C(38)	-142.0(4)
C(36)-O(7)-C(33)-C(37)	95.9(4)
C(36)-O(7)-C(33)-C(34)	-19.3(4)
O(7)-C(33)-C(34)-O(9)	-131.6(4)
C(38)-C(33)-C(34)-O(9)	-13.4(6)
C(37)-C(33)-C(34)-O(9)	113.0(5)
O(7)-C(33)-C(34)-C(35)	-9.6(5)
C(38)-C(33)-C(34)-C(35)	108.7(5)
C(37)-C(33)-C(34)-C(35)	-125.0(4)
O(9)-C(34)-C(35)-C(36)	155.7(4)
C(33)-C(34)-C(35)-C(36)	32.4(5)
O(9)-C(34)-C(35)-C(39)	38.7(6)
C(33)-C(34)-C(35)-C(39)	-84.6(5)
O(9)-C(34)-C(35)-Si(3)	-83.6(5)
C(33)-C(34)-C(35)-Si(3)	153.1(3)
C(42)-Si(3)-C(35)-C(36)	47.2(5)
C(41)-Si(3)-C(35)-C(36)	167.2(4)
C(43)-Si(3)-C(35)-C(36)	-74.3(5)
C(42)-Si(3)-C(35)-C(39)	174.2(4)
C(41)-Si(3)-C(35)-C(39)	-65.7(5)
C(43)-Si(3)-C(35)-C(39)	52.7(4)
C(42)-Si(3)-C(35)-C(34)	-62.7(4)
C(41)-Si(3)-C(35)-C(34)	57.4(4)
C(43)-Si(3)-C(35)-C(34)	175.8(3)
C(33)-O(7)-C(36)-C(40)	167.8(4)
C(33)-O(7)-C(36)-C(35)	41.8(5)
C(39)-C(35)-C(36)-O(7)	72.2(5)
C(34)-C(35)-C(36)-O(7)	-44.8(5)
Si(3)-C(35)-C(36)-O(7)	-161.5(3)
C(39)-C(35)-C(36)-C(40)	-47.2(6)
C(34)-C(35)-C(36)-C(40)	-164.2(4)
Si(3)-C(35)-C(36)-C(40)	79.1(5)
O(7)-C(33)-C(37)-O(8)	68.8(5)
C(38)-C(33)-C(37)-O(8)	-50.6(5)
C(34)-C(33)-C(37)-O(8)	-178.2(4)
C(42)-Si(3)-C(43)-C(48)	145.0(5)
C(41)-Si(3)-C(43)-C(48)	26.7(6)
C(35)-Si(3)-C(43)-C(48)	-93.2(6)

C(42)-Si(3)-C(43)-C(44)	-32.4(6)
C(41)-Si(3)-C(43)-C(44)	-150.8(5)
C(35)-Si(3)-C(43)-C(44)	89.3(6)
C(48)-C(43)-C(44)-C(45)	1.3(10)
Si(3)-C(43)-C(44)-C(45)	178.9(5)
C(43)-C(44)-C(45)-C(46)	-2.3(10)
C(44)-C(45)-C(46)-C(47)	2.3(11)
C(45)-C(46)-C(47)-C(48)	-1.3(11)
C(44)-C(43)-C(48)-C(47)	-0.3(9)
Si(3)-C(43)-C(48)-C(47)	-177.9(5)
C(46)-C(47)-C(48)-C(43)	0.3(10)
C(52)-O(10)-C(49)-C(54)	-132.1(4)
C(52)-O(10)-C(49)-C(53)	106.8(4)
C(52)-O(10)-C(49)-C(50)	-9.1(5)
O(10)-C(49)-C(50)-O(12)	-139.3(4)
C(54)-C(49)-C(50)-O(12)	-20.8(6)
C(53)-C(49)-C(50)-O(12)	104.2(5)
O(10)-C(49)-C(50)-C(51)	-16.6(5)
C(54)-C(49)-C(50)-C(51)	101.9(5)
C(53)-C(49)-C(50)-C(51)	-133.1(4)
O(12)-C(50)-C(51)-C(52)	157.3(4)
C(49)-C(50)-C(51)-C(52)	33.2(5)
O(12)-C(50)-C(51)-C(55)	40.7(6)
C(49)-C(50)-C(51)-C(55)	-83.4(5)
O(12)-C(50)-C(51)-Si(4)	-83.5(5)
C(49)-C(50)-C(51)-Si(4)	152.4(3)
C(57)-Si(4)-C(51)-C(52)	48.3(5)
C(58)-Si(4)-C(51)-C(52)	168.8(4)
C(59)-Si(4)-C(51)-C(52)	-72.8(4)
C(57)-Si(4)-C(51)-C(50)	-61.7(5)
C(58)-Si(4)-C(51)-C(50)	58.8(4)
C(59)-Si(4)-C(51)-C(50)	177.2(3)
C(57)-Si(4)-C(51)-C(55)	173.6(4)
C(58)-Si(4)-C(51)-C(55)	-65.9(4)
C(59)-Si(4)-C(51)-C(55)	52.5(4)
C(49)-O(10)-C(52)-C(56)	159.1(4)
C(49)-O(10)-C(52)-C(51)	31.6(5)
C(50)-C(51)-C(52)-O(10)	-39.3(5)
C(55)-C(51)-C(52)-O(10)	77.7(5)
Si(4)-C(51)-C(52)-O(10)	-157.1(3)
C(50)-C(51)-C(52)-C(56)	-160.7(4)
C(55)-C(51)-C(52)-C(56)	-43.7(6)
Si(4)-C(51)-C(52)-C(56)	81.5(5)
O(10)-C(49)-C(53)-O(11)	67.5(5)
C(54)-C(49)-C(53)-O(11)	-51.7(5)
C(50)-C(49)-C(53)-O(11)	-178.9(4)

C(57)-Si(4)-C(59)-C(64)	154.7(5)
C(58)-Si(4)-C(59)-C(64)	36.4(6)
C(51)-Si(4)-C(59)-C(64)	-82.9(5)
C(57)-Si(4)-C(59)-C(60)	-25.3(6)
C(58)-Si(4)-C(59)-C(60)	-143.6(5)
C(51)-Si(4)-C(59)-C(60)	97.2(5)
C(64)-C(59)-C(60)-C(61)	1.1(9)
Si(4)-C(59)-C(60)-C(61)	-178.9(5)
C(59)-C(60)-C(61)-C(62)	-1.7(10)
C(60)-C(61)-C(62)-C(63)	1.2(9)
C(61)-C(62)-C(63)-C(64)	0.0(9)
C(62)-C(63)-C(64)-C(59)	-0.5(10)
C(60)-C(59)-C(64)-C(63)	0.0(9)
Si(4)-C(59)-C(64)-C(63)	-180.0(5)

V. X-ray Analysis of compound 40 (the Enantiomer of the 5-Ethyl Analogue of 24):

X-ray Data Collection, Structure Solution and Refinement for 40. A colorless crystal of approximate dimensions 0.31 x 0.36 x 0.45 mm was mounted on a glass fiber and transferred to a Bruker CCD platform diffractometer. The SMART¹ program package was used to determine the unit-cell parameters and for data collection (25 sec/frame scan time for a sphere of diffraction data). The raw frame data was processed using SAINT² and SADABS³ to yield the reflection data file. Subsequent calculations were carried out using the SHELXTL⁴ program. The diffraction symmetry was *mmm* and the systematic absences were consistent with the orthorhombic space group *P*2₁2₁2₁ which was 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 factors⁵ 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, $wR2 = 0.0677$ and $Goof = 1.045$ for 443 variables refined against 6145 data. As a comparison for refinement on F , $R1 = 0.0257$ for those 5831 data with $I > 2.0\sigma(I)$. The absolute structure was assigned by refinement of the Flack parameter⁶.

References.

1. SMART Software Users Guide, Version 5.1, 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, Version 2.03, Bruker Analytical X-Ray Systems, Inc.; Madison, WI 2000.
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.
6. Flack, H. D. Acta. Cryst., A39, 876-881, 1983.

Definitions:

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

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

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

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

Table 1. Crystal data and structure refinement for 40.

Identification code	kaw32 (Zhihui Peng)	
Empirical formula	$C_{25} H_{36} O_7 Si$	
Formula weight	476.63	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	$P2_12_12_1$	
Unit cell dimensions	$a = 11.3139(5) \text{ Å}$	$a = 90^\circ$.
	$b = 14.8824(6) \text{ Å}$	$b = 90^\circ$.
	$c = 15.0063(6) \text{ Å}$	$c = 90^\circ$.
Volume	$2526.73(18) \text{ Å}^3$	
Z	4	
Density (calculated)	1.253 Mg/m^3	
Absorption coefficient	0.134 mm^{-1}	
$F(000)$	1024	
Crystal size	$0.45 \times 0.36 \times 0.31 \text{ mm}^3$	

Theta range for data collection	1.93 to 28.28°
Index ranges	$-15 \leq h \leq 14$, $-19 \leq k \leq 19$, $-19 \leq l \leq 19$
Reflections collected	26940
Independent reflections	6145 [R(int) = 0.0244]
Completeness to theta = 28.28°	99.4 %
Absorption correction	None
Max. and min. transmission	0.9596 and 0.9421
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	6145 / 0 / 443
Goodness-of-fit on F ²	1.045
Final R indices [I > 2sigma(I)]	R1 = 0.0257, wR2 = 0.0657
R indices (all data)	R1 = 0.0282, wR2 = 0.0677
Absolute structure parameter	-0.05(7)
Extinction coefficient	0.0015(5)
Largest diff. peak and hole	0.261 and -0.139 e.Å ⁻³

Table 2. Atomic coordinates (x 10⁴) and equivalent isotropic displacement parameters (Å²x 10³) for 40. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Si(1)	6821(1)	3298(1)	2458(1)	16(1)
O(1)	6816(1)	597(1)	2947(1)	20(1)
O(2)	7268(1)	1593(1)	881(1)	21(1)
O(3)	9198(1)	1375(1)	593(1)	37(1)
O(4)	9396(1)	-232(1)	2594(1)	43(1)
O(5)	9239(1)	1193(1)	3088(1)	20(1)
O(6)	9175(1)	1579(1)	4941(1)	39(1)
O(7)	11128(1)	1796(1)	4841(1)	32(1)
C(1)	6535(1)	1513(1)	3214(1)	18(1)
C(2)	6490(1)	2042(1)	2330(1)	16(1)
C(3)	7522(1)	1597(1)	1824(1)	17(1)
C(4)	7634(1)	625(1)	2218(1)	19(1)
C(5)	5417(1)	1481(1)	3773(1)	22(1)
C(6)	5620(1)	1022(1)	4669(1)	29(1)
C(7)	5309(1)	1899(1)	1840(1)	21(1)

C(8)	8016(1)	3469(1)	3302(1)	21(1)
C(9)	5463(1)	3935(1)	2779(1)	25(1)
C(10)	7353(1)	3711(1)	1341(1)	19(1)
C(11)	8556(1)	3699(1)	1113(1)	22(1)
C(12)	8944(1)	3994(1)	283(1)	26(1)
C(13)	8134(1)	4321(1)	-336(1)	29(1)
C(14)	6938(1)	4338(1)	-126(1)	29(1)
C(15)	6556(1)	4034(1)	704(1)	24(1)
C(16)	8209(1)	1507(1)	332(1)	27(1)
C(17)	7829(2)	1585(1)	-623(1)	40(1)
C(18)	7333(1)	-130(1)	1579(1)	23(1)
C(19)	8861(1)	460(1)	2627(1)	22(1)
C(20)	10280(1)	1086(1)	3618(1)	20(1)
C(21)	10076(1)	1506(1)	4531(1)	27(1)
C(22)	12046(1)	1652(1)	4169(1)	28(1)
C(23)	11388(1)	1565(1)	3278(1)	20(1)
C(24)	12078(1)	1007(1)	2606(1)	29(1)
C(25)	11101(1)	2493(1)	2897(1)	29(1)

Table 3. Bond lengths [Å] and angles [°] for 40.

Si(1)-C(9)	1.8679(12)
Si(1)-C(8)	1.8692(12)
Si(1)-C(10)	1.8839(11)
Si(1)-C(2)	1.9160(11)
O(1)-C(4)	1.4335(14)
O(1)-C(1)	1.4559(13)
O(2)-C(16)	1.3525(14)
O(2)-C(3)	1.4440(13)
O(3)-C(16)	1.2022(17)
O(4)-C(19)	1.1957(15)
O(5)-C(19)	1.3604(13)
O(5)-C(20)	1.4305(13)
O(6)-C(21)	1.1958(16)
O(7)-C(21)	1.3486(16)

O(7)-C(22)	1.4630(15)
C(1)-C(5)	1.5187(15)
C(1)-C(2)	1.5426(14)
C(2)-C(7)	1.5399(15)
C(2)-C(3)	1.5432(15)
C(3)-C(4)	1.5678(15)
C(4)-C(18)	1.5159(15)
C(4)-C(19)	1.5375(16)
C(5)-C(6)	1.5261(17)
C(10)-C(15)	1.3990(17)
C(10)-C(11)	1.4030(16)
C(11)-C(12)	1.3927(17)
C(12)-C(13)	1.3922(19)
C(13)-C(14)	1.390(2)
C(14)-C(15)	1.3934(17)
C(16)-C(17)	1.5003(19)
C(20)-C(21)	1.5229(17)
C(20)-C(23)	1.5295(16)
C(22)-C(23)	1.5352(16)
C(23)-C(24)	1.5218(17)
C(23)-C(25)	1.5300(17)
C(9)-Si(1)-C(8)	110.57(6)
C(9)-Si(1)-C(10)	109.06(6)
C(8)-Si(1)-C(10)	109.12(5)
C(9)-Si(1)-C(2)	111.06(5)
C(8)-Si(1)-C(2)	109.98(5)
C(10)-Si(1)-C(2)	106.97(5)
C(4)-O(1)-C(1)	108.86(8)
C(16)-O(2)-C(3)	116.13(9)
C(19)-O(5)-C(20)	116.94(9)
C(21)-O(7)-C(22)	109.98(9)
O(1)-C(1)-C(5)	107.70(9)
O(1)-C(1)-C(2)	104.40(8)
C(5)-C(1)-C(2)	117.57(9)
C(7)-C(2)-C(1)	111.66(9)
C(7)-C(2)-C(3)	111.22(9)

C(1)-C(2)-C(3)	100.32(8)
C(7)-C(2)-Si(1)	110.62(7)
C(1)-C(2)-Si(1)	113.92(7)
C(3)-C(2)-Si(1)	108.65(7)
O(2)-C(3)-C(2)	109.50(8)
O(2)-C(3)-C(4)	112.47(9)
C(2)-C(3)-C(4)	105.73(8)
O(1)-C(4)-C(18)	108.43(9)
O(1)-C(4)-C(19)	105.87(9)
C(18)-C(4)-C(19)	109.70(9)
O(1)-C(4)-C(3)	105.23(8)
C(18)-C(4)-C(3)	115.25(9)
C(19)-C(4)-C(3)	111.80(9)
C(1)-C(5)-C(6)	112.07(10)
C(15)-C(10)-C(11)	117.57(11)
C(15)-C(10)-Si(1)	120.96(9)
C(11)-C(10)-Si(1)	121.47(9)
C(12)-C(11)-C(10)	121.32(11)
C(13)-C(12)-C(11)	119.97(12)
C(14)-C(13)-C(12)	119.73(12)
C(13)-C(14)-C(15)	119.91(12)
C(14)-C(15)-C(10)	121.50(12)
O(3)-C(16)-O(2)	123.33(11)
O(3)-C(16)-C(17)	126.23(12)
O(2)-C(16)-C(17)	110.43(12)
O(4)-C(19)-O(5)	123.52(11)
O(4)-C(19)-C(4)	125.32(11)
O(5)-C(19)-C(4)	111.03(9)
O(5)-C(20)-C(21)	109.24(9)
O(5)-C(20)-C(23)	115.98(9)
C(21)-C(20)-C(23)	103.51(10)
O(6)-C(21)-O(7)	123.10(11)
O(6)-C(21)-C(20)	128.98(12)
O(7)-C(21)-C(20)	107.92(10)
O(7)-C(22)-C(23)	105.60(9)
C(24)-C(23)-C(20)	112.79(10)

C(24)-C(23)-C(25)	110.67(11)
C(20)-C(23)-C(25)	111.80(10)
C(24)-C(23)-C(22)	112.07(10)
C(20)-C(23)-C(22)	98.39(9)
C(25)-C(23)-C(22)	110.60(11)

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **40**. The anisotropic displacement factor exponent takes the form: $-2p^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Si(1)	17(1)	17(1)	16(1)	-1(1)	0(1)	2(1)
O(1)	22(1)	16(1)	23(1)	0(1)	4(1)	-1(1)
O(2)	21(1)	26(1)	16(1)	-2(1)	2(1)	1(1)
O(3)	26(1)	50(1)	37(1)	-7(1)	11(1)	4(1)
O(4)	38(1)	26(1)	65(1)	-16(1)	-25(1)	13(1)
O(5)	17(1)	18(1)	25(1)	-2(1)	-5(1)	1(1)
O(6)	31(1)	61(1)	26(1)	-4(1)	7(1)	1(1)
O(7)	28(1)	44(1)	23(1)	-7(1)	-4(1)	2(1)
C(1)	18(1)	17(1)	18(1)	0(1)	-1(1)	0(1)
C(2)	14(1)	18(1)	16(1)	0(1)	0(1)	0(1)
C(3)	16(1)	18(1)	16(1)	-1(1)	-1(1)	-1(1)
C(4)	17(1)	17(1)	22(1)	-1(1)	0(1)	0(1)
C(5)	21(1)	25(1)	20(1)	1(1)	3(1)	-1(1)
C(6)	36(1)	30(1)	21(1)	4(1)	6(1)	-2(1)
C(7)	15(1)	26(1)	21(1)	-1(1)	-3(1)	-1(1)
C(8)	23(1)	23(1)	19(1)	-2(1)	-3(1)	-2(1)
C(9)	25(1)	26(1)	24(1)	-1(1)	2(1)	8(1)
C(10)	23(1)	16(1)	18(1)	0(1)	1(1)	-2(1)
C(11)	23(1)	21(1)	22(1)	-1(1)	-2(1)	-1(1)
C(12)	24(1)	28(1)	26(1)	-4(1)	5(1)	-3(1)
C(13)	36(1)	32(1)	20(1)	1(1)	6(1)	-3(1)
C(14)	32(1)	36(1)	20(1)	4(1)	-3(1)	3(1)
C(15)	22(1)	27(1)	22(1)	1(1)	0(1)	1(1)
C(16)	30(1)	26(1)	24(1)	-3(1)	8(1)	-1(1)

C(17)	48(1)	53(1)	21(1)	0(1)	10(1)	1(1)
C(18)	23(1)	20(1)	27(1)	-6(1)	0(1)	-3(1)
C(19)	20(1)	19(1)	26(1)	-2(1)	-1(1)	1(1)
C(20)	18(1)	21(1)	21(1)	2(1)	-3(1)	1(1)
C(21)	29(1)	31(1)	20(1)	2(1)	-2(1)	1(1)
C(22)	21(1)	35(1)	26(1)	-3(1)	-6(1)	2(1)
C(23)	17(1)	21(1)	22(1)	-1(1)	-3(1)	-1(1)
C(24)	23(1)	33(1)	32(1)	-7(1)	5(1)	0(1)
C(25)	24(1)	25(1)	38(1)	6(1)	-3(1)	-3(1)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^{-3}$) for 40.

	x	y	z	U(eq)
H(1)	7175(11)	1713(9)	3573(8)	11(3)
H(3)	8263(12)	1899(8)	1921(8)	10(3)
H(5A)	5174(14)	2103(11)	3855(10)	24(4)
H(5B)	4805(14)	1187(10)	3437(10)	25(4)
H(6A)	6320(15)	1282(11)	5017(11)	35(4)
H(6B)	4914(17)	1046(13)	5020(13)	43(5)
H(6C)	5849(15)	394(12)	4610(11)	37(4)
H(7A)	4667(13)	2154(10)	2180(10)	23(4)
H(7B)	5307(13)	2187(10)	1254(10)	24(4)
H(7C)	5176(14)	1239(12)	1750(11)	34(4)
H(8A)	7696(16)	3446(13)	3882(13)	44(5)
H(8B)	8387(18)	4025(14)	3206(13)	51(5)
H(8C)	8642(16)	3024(13)	3240(12)	43(5)
H(9A)	5691(16)	4580(13)	2770(12)	47(5)
H(9B)	4824(16)	3915(11)	2360(12)	36(4)
H(9C)	5148(16)	3768(12)	3336(12)	43(5)
H(11)	9127(14)	3504(10)	1543(10)	26(4)
H(12)	9732(15)	4013(11)	146(10)	26(4)
H(13)	8362(15)	4511(11)	-921(11)	32(4)

H(14)	6369(15)	4533(12)	-548(11)	37(4)
H(15)	5703(13)	4037(10)	834(9)	20(3)
H(17A)	7190(30)	2080(20)	-688(19)	101(9)
H(17B)	7570(30)	1090(20)	-814(19)	98(10)
H(17C)	8470(20)	1746(17)	-972(17)	77(7)
H(18A)	6622(15)	-37(11)	1315(11)	32(4)
H(18B)	7900(14)	-167(10)	1129(10)	24(4)
H(18C)	7324(13)	-681(10)	1881(10)	20(3)
H(20)	10448(12)	463(9)	3712(9)	16(3)
H(22A)	12452(13)	1101(10)	4294(9)	20(3)
H(22B)	12577(14)	2150(11)	4223(10)	25(4)
H(24A)	12250(14)	430(11)	2871(10)	28(4)
H(24B)	12820(18)	1339(13)	2415(14)	56(5)
H(24C)	11595(15)	930(11)	2082(11)	32(4)
H(25A)	10728(15)	2894(11)	3346(11)	33(4)
H(25B)	11814(16)	2769(11)	2717(11)	37(4)
H(25C)	10613(15)	2439(10)	2403(11)	30(4)

Table 6. Torsion angles [°] for 40.

C(4)-O(1)-C(1)-C(5)	162.98(9)
C(4)-O(1)-C(1)-C(2)	37.29(10)
O(1)-C(1)-C(2)-C(7)	79.53(10)
C(5)-C(1)-C(2)-C(7)	-39.68(13)
O(1)-C(1)-C(2)-C(3)	-38.39(10)
C(5)-C(1)-C(2)-C(3)	-157.60(10)
O(1)-C(1)-C(2)-Si(1)	-154.26(7)
C(5)-C(1)-C(2)-Si(1)	86.53(11)
C(9)-Si(1)-C(2)-C(7)	42.19(9)
C(8)-Si(1)-C(2)-C(7)	164.91(7)
C(10)-Si(1)-C(2)-C(7)	-76.71(8)
C(9)-Si(1)-C(2)-C(1)	-84.56(8)
C(8)-Si(1)-C(2)-C(1)	38.16(9)
C(10)-Si(1)-C(2)-C(1)	156.53(7)
C(9)-Si(1)-C(2)-C(3)	164.55(7)

C(8)-Si(1)-C(2)-C(3)	-72.73(8)
C(10)-Si(1)-C(2)-C(3)	45.65(8)
C(16)-O(2)-C(3)-C(2)	158.97(9)
C(16)-O(2)-C(3)-C(4)	-83.79(12)
C(7)-C(2)-C(3)-O(2)	29.81(12)
C(1)-C(2)-C(3)-O(2)	148.05(9)
Si(1)-C(2)-C(3)-O(2)	-92.18(9)
C(7)-C(2)-C(3)-C(4)	-91.59(10)
C(1)-C(2)-C(3)-C(4)	26.65(10)
Si(1)-C(2)-C(3)-C(4)	146.41(7)
C(1)-O(1)-C(4)-C(18)	-142.99(9)
C(1)-O(1)-C(4)-C(19)	99.36(9)
C(1)-O(1)-C(4)-C(3)	-19.16(11)
O(2)-C(3)-C(4)-O(1)	-125.55(9)
C(2)-C(3)-C(4)-O(1)	-6.08(10)
O(2)-C(3)-C(4)-C(18)	-6.17(13)
C(2)-C(3)-C(4)-C(18)	113.30(10)
O(2)-C(3)-C(4)-C(19)	119.99(10)
C(2)-C(3)-C(4)-C(19)	-120.55(9)
O(1)-C(1)-C(5)-C(6)	68.49(12)
C(2)-C(1)-C(5)-C(6)	-174.05(10)
C(9)-Si(1)-C(10)-C(15)	-30.87(11)
C(8)-Si(1)-C(10)-C(15)	-151.74(10)
C(2)-Si(1)-C(10)-C(15)	89.32(10)
C(9)-Si(1)-C(10)-C(11)	149.76(10)
C(8)-Si(1)-C(10)-C(11)	28.89(11)
C(2)-Si(1)-C(10)-C(11)	-90.05(10)
C(15)-C(10)-C(11)-C(12)	-0.24(17)
Si(1)-C(10)-C(11)-C(12)	179.15(9)
C(10)-C(11)-C(12)-C(13)	0.95(18)
C(11)-C(12)-C(13)-C(14)	-0.99(19)
C(12)-C(13)-C(14)-C(15)	0.3(2)
C(13)-C(14)-C(15)-C(10)	0.4(2)
C(11)-C(10)-C(15)-C(14)	-0.42(18)
Si(1)-C(10)-C(15)-C(14)	-179.82(10)
C(3)-O(2)-C(16)-O(3)	5.22(17)

C(3)-O(2)-C(16)-C(17)	-175.57(12)
C(20)-O(5)-C(19)-O(4)	-5.65(18)
C(20)-O(5)-C(19)-C(4)	170.57(9)
O(1)-C(4)-C(19)-O(4)	102.93(15)
C(18)-C(4)-C(19)-O(4)	-13.87(17)
C(3)-C(4)-C(19)-O(4)	-143.00(13)
O(1)-C(4)-C(19)-O(5)	-73.21(11)
C(18)-C(4)-C(19)-O(5)	169.99(10)
C(3)-C(4)-C(19)-O(5)	40.86(12)
C(19)-O(5)-C(20)-C(21)	-134.40(10)
C(19)-O(5)-C(20)-C(23)	109.16(11)
C(22)-O(7)-C(21)-O(6)	-177.13(13)
C(22)-O(7)-C(21)-C(20)	3.53(14)
O(5)-C(20)-C(21)-O(6)	30.25(18)
C(23)-C(20)-C(21)-O(6)	154.37(14)
O(5)-C(20)-C(21)-O(7)	-150.47(10)
C(23)-C(20)-C(21)-O(7)	-26.34(12)
C(21)-O(7)-C(22)-C(23)	20.72(14)
O(5)-C(20)-C(23)-C(24)	-86.13(13)
C(21)-C(20)-C(23)-C(24)	154.26(10)
O(5)-C(20)-C(23)-C(25)	39.34(14)
C(21)-C(20)-C(23)-C(25)	-80.27(12)
O(5)-C(20)-C(23)-C(22)	155.57(10)
C(21)-C(20)-C(23)-C(22)	35.96(11)
O(7)-C(22)-C(23)-C(24)	-153.59(11)
O(7)-C(22)-C(23)-C(20)	-34.76(12)
O(7)-C(22)-C(23)-C(25)	82.40(12)

Racemic:

Sample Name: ZPII-163-B

Results obtained with enhanced integrator!

(-)-10

Injection Date : 11/18/01 11:04:59 PM
 Sample Name : ZPIV-111-B
 Acq. Operator : peng
 Location : Vial 43
 Inj Volume : 10 µl
 Method : D:\METHODS\PENG.M
 Last changed : 11/18/01 10:33:09 PM by peng
 (modified after loading)
 2% IPA/Hexanes
 1mL/min;

Area Percent Report

Sorted By : Signal
 Multiplier : 1.0000
 Dilution : 1.0000

Signal 1: DAD1 A, Sig=250,100 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.779	BB	0.1264	28.00249	3.38897	0.1862
2	4.194	BB	0.2384	146.92838	8.46762	0.9770
3	20.042	BB	1.2618	1.48638e4	178.07959	98.8368

Totals : 1.50388e4 189.93618

Results obtained with enhanced integrator!

(4)-10

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Injection Date   : 11/18/01 11:33:55 PM
Sample Name      : 2PIV-108-C
Acq. Operator    : peng
Location         : Vial 44
Inj Volume       : 10 µl

Method           : D:\METHODS\PENG.M
Last changed      : 11/18/01 10:33:09 PM by peng
                  (modified after loading)

2% IPA/Hexanes
1ml/min;
DAD1 A, Sig=250,100 Ref=360,100 (PENG-PENG-3.D)

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Area Percent Report
-----

```

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Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000

```

Signal 1: DAD1 A, Sig=250,100 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.812	BB	0.1420	35.61209	3.70878	0.0865
2	4.237	BB	0.2554	167.44833	9.07104	0.4065
3	24.051	BB	1.3662	4.09889e4	399.10303	99.5070

Totals : 4.11920e4 411.88285

Results obtained with enhanced integrator!