Supporting Information

Quasi-Biomimetic Ring Contraction Catalyzed by a Cysteine-Based Nucleophile: Total Synthesis of Sch-642305, Some Analogs and their Putative anti-HIV Activities

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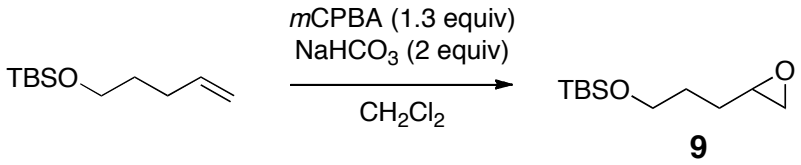
Supporting Information

I. General Procedures. Proton NMR spectra were recorded on a 400 or 500 MHz spectrometer. Proton chemical shifts were reported in ppm (δ) relative to internal tetramethylsilane (TMS, δ, 0.00 ppm), or with the solvent reference relative to TMS employed as the internal standard ($CDCl_3$, δ 7.26; d_6-DMSO, δ 2.50). Spectral data are reported as follows: chemical shift (multiplicity [singlet (s), doublet (d), triplet (t), quartet (q), and multiplet (m)], coupling constants [Hz], integration). Carbon NMR spectra were recorded on a 400 (100) or 500 (125) MHz spectrometer with complete proton decoupling. Carbon chemical shifts are reported in ppm (δ) relative to the residual solvent signal ($CDCl_3$, δ 77.0; DMSO, δ 39.5). NMR data were collected at ambient temperature unless otherwise indicated. Infrared spectra were obtained on a Nicolet 6700 FT-IR spectrometer, υ_{max} (cm^{-1}) and are partially reported. Analytical thin-layer chromatography (TLC) was performed using Silica Gel 60 Å F254 pre-coated plates (0.25 mm thickness). TLC R_f values are reported and visualization was accomplished by irradiation with a UV lamp and/or staining with cerium ammonium molybdate (CAM) or $KMnO_4$ solutions. Flash column chromatography was performed using Silica Gel 60 Å (32-63 micron). Optical rotations were recorded on a Rudolf Research Analytical Autopol IV Automatic polarimeter at the sodium D line (100 mm path length). High resolution mass spectra were acquired from the Mass Spectrometry Facility of the Univeristy of Illinois (Urbana-Champaign, IL) or at the Keck Center of Yale University. The method of ionization is given in parentheses. Chiral analytical normal phase HPLC was performed at a column temperature of 20 °C on a Hewlett-Packard 1100 Series chromatograph equipped with a diode array detector (210 nm, 230 nm or 254 nm).

All reactions were carried out under a nitrogen atmosphere employing oven- and flame-dried glassware. IBX was prepared following literature procedure.[1] (Iodomethyl)triphenylphosphium iodide was prepared following literature procedure.[2] Solvents were purified using a Seca Solvent Purification System by GlassContour unless indicated otherwise. All other chemicals were purchased commercially and used as received unless indicated otherwise. Compounds lacking hi-res mass spectra failed to ionize under positive as well as negative modes after several attempts; subsequent compounds, however, gave the correct hi-res mass spectra.

[1] Frigerio, M.; Santagostino, M.; Sputore, S. *J. Org. Chem.* **1999,** *64*, 4537-4538.
[2] Goundry, W. R. F.; Baldwin, J. E.; Lee, V. *Tetrahedron* **2003***, 59*, 1719-1729.

II. Experimental Procedures



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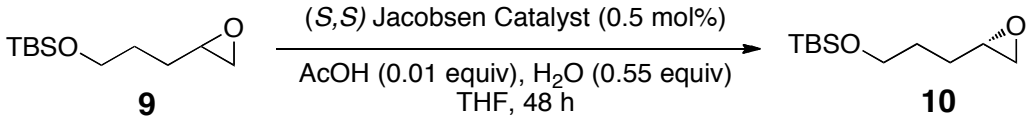
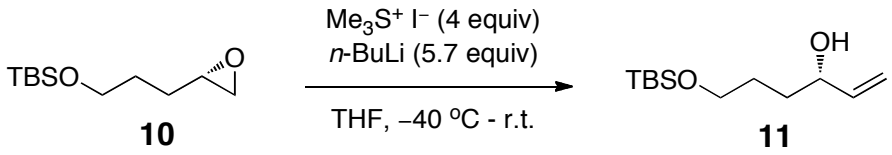
To a stirring solution of terminal alkene (47.0 g, 23.0 mmol) in CH_2Cl_2 (820 mL, 0.28 M) was added $NaHCO_3$ (38.6 g, 46.0 mol) and the reaction vessel was cooled in an ice bath. In several portions, mCPBA (52.6 g, 30.5 mmol) was added and the ice bath was removed. After stirring overnight the heterogeneous reaction mixture was vacuumed filtered through a Buchner funnel (to remove *m*-chlorobenzoic acid side-product) and rinsed five times with CH_2Cl_2 (100 mL). The organic layer was then washed once with sat. $NaHCO_3$, twice with H_2O (500 mL), dried over $NaSO_4$ and concentrated under reduced pressure to obtain the desired product as a clear liquid in quantitative yield (49.8 g, 23.0 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.72 – 3.54 (m, 2H), 3.00 – 2.89 (m, 1H), 2.75 (dd, *J* = 5.0, 4.1 Hz, 1H), 2.48 (dd, *J* = 5.0, 2.7 Hz, 1H), 1.76 – 1.49 (m, 4H), 0.89 (s, 9H), 0.04 (s, 6H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 77.3, 77.0, 76.8, 62.6, 52.2, 47.1, 29.1, 29.0, 25.9, 18.3, -5.3; **IR** (NaCl, cm^{-1}) 3040, 2954, 2925, 2889, 2852, 1471, 1409, 1389, 1360, 1250, 1205, 1102, 1005; $\mathbf{R}_f$ = 0.32 (9:1 Hexanes:EtOAc); Exact mass calc'd for $C_{11}H_{25}O_2Si$ $[M+H]^+$ requires *m/z* 217.1618. Found 217.1617 (Hi-Res ESI).



9 **10**

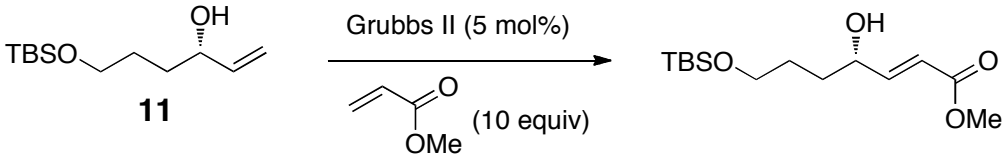
To a solution of (*S,S*)-Jacobsen's catalyst (405 mg, 0.67 mmol) in toluene (38 mL, 0.016 M) was added glacial acetic acid (154 μL, 2.68 mmol). The reaction mixture was allowed to stir open to air for 1 h after which it was concentrated under reduced pressure to yield a dark red solid. Epoxide (29.1 g, 134 mmol) was added and dissolved in THF (29 mL, 1:1 v/v epoxide/solvent). The resulting mixture was cooled in an ice bath and degassed H_2O (1.33 mL, 67.6 mmol) was added slowly. The reaction was allowed to reach room temperature and stirred for 48 h. The reaction mixture was loaded directly onto a silica gel column and chromatographed (19:1

hexanes/EtOAc) to yield the resolved epoxide as a red-brown liquid in 46% yield (13.2 g, 61.0 mmol). All spectral data are identical to the racemic epoxide; $[\alpha]_D$ –5.4 (*c* 1.0, $CHCl_3$).



10 **11**

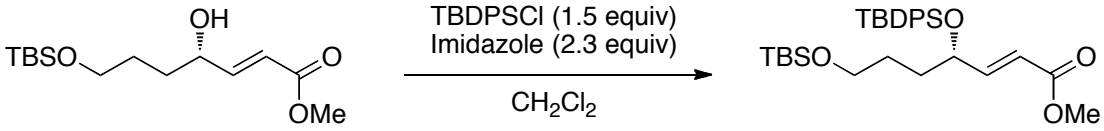
A suspension of trimethylsulfonium iodide (25.9 g, 127 mmol) in THF (500 ml) was cooled to –40° C. 2.5 M *n*-BuLi (73 mL, 183 mmol) was added slowly via addition funnel and the mixture was allowed to stir at –40° C for 30 minutes after which a 30 mL solution of epoxide (6.85 g, 31.6 mmol) in THF (0.055 M total) was added dropwise over 10 minutes. This solution was stirred at –40° C for one hour then allowed to warm to room temperature. After 12 hours the reaction was slowly quenched with water (100 mL). The resulting heterogeneous mixture was diluted with EtOAc (400 mL), and washed thrice with water (200 mL), twice with sat. NH_4Cl (200 mL), dried over Na_2SO_4, and concentrated under reduced pressure. The crude product was purified via silica gel chromatography (9:1 hexanes:EtOAc) to yield a clear oil (6.34 g, 87% yield). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 5.88 (ddd, *J* = 17.0, 10.4, 5.9 Hz, 1H), 5.24 (dt, *J* = 17.2, 1.5 Hz, 1H), 5.10 (dt, *J* = 10.4, 1.4 Hz, 1H), 4.12 (d, *J* = 9.4 Hz, 1H), 3.66 (t, *J* = 5.4 Hz, 2H), 2.58 (d, *J* = 2.1 Hz, 1H), 1.73 – 1.54 (m, 5H), 0.90 (s, 10H), 0.07 (s, 6H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 141.3, 114.3, 72.7, 63.4, 34.4, 28.8, 25.9, 18.3, -5.4; **IR** (NaCl, cm^{-1}) 3436, 2946, 2925, 2884, 2856, 1724, 1654, 1471, 1434, 1389, 1356, 1307, 1254, 1098, 1001; $\mathbf{R}_f$ = 0.38 (4:1 hexanes:EtOAc); $[\alpha]_D$ + 2.6 (*c* 1.0, $CHCl_3$). Exact mass calc'd for $[C_{12}H_{26}O_2Si]^+$ requires *m/z* 231.1775. Found 231.1772 (Hi-Res ESI).



11

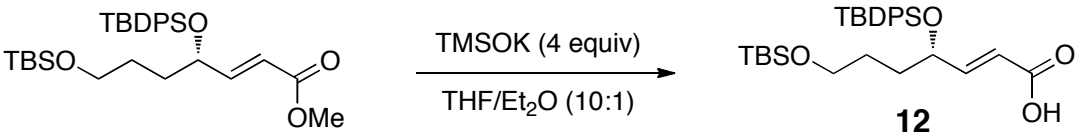
To a flame-dried round bottom flask equipped with a condenser was added allylic alcohol (100 mg, 0.434 mmol) and CH_2Cl_2 (1.1 mL, 0.4 M). To this mixture was added methyl acrylate (220 μL, 2.17 mmol) and Grubbs 2nd Generation catalyst (18.4 mg, 21.7 μmol). The reaction was stirred for one hour at room temperature and over a 30 minute period increased to 35 °C at which

point the reaction was allowed to cool back to room temperature and concentrated under reduced pressure. The crude reaction mixture was purified by silica gel chromatography (9:1 Hexanes/EtOAc) to yield a clear oil in 99% yield (124 mg, 0.43 mmol). Larger scale: To a round-bottom-flask equipped with a condenser was added allylic alcohol (5.4 g, 23.4 mmol) and CH_2Cl_2 (59.0 mL, 0.4 M). To this mixture was added methyl acrylate (21.0 mL, 234 mmol) and three portions of Grubbs 2nd Generation catalyst in 4 hr intervals (1.2 g, 1.38 mmol). The reaction mixture was then heated to 30 ^{o}C and allowed to stir overnight. After 24 hr the reaction was concentrated under reduced pressure and purified via silica gel chromatography (9:1-3:1 hexanes:EtOAc) to yield the desired product as a clear oil in 80% yield (5.4 g, 18.7 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.95 (dd, *J* = 15.6, 4.4 Hz, 1H), 6.09 (dd, *J* = 15.6, 1.8 Hz, 1H), 4.34 (m, 1H), 3.74 (s, 3H), 3.67 (t, *J* = 4.7 Hz, 2H), 3.59 (d, *J* = 4.4 Hz, 1H), 1.82 (m, 1H), 1.66 (m, 3H), 0.90 (s, 9H), –0.08 (s, 6H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 167.5, 151.1, 120.1, 70.9, 51.9, 34.8, 29.0, 26.3, 18.7, -5.1; **IR** (NaCl, cm^{-1}) 3440, 2953, 2925, 2892, 2856, 1724, 1704, 1654, 1471, 1434, 1381, 1307, 1258, 1197, 1164, 1095; $\mathbf{R}_f$ = 0.31 (3:1 Hexanes:EtOAc); $\mathbf{[\alpha]_D}$ –0.25 (*c* 1.0, $CHCl_3$, 95% ee). Exact mass calc'd for $C_{14}H_{29}O_4Si$ $[M+H]^+$ requires *m/z* 289.1835. Found 289.1835 (Hi-Res ESI); Assay of enantiomeric purity: Enantiomers of product were separated by chiral HPLC employing a Chiralcel OD column (Daicel). Conditions: 96:4 hexanes/isopropanol; Flow rate 0.75 mL/min; 12.2 min (major ent), 14.3 min (minor ent).



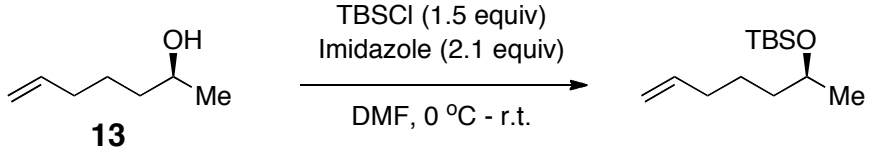
A solution of alcohol (10.09 g, 34.9 mmol) in CH_2Cl_2 (175 mL, 0.2 M) was cooled in an ice bath. Imidazole (5.5 g, 80.3 mmol) and TBDPSCl (13.6 mL, 52.5 mmol) were added to the reaction mixture and allowed to stir for 3.5 hours. The reaction was quenched with H_2O (200 mL) and further diluted with CH_2Cl_2 (300 mL). The layers were separated and the aqueous layer was extracted twice with CH_2Cl_2 (200 mL). The combined organic layers were washed once with sat. NH_4Cl (200 mL), H_2O (200 mL), brine (200 mL), dried over Na_2SO_4 and concentrated under reduced pressure. The crude product was purified via silica gel chromatography (19:1 Hex/EtOAc) to yield a pale yellow oil in 92% yield (16.9 g, 32.1 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.68 – 7.58 (m, 4H), 7.44 – 7.32 (m, 6H), 6.87 (dd, *J* = 15.6, 5.1 Hz, 1H), 5.92 (dd, *J* =

15.6, 1.5 Hz, 1H), 4.39 (dt, *J* = 6.2, 3.1 Hz, 1H), 3.72 (s, 3H), 3.50 – 3.37 (m, 2H), 1.53 – 1.34 (m, 4H), 1.08 (s, 9H), 0.85 (s, 9H), -0.02 (d, *J* = 2.4 Hz, 6H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 166.9, 150.3, 135.8, 133.8, 133.4, 129.8, 129.7, 127.6, 127.6, 119.9, 72.2, 51.5, 33.2, 27.3, 27.0, 25.9, 19.3, 18.3, -5.4; **IR** (NaCl, cm^{-1}) 3072, 3044, 2954, 2929, 2889, 2852, 1724, 1659, 1471, 1426, 1389, 1356, 1299, 1254, 1193, 1160, 1099; $\mathbf{R}_f$ = 0.69 (4:1 hexanes:EtOAc); **[α]$_D$** –17.1 (*c* 1.0, $CHCl_3$). Exact mass calc'd for $C_{30}H_{47}O_4Si_2$ $[M+H]^+$ requires *m/z* 549.2827. Found 549.2871 (Hi-Res ESI).



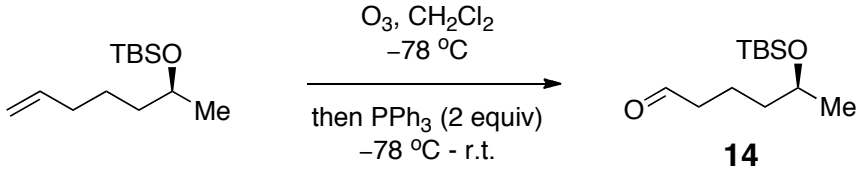
12

To a solution of methyl ester (1.0 g, 1.9 mmol) in THF/Et_2O (10:1 v/v, 0.037 M) was added TMSOK (974 mg, 7.6 mmol) in two portions. The reaction was allowed to stir overnight after which it was quenched with sat. $KHSO_4$ (25 mL) until pH 6. The layers were separated and the aqueous layer was extracted twice with EtOAc (50 mL). The combined organic layers were washed twice with brine (50 mL), dried over Na_2SO_4, and concentrated under reduced pressure. The crude material was purified via silica gel chromatography (4:1 hexanes:EtOAc) to yield a clear oil in quantitative yield (1.0 g, 1.9 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 11.76 (s, 1H) 7.67, 7.62 (dd, *J* = 7.9, 1.4 Hz, 4H), 7.44-7.36 (m, 6H), 6.98 (dd, *J* = 15.5, 5.0 Hz, 1H), 5.93 (dd, *J* = 15.6, 1.4 Hz, 1H), 4.45-4.42 (m, 1H), 3.52-3.40 (m, 2H), 1.53-1.40 (m, 4H), 1.10 (s, 9H), 0.86 (s, 9H), 0.0 (s, 3H), –0.01 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 171.69, 152.89, 135.84, 133.79, 133.35, 129.87, 127.68, 119.60, 72.17, 72.13, 62.97, 33.16, 27.30, 27.06, 25.97, 19.39, 18.34, –5.30; **IR** (NaCl, cm^{-1}) 3072, 3048, 2958, 2925, 2893, 2856, 2733, 2680, 2570, 1957, 1883, 1818, 1691, 1650, 1585, 1467, 1422, 1385, 1352, 1307, 1250, 1193, 1111, 1001; $\mathbf{R}_f$ = 0.27 (3:1 hexanes:EtOAc); **[α]$_D$** –14.6 (*c* 1.0, $CHCl_3$). Exact mass calc'd for $C_{29}H_{44}NaO_4Si_2$ $[M+Na]^+$ requires *m/z* 535.2670. Found 535.2690 (Hi-Res ESI).



13

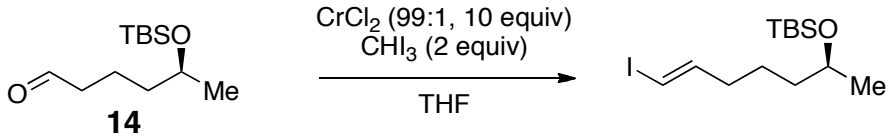
Alcohol (4.0 g, 35.0 mmol) was dissolved in DMF (175 mL, 0.2 M) and cooled in an ice bath. Imidazole (4.77g, 75.0 mmol) was added followed by TBSCl (7.91g, 52.5 mmol). The reaction was stirred in the ice bath for 30 minutes and then allowed to stir at room temperature for 2 hours. The reaction was quenched with H_2O (20 mL) and then additional H_2O (200 mL) was added. The aqueous layer was extracted thrice with Et_2O (200 mL), and the combined organic layers were washed thrice with H_2O (200 mL), once with brine (200 mL), dried over $MgSO_4$ and concentrated under reduced pressure. The crude product was purified via silica gel chromatography (9:1 hexanes/Et_2O) to yield a pale yellow liquid in 95% yield (7.56 g, 33.3 mmol). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 5.81 (ddt, J = 16.9, 10.2, 6.6 Hz, 1H), 5.00 (ddd, J = 17.1, 3.5, 1.6 Hz, 1H), 4.97 – 4.91 (m, 1H), 3.83 –3.74 (m, 1H), 2.04 (q, J = 6.6 Hz, 2H), 1.52 – 1.32 (m, 4H), 1.12 (d, J = 6.1 Hz, 3H), 0.9 (s, 9H), 0.0 (s, 6H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 139.0, 114.3, 68.5, 39.2, 33.8, 25.9, 25.1, 23.8, 18.2, –4.4, –4.7; **IR** (NaCl, cm^{-1}) 3081, 2954, 2929, 2897, 2860, 1822, 1638, 1471, 1458, 1409, 1368, 1364, 1250, 1131, 1091, 1054, 1038, 1001; $\mathbf{R}_f$ = +0.81 (9:1 hexanes:Et_2O); $[\alpha]_D$ +11.5 (*c* 1.0, $CHCl_3$).



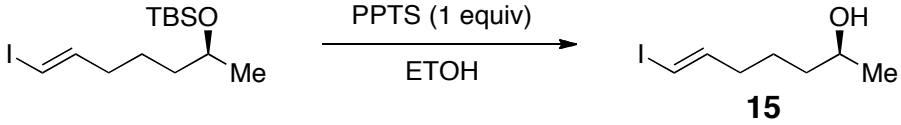
14

A solution of terminal alkene (2.0 g, 8.75 mmol) dissolved in CH_2Cl_2 (44 mL, 0.2 M) was cooled in a –78° C bath and stirred for at least 10 minutes. Ozone was added until the reaction mixture turned light blue and then immediately purged with N_2 until the solution became clear again. In one portion was added PPh_3 (4.59 g, 17.5 mmol) and the cooling bath was removed. After 2 hours of stirring the reaction mixture was concentrated and purified via silica gel chromatography (9:1 hexanes: Et_2O) to yield a clear liquid in quantitative yield (2.02 g, 8.75 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.76 (t, J = 1.8 Hz, 1H) 3.83-3.78 (m, 1H) 2.43 (dt, J =7.3, 1.8 Hz, 2H) 1.73-1.56 (m, 2H) 1.47-1.40 (m, 2H) 1.13 (d, J = 6.1 Hz, 3H) 0.88 (s, 9H) 0.05 (d, J = 1.9 Hz, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 202.7, 68.1, 43.9, 38.9, 25.9, 25.6, 23.7, 18.3, 18.1, -4.4, -4.8; **IR** (NaCl, cm^{-1}) 2954, 2925, 2884, 2815, 2713, 1728, 1467, 1462, 1409, 1373, 1356, 1250, 1185, 1136, 1099, 1001; $\mathbf{R}_f$ = 0.39 (9:1 hexanes:Et_2O); $[\alpha]_D$ +13.4 (*c* 1.0,

$CHCl_3$). Exact mass calc'd for $C_{12}H_{27}O_2Si$ $[M+H]^+$ requires *m/z* 231.1774. Found 230.1772 (Hi-Res ESI).

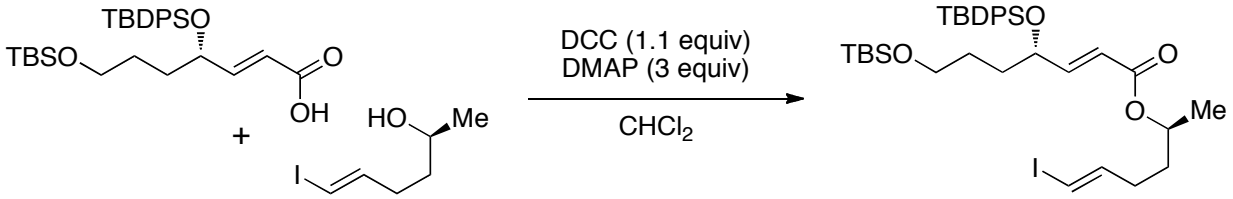


In a glovebox was added $CrCl_2$ (533 mg, 4.34mmol) to a 100 mL round bottom flask. The flask was removed from the glovebox and kept under an inert atmosphere. To this round bottom flask was added THF (0.90 mL) followed by a mixture of aldehyde (100 mg, 0.434mmol) and iodoform (342 mg, 0.868 mmol) in THF (7.2 mL) via cannula to yield a dark brown solution. The reaction was allowed to stir at room temperature for 2 hours and then diluted with Et_2O (50 mL). The solution was washed once with H_2O (50 mL), the layers were separated and the aqueous layer was extracted twice with Et_2O (50 mL). The combined organic layers were washed twice with brine (50 mL), dried over Na_2SO_4 and concentrated. The crude product was purified via silica gel chromatography (49:1, hexanes → EtOAc) to yield the desired product in 64% yield (99 mg, 0.28 mmol) as a 5:1 ratio of E/Z olefins. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.20-6.13 (m, 2H) 3.83-3.76 (m, 1H) 2.16-2.11 (m, 2H) 1.55-1.38 (m, 4H) 1.12 (d, *J* = 6.0 Hz, 3H) 0.89 (s, 9H) 0.05 (s, 6H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 141.3, 82.3, 68.3, 39.0, 34.7, 25.9, 24.1, 23.8, 18.1, –4.4, –4.7; **IR** (NaCl, cm^{-1}) 3060, 2954, 2925, 2893, 2852, 2729, 2705, 2643, 1605, 1471, 1458, 1405, 1368, 1356, 1270, 1250, 1213, 1131, 1091, 1033, 1001; $\mathbf{R_f}$ = 0.77 (19:1 hexanes:Et_2O); $\mathbf{[\alpha]_D}$ +5.6 (*c* 1.0, $CHCl_3$).

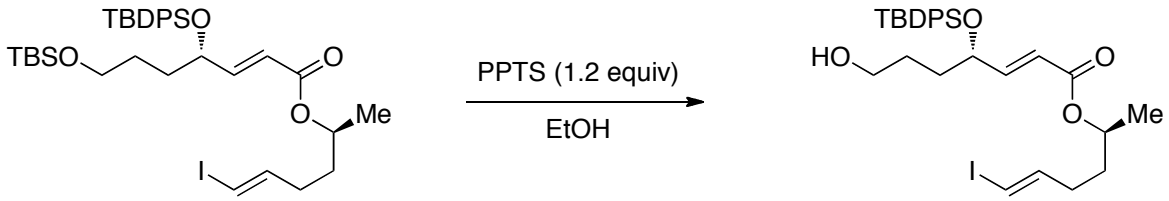


The protected alcohol (68 mg, 0.195 mmol) was dissolved in EtOH (1.95 mL, 0.10 M) and to this solution was added PPTS (49 mg, 0.195 mmol) and allowed to stir overnight. The mixture was diluted with EtOAc (20 mL), washed thrice with saturated $NaHCO_3$ (20 mL), washed once with brine (20 mL), dried over Na_2SO_4 and concentrated under reduced pressure. The crude material was purified via silica gel chromatography to yield a clear oil in quantitative yield (47 mg, 0.195 mol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.51 (dt, *J* = 7.2, 14.3, 1H), 6.00 (dt, *J* = 1.3,

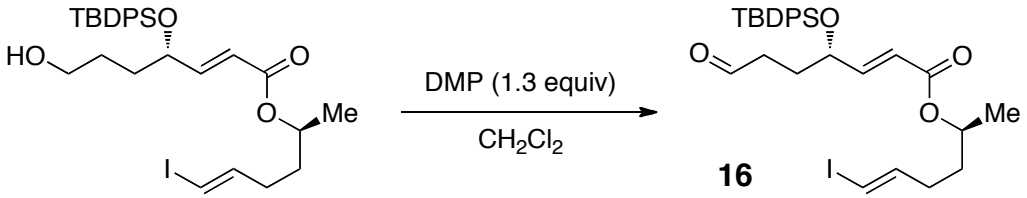
14.3, 1H), 3.79 (m, 1H), 2.12 – 2.04 (m, 2H), 1.60 – 1.38 (m, 4H), 1.19 (d, J = 6.2, 3H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 146.27, 74.75, 67.85, 38.42, 35.93, 24.52, 23.61; **IR** (NaCl, cm^{-1}) 3354, 2962, 2929, 2852, 1601, 1454, 1368, 1274, 1123, 1078; **R_f** = 0.37 (2:1 hexanes:EtOAc); **$[\alpha]_D$** +5.6 (*c* 1.0, $CHCl_3$). Exact mass calc'd for $[C_7H_{13}IONa]^+$ requires *m/z* 262.9903. Found 262.9903 (Hi-Res ESI).



To a stirring solution of carboxylic acid (630 mg, 1.23 mmol) and secondary alcohol (340 mg, 1.42 mmol) in CH_2Cl_2 (6.2 mL, 0.2 M) was added DMAP (520 mg, 4.26 mmol). After cooling the reaction mixture in an ice bath a 1 mL solution (CH_2Cl_2) of DCC (280 mg, 1.35 mmol) was slowly added to the reaction mixture upon which it was warmed to room temperature and stirred for 12 hours. The reaction was diluted with CH_2Cl_2 (20 mL), washed once with H_2O (20 mL), twice with brine (20 mL), dried over Na_2SO_4 and concentrated under reduced pressure. The crude product was purified by silica gel chromatography (19:1 hexanes:EtOAc) to obtain a clear oil in 91% yield (820 mg, 1.12 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.63 (dd, J = 19.7, 6.7 Hz, 4H), 7.39 (dq, J = 15.6, 7.3 Hz, 7H), 6.82 (dd, J = 15.6, 5.3 Hz, 1H), 6.49 (dt, J = 14.3, 7.1 Hz, 1H), 6.00 (d, J = 14.4 Hz, 1H), 5.82 (dd, J = 15.6, 1.2 Hz, 1H), 4.92 (dt, J = 12.9, 6.5 Hz, 1H), 4.38 (d, J = 4.7 Hz, 1H), 3.47 (tt, J = 16.7, 8.3 Hz, 2H), 2.06 (dd, J = 14.3, 7.1 Hz, 2H), 1.65 – 1.33 (m, 8H), 1.23 (d, J = 6.2 Hz, 3H), 1.08 (s, 9H), 0.85 (s, 9H), –0.01 (d, J = 2.1 Hz, 6H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 166.1, 149.7, 146.0, 135.8, 135.8, 133.8, 133.5, 129.8, 129.7, 127.6, 127.5, 120.7, 74.9, 72.3, 70.4, 63.0, 35.7, 35.2, 34.9, 33.3, 27.4, 27.0, 25.9, 25.4, 24.7, 24.2, 20.0, 19.3, 18.3, -5.3, -5.3; **IR** (NaCl, cm^{-1}) 3068, 3044, 2934, 2856, 2733, 2705, 2116, 2067, 1953, 1892, 1822, 1712, 1659, 1601, 1586, 1467, 1430, 1360, 1262, 1164, 1099, 1001; **R_f** = 0.68 (4:1 hexanes:Et_2O); **$[\alpha]_D$** –14.1 (*c* 1.0, $CHCl_3$).



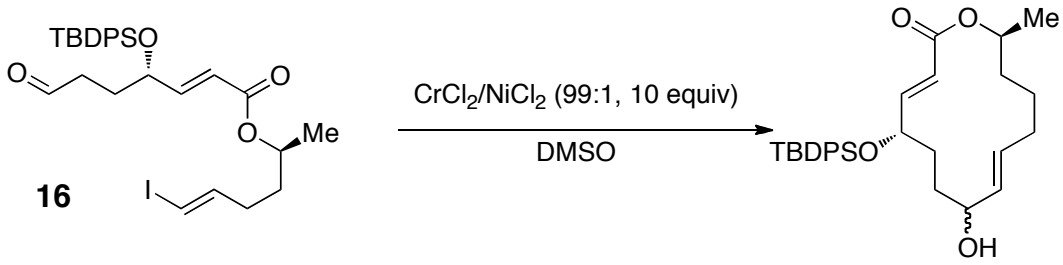
A stirring solution of the TBS protected compound (780 mg, 1.06 mmol) in ethanol (10.6 mL, 0.01 M) was cooled in an ice bath. After cooling in an ice bath for 10 minutes was added PPTS (319 mg, 1.27 mmol). The reaction was slowly warmed to room temperature and allowed to stir for 36 hours. The reaction mixture was diluted with EtOAc (25 mL), washed twice with sat. $NaHCO_3$ (25 mL), once with brine (25 mL), dried over Na_sSO_4 and concentrated under reduced pressure. The crude material was purified via silica gel chromatography to obtain a clear oil in 83% yield (548 mg, 0.88 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.67, 7.61 (dd, *J* = 7.9, 1.3 Hz, 1H), 7.47 – 7.31 (m, 6H), 6.82 (dd, *J* = 15.6, 5.3 Hz, 1H), 6.49 (dt, *J* = 14.3, 7.1 Hz, 1H), 6.01 (d, *J* = 14.4 Hz, 1H), 5.84 (dd, *J* = 15.6, 1.4 Hz, 1H), 4.93 (dq, *J* = 12.4, 6.2 Hz, 1H), 4.42 (q, *J* = 3.9 Hz, 1H), 3.54 – 3.43 (m, 2H), 2.10 – 2.02 (m, 2H), 1.66 – 1.33 (m, 8H), 1.30 (t, *J* = 5.5 Hz, 1H), 1.23 (d, *J* = 6.2 Hz, 3H), 1.09 (s, 9H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 166.0, 149.4, 146.0, 135.8, 135.8, 133.7, 133.3, 129.8, 129.8, 127.6, 127.6, 120.9, 74.9, 72.1, 70.4, 62.6, 35.7, 35.1, 33.0, 27.2, 27.0, 24.1, 20.0, 19.3; **IR** (NaCl, cm^{-1}) 3432, 3068, 3045, 2934, 2856, 1961, 1892, 1822, 1712, 1655, 1605, 1471, 1426, 1356, 1275, 1168, 1111, 1082, 1054; $\mathbf{R}_f$ = 0.44 (2:1 hexanes:EtOAc); $[\alpha]_D$ –22.6 (*c* 1.0, $CHCl_3$). Exact mass calc'd for $C_{30}H_{42}IOSi$ $[M+H]^+$ requires *m/z* 621.1892 Found 621.1883 (Hi-Res ESI).



To a stirring solution of alcohol (250 mg, 0.403 mmol) in CH_2Cl_2 (5.0 mL, 0.08 M) was added Dess-Martin periodinane[3] (222 mg, 0.52 mmol). After stirring for 3 h, the reaction was concentrated and purified via silica gel chromatography (4:1 hexanes:EtOAc) to give the desired product in 82% yield (205 mg, 0.33 mmol) as a clear oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.62 (t, *J* = 1.3 Hz, 1H), 7.69 – 7.63 (m, 2H), 7.63 – 7.57 (m, 2H), 7.49 – 7.31 (m, 6H), 6.75 (dd, *J* =

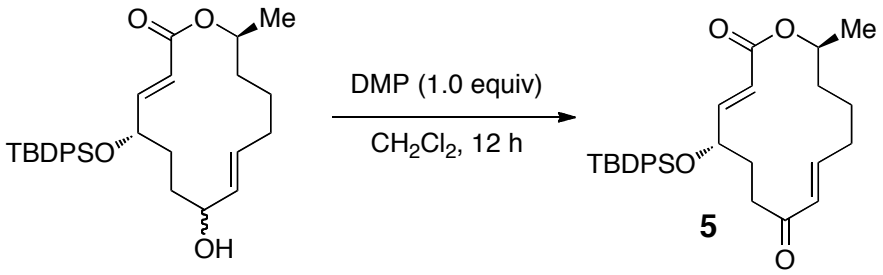
[3] D. B. Dess, J. C. Martin *J. Org. Chem.* 1983, ***48***, 4155.

15.6, 5.2 Hz, 1H), 6.49 (dt, *J* = 14.3, 7.1 Hz, 1H), 6.01 (dt, *J* = 14.4, 1.4 Hz, 1H), 5.85 (dd, *J* = 15.6, 1.5 Hz, 1H), 4.98 – 4.88 (m, 1H), 4.48 (td, *J* = 5.8, 1.3, 1H), 2.54 – 2.31 (m, 2H), 2.11 – 2.01 (m, 2H), 1.87 – 1.71 (m, 2H), 1.63 – 1.34 (m, 4H), 1.23 (d, *J* = 6.3 Hz, 3H), 1.09 (s, 9H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 201.5, 165.8, 148.6, 146.0, 135.8, 135.7, 133.4, 133.1, 130.0, 129.9, 127.8, 127.7, 121.6, 74.9, 71.1, 70.6, 38.4, 35.7, 35.1, 28.6, 27.0, 24.1, 20.0, 19.3; **IR** (NaCl, cm^{-1}) 3072, 3044, 2929, 2889, 2856, 2717, 1715, 1646, 1474, 1425, 1360, 1270, 1107, 1078; $\mathbf{R}_f$= 0.51 (3:1 hexanes:EtOAc); $\mathbf{[\alpha]_D}$ –26.4 (*c* 1.0, $CHCl_3$). Exact mass calc'd for $C_{30}H_{40}IO_4Si$ $[M+H]^+$ requires *m/z* 619.1735. Found 619.1733 (Hi-Res ESI).



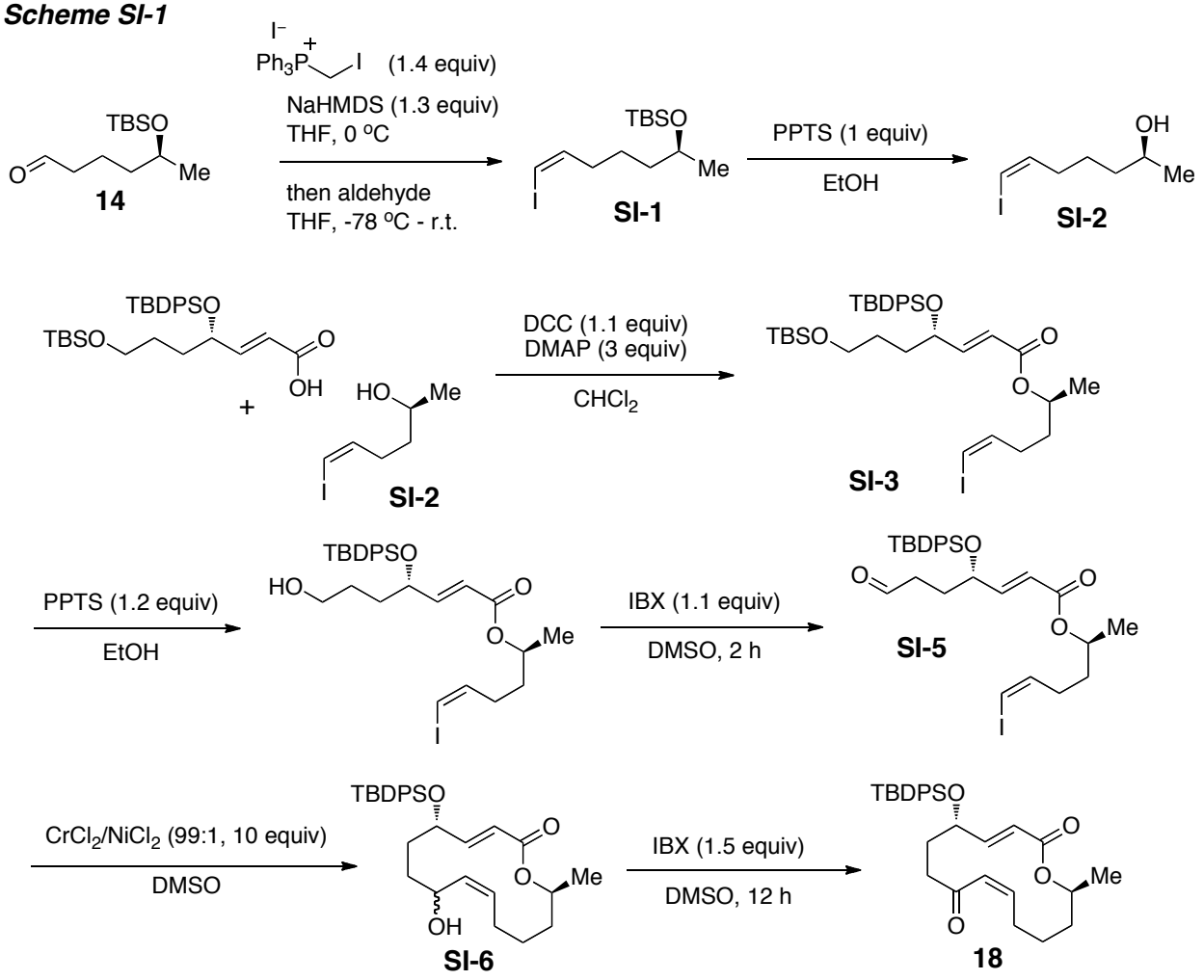
In a glovebox was added $CrCl_2/NiCl_2$ (99:1, 973 mg, 7.11 mmol) portionwise to anhydrous DMSO (350 mL) to produce a pale green color. The reaction vessel was capped with a septum, removed from the glovebox, and immediately put under an inert atmosphere (N_2) after which it was stirred for 30 minutes at room temperature (to further dissolve the $CrCl_2/NiCl_2$ mixture). A 20 mL solution of substrate (440 mg, 0.711 mmol) was added via cannula (370 mL total, 2 mM) and the reaction was allowed to stir at room temperature for 24 hours until it was dark green and complete by TLC. The reaction was diluted with EtOAc (300 mL), and water (30 mL) was slowly added to quench the reaction. Additional water (200 mL) was added and the layers were separated. The aqueous layer was extracted once with EtOAc (250 mL). The combined organic layers were washed twice with water (250 mL), dried over Na_2SO_4, and concentrated under reduced pressure. The crude product was purified by silica gel (5:1-2:1 Hex/EtOAc) to yield a 1:1 ratio of α/β isomers in 74% yield as a white foam (260 mg, 0.53 mmol). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.61 (d, *J* = 6.7 Hz, 4H), 7.53 (d, *J* = 7.3 Hz, 4H), 7.37-7.27 (m, 12H), 6.76 (dd, *J* = 15.6, 3.4, 2H), 6.10 (dd, *J* = 15.5, 1.9, 1H), 6.01 (dd, *J* = 15.6, 1.8, 1H), 5.63 (m, 1H), 5.55 (m, 1H), 5.34 (m, 2H), 5.05 (m, 1H), 4.95 (m, 1H), 4.44 (m, 1H), 4.38 (m, 1H), 4.10 (m, 1H), 3.85 (m, 1H), 2.01-1.85 (m, 4H) 1.66-1.41 (m, 16H), 1.29 (m, 2H), 1.21 (d, *J* = 6.3 Hz, 3H), 1.53 (d, *J* = 6.5 Hz, 3H), 1.26 (s, 9H), 1.02 (s, 9H); **^{13}C NMR** (126 MHz, $CDCl_3$) 166.6, 166.5, 152.0,

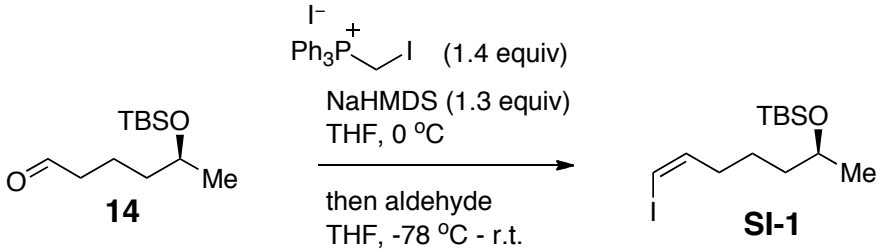
151.6, 136.2, 136.2, 136.1, 134.3, 134.3, 133.7, 133.6, 133.4, 133.1, 132.8, 132.7, 130.3, 130.2, 128.1, 120.9, 120.6, 72.9, 72.2, 71.8, 71.8, 70.7, 70.4, 34.3, 33.2, 30.9, 30.7, 30.5, 30.5, 30.4, 30.1, 29.9, 27.5, 27.4, 24.7, 23.7, 20.7, 19.8, 19.8, 19.5; **IR** (NaCl, cm^{-1}) 3436,m 3072, 3044, 2934, 2856, 1961, 1887, 1826, 1708, 1646, 1585, 1471, 1454, 1426, 1389, 1356, 1254, 1189, 1111, 1074, 1001; $\mathbf{R}_f$ = 0.30 (2:1 hexanes:EtOAc); $[\alpha]_D$ +15.0 (*c* 1.0, $CHCl_3$). Exact mass calc'd for $C_{30}H_{40}NaO_4Si$ $[M+Na]^+$ requires *m/z* 515.2588. Found 515.2565 (Hi-Res ESI).



Allylic alcohol (236 mg, 0.480 mmol) was dissolved in CH_2Cl_2 (6 ml, 0.08 M) and cooled in an ice bath. Dess-Martin periodinane (204 mg, 0.480 mmol) was added and the mixture was stirred and allowed to reach room temperature. Once complete by TLC the reaction mixture was concentrated and purified by silica gel chromatography (10:1 hexanes/EtOAc) to yield a clear solid in 81% yield (191 mg, 0.389 mmol), which was crystallized from 10:1 hexanes/EtOAc. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.63 (m, 4H), 7.49 – 7.31 (m, 6H), 6.72 (dt, *J* = 15.6, 7.1 Hz, 1H), 6.59 (dd, *J* = 15.6, 5.5 Hz, 1H), 6.05 (d, *J* = 15.7 Hz, 1H), 5.89 (dd, *J* = 15.6, 1.4 Hz, 1H), 5.10 – 4.97 (m, 1H), 4.33 (ddd, *J* = 7.1, 5.7, 2.8 Hz, 1H), 2.46 (ddd, *J* = 15.0, 8.5, 2.3 Hz, 1H), 2.29 – 2.19 (m, 1H), 2.19 – 2.08 (m, 2H), 2.08 – 1.97 (m, 1H), 1.91 – 1.80 (m, 1H), 1.76 – 1.52 (m, 4H), 1.20 (d, *J* = 6.5 Hz, 3H), 1.08 (s, 9H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 200.4, 165.4, 148.9, 147.3, 135.8, 133.8, 133.2, 131.4, 129.9, 129.8, 127.7, 127.7, 121.9, 72.5, 70.5, 34.6, 32.6, 31.9, 30.9, 27.0, 22.8, 19.3; **IR** (NaCl, cm^{-1}) 3072, 2933, 2856, 1720, 1691, 1667, 1471, 1426, 1368, 1291, 1258, 1205, 1189, 1160, 1107, 1070, 980; $\mathbf{R}_f$ = 0.31 (4:1 hexanes:EtOAc); $[\alpha]_D$ –8.4 (*c* 1.0, $CHCl_3$). **Mp** = 94-96 ^oC. Exact mass calc'd for $C_{30}H_{39}O_4Si$ $[M+H]^+$ requires *m/z* 491.2618. Found 491.2607 (Hi-Res ESI).

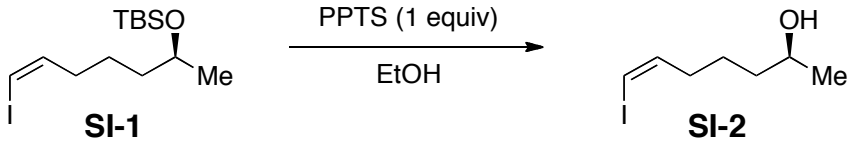
Scheme SI-1





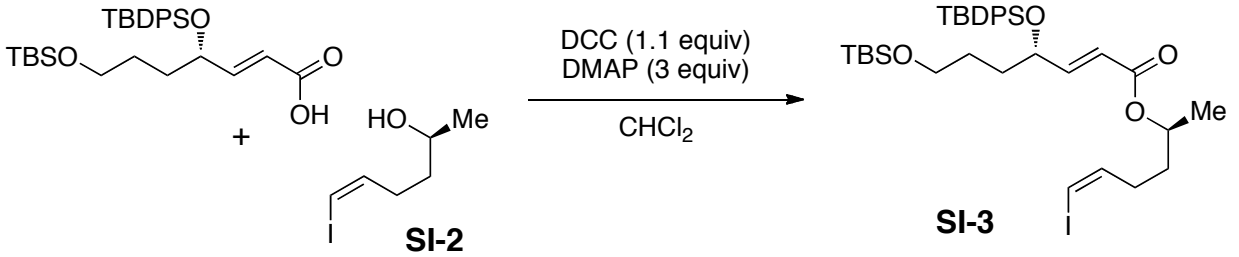
A suspension of the Wittig salt (6.51 g, 12.3 mmol) in THF (100 mL, 0.12 M) was cooled in an ice bath. NaHMDS (11.4 mL, 1 M THF) was added slowly and the resulting orange solution was allowed to stir for 20 minutes after which it was cooled in a –78°C bath. A THF solution (35 mL, 0.25 M) of aldehyde (2.02 g, 8.77 mmol) was added slowly and the reaction was allowed to stir overnight while slowly warming to room temperature. The dark orange solution was cooled in an ice bath and quenched with sat. NH_4Cl (50 mL). The resulting mixture was

extracted twice with Et_2O (200 mL) and the combined organic layers were dried over $MgSO_4$ and concentrated under reduced pressure. The crude orange material was purified via silica gel chromatography (49:1 hexanes:Et_2O) to yield a pale yellow liquid in 65% yield (1.95 g, 5.50 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.20-6.13 (m, 2H) 3.83-3.76 (m, 1H) 2.16-2.11 (m, 2H) 1.55-1.38 (m, 4H) 1.12 (d, *J* = 6.0 Hz, 3H) 0.89 (s, 9H) 0.05 (s, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 141.3, 82.3, 68.3, 39.0, 34.7, 25.9, 24.1, 23.8, 18.1, –4.4, –4.7; **IR** (NaCl, cm^{-1}) 3060, 2954, 2925, 2893, 2852, 2729, 2705, 2643, 1605, 1471, 1458, 1405, 1368, 1356, 1270, 1250, 1213, 1131, 1091, 1033, 1001; $\mathbf{R}_f$ = 0.77 (19:1 hexanes:Et_2O); $[\alpha]_D$ +5.6 (*c* 1.0, $CHCl_3$).



SI-1 **SI-2**

The protected alcohol (3.87 g, 10.9 mmol) was dissolved in EtOH (110 mL, 0.10 M) and to this solution was added PPTS (2.7 g, 10.9 mmol) and allowed to stir overnight. Upon completion by TLC the mixture was diluted with 200 mL Et_2O, washed thrice with sat. $NaHCO_3$ (150 mL), washed once with brine (150 mL), dried over Na_2SO_4 and concentrated under reduced pressure. The crude material was purified via silica gel chromatography to yield a clear oil in 85% yield (2.05 g). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.22-6.15 (m, 2H) 3.86-3.80 (m, 1H) 2.20-2.15 (m, 2H) 1.60-1.44 (m, 4H) 1.29 (d, *J* = 4.8 Hz, 1H) 1.20 (d, *J* = 6.2 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 141.99, 82.65, 67.86, 38.56, 34.54, 24.14, 23.58; **IR** (NaCl, cm^{-1}) 3346, 2966, 2925, 2856, 1610, 1454, 1368, 1283, 1123, 1082, 1005; $\mathbf{R}_f$ = 0.37 (2:1 hexanes:EtOAc); $[\alpha]_D$ +2.5 (*c* 1.0, $CHCl_3$). Exact mass calc'd for $C_7H_{13}INaO$ $[M+Na]^+$ requires *m/z* 262.9903. Found 262.9903 (Hi-Res).



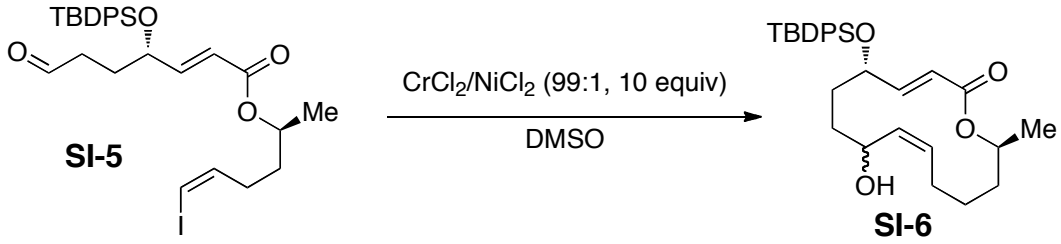
SI-2 **SI-3**

To a stirring solution of carboxylic acid (1.32 g, 2.6 mmol) and secondary alcohol (655 mg, 2.73 mmol) in CH_2Cl_2 (13 mL, 0.2 M) was added DMAP (952 mg, 7.8 mmol). After cooling the

reaction mixture in an ice bath, a solution of DCC (590 mg, 2.86 mmol) in CH_2Cl_2 (1 mL) was slowly added. The reaction was stirred for 12 hours while warming to room temperature and diluted with CH_2Cl_2 (20 mL). The organic layer was washed with H_2O (30 mL), twice with brine (40 mL), dried over Na_2SO_4 and concentrated under reduced pressure. The crude product was purified by silica gel chromatography (19:1 hexanes:EtOAc) to obtain a clear oil in 84% yield (1.60 g, 2.18 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.69 – 7.58 (m, 4H), 7.46 – 7.30 (m, 6H), 6.82 (dd, *J* = 15.6, 5.4 Hz, 1H), 6.18 (dt, *J* = 20.7, 7.1 Hz, 2H), 5.83 (dd, *J* = 15.6, 1.4 Hz, 1H), 5.01 – 4.89 (m, 1H), 4.38 (dd, *J* = 9.9, 4.8 Hz, 1H), 3.46 (pd, *J* = 10.2, 6.1 Hz, 2H), 2.16 (q, *J* = 6.7 Hz, 2H), 1.69 – 1.29 (m, 10H), 1.24 (d, *J* = 6.2 Hz, 3H), 1.08 (s, 9H), 0.86 (s, 9H), -0.01 (d, *J* = 2.2 Hz, 6H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 166.1, 149.6, 140.8, 135.9, 135.8, 133.9, 133.6, 129.8, 129.7, 127.6, 127.6, 120.9, 82.8, 77.3, 77.2, 77.0, 76.8, 72.4, 70.6, 63.0, 55.8, 35.4, 34.9, 34.4, 33.4, 27.5, 27.1, 26.0, 25.5, 24.7, 23.8, 20.0, 19.4, 18.3, -5.3, –5.3; **IR** (NaCl, cm^{-1}) 3068, 3044, 2934, 2856, 2733, 2705, 2116, 2067, 1953, 1892, 1822, 1712, 1659, 1601, 1586, 1467, 1430, 1360, 1262, 1164, 1099, 1001; $\mathbf{R}_f$ = 0.68 (4:1 hexanes:EtOAc); $[\alpha]_D$ –13.5 (*c* 1.0, $CHCl_3$).

The alcohol (1.3 g, 2.09 mmol) was dissolved in DMSO (21.0 mL, 0.1 M) and stirred at room temperature. To this solution was added IBX (645 mg, 2.30 mmol) and the reaction was allowed to stir for two hours at which point the reaction was diluted with EtOAc (100 mL) and washed thrice with H_2O (50 mL). The organic layer was dried over Na_2SO_4 and concentrated under reduced pressure. The crude material was purified via silica gel chromatography (4:1 hexanes:EtOAc) to yield the desired product in 83% yield as a clear oil (1.07 g, 1.73 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.62 (t, *J* = 1.3 Hz, 1H), 7.69 – 7.56 (m, 4H), 7.48 – 7.31 (m, 6H), 6.75 (dd, *J* = 15.6, 5.2 Hz, 1H), 6.24 – 6.20 (m, 1H), 6.15 (dd, *J* = 13.8, 6.8 Hz, 1H), 5.85 (dd, *J* = 15.6, 1.5 Hz, 1H), 5.01 – 4.89 (m, 1H), 4.47 (q, *J* = 4.6 Hz, 1H), 2.54 – 2.42 (m, 1H), 2.42 – 2.30 (m, 1H), 2.21 – 2.11 (m, 2H), 1.80 (m, 2H), 1.71 – 1.34 (m, 4H), 1.24 (d, *J* = 6.2 Hz, 3H), 1.09 (s, 9H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 201.5, 165.7, 148.4, 140.7, 135.8, 135.7, 133.4,

133.0, 130.0, 130.0, 127.7, 127.6, 121.6, 82.8, 71.1, 70.7, 38.3, 35.3, 34.4, 28.6, 27.0, 27.0, 27.0, 23.8, 19.3; **IR** (NaCl, cm^{-1}) 3072, 3044, 2929, 2889, 2856, 2717, 1715, 1646, 1474, 1425, 1360, 1270, 1107, 1078, 980; $\mathbf{R}_f$= 0.51 (3:1 hexanes:EtOAc); $[\alpha]_D$ –29.4 (*c* 1.0, $CHCl_3$). Exact mass calc'd for $C_{30}H_{40}IO_4Si$ $[M+H]^+$ requires *m/z* 619.1735. Found 619.1733 (Hi-Res ESI).

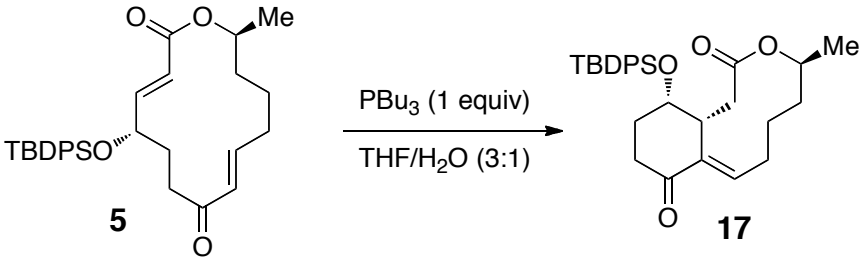


In a glovebox was added $CrCl_2/NiCl_2$ (99:1) (1.59 g, 12.9 mmol) portionwise to DMSO (645 mL). The reaction vessel was capped with a septum, carefully removed from the glovebox, and immediately placed under an inert atmosphere (N_2). This mixture was allowed to stir for 30 minutes at room temperature (to further dissolve the $CrCl_2/NiCl_2$ mixture). At this point a DMSO solution (15 mL) of substrate (800 mg, 1.29 mmol) was transferred via cannula into the reaction mixture. The reaction was allowed to stir at room temperature for 24 hours until it was dark green. The reaction was diluted with EtOAc (500 mL) and water (50 mL) was slowly added to quench the reaction. The organic and aqueous layers were separated and the aqueous layer was extracted with EtOAc (250 mL) and the organic layers were combined and washed twice with water (250 mL). The organic layer was dried over Na_2SO_4 and concentrated under reduced pressure. The crude material was purified via silica chromatography (4:1 Hex:EtOAc) to produce the product in 74% yield as a white foam (469 mg, 0.95 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.71 – 7.55 (m, 4H), 7.47 – 7.31 (m, 6H), 6.83 (dd, *J* = 15.6, 3.8 Hz, 1H), 6.15 (dd, *J* = 15.6, 1.8 Hz, 1H), 5.47 – 5.36 (m, 1H), 5.36 – 5.28 (m, 1H), 5.05 (ddd, *J* = 9.4, 6.3, 3.1 Hz, 1H), 4.60 (dt, *J* = 7.4, 2.8 Hz, 1H), 4.27 – 4.17 (m, 1H), 2.20 – 2.08 (m, 1H), 1.96 – 1.85 (m, 1H), 1.81 (dddd, *J* = 14.1, 8.6, 5.8, 3.1 Hz, 1H), 1.73 – 1.48 (m, 4H), 1.48 – 1.37 (m, 3H), 1.29 (d, *J* = 6.4 Hz, 3H), 1.19 (d, *J* = 3.8 Hz, 1H), 1.09 (s, 9H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 166.9, 166.5, 152.1, 151.3, 149.7, 136.2, 136.2, 136.2, 136.1, 136.1, 134.3, 134.3, 133.7, 133.7, 132.9, 132.8, 132.5, 131.3, 130.3, 130.2, 130.2, 130.2; **IR** (NaCl, cm^{-1}) 3444, 3068, 3044, 3007, 2934, 2852, 1716, 1650, 1585, 1467, 1422, 1356, 1258, 1201, 1107, 1058, 993; $\mathbf{R}_f$ = 0.51 (2:1

hexanes:EtOAc); **[α]$_D$** −8.8 (*c* 1.0, $CHCl_3$). Exact mass calc'd for $C_{30}H_{40}NaO_4Si$ $[M+Na]^+$ requires *m/z* 515.2588. Found 515.2565 (Hi-Res ESI).

SI-6 **18**

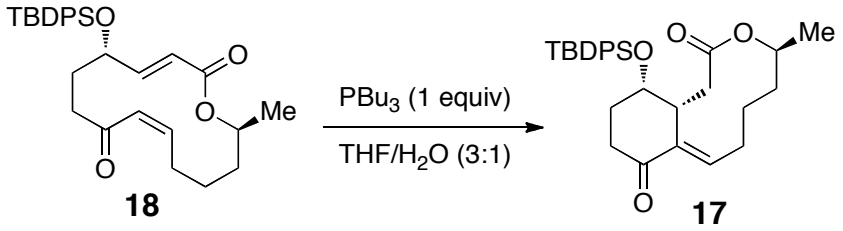
The substrate (450 mg, 0.913 mmol) was dissolved in DMSO (9.1 mL, 0.1 M) and to this solution was added IBX (383 mg, 1.37 mmol) in one portion. The reaction was allowed to stir overnight at room temperature after which it was diluted with EtOAc (30 mL), washed thrice with water (30 mL), dried over Na_2SO_4, and concentrated under reduced pressure. The crude material was purified via silica gel chromatography (10:1 Hex:EtOAc) to provide the product as a white foam in 85% yield (379 mg, 0.77 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.70 – 7.64 (m, 2H), 7.61 – 7.56 (m, 2H), 7.48 – 7.32 (m, 7H), 6.49 (dd, *J* = 15.6, 3.0 Hz, 1H), 6.17 (d, *J* = 2.0 Hz, 1H), 6.14 (dd, *J* = 4.6, 2.2 Hz, 1H), 6.02 (ddd, *J* = 11.3, 7.9, 4.8 Hz, 1H), 5.08 (pd, *J* = 6.6, 1.9 Hz, 1H), 4.51 (td, *J* = 5.1, 3.1 Hz, 1H), 3.01 – 2.88 (m, 1H), 2.69 (dd, *J* = 17.3, 10.4 Hz, 1H), 2.46 – 2.33 (m, 1H), 2.15 – 1.98 (m, 2H), 1.89 – 1.75 (m, 2H), 1.68 – 1.38 (m, 4H), 1.23 (d, *J* = 6.6 Hz, 3H), 1.10 (s, 9H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 199.7, 171.1, 140.9, 137.8, 135.7, 135.7, 133.9, 133.6, 130.0, 129.9, 127.9, 127.8, 70.8, 70.3, 38.6, 37.2, 33.9, 29.8, 27.2, 27.0, 26.5, 21.4, 19.2, 17.1; **IR** (NaCl, cm^{-1}) 3068, 2929, 2852, 1708, 1646, 1605, 1467, 1426, 1356, 1266, 1189, 1102, 1054; **R_f** = 0.70 (2:1 hexanes:EtOAc); **[α]$_D$** +26.8 (*c* 1.0, $CHCl_3$). Exact mass calc'd for $C_{30}H_{39}O_4Si$ $[M+H]^+$ requires *m/z* 491.2618. Found 491.2617 (Hi-Res ESI).



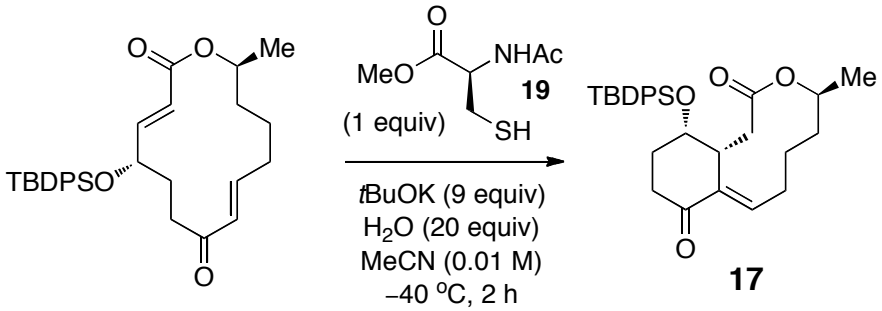
5 **17**

To a round bottom flask was added THF (9.5 mL), H_2O (4.8 mL) and PBu_3 (189 μL, 0.76 mmol). At room temperature the substrate (375 mg, 0.76 mmol) was added as a solution in THF

(5.0 mL). After stirring for 12 h at room temperature the reaction was complete by TLC. The yellow reaction mixture was diluted with EtOAc (50 mL), washed with brine (50 mL), dried over Na_2SO_4, and concentrated under reduced pressure. Purification by silica gel chromatography (9:1 hexanes:EtOAc) afforded a white foam in 80% yield (293 mg, 0.60 mmol).



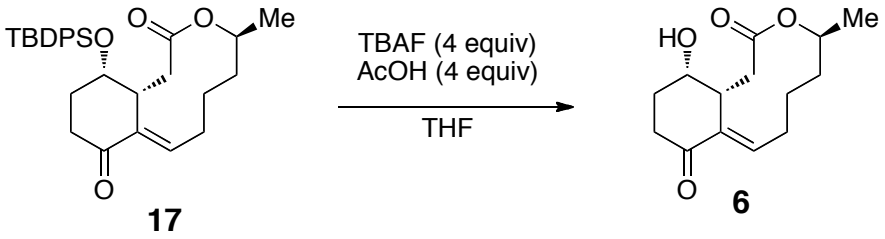
To a round bottom flask was added THF (2.6 mL), H_2O (1.3 mL), and PBu_3 (50.0 μL, 0.204 mmol). At room temperature the substrate (100 mg, 0.204 mmol) was added as a solution in THF (1.2 mL). After stirring for 12h at room temperature the reaction was complete by TLC. The yellow reaction mixture was diluted with EtOAc (20 mL), washed with brine (20 mL), dried over Na_2SO_4, and concentrated under reduced pressure. Purification by silica gel chromatography (9:1 hexanes:EtOAc) afforded a white foam in 62% yield (62 mg, 0.13 mmol).



To a stirring solution of substrate (15.0 mg, 3.06 x 10^{-2} mmol) in H_2O (11.0 μL, 0.61 mmol) and MeCN (3.0 mL, 0.01 M) stirring at –40 °C was added cysteine catalyst **19** (5.40 mg, 3.06 x 10^{-2} mmol).[4] After thoroughly stirring for 5-10 minutes, *t*-BuOK (30.9 mg, 0.27 mmol) was added to the reaction mixture and allowed to stir vigorously until complete by TLC (2 h). The reaction was immediately plugged through silica gel (EtOAc) and concentrated under reduced pressure. The crude product was purified via silica gel chromatography (9:1 hexanes:EtOAc) to afford the

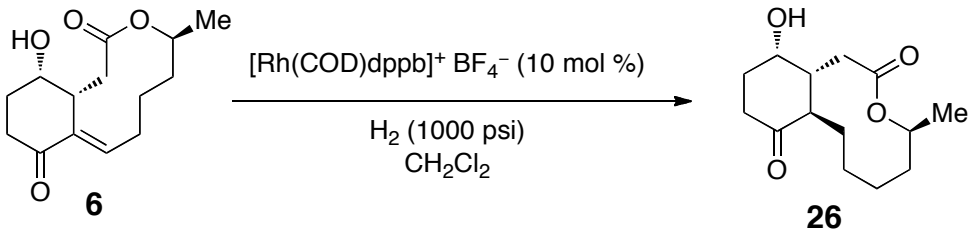
[4] MeCN was freshly distilled over CaH_2 under Ar, H_2O was thoroughly degassed with Ar, *t*-BuOK was freshly sublimed, and the substrate was purified via silica gel chromatography prior to use.

desired product in 66% yield (9.9 mg, 2.02 x 10^{-2} mmol); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.74-7.66 (m, 4H), 7.49-7.38 (m, 6H), 6.21 (dd, *J* = 12.9, 4.1 Hz, 1H), 5.10 (m, 1H), 4.16 (dt, *J* = 11.0, 4.8 Hz, 1H), 3.37 (dt, *J* = 12.6, 4.6 Hz, 1H), 2.97 (dd, *J* = 14.8, 4.6 Hz, 1H), 2.52-2.46 (m, 1H), 2.25 (dd, *J* = 14.8, 12.8 Hz, 1H), 2.21-2.03 (m, 3H), 2.00-1.94 (m, 1H), 1.88-1.76 (m, 2H), 1.71 (dt, *J* = 12.7, 2.7 Hz, 1H), 1.32-1.26 (m, 1H), 1.23 (d, *J* = 6.7 Hz, 3H), 1.10 (s, 9H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 199.7, 171.1, 140.9, 137.8, 135.7, 133.7, 133.6, 130.0, 129.9, 127.9, 127.8, 77.2, 70.8, 70.3, 38.6, 37.2, 33.9, 29.8, 27.2, 27.0, 26.5, 21.4, 19.2, 17.1; **IR** (NaCl, cm^{-1}) 3064, 3048, 2938, 2856, 2247, 1961, 1887, 1822, 1728, 1691, 1618, 1467, 1438, 1426, 1385, 1356, 1299, 1270, 1250, 1197, 1181, 1148, 1107, 1062, 1038, 1001; $\mathbf{R}_f$ = 0.54 (2:1 hexanes:EtOAc); $[\alpha]_D$ –39.8 (*c* 1.0, $CHCl_3$). Exact mass calc'd for $C_{30}H_{38}NaO_4Si$ $[M+Na]^+$ requires *m/z* 513.2432. Found 513.2433 (Hi-Res ESI).



To a solution of **17** (140 mg, 0.29 mmol) in THF (28.5 mL) was added AcOH (65.0 μL, 1.14 mmol) followed by addition of 1M TBAF (1.14 mL, 1.14 mmol). After stirring for 12 hours at room temperature the reaction was quenched with saturated NH_4Cl (50 mL) and then extracted thrice with EtOAc (50 mL). The combined organic layers were washed with brine (50 mL), dried over Na_2SO_4, and concentrated under reduced pressure. The crude material was purified via silica gel chromatography (2:1 EtOAc:hexanes) to yield the desired product as a clear oil in 86% yield (63.0 mg, 0.25 mmol), which can be crystallized from $CDCl_3$. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.41 (dd, *J* = 4.2, 12.8 Hz, 1H), 5.12 (qt, *J* = 3.5, 6.7 Hz, 1H), 4.29 – 4.17 (m, 1H), 3.71 (dt, *J* = 4.7, 12.5 Hz, 1H), 2.92 (dd, *J* = 4.7, 14.7 Hz, 1H), 2.71 (qd, *J* = 3.6, 12.9 Hz, 1H), 2.64 – 2.53 (m, 1H), 2.41 – 2.28 (m, 1H), 2.23 (dd, *J* = 12.9, 14.6 Hz, 1H), 2.13 (ddd, *J* = 4.4, 8.8, 13.3 Hz, 1H), 2.06 – 1.92 (m, 4H), 1.89 – 1.78 (m, 1H), 1.66 – 1.54 (m, 1H), 1.36 (ddd, *J* = 3.9, 7.5, 11.8 Hz, 1H), 1.25 (d, *J* = 6.7 Hz, 3H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 199.57, 170.88, 141.39, 137.60, 71.12, 68.92, 38.84, 37.01, 33.62, 29.80, 26.93, 26.70, 21.51, 17.05; **IR** (NaCl, cm^{-1}) 3424, 2974, 2942, 2889, 1716, 1687, 1614, 1467, 1438, 1364, 1299, 1246, 1046, 1029; $\mathbf{R}_f$

= 0.34 (4:1 EtOAc:hexanes); **[α]$_D$** +17.0 (*c* 1.0, $CHCl_3$). **Mp** = 131 – 132 °C. Exact mass calc'd for $C_{14}H_{21}O_4$ $[M+H]^+$ requires 253.1434 *m/z*. Found 253.1434 (Hi-Res ESI).



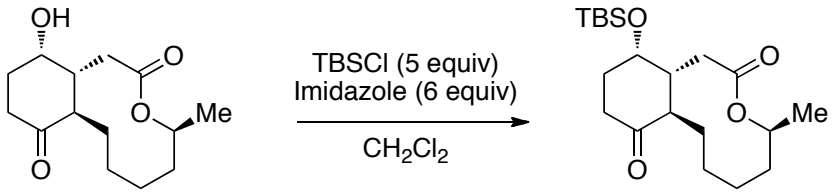
6 **26**

In a flamed-dried glass vial was added the substrate (40.0 mg, 15.9 mmol) and dissolved in CH_2Cl_2 (6.4 mL). To this solution was added $[RhCOD(dppb)]^+BF_4^-$ (23.0 mg, 3.2 mmol), and the vessel was purged thrice with H_2 (200 psi) and then with H_2 (1000 psi) using a Parr apparatus. After stirring for 24 hours the pressure was released and the reaction vessel was removed from the Parr apparatus and plugged through silica gel with EtOAc. The crude material (12:1 ratio of isomers) was purified via silica gel chromatography (1:1 hexanes:EtOAc) to yield the desired products in 81% combined yield as white foams (32.4 mg, 0.13 mmol), which can be crystallized from $CDCl_3$.

Major Isomer: **^{1}H NMR** (500 MHz, $CDCl_3$) δ 5.18 – 5.03 (m, 1H), 4.12 (s, 1H), 3.17 (s, 1H), 2.77 (ddd, *J* = 5.8, 14.6, 28.3 Hz, 2H), 2.70 – 2.55 (m, 2H), 2.44 – 2.33 (m, 1H), 2.23 (ddd, *J* = 2.2, 4.6, 14.4 Hz, 1H), 2.14 (dddd, *J* = 2.4, 3.4, 6.1, 13.9 Hz, 1H), 2.06 – 1.93 (m, 1H), 1.91 – 1.71 (m, 3H), 1.71 – 1.56 (m, 1H), 1.45 (m, 1H), 1.40 – 1.30 (m, 2H), 1.29 – 1.16 (m, 4H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 210.8, 173.0, 73.9, 70.8, 47.0, 41.6, 39.6, 35.6, 32.9, 32.3, 24.8, 22.5, 22.0, 19.5; **IR** (NaCl, cm^{-1}) 3485, 2954, 2929, 1720, 1695, 1458, 1413, 1356, 1279, 1250, 1193, 1131, 1062; **R_f** = 0.48 (2:1 EtOAc:hexanes); **[α]$_D$** 17.2 (*c* 0.5, $CHCl_3$); **Mp** = 132 – 134 °C. Exact mass calc'd for $C_{30}H_{41}O_4Si$ $[M+H]^+$ requires *m/z* 255.1591. Found 255.1585 (Hi-Res ESI).

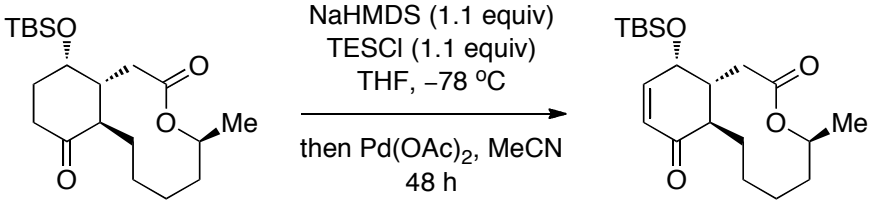
Minor Isomer: **^{1}H NMR** (500 MHz, $CDCl_3$) δ 5.11 – 5.02 (m, 1H), 4.35 (dt, *J* = 11.8, 4.5, 1H), 3.18 – 3.10 (m, 1H), 2.85 (dd, *J* = 17.0, 2.0 Hz, 1H), 2.48 – 2.41 (m, 1H), 2.36 (dd, *J* = 9.2, 4.0 Hz, 2H), 2.14 – 2.02 (m, 2H), 1.91 (dd, *J* = 17.0, 12.6 Hz, 2H), 1.87 – 1.73 (m, 3H), 1.55 (dd, *J* = 18.9, 7.5, Hz, 1H), 1.46 – 1.28 (m, 3H), 1.26 (d, *J* = 6.6 Hz, 3H), 1.06 – 0.96 (m, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 210.3, 171.6, 77.3, 77.2, 77.0, 76.8, 72.8, 70.5, 51.3, 40.0, 38.6, 30.2, 29.8, 29.4, 23.0, 22.2, 18.2; **IR** (NaCl, cm^{-1}) 3485, 2954, 2929, 1720, 1695, 1458, 1413, 1356, 1279,

1250, 1193, 1131, 1062; $\mathbf{R}_f$ = 0.37 (2:1 EtOAc:hexanes); $[\alpha]_D$ +4.0 (*c* 0.50, $CHCl_3$). Exact mass calc'd for $C_{30}H_{41}O_4Si$ $[M+H]^+$ requires *m/z* 255.1591. Found 255.1587 (Hi-Res ESI).



26

To a solution of substrate (50.0 mg, 0.20 mmol) in CH_2Cl_2 (2.0 mL, 0.1 M) was added imidazole (80 mg, 1.18 mmol) and TBSCl (150 mg, 1.0 mmol). The reaction was stirred for 48 h at room temperature after which it was quenched with water (10 mL). The aqueous layer was extracted twice with EtOAc (10 mL), dried over Na_2SO_4, and concentrated under reduced pressure. The crude matieral was purified via silica gel chromatography to give the desired product in 71% yield as white needles (52.0 mg, 0.14 mmol). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 5.06 (m, 1H), 4.02 (s, 1H), 2.67 (td, *J* = 14.7, 6.2 Hz, 1H), 2.62 – 2.43 (m, 4H), 2.20 (ddd, *J* = 15.1, 4.5, 2.1, Hz, 1H), 2.14 – 1.96 (m, 3H), 1.88 – 1.75 (m, 3H), 1.39 – 1.27 (m, 3H), 1.25 (d, *J* = 6.6 Hz, 3H), 0.91 (s, 9H), 0.91 (s, 9H), 0.11 (s, 3H), 0.09 (s, 3H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 211.0, 171.7, 73.0, 71.0, 50.1, 40.9, 40.3, 35.1, 32.0, 29.5, 26.0, 23.2, 23.0, 21.6, 18.3, 18.1, –4.4, –4.9. The spectral data matched that which had been previously reported.[5]

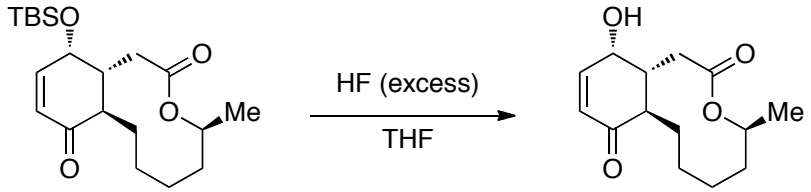


27

To a solution of substrate (50.0 mg, 0.14 mmol) in THF (1.4 mL, 0.10 M) was added TESCl (162 μL, 1 M in THF, 0.16 mmol). The reaction mixture was cooled to -78 °C and NaHMDS (162 μL, 1 M in THF, 0.16 mmol) was added and stirred for 30 mins. The reaction was plugged through silica gel (4:1 hexanes:EtOAc) and concentrated under reduced pressure. The silyl enol ether was dissolved in MeCN (5.4 mL, 0.025 M) to which was added $Pd(OAc)_2$ (153 mg, 0.68

[5] Wilson, E. M.; Trauner, D. *Org. Lett.* **2007**, *9*, 1327-1329.

mmol). The reaction was stirred for 48 h and then plugged through celite with MeCN and concentrated under reduced pressure. The crude material was purified via silica gel chromatography (20:1 hexanes:EtOAc) to yield the desired product in 62% yield as a white solid (31.0 mg, 0.084 mmol). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 6.85 (dd, *J* = 10.0, 5.6 Hz, 1H), 5.97 (d, *J* = 10.0 Hz, 1H), 5.13 – 5.03 (m, 1H), 4.22 (dd, *J* = 5.5, 3.1 Hz, 1H), 2.81 (tt, *J* = 11.2, 2.7 Hz, 1H), 2.72 (dt, *J* = 11.1, 3.7 Hz, 1H), 2.59 (dd, *J* = 16.9, 11.3 Hz, 1H), 2.48 (dd, *J* = 16.9, 2.3 Hz, 1H), 2.24 – 2.06 (m, 2H), 1.80 (m, 1H), 1.65 – 1.57 (m, 1H), 1.38 – 1.30 (m, 1H), 1.31-1.21 (m, 3H), 1.26 (d, *J* = 6.7 Hz, 3H), 1.12 – 1.04 (m, 1H), 0.87 (s, 9H), 0.10 (s, 3H), 0.07 (s, 3H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 200.5, 171.5, 146.7, 130.1, 73.1, 67.5, 46.5, 39.3, 37.3, 29.7, 25.8, 25.7, 22.8, 21.5, 18.2, 18.0, -3.9, -4.8; Exact mass calc'd for $C_{20}H_{34}O_4Si$ $[M+H]^+$ requires *m/z* 366.2299. Found 367.2294 (Hi-Res ESI). The spectral data matched that which had been previously reported.[5]



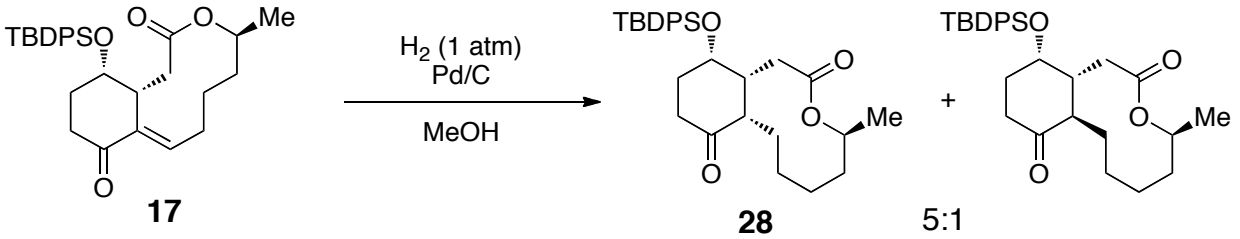
27 Sch-642305

To a solution of substrate (31.0 mg, 0.085 mmol) in THF (8.5 mL, 0.01 M) was added HF-Pyr (0.93 mL, excess) and stirred for 36 h (Note: a polyethylene tube was used). Upon completion the reaction mixture was slowly poured into sat. $NaHCO_3$ (300 mL) and then extracted thrice with EtOAc (100 mL). The combined organic layers were dried over Na_2SO_4 and concentrated under reduced pressure. The crude material was purified via silica gel chromatography (1.5:1 hexanes:EtOAc) to yield the desired product as a white solid in 69% yield (14.8 mg, 0.059 mmol), which can be crystallized from 3:2 hexanes:EtOAc. **^{1}H NMR** (500 MHz, *d_4*-MeOH) δ 7.06 (dd, *J* = 10.0, 5.7 Hz, 1H), 5.99 (d, *J* = 10.0 Hz, 1H), 5.13 – 5.03 (m, 1H), 4.25 (dd, *J* = 5.5, 3.6 Hz, 1H), 2.86 (dddd, *J* = 11.5, 11.5, 3.0, 3.0 Hz, 1H), 2.71 (dd, *J* = 17.0, 2.5 Hz, 2H), 2.69 (dt, *J* = 11.5, 4.0 Hz, 1H), 2.56 (dd, *J* = 16.9, 11.5 Hz, 1H), 2.25 – 2.09 (m, 2H), 1.93 – 1.82 (m, 1H), 1.64 – 1.52 (m, 1H), 1.48 – 1.33 (m, 3H), 1.31 (d, *J* = 6.6 Hz, 3H), 1.28 – 1.20 (m, 1H), 1.12 (ddd, *J* = 14.0, 11.0, 3.0 Hz, 1H). **^{13}C NMR** (126 MHz, *d_4*-MeOH) δ 203.2, 174.6, 150.3, 131.4, 75.5, 67.9, 48.5, 40.6, 38.6, 31.6, 25.0, 24.9, 23.4, 19.4; $\mathbf{R}_f$ = 0.42 (2:1 EtOAc:hexanes);

$[\alpha]_D$ +73.6 (*c* 1.0, MeOH). Exact mass calc'd for $C_{14}H_{21}O_4$ $[M+H]^+$ requires *m/z* 253.1434. Found 253.1434 (Hi-Res ESI). All spectral and analytical data matched that which had previously been reported.[6]

Natural	Synthetic	Natural	Synthetic
^{13}C NMR[a]	^{13}C NMR[a]	^{1}H NMR[b]	^{1}H NMR[b]
202.6	203.2	7.03 dd, *J* = 9.9, 5.6	7.06 dd, *J* = 10.0, 5.7
173.9	174.6	5.96 d, *J* = 9.9	5.99 d, *J* = 10.0
149.6	150.3	5.05 m	5.13 – 5.03 m
130.7	131.4	4.22 dd, *J* = 5.6, 3.7	4.25 dd, *J* = 5.5, 3.6
74.8	75.5	2.82 dddd, *J* = 11.5, 11.5, 3.7, 2.4	2.86 dddd, *J* = 11.5, 11.5, 3.0, 3.0
67.2	67.9	2.68 dd, *J* = 16.8, 2.4	2.71 dd, *J* = 17.0, 2.5
47.8	48.5	2.65 dt, *J* = 11.5, 3.7	2.69 dt, *J* = 11.5, 4.0
39.9	40.6	2.53 dd, *J* = 16.8, 11.5	2.56 dd, *J* = 16.9, 11.5
37.9	38.6	2.16 m	2.25 – 2.09 m
30.8	31.6	2.10 m	1.93 – 1.82 m
24.2	25.0	1.83 m	1.64 – 1.52 m
24.1	24.9	1.39 m	1.48 – 1.33 m
22.6	23.4	1.33 m	1.31 d, *J* = 6.6
18.7	19.4	1.27 d, *J* = 6.6	1.28 – 1.20 m
		1.23 m	1.12 ddd, *J* = 14.0, 11.0, 3.0
		1.08 m	

[a] Isolated: δ d_4-MeOH, 50.3; Synthetic: δ d_4-MeOH, 49.9; [b] Isolated: δ d_4-MeOH, 7.29; δ d_4-MeOH, 7.26

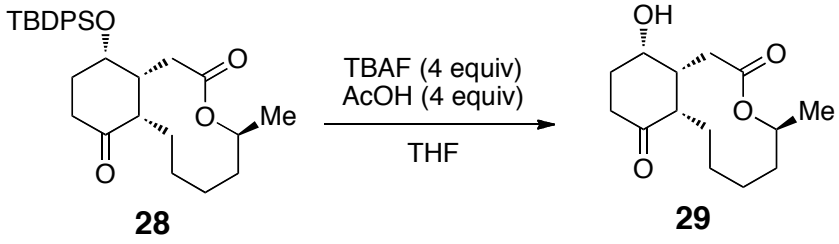


In a round bottom flask was dissolved Rauhut-Currier product (100.0 mg, 0.20 mmol) in degassed MeOH (N_2, 6.8 mL, 0.03 M). To this mixture was added a Pd/C slurry (MeOH, 1 mL) and the reaction vessel was purged with H_2 (1 atm). After 24 h the reaction was plugged through

[6] a) Chu, M.; Mierzwa, R.; Xu, L.; He, L.; Terraciano, J.; Patel, M.; Gullo, V.; Black, T.; Zhao, W.; Chan, T.-M.; McPhail, A. T. *J. Nat. Prod.* **2003**, *66*, 1527-1530. b) Jayasuriya, H.; Zink, D. L.; Polishook, J. D.; Bills, G. F.; Dombrowski, A. W.; Genilloud, O.; Pelaez, F. F.; Herranz, L.; Quamina, D.; Lingham, R. B.; Danzeizen, R.; Graham, P. L.; Tomassini, J. E.; Singh, S. B. *Chem. Biodiversity* **2005**, *2*, 112-122.

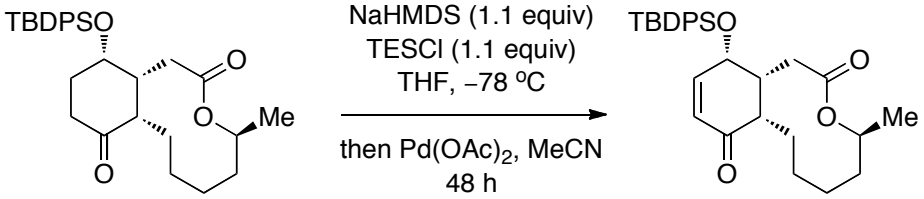
celite using MeOH and concentrated under reduced pressure. The crude material (5:1 ratio of isomers) was purified via silica gel chromatography (10:1 hexanes:EtOAc) to provide the separate diastereomeric products in a combined 69% yield as white foams (69 mg, 0.14 mmol). **MAJOR ISOMER**: **^{1}H NMR** (500 MHz, $CDCl_3$) d 7.74 – 7.64 (m, 4H), 7.50 – 7.35 (m, 6H), 5.10 – 4.99 (m, 1H), 4.17 (dt, *J* = 4.5, 11.6 Hz, 1H), 3.03 (dd, *J* = 1.8, 17.0 Hz, 1H), 2.95 – 2.86 (m, 1H), 2.24 (ddd, *J* = 1.9, 5.3, 14.4 Hz, 1H), 2.18 – 2.02 (m, 3H), 2.02 – 1.97 (m, 1H), 1.93 (dd, *J* = 12.5, 17.0 Hz, 1H), 1.90 – 1.70 (m, 2H), 1.64 (ddd, *J* = 7.4, 9.8, 16.7 Hz, 1H), 1.36 – 1.29 (m, 1H), 1.28 (d, *J* = 6.6 Hz, 3H), 1.22 (dd, *J* = 8.1, 17.5 Hz, 3H), 1.08 (s, 9H), 0.91 (ddt, *J* = 4.5, 11.7, 17.7 Hz, 1H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 210.4, 171.8, 135.8, 135.7, 133.7, 129.9, 129.9, 127.8, 127.7, 77.2, 72.5, 71.8, 51.3, 40.0, 38.8, 30.5, 30.4, 29.3, 26.9, 22.8, 22.7, 22.1, 19.2, 18.2.; **IR** (NaCl, cm^{-1}) 2930, 2858, 1723, 1462, 1427, 1361, 1255, 1200, 1140, 1112, 1075; $\mathbf{R}_f$ = 0.49 (4:1 hexanes:EtOAc); $\mathbf{[\alpha]_D}$ –36.6 (*c* 1.0, $CHCl_3$). Exact mass calc'd for $C_{30}H_{40}NaO_4Si$ $[M+Na]^+$ requires *m/z* 515.2588. Found 515.2567 (Hi-Res ESI).

MINOR ISOMER: **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.67 (ddd, *J* = 1.3, 7.9, 22.7 Hz, 4H), 7.49 – 7.35 (m, 6H), 5.05 – 4.97 (m, 1H), 4.08 (s, 1H), 2.77 – 2.67 (m, 2H), 2.55 (dd, *J* = 11.6, 16.2 Hz, 1H), 2.47 (m, 1H), 2.21 (dd, *J* = 1.3, 16.2 Hz, 1H), 2.15 (ddd, *J* = 2.4, 4.6, 15.4 Hz, 1H), 2.12 – 1.99 (m, 2H), 1.93 – 1.86 (m, 1H), 1.82 – 1.72 (m, 2H), 1.64 (tdd, *J* = 1.8, 4.8, 13.9 Hz, 1H), 1.35 – 1.27 (m, 1H), 1.27 – 1.22 (m, 3H), 1.21 (d, *J* = 6.6 Hz, 3H), 1.11 (s, 9H), 0.99 – 0.91 (m, 1H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 210.9, 171.5, 136.1, 135.9, 133.9, 133.1, 130.0, 129.9, 127.8, 127.7, 73.0, 72.0, 50.2, 40.5, 40.4, 35.3, 31.1, 27.3, 23.2, 23.0, 21.6, 19.6, 18.2; **IR** (NaCl, cm^{-1}) 2932, 2858, 1723, 1462, 1427, 1361, 1255, 1200, 1140, 1112, 1075; $\mathbf{R}_f$ = 0.53 (4:1 hexanes:EtOAc); $\mathbf{[\alpha]_D}$ –24.2 (*c* 0.52, $CHCl_3$). Exact mass calc'd for $[C_{30}H_{40}NaO_4Si]^+$ requires *m/z* 515.2588. Found 515.2567 (Hi-Res ESI).



28 **29**

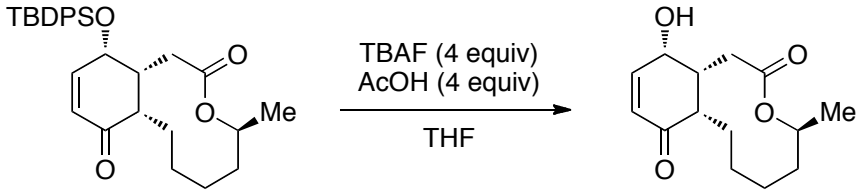
To a stirring solution of compound **28** (10 mg, 0.020 mmol) in THF (2 mL, 0.01 M) was added AcOH (4.6 μL, 0.081 mmol) and 1M TBAF (81 μL, 0.081 mmol). The reaction was allowed to stir at rt for 12 h before it was diluted with EtOAc and quenched with saturated NH_4Cl. The layers were separated and the aqueous layer was extracted thrice with EtOAc, dried over Na_2SO_4, and concentrated under reduced pressure. The crude material was prufied via silica gel chromatography (1:1 hexanes:EtOAc) to yield the desired product in quantitative yield (5 mg, 0.020 mml). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 5.07 (qt, *J* = 9.6, 4.8 Hz, 1H), 4.40 – 4.31 (m, 1H), 3.20 – 3.10 (m, 1H), 2.85 (dd, *J* = 17.0, 2.0 Hz, 1H), 2.49 – 2.41 (m, 1H), 2.37 (dd, *J* = 9.2, 4.1 Hz, 2H), 2.15 – 2.02 (m, 2H), 1.91 (dd, *J* = 15.1, 10.7 Hz, 1H), 1.89 – 1.73 (m, 4H), 1.65 – 1.50 (m, 1H), 1.47 – 1.28 (m, 3H), 1.26 (d, *J* = 6.6 Hz, 3H), 1.07 – 0.97 (m, 1H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 210.29, 171.60, 72.80, 70.49, 51.29, 40.01, 38.55, 30.22, 29.34, 22.21, 18.17; **IR** (NaCl, cm^{-1}) 3485, 2954, 2929, 1720, 1695, 1458, 1413, 1356, 1279, 1250, 1193, 1131, 1062; $\mathbf{R}_f$ = 0.37 (2:1 hexanes:EtOAc); $[\alpha]_D$ +4.0 (*c* 0.50, $CHCl_3$). Exact mass calc'd for $C_{30}H_{41}O_4Si$ $[M+H]^+$ requires *m/z* 255.1591. Found 255.1585 (Hi-Res ESI).



28

To a solution of saturated compound (22.0 mg, 0.045 mmol) in THF (0.45 mL, 0.10 M) was added TESCl (54 μL, 1.0 M in THF, 0.054 mmol). The solution was cooled to –78 °C and NaHMDS (0.054 mmol, 1.0 M in THF) was added dropwise. The reaction was stirred for 30 mins then plugged through silica gel (4:1 hexanes:EtOAc) and concentrated under reduced pressure. The TES enol ether was dissolved in MeCN (1.8 mL, 0.025 M) to which was added $Pd(OAc)_2$ (51 mg, 0.23 mmol). The reaction was stirred for 36 h and plugged through celite (MeCN) and concentrated under reduced pressure. The crude material was purified via silica gel chromatography (20:1-10:1 hexanes:EtOAc) to yield the desired product in 79% yield as a white foam (17.4 mg, 0.035 mmol). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.75 – 7.63 (m, 4H), 7.48 (m, 2H), 7.42 (m, 4H), 6.64 (dt, *J* = 10.3, 1.8 Hz, 1H), 5.88 (dd, *J* = 10.3, 2.6 Hz, 1H), 5.06 – 4.95 (m, 1H), 4.76 – 4.69 (m, 1H), 3.02 (d, *J* = 2.2 Hz, 1H), 2.99 (d, *J* = 2.2 Hz, 1H), 2.11 (dd, *J* = 12.5,

4.5 Hz, 1H), 2.09 – 2.01 (m, 3H), 1.37 – 1.23 (m, 4H), 1.28 (d, *J* = 6.5 Hz, 3H), 0.92 – 0.82 (m, 1H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 198.7, 172.0, 150.3, 135.7, 135.7, 133.1, 132.9, 130.2, 130.1, 128.9, 128.0, 127.9, 72.7, 71.2, 50.2, 40.3, 29.9, 26.9, 23.2, 23.2, 22.8, 22.6, 22.5, 22.5, 22.5, 19.2, 18.6, 18.6. The spectral data matched that which had been previously reported.[7]

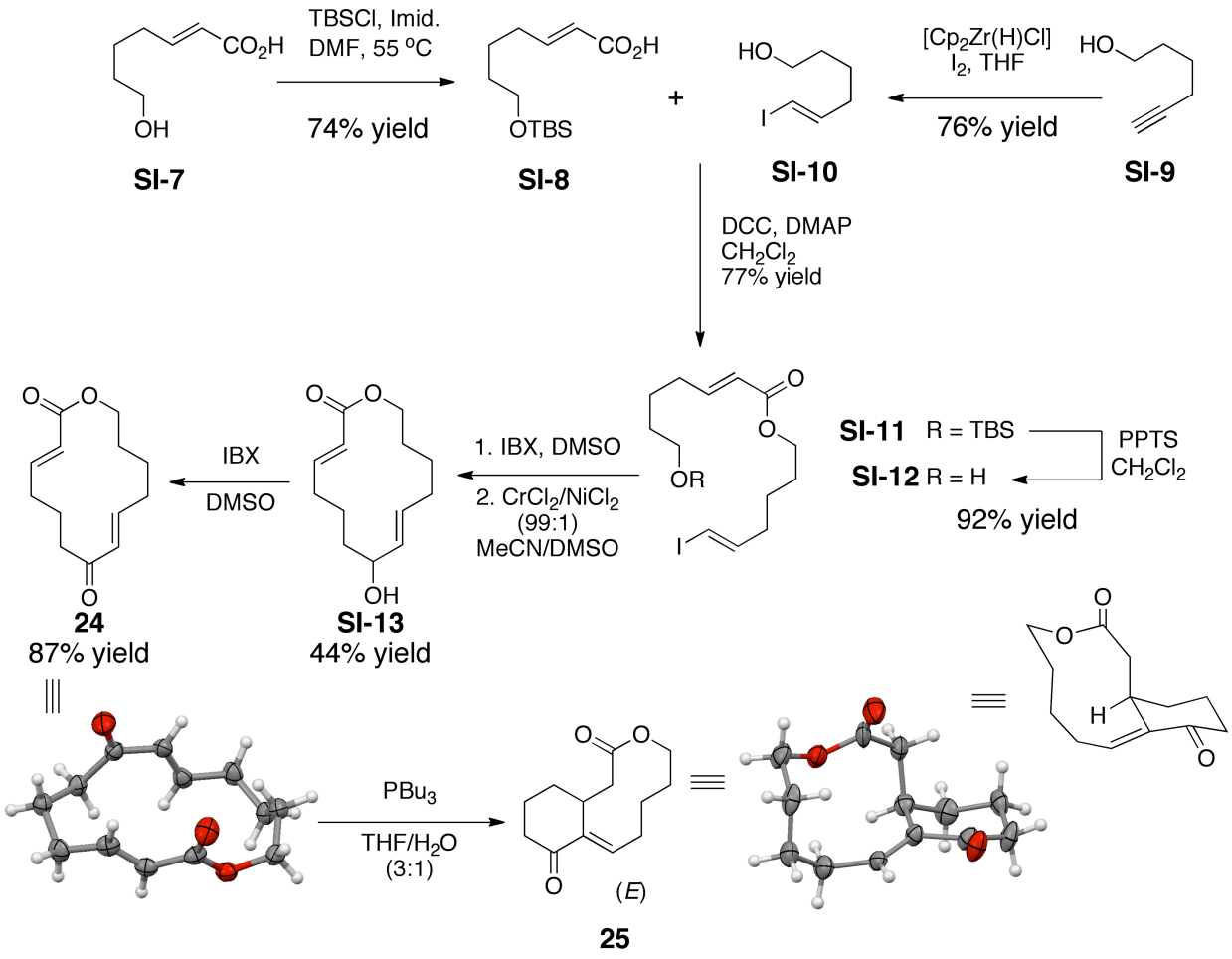


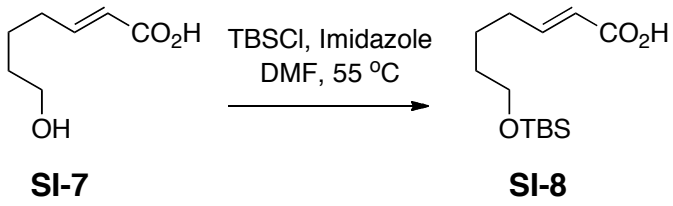
***epi*-Sch-642305**

To a solution of substrate (12.0 mg, 0.024 mmol) in THF (2.4 mL, 0.01 M) was added AcOH (5.6 μL, 0.098 mmol) and TBAF (98.0 μL, 1 M THF solution, 0.098 mmol). The reaction was stirred for 24 h and then quenched with sat. NH_4Cl (10 mL). The mixture was extracted thrice with EtOAc (5 mL), dried over Na_2SO_4, and concentrated under reduced pressure. The crude material was purified via silica gel chromatography (1.5:1 hexanes:EtOAc) to yield the desired product as a white solid in 80% yield (4.8 mg, 0.019 mmol). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 6.66 (dt, *J* = 10.2, 1.9 Hz, 1H), 5.98 (dd, *J* = 10.2, 2.7 Hz, 1H), 5.08 – 5.00 (m, 1H), 4.89 (s, 1H), 3.35 – 3.27 (m, 1H), 2.84 (dd, *J* = 17.0, 2.3 Hz, 1H), 2.36 (dt, *J* = 12.3, 3.2 Hz, 1H), 2.21 – 2.12 (m, 1H), 2.12 – 2.03 (m, 3H), 1.91 – 1.81 (m, 1H), 1.66 – 1.56 (m, 2H), 1.56 – 1.45 (m, 1H), 1.45 – 1.30 (m, 3H), 1.27 (d, *J* = 6.6 Hz, 4H), 0.99 (dt, J = 14.6, 11.8 Hz, 1H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 198.6, 171.7, 149.0, 129.4, 72.9, 69.9, 50.3, 40.2, 29.7, 29.6, 23.3, 23.1, 22.6, 18.6; $\mathbf{R}_f$ = 0.42 (2:1 EtOAc:hexanes); $\mathbf{[\alpha]_D}$ +56.6 (*c* 0.50, $CHCl_3$); **Mp** = 159 – 162 °C. Exact mass calc'd for $C_{14}H_{21}O_4$ $[M+H]^+$ requires *m/z* 253.1434. Found 253.1434 (Hi-Res ESI). The spectral data matched that which had been previously reported.[4]

[7] Snider, B. B.; Zhou, J. *Org. Lett.* **2006**, *8* 1283-1286.

Scheme SI-2

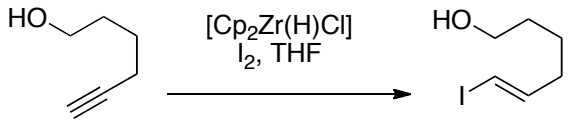




Alcohol **SI-7**[8] (1.00 g, 6.94 mmol) and imidazole (1.06 g, 15.6 mmol, 2.25 equiv.) were dissolved in DMF, TBS-Cl (1.15 g, 7.63 mmol, 1.1 equiv.) added, and the mixture stirred at 50 °C for 18 h. The mixture was poured into brine (150 mL) and extracted with EtOAc (4×50 mL). The combined organic extracts were washed with brine (50 mL), dried over Na_2SO_4, filtered, and the solvent evaporated. Silica gel chromatography (2:1 hexanes:EtOAc) of the residue gave *O*-

[8] (a) Woods, G. F. *Org. Synth. Coll. Vol. 3* **1955**, 470, (b) Kennedy, J.; McCorkindale, J.; Raphael, R. A. *J. Chem. Soc.* **1961**, 3813-3815.

TBS protected alcohol (1.32 g, 5.11 mmol, 74%) as a colorless oil.[9] **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.08 (dt, *J* = 15.6, 6.9 Hz, 1 H), 5.83 (dt, *J* = 15.6, 1.5 Hz, 1 H), 3.62 (t, *J* = 5.9 Hz, 2 H), 2.25 (dq, *J* = 6.9, 1.5 Hz, 2 H), 1.56-1.53 (m, 4 H), 0.89 (s, 9 H), 0.04 (s, 6 H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ 171.6, 152.5, 121.0, 63.1, 32.6, 32.5, 26.3, 24.7, 18.7, -4.9. The spectral data matched that which had been previously reported.[10]



SI-9 **SI-10**

Cp_2ZrCl_2 (8.35 g, 28.8 mmol) was dissolved in THF (30 mL), $LiEt_4BH$ (28.8 mL, 1.0 M THF, 28.8 mmol) was added and the mixture was stirred at rt (protected from light) for 1 h, during which time the beige suspension of Cp_2ZrCl_2 turned into a white suspension of $Cp_2Zr(H)Cl$. In a second flask, 5-hexynol **SI-9** (2.50 mL, 2.26 g, 23.1 mmol) was dissolved in THF (20 mL) and $LiEt_4BH$ (23.1 mL 1.0 M THF, 23.1 mmol) was added. The resulting alkoxide solution was added with a syringe into the suspension of $Cp_2Zr(H)Cl$ and the resulting solution stirred for 10 min. at rt, then cooled to 0 °C and solid I_2 (7.50 g, 29.5 mmol) was added. The mixture was allowed to stir at rt for 1 h, then quenched with saturated $NaHCO_3$ (sat. aq., 75 mL). The phases were separated and the aqueous phase extracted with EtOAc (3×30 mL). The combined organic extracts were washed with brine (3×30 mL), dried over Na_2SO_4, filtered from Na_2SO_4 and inoluble Zr-salts, and concentrated under reduced pressure. Purification of the crude material via silica gel chromatography (2:1 → 3:2 hexanes:EtOAc) gave the vinyl-iodide alcohol **SI-10** (3.96 g, 17.5 mmol) as an dark orange liquid in 76% yield.[11] **^{1}H NMR** (400 MHz, $CDCl_3$): *δ* 6.49 (*J* = 14.4, 7.2 Hz, 1 H), 5.98 (dt, *J* = 14.4 Hz, 1.4 Hz, 1 H), 3.60 (t, *J* = 6.4 Hz, 2 H), 2.07 (qd, *J* = 7.2 Hz, 1.4 Hz, 2 H), 1.87 (br. s, 1 H), 1.62-1.50 (m, 2 H), 1.50-1.40 (m, 2 H); **^{13}C NMR** (126 MHz, $CDCl_3$): *δ* 146.2, 74.4, 62.4, 35.6, 31.8, 34.5. The spectral data matched that which had been previously reported.[12]

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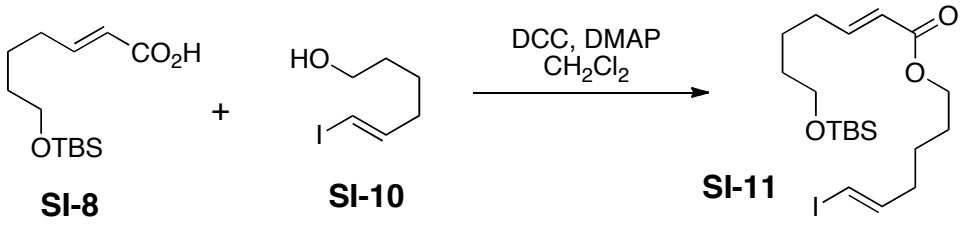
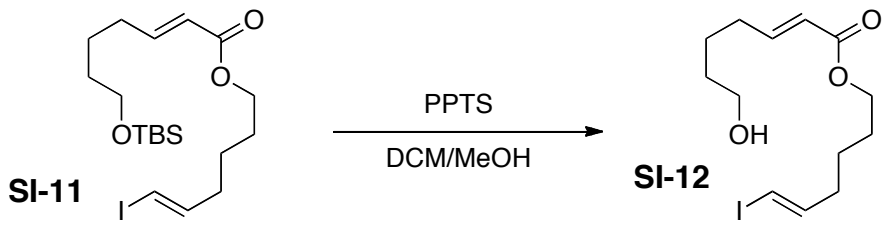
[9] K. Takizawa, C. Tang, C. J. Hawker *J. Am. Chem. Soc.* 2008, ***130***, 1718.
[10] K. C. Nicolaou, C. K. Hwang, B. E. Marron, S. A. DeFrees, E. A. Couladouros, Y. Abe, P. J. Carroll, J. P. Snyder *J. Am. Chem. Soc.* 1990, ***112***, 3040.
[11] B. H. Lipshutz, R. Kell, E. L. Ellsworth, *Tetrahedron Lett.* 1990, ***31***, 7257.
[12] A. Nishida, F. Shirato, M. Nakagawa, *Tetrahedron: Asymmetry* 2000, ***11***, 3789.



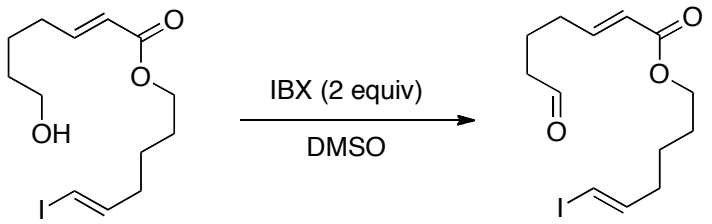
SI-8 **SI-10** **SI-11**

Carboxylic acid **SI-8** (5.70 g, 22.0 mmol, 1.22 equiv.) and alcohol **SI-10** (4.10 g, 18.1 mmol) were dissolved in CH_2Cl_2 (50 mL), DMAP (8.45 g, 72.4 mmol) and DCC (5.60 g, 27.2 mmol, 1.5 equiv.) were added and the mixture stirred over night at rt. After 24 h the reaction was quenched with AcOH (4.14 mL, 4.35 g, 72.4 mmol) and MeOH (0.73 mL, 580 mg, 18.1 mmol), stirred for 30 min. and filtered from precipitants. The filtrate was washed with H_2O (100 mL) and the aqueous phase re-extracted with CH_2Cl_2 (3×30 mL). The combined organic extracts were washed with saturated $NaHCO_3$ (100 mL), saturated NH_4Cl (100 mL), H_2O (100 mL), brine (100 mL), dried over Na_2SO_4, filtered and concentrated under reduced pressure. Purification of the residue by silica gel chromatography (25:1 hexanes:EtOAc) gave the desired ester **SI-11** (6.26 g, 14.0 mmol) as a slightly yellow oil in 77% yield. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 6.95 (dt, *J* = 15.6, 6.9 Hz, 1 H), 6.50 (dt, *J* = 14.4 Hz, 7.2 Hz, 1 H), 6.01 (dt, *J* = 14.4 Hz, 1.4 Hz, 1 H), 5.81 (dt, *J* = 15.6 Hz, 1.5 Hz, 1 H), 4.11 (t, *J* = 6.6 Hz, 2 H), 3.61 (t, *J* = 5.9 Hz, 2 H), 2.28-2.17 (m, 2 H, C-4H_2), 2.09 (qd, *J* = 7.3 Hz, 1.4 Hz, 2 H), 1.70-1.64 (m, 2 H), 1.59-1.42 (m, 6 H), 0.88 (s, 9 H), 0.04 (s, 6 H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ 166.7, 149.3, 145.9, 121.2, 74.9, 63.8, 62.7, 35.5, 32.2, 31.9, 28.0, 25.9, 24.8, 24.4, 18.3, -5.3; $\boldsymbol{R_f}$ = 0.51 (4:1 hexanes:EtOAc); **IR** (film, cm^{-1}): 2930, 2858, 1722, 1654, 1472, 1257, 1163, 1100, 836, 776; Exact mass calc'd for $C_{19}H_{36}IO_3Si$ $[M+H]^+$ requires *m/z* 467.14729. Found 467.14763 (Hi-Res ESI).



SI-11 **SI-12**

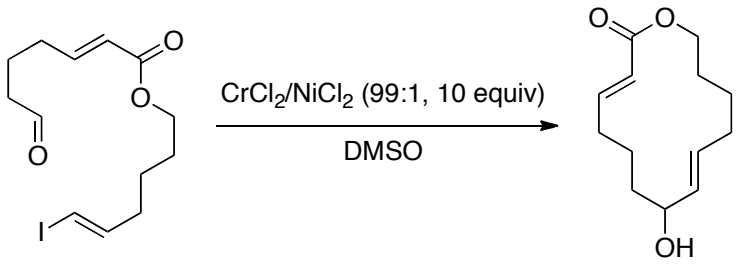
To a stirring solution of **SI-12** (264 mg, 0.57 mmol) in 5 mL DCM/MeOH (1:1, 0.1 M) was added PPTS (356 mg, 1.4 mmol). The reaction was allowed to stir overnight after which it was diluted with EtOAc (50 mL), washed thrice with H_2O (30 mL), once with saturated $NaHCO_3$ (30 mL), dried over Na_2SO_4 and concentrated under reduced pressure. The crude material was purified via silica gel chromatography (3:2 → 1:1 hexanes:EtOAc) to yield the desired product as a colorless oil in 92% yield (185 mg, 0.53 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.95 (dt, *J* = 15.6, 6.9 Hz, 1H), 6.50 (dt, *J* = 14.3, 7.1 Hz, 1H), 6.01 (dt, *J* = 14.3, 1.4 Hz, 1H), 5.82 (dt, *J* = 15.6, 1.6 Hz, 1H), 4.11 (t, *J* = 6.5 Hz, 2H), 3.66 (t, *J* = 6.1 Hz, 2H), 2.24 (qd, *J* = 7.2, 1.5 Hz, 2H), 2.09 (qd, *J* = 7.3, 1.4 Hz, 2H), 1.70 – 1.53 (m, 7H), 1.53 – 1.43 (m, 2H), 1.36 (s, 1H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 166.6, 149.0, 145.9, 121.4, 74.9, 63.8, 62.5, 35.5, 32.1, 31.9, 27.9, 24.8, 24.2; $\boldsymbol{R_f}$ = 0.28 (1:1 hexanes:EtOAc); **IR** (film, cm^{-1}) 3403, 2936, 2862, 1717, 1653, 1454, 1270, 1188, 1059; Exact mass calc'd for $C_{13}H_{22}IO_3$ $[M+H]^+$ requires *m/z* 353.06081. Found 353.06014 (Hi-Res ESI).



SI-12

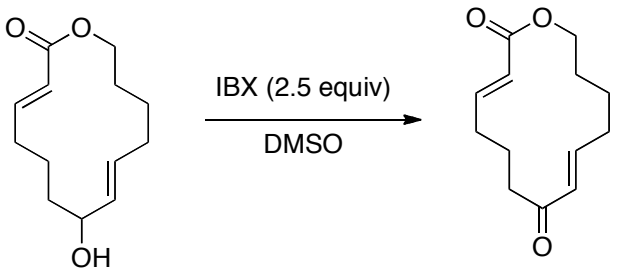
Alcohol **SI-6** (4.30 g, 12.2 mmol) and IBX (6.83 g, 24.4 mmol) were stirred in DMSO (25 mL) for 2 h, then poured into H_2O (300 mL). The obtained suspension was extracted with EtOAc (2×100 mL). The organic extracts were washed with H_2O (3×250 mL) and brine (2×100 mL), dried over Na_2SO_4, filtered and concentrated under reduced pressure. Purification of the residue via silica gel chromatography (3:1 → 2:1 hexanes:EtOAc) gave the desired aldehyde (3.63 g, 10.4 mmol, 85%) as a slightly yellow oil in 85% yield. **^{1}H NMR** (400 MHz, $CDCl_3$): *δ* 9.77 (t, *J* = 1.4 Hz, 1 H), 6.91 (dt, *J* = 15.6, 7.0 Hz, 1 H), 6.49 (dt, *J* = 14.4, 7.2 Hz, 1 H), 6.01 (dt, *J* = 14.4 , 1.4 Hz, 1 H), 5.83 (dt, *J* = 15.6, 1.5 Hz, 1 H), 4.11 (t, *J* = 6.6 Hz, 2 H), 2.48 (td, *J* = 7.2, 1.4 Hz, 2 H), 2.25 (qd, *J* = 7.4, 1.5 Hz, 2 H), 2.09 (qd, *J* = 7.3, 1.4 Hz, 2 H), 1.80 (p, *J* = 7.4 Hz, 2 H), 1.70-1.58 (m, 2 H), 1.52-1.42 (m, 2 H); **^{13}C NMR** (126 MHz, $CDCl_3$): *δ* 201.5, 166.4, 147.7, 145.9, 122.1, 75.0, 63.9, 42.9, 35.5, 32.2, 27.9, 24.7, 20.3; $\boldsymbol{R_f}$ = 0.56 (1:1 hexanes:EtOAc);

IR (film, cm^{-1}): 2942, 2722, 1721, 1653, 1268, 1191, 1160, 983, 952; Exact mass calc'd for $C_{13}H_{20}IO_3$ $[M+H]^+$ requires *m/z* 351.04516. Found 351.04553 (Hi-Res ESI).



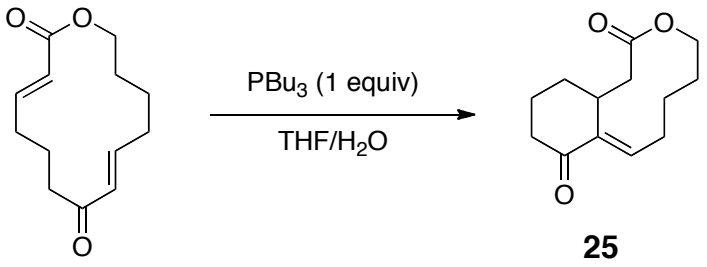
SI-13

A 2 L, three-neck flask was loaded with MeCN (1000 mL) and anhydrous DMSO (350 mL). To this was added, while stirring, a mixture of $CrCl_2/NiCl_2$ (99:1) (13.4 g mixture, 103 mmol) giving an almost black solution. To this was added with an addition funnel a solution of substrate aldehyde (3.60 g, 10.28 mmol) in dry MeCN (500 mL) dropwise over one hour. The mixture was stirred for 3 h at rt and then slowly poured into a mixture of H_2O (2.50 L) and brine (750 mL). The resulting mixture was extracted with EtOAc (4×300 mL). The combined organic extracts were washed once with H_2O (1.50 L), then concentrated to ~500 mL volume. This concentrate was washed again with H_2O (2×1000 mL) and brine (2×200 mL), dried over Na_2SO_4, filtered and concentrated under reduced pressure. Purification of the residue via silica gel chromatograph (4:1 hexanes:EtOAc) gave the macrocyclic allylic alcohol **SI-13** (1.21 g, 5.39 mmol) as a colorless oil, which slowly crystallized, in 52% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.90 (ddd, J = 15.5, 7.9 Hz, 7.4 Hz, 1 H), 5.74 (dt, J = 15.5, 1.4 Hz, 1 H), 5.69 (dddd, J = 15.4, 8.3, 5.3, 0.7 Hz, 1 H), 5.51 (dddd, J = 15.4, 7.4, 1.4, 1.1 Hz, 1 H), 4.37 (dt, J = 11.0, 4.2 Hz, 1 H), 4.12 (dddd, J = 7.4, 7.1, 4.5, 0.7 Hz, 1 H), 4.05 (ddd, J = 11.0, 7.5, 4.7 Hz, 1 H), 2.34-2.24 (m, 1 H), 2.23-1.99 (m, 3 H), 1.74-1.60 (m, 7 H), 1.58-1.48 (m, 1 H), 1.45 (br s, 1 H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 166.2, 151.1, 133.4, 132.5, 122.2, 72.6, 63.7, 35.4, 31.9, 30.5, 26.6, 25.4, 22.5; $\boldsymbol{R_f}$ = 0.28 (1:1 hexanes:EtOAc); **IR** (film, cm^{-1}) 3422, 2934, 2861, 1714, 1454, 1255, 1157, 970; **Mp** = 32-35 °C; Exact mass calc'd for $C_{13}H_{20}O_3Na$ $[M+Na]^+$ requires *m/z* 247.13047. Found 247.13053 (Hi-Res ESI).



SI-13 **24**

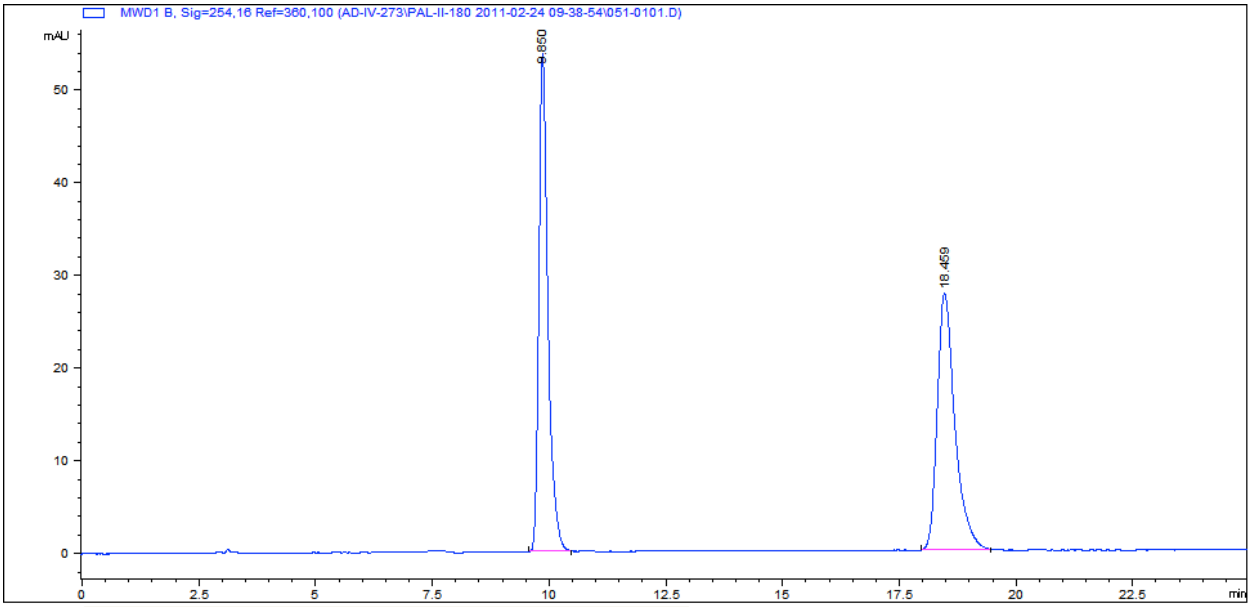
Allylic alcohol **SI-13** (550 mg, 2.45 mmol) and IBX (1.72 g, 6.13 mmol) were stirred in DMSO (7.5 mL) for 1 h, then poured into H_2O (150 mL). The obtained suspension was extracted with EtOAc (3×40 mL). The organic extracts were washed with H_2O (1×100 mL), brine (100 mL), dried over Na_2SO_4, filtered and concentrated under reduced pressure. Purification of the residue via silica gel chromatography (3:1 hexanes:EtOAc) gave the macrocyclic **24** (475 mg, 2.14 mmol, 87%) as a colorless oil in 87% yield, which crystallized into block-crystals from Et_2O. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.75 (dt, J = 15.6, 7.1 Hz, 1 H), 6.63 (dt, J = 15.7, 8.0 Hz, 1 H), 6.13 (dt, J = 15.6, 1.5 Hz, 1 H), 5.76 (dt, J = 15.7, 1.2 Hz, 1 H), 4.20-4.02 (m, 2 H), 2.44-2.38 (m, 2 H), 2.30-2.21 (m, 4 H), 2.07-1.97 (m, 2 H), 1.79-1.64 (m, 4 H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 200.5, 166.0, 148.1, 146.9, 131.7, 123.9, 64.3, 39.0, 32.4, 31.1, 26.4, 25.2, 23.1; IR (film, cm^{-1}) 2935, 2862, 1719, 1690, 1663, 1622, 1441, 1341, 1307, 1196, 1177, 990, 837, 709; $\boldsymbol{R_f}$ = 0.49 (1:1 hexanes:EtOAc); **Mp** = 58-61 °C; Exact mass calc'd for $C_{13}H_{19}O_3$ $[M+H]^+$ requires *m/z* 223.13287. Found 223.13255 (Hi-Res ESI).



24 **25**

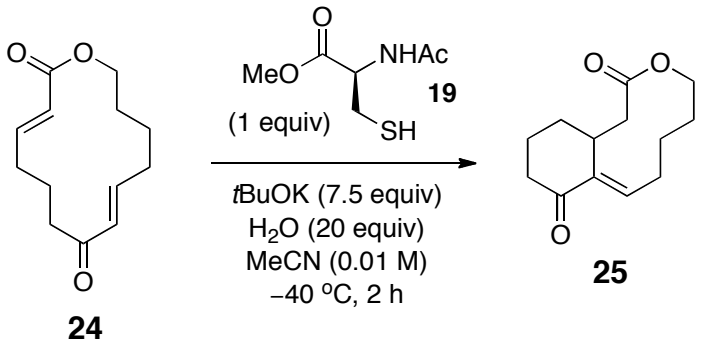
To a stirring solution of PBu_3 (16.6 μL, 0.068 mmol) in THF/H_2O (3:1, 1.7 mL) was added substrate **24**. After stirring for 3 h the reaction was complete by TLC and diluted with EtOAc. This mixture was washed twice with H_2O, dried over Na_2SO_4 and concentrated under reduced

pressure. The crude material was purified via silica gel chromatography (9:1 → 4:1 hexanes:EtOAc) to yield the desired product in 92% yield (13.8 mg, 0.062 mmol) and clear crystals (upon concentration).[13] **^{1}H NMR** (400 MHz, $CDCl_3$): δ 6.35 (dd, J = 12.9, 4.0 Hz, 1 H), 4.75-4.63 (m, 1 H), 3.83-3.73 (m, 1 H), 3.69-3.59 (m, 1 H), 2.80-2.64 (m, 1 H), 2.60-2.51 (m, 1 H), 2.47-2.36 (m, 2 H), 2.34-2.23 (m, 1 H), 2.19-2.10 (m, 1 H), 1.98-1.86 (m, 3 H), 1.84-1.69 (m), 1.54-1.48 (m, 1 H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ 201.3, 170.7, 139.6, 139.4, 66.1, 40.2, 39.1, 32.5, 28.9, 26.9, 26.4, 25.5, 19.1; **IR** (film, cm^{-1}) 2990, 2919, 2854, 1733, 1687, 1620, 1442, 1310, 1290, 1245, 1154, 1061, 1030; $\boldsymbol{R_f}$ = 0.45 (1:1 hexanes:EtOAc); **Mp** = 90-92 °C; Exact mass calc'd for $C_{13}H_{19}O_3$ $[M+H]^+$ requires *m/z* 223.13287. Found 223.13284 (Hi-Res ESI); **HPLC** (Chiralpak AD-H): 90:10 hexanes:isopropanol, 1.0 mL/min; 9.85 min, 18.5 min.

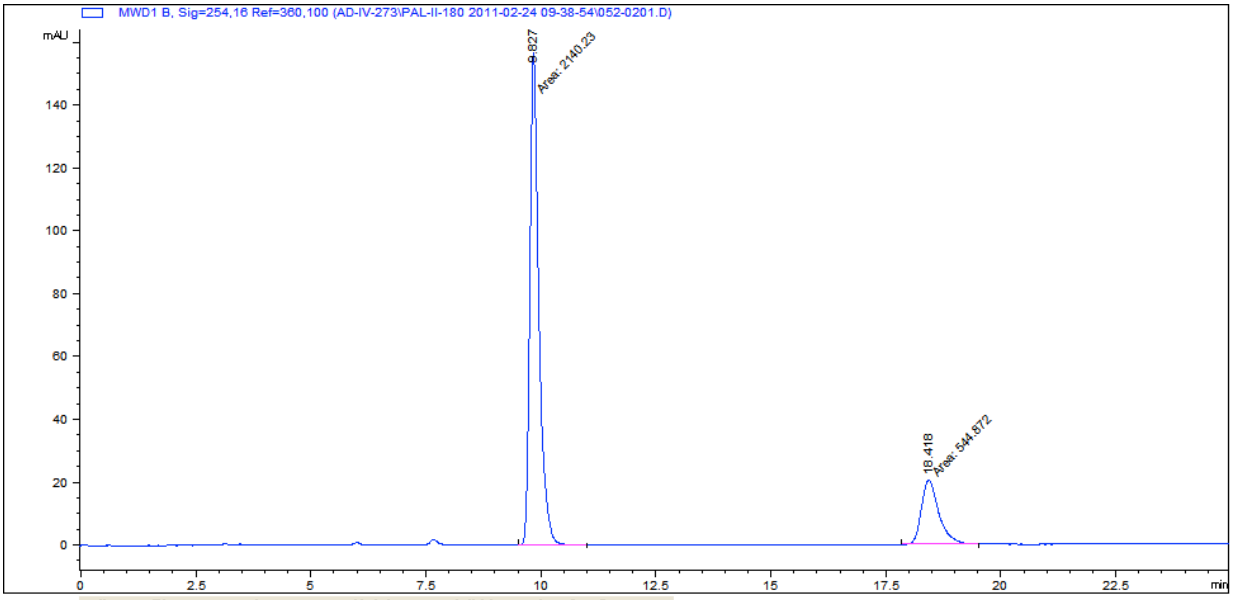


#	Time	Area	Height	Width	Area%	Symmetry
1	9.85	730.3	53.7	0.2027	50.024	0.656
2	18.459	729.5	27.7	0.3934	49.976	0.661

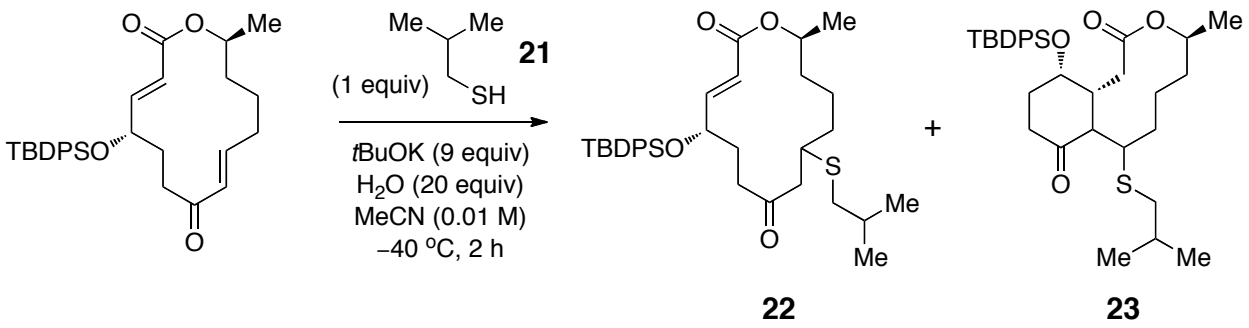
[13] The crystal structure shown on page S-25 was solved at low probability (12%), but was consistent with the illustrated structure. The olefin geometry of **25** was also assigned by spectroscopic analogy to compound **17** (J = 12.9, 4.0 Hz and J = 12.9, 4.1 Hz, respectively).



To a stirring solution of substrate (15.0 mg, 0.068 mmol) in H_2O (24.3 μL, 1.35 mmol) and MeCN (6.8 mL, 0.01 M) stirring at –40 °C was added cysteine catalyst **19** (12.0 mg, 0.068 mmol).[3] After thoroughly stirring for 5-10 minutes, *t*-BuOK (56.7 mg, 0.506 mmol) was added to the reaction mixture and allowed to stir vigorously until reaction was complete or before any decomposition took place to a large extent (10 h). The reaction was immediately plugged through silica gel (EtOAc) and concentrated under reduced pressure. The crude product was purified via silica gel chromatography (9:1 hexanes:EtOAc) to afford the desired product in 21% yield or 35% (based on recovered starting material) (3.2 mg, 1.4 x 10^{-2} mmol); **HPLC** (Chiralpak AD-H): 90:10 hexanes:isopropanol, 1.0 mL/min; 9.83 min (major), 18.4 min (minor).



#	Time	Area	Height	Width	Area%	Symmetry
1	9.827	2140.2	156.5	0.2279	79.708	0.659
2	18.418	544.9	20.6	0.4416	20.292	0.692



To a stirring solution of substrate (15.0 mg, 0.0306 mmol) in H_2O (24.3 μL, 1.35 mmol) and MeCN (6.8 mL, 0.01 M) stirring at –40 °C was added isobutyl thiol **21** as a 100 μL solution in MeCN (2.7 mg, 3.3 μL, 0.0306 mmol).[3] After thoroughly stirring for 5-10 minutes, *t*-BuOK (30.9 mg, 0.275 mmol) was added to the reaction mixture and allowed to stir vigorously until reaction was complete (2 h). The reaction was immediately plugged through silica gel (EtOAc) and concentrated under reduced pressure. The crude material was purified via preparative plate silica gel chromatography (4:1 hexanes:EtOAc) to yield products **22** (1.5:1 d.r.) and **23** in 81% combined yield (14.3 mg). The products were further separated by preparative plate silica gel chromatography to obtain each compound.

Compound **22a**: **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.66 (dd, J = 8.0, 1.5 Hz, 2H), 7.59 (dd, J = 8.0, 1.2 Hz, 2H), 7.46 – 7.41 (m, 2H), 7.40 – 7.35 (m, 4 H), 6.58 (dd, J = 15.5, 2.4 Hz, 1H), 6.22 (dd, J = 15.5, 2.2 Hz, 1H), 5.11-5.04 (m, 1H), 4.64 (m, 1H), 3.21 (quin, 5.4 Hz, 1H), 2.66-2.59 (m, 3H), 2.34 (dd, J = 6.8, 2.9 Hz, 2H), 2.20-2.07 (m, 2H), 1.75 (sex, J = 6.8 Hz), 1.69 – 1.60 (m, 1H), 1.60 – 1.50 (m, 3H), 1.50 – 1.42 (m, 3H), 1.42-1.35 (m, 1H), 1.26 – 1.24 (m, 7H), 1.10 (s, 6H), (dd, J = 6.8, 2.0 Hz, 6H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 208.1, 165.6, 148.9, 135.7, 135.6, 133.9, 132.9, 130.0, 129.9, 127.8, 127.7, 121.7, 71.0, 70.6, 49.4, 40.0, 38.6, 35.8, 34.7, 33.6, 29.7, 28.7, 27.4, 27.1, 22.2, 22.1, 22.1, 20.3, 19.4; **IR** (film, cm^{-1}) 2954, 2925, 2860, 1711, 1655, 1462, 1426, 1360, 1258, 1201, 1156, 1111; $\boldsymbol{R_f}$ = 0.70 (4:1 hexanes:EtOAc); $\boldsymbol{[\alpha]_D}$ 0.6 (*c* 0.33, $CHCl_3$); Exact mass calc'd for $C_{34}H_{48}O_4SSi$ $[M+H]^+$ requires *m/z* 581.3117. Found 581.3116 (Hi-Res ESI).

Compound **22b**: **^{1}H NMR** (400 MHz, $CDCl_3$) 7.67 (dd, 6.6, 1.4 Hz, 2H), 7.44 – 7.33 (m, 6H), 6.62 (dd, J = 15.6, 5.6 Hz, 1H), 5.82 (dd, J = 15.5, 1.4 Hz), 5.02 – 4.96 (m, 1H), 4.19 – 4.15 (m 1H), 3.07 – 3.00 (m, 1H), 2.55 – 2.53 (m, 2H), 2.43 – 2.36 (m, 1H), 2.33 (dd, 6.8, 3.0 Hz, 2H), 1.77 – 1.64 (m, 3H), 1.58 – 1.53 (m, 4H), 1.49 – 1.35 (m, 3H), 1.30 – 1.20 (m, 4H) 1.21 (d, J =

11.6 Hz, 3H), 1.07 (s, 9H), 0.95 (d, J = 6.8 Hz, 6H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 206.44, 165.79, 148.58, 135.78, 135.77, 133.91, 133.14, 129.82, 127.66, 127.65, 120.75, 72.87, 71.21, 48.42, 40.40, 40.29, 37.60, 34.42, 33.66, 29.69, 28.72, 28.56, 26.93, 22.15, 22.11, 22.01, 20.48, 19.26; **$[\alpha]_D$** 4.5 (*c* 0.33, $CHCl_3$); $\boldsymbol{R_f}$ = 0.65 (4:1 hexanes:EtOAc); **IR** (film, cm^{-1}) 2958, 2929, 2860, 2635, 1716, 1646, 1458, 1425, 1364, 1299, 1262, 1201, 1156, 1107; **$[\alpha]_D$** 4.5 (*c* 0.33, $CHCl_3$); Exact mass calc'd for $C_{34}H_{48}O_4SSi$ $[M+H]^+$ requires *m/z* 581.3117. Found 581.3116 (Hi-Res ESI).

Compound **23**: **^{1}H NMR** (500 MHz, $CDCl_3$) 7.4 (dt, J = 8.5, 1.5 Hz, 2H), 7.68 (dt, J = 8.5, 1.5 Hz, 2H), 7.49 – 7.39 (m, 6H), 5.08 – 5.04 (m, 1H), 4.15 (dt, J = 14.5, 6.0 Hz, 1H), 2.90 (dd, J = 20.0, 3.0 Hz, 1H), 2.82 – 2.79 (m, 1H), 2.59 – 2.55 (m, 3H), 2.32 – 2.23 (m, 2H), 2.20 – 2.16 (m, 1H), 2.08 – 2.02 (m, 2H), 1.95 (dd, J = 20.0, 16.0 Hz, 1H), 1.88 – 1.80 (m, 1H), 1.71 – 1.70 (m, 3H), 1.64 (sep, J = 8.5 Hz, 1H), 1.39 – 1.34 (m, 1H), 1.28 (d, J = 8 Hz, 3H), 1.23 – 1.21 (m, 1H), 1.1 (s, 9H), 1.01 – 0.97 (m, 1H), 0.91 (t, J = 8.5 Hz, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 209.1, 171.4, 135.7, 135.7, 133.6, 133.5, 130.0, 130.0, 127.9, 127.8, 72.3, 71.8, 58.9, 43.6, 43.1, 40.9, 40.0, 31.9, 30.9, 30.0, 29.2, 27.0, 26.9, 22.4, 21.8, 19.1, 18.2; $\boldsymbol{R_f}$ = 0.62 (4:1 hexanes:EtOAc); **IR** (film, cm^{-1}) 3072, 2954, 2860, 1724, 1458, 1426, 1373, 1246, 1164, 1107; **$[\alpha]_D$** −24.4 (*c* 0.50, $CHCl_3$); Exact mass calc'd for $C_{13}H_{19}O_3$ $[M+H]^+$ requires *m/z* 581.3114. Found 581.3114 (Hi-Res ESI).

III. Crystallographic Data

YALE CHEMICAL INSTRUMENTATION CENTER

Compound **5**

December 11, 2009

Experimental

Data Collection

A colorless block crystal of $C_{30}H_{38}O_4Si$ having approximate dimensions of 0.26 x 0.15 x 0.10 mm was mounted in a loop. All measurements were made on a Rigaku RAXIS RAPID imaging plate area detector with filtered Cu-Ka radiation.

Indexing was performed from 5 oscillations that were exposed for 300 seconds. The crystal-to-detector distance was 127.40 mm.

Cell constants and an orientation matrix for data collection corresponded to a primitive monoclinic cell with dimensions:

a = 9.7467(2) Å
b = 14.1261(3) Å b = 95.8453(14)°
c = 20.0486(4) Å
V = 2746.01(10) Å^3

For Z = 4 and F.W. = 490.71, the calculated density is 1.187 g/cm^3. Based on the systematic absences of:

0k0: k ± 2n

packing considerations, a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be:

$P2_1$ (#4)

The data were collected at a temperature of -180 ± 1°C to a maximum 2q value of 133.2°. A total of 154 oscillation images were collected. A sweep of data was done using w scans from 20.0 to 200.0° in 5.0° step, at c=0.0° and f = 270.0°. The exposure rate was 60.0 [sec./°]. A second sweep was performed using w scans from 20.0 to 200.0° in 5.0° step, at c=0.0° and f = 90.0°. The exposure rate was 60.0 [sec./°]. Another sweep was performed using w scans from 24.0 to 144.0° in 5.0° step, at c=54.0° and f = 180.0°. The exposure rate was 60.0 [sec./°]. Another sweep was performed using w scans from 24.0 to 134.0° in 5.0° step, at c=54.0° and f = 0.0°. The exposure rate was 60.0 [sec./°]. Another sweep was performed using w scans from 42.0 to 132.0° in 5.0° step, at c=54.0° and f = 270.0°. The exposure rate was 60.0 [sec./°]. Another sweep was performed using w scans from 42.0 to 132.0° in 5.0° step, at c=54.0° and f = 90.0°. The exposure rate was 60.0 [sec./°]. The crystal-to-detector distance was 127.40 mm. Readout was performed in the 0.100 mm pixel mode.

Data Reduction

Of the 24830 reflections that were collected, 8748 were unique (R_{int} = 0.033).

The linear absorption coefficient, m, for Cu-Ka radiation is 10.074 cm^{-1}. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods[1] and expanded using Fourier techniques[2]. Some non-hydrogen atoms were refined anisotropically, while the rest were refined isotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement[3] on F^2 was based on 8710 observed reflections and 622 variable parameters and converged (largest parameter shift was 0.30 times its esd) with unweighted and weighted agreement factors of:

$$R1 = S \; ||Fo| - |Fc|| \; / \; S \; |Fo| = 0.0766$$

$$wR2 = [\; S \; (\; w \; (Fo^2 - Fc^2)^2 \;) / \; S \; w(Fo^2)^2]^{1/2} = 0.2122$$

The standard deviation of an observation of unit weight[4] was 1.05. Unit weights were used. The maximum and minimum peaks on the final difference Fourier map corresponded to 1.07 and -0.41 $e^-/Å^3$, respectively. The absolute structure was deduced based on Flack parameter, 0.02(3), using 4037 Friedel pairs.[5]

Neutral atom scattering factors were taken from Cromer and Waber[6]. Anomalous dispersion effects were included in Fcalc[7]; the values for Df' and Df'' were those of Creagh and McAuley[8]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[9]. All calculations were performed using the CrystalStructure[10] crystallographic software package except for refinement, which was performed using SHELXL-97[11].

References

(1) SHELX97: Sheldrick, G.M. (1997).

(2) DIRDIF99: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M.(1999). The DIRDIF-99 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(3) Least Squares function minimized: (SHELXL97)

$$Sw(F_o^2-F_c^2)^2$$ where w = Least Squares weights.

(4) Standard deviation of an observation of unit weight:

$$[Sw(F_o^2-F_c^2)^2/(N_o-N_v)]^{1/2}$$

where: N_o = number of observations

N_V = number of variables

(5) Flack, H. D. (1983), Acta Cryst. A39, 876-881.

(6) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(7) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(8) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(9) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(10) CrystalStructure 3.8: Crystal Structure Analysis Package, Rigaku and Rigaku Americas (2000-2007). 9009 New Trails Dr. The Woodlands TX 77381 USA.

(11) SHELX97: Sheldrick, G.M. (1997).

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{30}H_{38}O_4Si$
Formula Weight	490.71
Crystal Color, Habit	colorless, block
Crystal Dimensions	0.26 X 0.15 X 0.10 mm
Crystal System	monoclinic
Lattice Type	Primitive
Indexing Images	5 oscillations @ 300.0 seconds
Detector Position	127.40 mm
Pixel Size	0.100 mm

Lattice Parameters	a = 9.7467(2) Å b = 14.1261(3) Å c = 20.0486(4) Å b = 95.8453(14) ^{o} V = 2746.01(10) Å^{3}
Space Group	$P2_1$ (#4)
Z value	4
D_{calc}	1.187 g/cm^{3}
F_{000}	1056.00
m(CuKa)	10.074 cm^{-1}

B. Intensity Measurements

Diffractometer	Rigaku RAXIS-RAPID
Radiation	CuKa (l = 1.54187 Å)
Detector Aperture	280 mm x 256 mm
Data Images	154 exposures
w oscillation Range (c=0.0, f=270.0)	20.0 - 200.0^{o}
Exposure Rate	60.0 sec./^{o}
w oscillation Range (c=0.0, f=90.0)	20.0 - 200.0^{o}
Exposure Rate	60.0 sec./^{o}
w oscillation Range (c=54.0, f=180.0)	24.0 - 144.0^{o}
Exposure Rate	60.0 sec./^{o}
w oscillation Range (c=54.0, f=0.0)	24.0 - 134.0^{o}
Exposure Rate	60.0 sec./^{o}

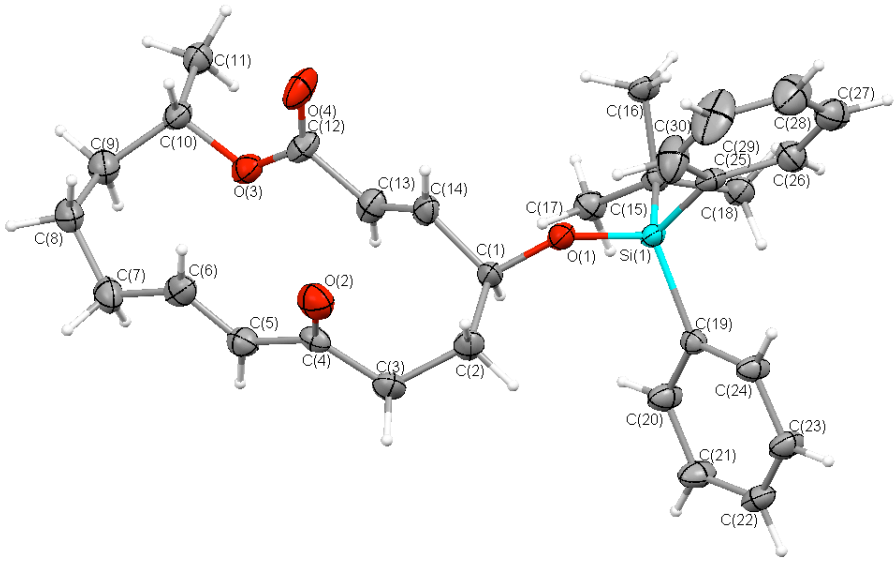
w oscillation Range (c=54.0, f=270.0)	42.0 - 132.0°
Exposure Rate	60.0 sec./°
w oscillation Range (c=54.0, f=90.0)	42.0 - 132.0°
Exposure Rate	60.0 sec./°
Detector Position	127.40 mm
Pixel Size	0.100 mm
$2q_{max}$	133.2°
No. of Reflections Measured	Total: 24830 Unique: 8710 (R_{int} = 0.033) Friedel pairs: 4037
Corrections	Lorentz-polarization

C. Structure Solution and Refinement

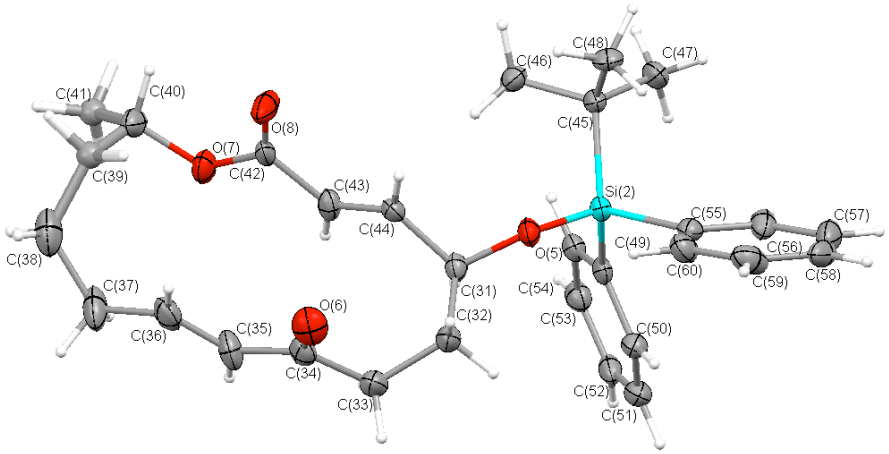
Structure Solution	Direct Methods (SHELX97)
Refinement	Full-matrix least-squares on F^2
Function Minimized	S w $(Fo^2 - Fc^2)^2$
Least Squares Weights	w = 1/ [$s^2(Fo^2)$ + $(0.1285 \cdot P)^2$ + 2.6363 · P] where P = $(Max(Fo^2,0) + 2Fc^2)/3$
$2q_{max}$ cutoff	133.2°
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	8710
No. Variables	622
Reflection/Parameter Ratio	14.00

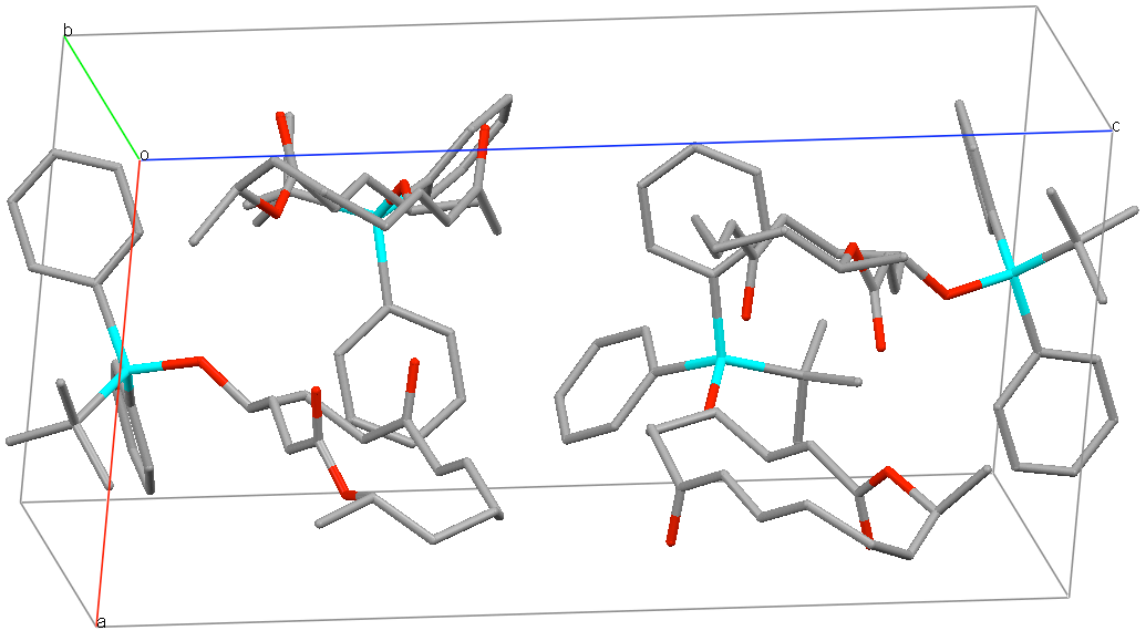
Residuals: R1 (I>2.00s(I))	0.0766
Residuals: R (All reflections)	0.0846
Residuals: wR2 (All reflections)	0.2122
Goodness of Fit Indicator	1.051
Flack Parameter	0.02(3)
Max Shift/Error in Final Cycle	0.305
Maximum peak in Final Diff. Map	1.07 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.41 $e^-/Å^3$

First independent molecule

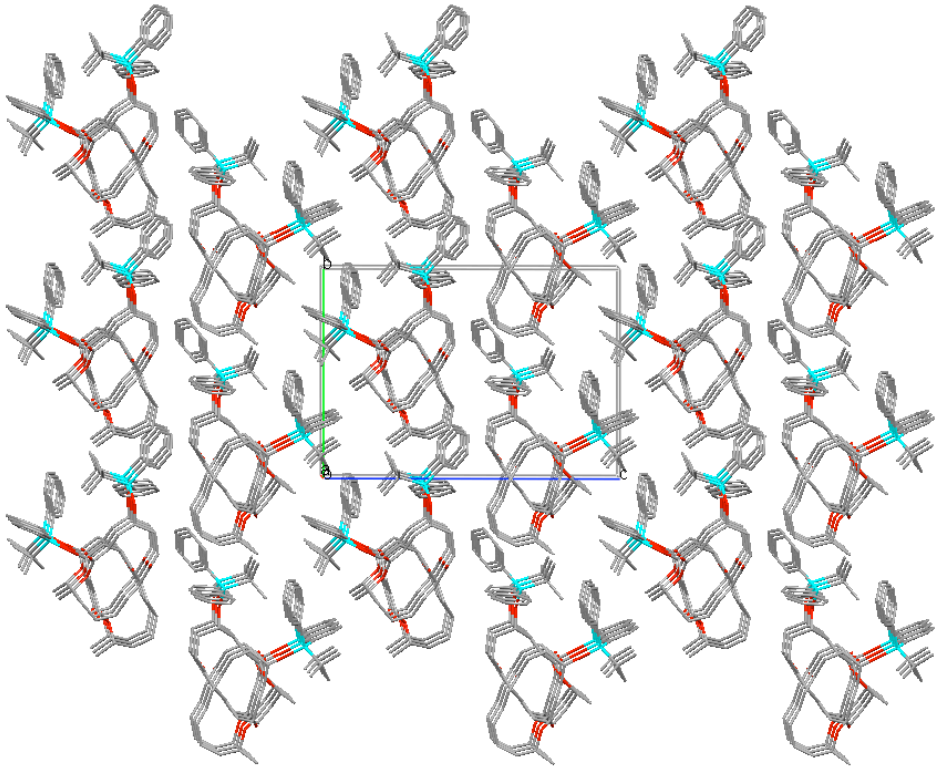


Second independent molecule

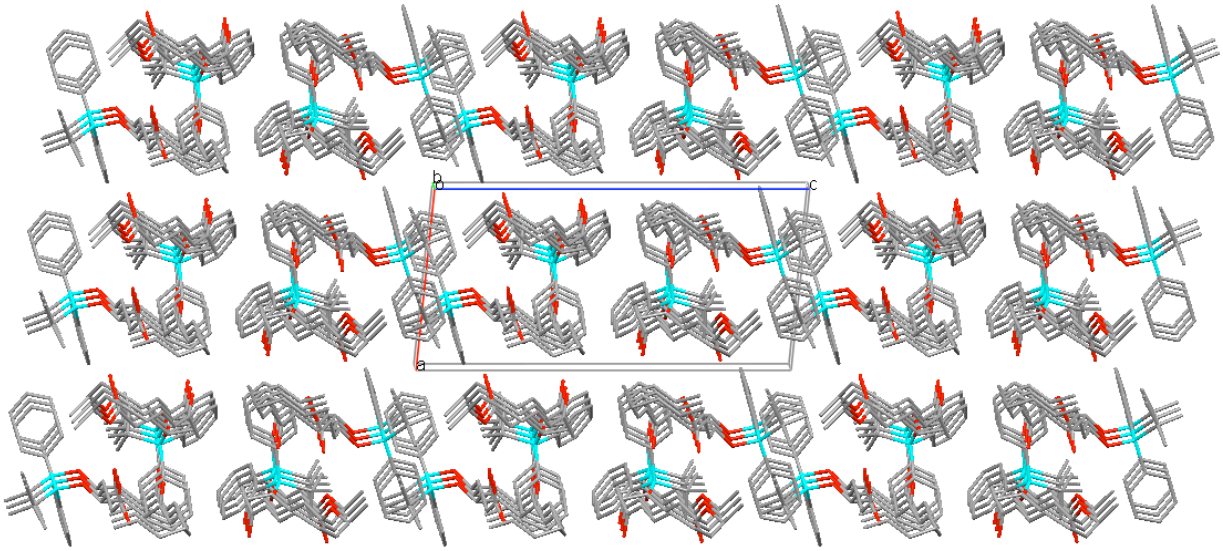

C(48)
C(46)
C(47)
C(45)
C(41)
C(40)
O(8)
O(7)
C(42)
C(39)
C(43)
Si(2)
C(55)
C(57)
C(44)
C(49)
C(56)
C(38)
O(5)
C(60)
C(59)
C(58)
C(37)
C(54)
C(31)
C(53)
C(32)
O(6)
C(50)
C(36)
C(35)
C(34)
C(52)
C(33)
C(51)



Packing diagram – View down the *a*-axis



Packing diagram – View down the *b*-axis



Packing diagram – View down the *c*-axis

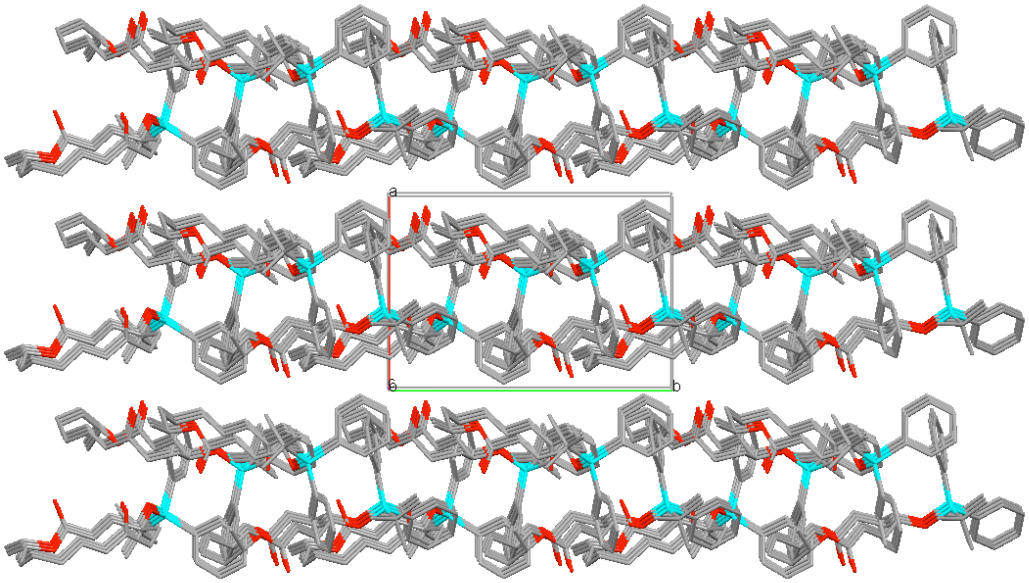


Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
Si(1)	0.64369(15)	0.70823(8)	0.07078(6)	2.52(2)
Si(2)	0.40449(15)	0.98122(8)	0.33782(6)	2.69(2)
O(1)	0.6143(3)	0.6572(2)	0.14111(16)	3.24(6)
O(2)	0.6075(5)	0.5678(3)	0.3539(2)	5.27(10)
O(3)	0.8184(4)	0.3148(2)	0.27329(18)	3.81(7)
O(4)	0.5965(5)	0.3293(3)	0.2332(2)	5.50(10)
O(5)	0.3103(3)	0.8895(2)	0.35498(17)	3.08(6)
O(6)	0.1189(5)	0.5995(3)	0.4071(2)	5.22(9)
O(7)	0.2722(4)	0.5424(2)	0.19486(19)	4.03(7)
O(8)	0.0884(6)	0.6387(3)	0.1982(2)	6.59(14)
C(1)	0.7099(6)	0.6232(3)	0.1944(2)	3.30(9)
C(2)	0.7024(7)	0.6881(3)	0.2548(2)	3.90(11)
C(3)	0.7925(7)	0.6532(4)	0.3170(2)	4.22(12)
C(4)	0.7328(5)	0.5713(3)	0.3514(2)	2.76(8)
C(5)	0.8242(6)	0.4987(4)	0.3838(2)	3.79(10)
C(6)	0.7829(7)	0.4139(4)	0.4037(2)	4.46(12)
C(7)	0.8804(7)	0.3417(4)	0.4356(3)	4.50(12)
C(8)	0.8435(7)	0.2404(4)	0.4207(2)	4.54(12)
C(9)	0.8891(7)	0.1968(5)	0.3563(2)	4.51(12)
C(10)	0.7987(6)	0.2168(3)	0.2921(2)	3.55(10)
C(11)	0.8357(6)	0.1570(4)	0.2342(2)	4.08(11)
C(12)	0.7119(7)	0.3600(3)	0.2434(2)	3.68(11)
C(13)	0.7559(7)	0.4560(4)	0.2235(2)	3.88(11)
C(14)	0.6671(7)	0.5232(4)	0.2072(2)	3.81(11)
C(15)	0.7302(5)	0.6232(3)	0.0145(2)	2.82(8)
C(16)	0.6237(6)	0.5492(3)	-0.0107(2)	3.43(10)
C(17)	0.8531(6)	0.5716(4)	0.0510(2)	3.62(10)
C(18)	0.7798(6)	0.6763(3)	-0.0459(2)	3.55(10)
C(19)	0.7451(5)	0.8204(3)	0.0877(2)	2.53(8)
C(20)	0.8846(6)	0.8202(4)	0.1129(3)	4.02(11)
C(21)	0.9563(6)	0.9035(4)	0.1239(3)	4.36(11)
C(22)	0.8927(6)	0.9898(4)	0.1098(2)	3.80(10)
C(23)	0.7550(6)	0.9915(3)	0.0865(3)	4.15(11)
C(24)	0.6834(5)	0.9078(3)	0.0768(2)	3.41(9)
C(25)	0.4641(5)	0.7397(3)	0.0337(2)	2.86(8)
C(26)	0.4341(6)	0.7765(4)	-0.0304(2)	4.07(11)
C(27)	0.2981(7)	0.7985(4)	-0.0558(3)	4.89(13)

Table 1. Atomic coordinates and B_{iso}/B_{eq} (continued)

atom	x	y	z	B_{eq}
C(28)	0.1926(8)	0.7821(6)	-0.0178(4)	6.41(18)
C(29)	0.2178(8)	0.7469(7)	0.0453(6)	8.2(2)
C(30)	0.3525(8)	0.7255(6)	0.0714(4)	6.16(17)
C(31)	0.3423(5)	0.7911(3)	0.3559(2)	2.84(8)
C(32)	0.3148(6)	0.7528(3)	0.4246(2)	3.49(10)
C(33)	0.3506(7)	0.6480(4)	0.4337(2)	4.23(12)
C(34)	0.2403(6)	0.5805(3)	0.4032(2)	3.43(10)
C(35)	0.2880(7)	0.4972(3)	0.3680(3)	4.67(13)
C(36)	0.2082(8)	0.4404(4)	0.3304(3)	4.73(13)
C(37)	0.2555(8)	0.3586(4)	0.2917(4)	6.28(19)
C(38)	0.1717(9)	0.3367(5)	0.2239(4)	6.65(19)
C(39)	0.1235(12)	0.4167(9)	0.1765(6)	9.7(2)
C(40)	0.1975(7)	0.4800(4)	0.1466(2)	4.48(12)
C(41)	0.2975(18)	0.4365(14)	0.1026(8)	15.8(6)
C(42)	0.2057(7)	0.6190(3)	0.2163(2)	3.58(11)
C(43)	0.2984(6)	0.6710(3)	0.2657(2)	3.18(9)
C(44)	0.2544(5)	0.7425(3)	0.3009(2)	2.77(8)
C(45)	0.3530(5)	1.0191(3)	0.2481(2)	2.77(8)
C(46)	0.3582(6)	0.9347(4)	0.1995(2)	3.39(9)
C(47)	0.4407(7)	1.0991(3)	0.2263(2)	4.10(11)
C(48)	0.2032(5)	1.0522(4)	0.2449(2)	3.63(10)
C(49)	0.5925(5)	0.9565(3)	0.3567(2)	2.86(8)
C(50)	0.6533(5)	0.9622(3)	0.4233(2)	3.13(9)
C(51)	0.7923(6)	0.9455(3)	0.4398(2)	4.07(12)
C(52)	0.8776(6)	0.9230(3)	0.3910(3)	3.90(11)
C(53)	0.8210(6)	0.9165(3)	0.3253(2)	3.61(10)
C(54)	0.6830(5)	0.9325(3)	0.3076(2)	3.05(9)
C(55)	0.3541(5)	1.0760(3)	0.3970(2)	3.07(9)
C(56)	0.4212(6)	1.1617(3)	0.4027(2)	3.83(10)
C(57)	0.3767(8)	1.2332(4)	0.4442(3)	5.32(16)
C(58)	0.2684(9)	1.2188(4)	0.4804(2)	5.29(17)
C(59)	0.1994(8)	1.1328(5)	0.4760(3)	5.19(15)
C(60)	0.2445(7)	1.0616(4)	0.4345(2)	4.07(11)

$B_{eq} = 8/3\ p^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos g + 2U_{13}(aa^*cc^*)\cos b + 2U_{23}(bb^*cc^*)\cos a)$

Table 2. Atomic coordinates and B $_{iso}$ involving hydrogens/B_{eq}

atom	x	y	z	B_{eq}
H(1)	0.8053	0.6239	0.1802	3.96
H(2)	0.6054	0.6922	0.2652	4.68
H(3)	0.7323	0.7524	0.2433	4.68
H(4)	0.8837	0.6346	0.3037	5.07
H(5)	0.8072	0.7062	0.3493	5.07
H(6)	0.9198	0.5130	0.3912	4.55
H(7)	0.6874	0.3991	0.3973	5.35
H(8)	0.8875	0.3508	0.4847	5.40
H(9)	0.9728	0.3538	0.4209	5.40
H(10)	0.7419	0.2345	0.4188	5.44
H(11)	0.8833	0.2016	0.4591	5.44
H(12)	0.9832	0.2198	0.3509	5.41
H(13)	0.8949	0.1273	0.3624	5.41
H(14)	0.6998	0.2061	0.2993	4.26
H(15)	0.7538	0.1478	0.2023	4.90
H(16)	0.8700	0.0953	0.2510	4.90
H(17)	0.9075	0.1890	0.2117	4.90
H(18)	0.8514	0.4686	0.2227	4.66
H(19)	0.5716	0.5084	0.2034	4.58
H(20)	0.6277	0.4958	0.0207	4.12
H(21)	0.6436	0.5266	-0.0550	4.12
H(22)	0.5314	0.5774	-0.0141	4.12
H(23)	0.9371	0.6087	0.0476	4.34
H(24)	0.8629	0.5093	0.0305	4.34
H(25)	0.8384	0.5637	0.0983	4.34
H(26)	0.7062	0.6765	-0.0830	4.26
H(27)	0.8613	0.6444	-0.0601	4.26
H(28)	0.8035	0.7416	-0.0329	4.26
H(29)	0.9303	0.7617	0.1226	4.83
H(30)	1.0506	0.9018	0.1414	5.24
H(31)	0.9434	1.0470	0.1162	4.57
H(32)	0.7096	1.0502	0.0771	4.98
H(33)	0.5877	0.9103	0.0620	4.09
H(34)	0.5070	0.7871	-0.0575	4.89
H(35)	0.2799	0.8248	-0.0994	5.87
H(36)	0.1005	0.7953	-0.0355	7.69
H(37)	0.1436	0.7368	0.0717	9.79

Table 2. Atomic coordinates and B_{iso} involving hydrogens/B_{eq} (continued)

atom	x	y	z	B_{eq}
H(38)	0.3688	0.7009	0.1156	7.40
H(39)	0.4419	0.7822	0.3494	3.41
H(40)	0.2160	0.7620	0.4305	4.19
H(41)	0.3695	0.7898	0.4598	4.19
H(42)	0.4372	0.6354	0.4134	5.08
H(43)	0.3681	0.6345	0.4823	5.08
H(44)	0.3841	0.4840	0.3729	5.60
H(45)	0.1117	0.4520	0.3275	5.68
H(46)	0.2548	0.3014	0.3201	7.53
H(47)	0.3523	0.3703	0.2832	7.53
H(48)	0.2279	0.2934	0.1990	7.98
H(49)	0.0887	0.3010	0.2337	7.98
H(50)	0.0648	0.3858	0.1396	11.68
H(51)	0.0601	0.4546	0.2014	11.68
H(52)	0.1315	0.5199	0.1173	5.37
H(53)	0.3078	0.4785	0.0645	19.02
H(54)	0.2622	0.3750	0.0860	19.02
H(55)	0.3873	0.4279	0.1286	19.02
H(56)	0.3927	0.6531	0.2727	3.82
H(57)	0.1623	0.7637	0.2905	3.32
H(58)	0.4505	0.9304	0.1844	4.07
H(59)	0.2899	0.9441	0.1607	4.07
H(60)	0.3374	0.8761	0.2226	4.07
H(61)	0.3981	1.1598	0.2360	4.92
H(62)	0.4479	1.0942	0.1780	4.92
H(63)	0.5329	1.0954	0.2506	4.92
H(64)	0.1415	0.9985	0.2338	4.36
H(65)	0.1867	1.1013	0.2105	4.36
H(66)	0.1853	1.0782	0.2885	4.36
H(67)	0.5973	0.9779	0.4578	3.75
H(68)	0.8299	0.9496	0.4854	4.89
H(69)	0.9733	0.9122	0.4025	4.68
H(70)	0.8787	0.9007	0.2915	4.33
H(71)	0.6472	0.9275	0.2619	3.66
H(72)	0.4984	1.1727	0.3784	4.59
H(73)	0.4232	1.2924	0.4469	6.38
H(74)	0.2399	1.2675	0.5086	6.35

Table 2. Atomic coordinates and B_{iso} involving hydrogens/B_{eq} (continued)

atom	x	y	z	B_{eq}
H(75)	0.1227	1.1222	0.5008	6.23
H(76)	0.1986	1.0022	0.4321	4.88

$B_{eq} = 8/3\ p^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos g + 2U_{13}(aa^*cc^*)\cos b + 2U_{23}(bb^*cc^*)\cos a)$

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Si(1)	0.0394(8)	0.0236(6)	0.0330(5)	-0.0058(5)	0.0057(5)	-0.0019(4)
Si(2)	0.0452(8)	0.0226(5)	0.0331(5)	-0.0017(5)	-0.0018(5)	-0.0004(4)
O(1)	0.051(2)	0.0345(19)	0.0390(17)	-0.0076(16)	0.0087(15)	-0.0001(13)
O(2)	0.074(3)	0.078(3)	0.049(2)	-0.007(2)	0.010(2)	-0.004(2)
O(3)	0.056(2)	0.042(2)	0.049(2)	-0.0105(18)	0.0130(18)	-0.0001(16)
O(4)	0.052(3)	0.041(2)	0.115(3)	0.008(2)	0.005(2)	0.028(2)
O(5)	0.043(2)	0.0253(16)	0.0482(19)	0.0019(14)	0.0011(15)	-0.0040(13)
O(6)	0.070(3)	0.065(2)	0.067(2)	-0.003(2)	0.027(2)	-0.000(2)
O(7)	0.065(2)	0.037(2)	0.051(2)	0.0012(18)	0.0081(18)	-0.0147(16)
O(8)	0.101(4)	0.053(2)	0.084(3)	0.038(2)	-0.050(2)	-0.036(2)
C(1)	0.066(3)	0.031(2)	0.028(2)	-0.006(2)	0.001(2)	-0.0004(18)
C(2)	0.072(4)	0.035(2)	0.041(2)	0.004(2)	0.007(2)	-0.006(2)
C(3)	0.074(4)	0.046(3)	0.040(2)	-0.010(3)	0.002(2)	-0.011(2)
C(4)	0.030(3)	0.047(2)	0.030(2)	-0.001(2)	0.0060(19)	-0.0095(19)
C(5)	0.047(3)	0.055(3)	0.042(2)	-0.007(2)	0.005(2)	-0.004(2)
C(6)	0.083(4)	0.053(3)	0.036(2)	-0.015(3)	0.022(2)	-0.009(2)
C(7)	0.059(4)	0.067(4)	0.045(3)	-0.006(3)	0.004(2)	0.014(2)
C(8)	0.068(4)	0.063(3)	0.041(2)	-0.011(3)	0.000(2)	0.009(2)
C(9)	0.060(4)	0.059(3)	0.051(3)	-0.002(3)	0.002(2)	0.008(2)
C(10)	0.053(3)	0.035(2)	0.048(2)	-0.009(2)	0.009(2)	0.001(2)
C(11)	0.066(4)	0.039(3)	0.049(3)	0.001(2)	0.004(2)	0.003(2)
C(12)	0.053(4)	0.034(2)	0.056(3)	-0.006(2)	0.022(2)	0.000(2)
C(13)	0.066(4)	0.041(3)	0.044(2)	0.001(2)	0.022(2)	-0.002(2)
C(14)	0.075(4)	0.040(2)	0.031(2)	-0.007(2)	0.011(2)	0.003(2)
C(15)	0.042(3)	0.032(2)	0.034(2)	-0.005(2)	0.009(2)	-0.0073(18)
C(16)	0.059(3)	0.027(2)	0.045(2)	-0.004(2)	0.006(2)	-0.0098(19)
C(17)	0.048(3)	0.043(3)	0.047(2)	0.005(2)	0.003(2)	-0.006(2)
C(18)	0.059(3)	0.041(2)	0.037(2)	-0.004(2)	0.012(2)	-0.007(2)
C(19)	0.032(3)	0.034(2)	0.030(2)	-0.001(2)	0.0033(18)	-0.0011(17)
C(20)	0.048(3)	0.032(2)	0.071(3)	0.002(2)	-0.008(2)	-0.007(2)
C(21)	0.038(3)	0.045(3)	0.079(4)	-0.005(2)	-0.010(2)	-0.006(2)
C(22)	0.054(4)	0.034(2)	0.057(3)	-0.012(2)	0.009(2)	-0.009(2)

C(23)	0.055(4)	0.029(2)	0.072(3)	-0.002(2)	-0.000(2)	-0.006(2)
C(24)	0.039(3)	0.030(2)	0.059(3)	0.003(2)	-0.004(2)	-0.010(2)
C(25)	0.034(3)	0.025(2)	0.050(2)	-0.0046(19)	0.003(2)	-0.0060(19)
C(26)	0.058(4)	0.049(3)	0.047(3)	0.003(2)	-0.003(2)	-0.001(2)
C(27)	0.063(4)	0.044(3)	0.074(4)	0.007(3)	-0.015(3)	-0.006(2)
C(28)	0.052(5)	0.070(4)	0.118(6)	0.004(3)	-0.009(4)	0.018(4)
C(29)	0.033(4)	0.113(7)	0.168(9)	0.000(4)	0.029(4)	0.055(6)
C(30)	0.064(5)	0.085(5)	0.088(4)	0.003(4)	0.019(3)	0.035(4)
C(31)	0.037(3)	0.025(2)	0.046(2)	0.002(2)	0.003(2)	-0.0047(18)
C(32)	0.061(3)	0.033(2)	0.037(2)	-0.004(2)	-0.002(2)	-0.0022(19)
C(33)	0.081(4)	0.039(3)	0.039(2)	0.002(2)	-0.004(2)	0.003(2)
C(34)	0.056(4)	0.035(2)	0.041(2)	-0.009(2)	0.010(2)	0.006(2)
C(35)	0.070(4)	0.027(2)	0.082(4)	0.000(2)	0.018(3)	-0.005(2)
C(36)	0.088(5)	0.033(3)	0.060(3)	-0.002(3)	0.015(3)	0.010(2)
C(37)	0.071(5)	0.035(3)	0.128(6)	0.005(3)	-0.013(4)	-0.026(3)
C(38)	0.091(6)	0.045(3)	0.117(6)	-0.005(3)	0.013(5)	-0.023(3)
C(40)	0.080(4)	0.040(3)	0.046(2)	0.019(3)	-0.011(2)	-0.011(2)
C(42)	0.081(4)	0.020(2)	0.034(2)	0.003(2)	-0.001(2)	-0.0013(18)
C(43)	0.052(3)	0.028(2)	0.043(2)	-0.006(2)	0.016(2)	-0.0023(19)
C(44)	0.046(3)	0.026(2)	0.033(2)	0.003(2)	0.002(2)	0.0048(17)
C(45)	0.044(3)	0.026(2)	0.033(2)	-0.002(2)	-0.0060(19)	0.0009(17)
C(46)	0.049(3)	0.046(2)	0.033(2)	-0.009(2)	-0.003(2)	-0.003(2)
C(47)	0.073(4)	0.035(2)	0.045(2)	-0.010(2)	-0.007(2)	0.009(2)
C(48)	0.046(3)	0.050(3)	0.038(2)	0.004(2)	-0.012(2)	0.004(2)
C(49)	0.047(3)	0.020(2)	0.041(2)	-0.0069(19)	0.001(2)	0.0001(16)
C(50)	0.043(3)	0.035(2)	0.039(2)	0.005(2)	-0.004(2)	0.0008(19)
C(51)	0.066(4)	0.036(2)	0.049(3)	0.002(2)	-0.016(2)	0.003(2)
C(52)	0.046(3)	0.027(2)	0.071(3)	0.001(2)	-0.014(2)	-0.003(2)
C(53)	0.045(3)	0.034(2)	0.058(3)	-0.003(2)	0.003(2)	0.002(2)
C(54)	0.047(3)	0.026(2)	0.042(2)	-0.002(2)	0.003(2)	0.0013(19)
C(55)	0.046(3)	0.035(2)	0.034(2)	0.008(2)	-0.007(2)	0.0004(19)
C(56)	0.044(3)	0.037(2)	0.062(3)	0.003(2)	-0.011(2)	-0.007(2)
C(57)	0.088(5)	0.039(3)	0.068(4)	0.014(3)	-0.024(3)	-0.016(2)
C(58)	0.110(6)	0.046(3)	0.040(2)	0.036(3)	-0.016(3)	-0.014(2)
C(59)	0.093(5)	0.063(4)	0.043(3)	0.030(3)	0.016(3)	0.008(2)
C(60)	0.071(4)	0.037(3)	0.047(3)	0.012(2)	0.007(2)	0.002(2)

The general temperature factor expression: $\exp(-2p^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
Si(1)	O(1)	1.634(3)	Si(1)	C(15)	1.903(5)
Si(1)	C(19)	1.880(4)	Si(1)	C(25)	1.884(5)
Si(2)	O(5)	1.645(3)	Si(2)	C(45)	1.895(4)
Si(2)	C(49)	1.866(5)	Si(2)	C(55)	1.888(5)
O(1)	C(1)	1.427(6)	O(2)	C(4)	1.228(7)
O(3)	C(10)	1.453(6)	O(3)	C(12)	1.311(7)
O(4)	C(12)	1.203(8)	O(5)	C(31)	1.424(5)
O(6)	C(34)	1.223(8)	O(7)	C(40)	1.449(6)
O(7)	C(42)	1.353(6)	O(8)	C(42)	1.196(8)
C(1)	C(2)	1.526(7)	C(1)	C(14)	1.502(7)
C(2)	C(3)	1.532(7)	C(3)	C(4)	1.494(8)
C(4)	C(5)	1.466(7)	C(5)	C(6)	1.337(8)
C(6)	C(7)	1.493(9)	C(7)	C(8)	1.498(9)
C(8)	C(9)	1.537(8)	C(9)	C(10)	1.510(7)
C(10)	C(11)	1.509(8)	C(12)	C(13)	1.488(8)
C(13)	C(14)	1.304(8)	C(15)	C(16)	1.523(7)
C(15)	C(17)	1.524(7)	C(15)	C(18)	1.544(7)
C(19)	C(20)	1.401(7)	C(19)	C(24)	1.382(7)
C(20)	C(21)	1.375(8)	C(21)	C(22)	1.383(8)
C(22)	C(23)	1.376(8)	C(23)	C(24)	1.376(7)
C(25)	C(26)	1.388(7)	C(25)	C(30)	1.402(10)
C(26)	C(27)	1.406(9)	C(27)	C(28)	1.361(12)
C(28)	C(29)	1.358(14)	C(29)	C(30)	1.396(11)
C(31)	C(32)	1.528(7)	C(31)	C(44)	1.495(6)
C(32)	C(33)	1.529(7)	C(33)	C(34)	1.519(8)
C(34)	C(35)	1.472(8)	C(35)	C(36)	1.303(9)
C(36)	C(37)	1.492(10)	C(37)	C(38)	1.544(12)
C(38)	C(39)	1.519(15)	C(39)	C(40)	1.330(15)
C(40)	C(41)	1.511(19)	C(42)	C(43)	1.469(7)
C(43)	C(44)	1.327(7)	C(45)	C(46)	1.544(7)
C(45)	C(47)	1.509(7)	C(45)	C(48)	1.528(7)
C(49)	C(50)	1.408(6)	C(49)	C(54)	1.427(7)
C(50)	C(51)	1.382(8)	C(51)	C(52)	1.385(9)
C(52)	C(53)	1.377(8)	C(53)	C(54)	1.375(7)
C(55)	C(56)	1.375(7)	C(55)	C(60)	1.383(8)
C(56)	C(57)	1.404(9)	C(57)	C(58)	1.356(11)
C(58)	C(59)	1.388(10)	C(59)	C(60)	1.405(9)

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C(1)	H(1)	1.000	C(2)	H(2)	0.990
C(2)	H(3)	0.990	C(3)	H(4)	0.990
C(3)	H(5)	0.990	C(5)	H(6)	0.950
C(6)	H(7)	0.950	C(7)	H(8)	0.990
C(7)	H(9)	0.990	C(8)	H(10)	0.990
C(8)	H(11)	0.990	C(9)	H(12)	0.990
C(9)	H(13)	0.990	C(10)	H(14)	1.000
C(11)	H(15)	0.980	C(11)	H(16)	0.980
C(11)	H(17)	0.980	C(13)	H(18)	0.950
C(14)	H(19)	0.950	C(16)	H(20)	0.980
C(16)	H(21)	0.980	C(16)	H(22)	0.980
C(17)	H(23)	0.980	C(17)	H(24)	0.980
C(17)	H(25)	0.980	C(18)	H(26)	0.980
C(18)	H(27)	0.980	C(18)	H(28)	0.980
C(20)	H(29)	0.950	C(21)	H(30)	0.950
C(22)	H(31)	0.950	C(23)	H(32)	0.950
C(24)	H(33)	0.950	C(26)	H(34)	0.950
C(27)	H(35)	0.950	C(28)	H(36)	0.950
C(29)	H(37)	0.950	C(30)	H(38)	0.950
C(31)	H(39)	1.000	C(32)	H(40)	0.990
C(32)	H(41)	0.990	C(33)	H(42)	0.990
C(33)	H(43)	0.990	C(35)	H(44)	0.950
C(36)	H(45)	0.950	C(37)	H(46)	0.990
C(37)	H(47)	0.990	C(38)	H(48)	0.990
C(38)	H(49)	0.990	C(39)	H(50)	0.990
C(39)	H(51)	0.990	C(40)	H(52)	1.000
C(41)	H(53)	0.980	C(41)	H(54)	0.980
C(41)	H(55)	0.980	C(43)	H(56)	0.950
C(44)	H(57)	0.950	C(46)	H(58)	0.980
C(46)	H(59)	0.980	C(46)	H(60)	0.980
C(47)	H(61)	0.980	C(47)	H(62)	0.980
C(47)	H(63)	0.980	C(48)	H(64)	0.980
C(48)	H(65)	0.980	C(48)	H(66)	0.980
C(50)	H(67)	0.950	C(51)	H(68)	0.950
C(52)	H(69)	0.950	C(53)	H(70)	0.950
C(54)	H(71)	0.950	C(56)	H(72)	0.950
C(57)	H(73)	0.950	C(58)	H(74)	0.950
C(59)	H(75)	0.950	C(60)	H(76)	0.950

Table 6. Bond angles (º)

atom	atom	atom	angle	atom	atom	atom	angle
O(1)	Si(1)	C(15)	111.12(19)	O(1)	Si(1)	C(19)	110.56(19)
O(1)	Si(1)	C(25)	102.0(2)	C(15)	Si(1)	C(19)	112.5(2)
C(15)	Si(1)	C(25)	111.4(2)	C(19)	Si(1)	C(25)	108.8(2)
O(5)	Si(2)	C(45)	108.8(2)	O(5)	Si(2)	C(49)	111.59(19)
O(5)	Si(2)	C(55)	103.9(2)	C(45)	Si(2)	C(49)	113.9(2)
C(45)	Si(2)	C(55)	109.6(2)	C(49)	Si(2)	C(55)	108.6(2)
Si(1)	O(1)	C(1)	129.5(3)	C(10)	O(3)	C(12)	117.6(4)
Si(2)	O(5)	C(31)	130.2(3)	C(40)	O(7)	C(42)	118.2(4)
O(1)	C(1)	C(2)	108.1(4)	O(1)	C(1)	C(14)	105.9(4)
C(2)	C(1)	C(14)	113.0(4)	C(1)	C(2)	C(3)	112.6(4)
C(2)	C(3)	C(4)	114.0(5)	O(2)	C(4)	C(3)	119.0(5)
O(2)	C(4)	C(5)	121.0(5)	C(3)	C(4)	C(5)	119.9(5)
C(4)	C(5)	C(6)	124.8(5)	C(5)	C(6)	C(7)	122.9(6)
C(6)	C(7)	C(8)	116.0(5)	C(7)	C(8)	C(9)	117.7(5)
C(8)	C(9)	C(10)	116.6(5)	O(3)	C(10)	C(9)	108.6(4)
O(3)	C(10)	C(11)	106.6(4)	C(9)	C(10)	C(11)	112.7(5)
O(3)	C(12)	O(4)	125.8(5)	O(3)	C(12)	C(13)	109.3(5)
O(4)	C(12)	C(13)	124.9(5)	C(12)	C(13)	C(14)	122.0(6)
C(1)	C(14)	C(13)	122.7(6)	Si(1)	C(15)	C(16)	107.6(3)
Si(1)	C(15)	C(17)	113.1(3)	Si(1)	C(15)	C(18)	110.6(3)
C(16)	C(15)	C(17)	107.9(4)	C(16)	C(15)	C(18)	109.3(3)
C(17)	C(15)	C(18)	108.4(4)	Si(1)	C(19)	C(20)	122.5(3)
Si(1)	C(19)	C(24)	120.8(3)	C(20)	C(19)	C(24)	116.7(4)
C(19)	C(20)	C(21)	121.0(5)	C(20)	C(21)	C(22)	120.8(5)
C(21)	C(22)	C(23)	119.1(5)	C(22)	C(23)	C(24)	119.8(5)
C(19)	C(24)	C(23)	122.6(5)	Si(1)	C(25)	C(26)	123.6(4)
Si(1)	C(25)	C(30)	119.6(4)	C(26)	C(25)	C(30)	116.8(5)
C(25)	C(26)	C(27)	121.4(5)	C(26)	C(27)	C(28)	119.8(6)
C(27)	C(28)	C(29)	120.6(7)	C(28)	C(29)	C(30)	120.2(8)
C(25)	C(30)	C(29)	121.2(7)	O(5)	C(31)	C(32)	107.4(3)
O(5)	C(31)	C(44)	109.3(3)	C(32)	C(31)	C(44)	111.2(4)
C(31)	C(32)	C(33)	113.1(4)	C(32)	C(33)	C(34)	114.6(5)
O(6)	C(34)	C(33)	119.1(5)	O(6)	C(34)	C(35)	124.2(5)
C(33)	C(34)	C(35)	116.7(5)	C(34)	C(35)	C(36)	124.7(6)
C(35)	C(36)	C(37)	125.5(7)	C(36)	C(37)	C(38)	116.7(6)
C(37)	C(38)	C(39)	120.2(7)	C(38)	C(39)	C(40)	129.4(10)
O(7)	C(40)	C(39)	111.5(6)	O(7)	C(40)	C(41)	109.1(8)

Table 6. Bond angles (°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
C(39)	C(40)	C(41)	113.7(10)	O(7)	C(42)	O(8)	124.3(4)
O(7)	C(42)	C(43)	109.4(5)	O(8)	C(42)	C(43)	126.3(5)
C(42)	C(43)	C(44)	121.9(5)	C(31)	C(44)	C(43)	123.4(5)
Si(2)	C(45)	C(46)	111.0(3)	Si(2)	C(45)	C(47)	112.6(3)
Si(2)	C(45)	C(48)	106.6(3)	C(46)	C(45)	C(47)	109.9(4)
C(46)	C(45)	C(48)	107.6(4)	C(47)	C(45)	C(48)	109.0(4)
Si(2)	C(49)	C(50)	119.4(4)	Si(2)	C(49)	C(54)	124.6(3)
C(50)	C(49)	C(54)	116.0(4)	C(49)	C(50)	C(51)	121.7(5)
C(50)	C(51)	C(52)	121.0(5)	C(51)	C(52)	C(53)	118.7(5)
C(52)	C(53)	C(54)	121.5(5)	C(49)	C(54)	C(53)	121.2(4)
Si(2)	C(55)	C(56)	121.7(4)	Si(2)	C(55)	C(60)	120.1(3)
C(56)	C(55)	C(60)	118.2(5)	C(55)	C(56)	C(57)	120.6(5)
C(56)	C(57)	C(58)	120.9(6)	C(57)	C(58)	C(59)	119.8(6)
C(58)	C(59)	C(60)	119.0(6)	C(55)	C(60)	C(59)	121.5(5)

Table 7. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
O(1)	C(1)	H(1)	109.9	C(2)	C(1)	H(1)	109.9
C(14)	C(1)	H(1)	109.9	C(1)	C(2)	H(2)	109.1
C(1)	C(2)	H(3)	109.1	C(3)	C(2)	H(2)	109.1
C(3)	C(2)	H(3)	109.1	H(2)	C(2)	H(3)	107.8
C(2)	C(3)	H(4)	108.8	C(2)	C(3)	H(5)	108.7
C(4)	C(3)	H(4)	108.7	C(4)	C(3)	H(5)	108.7
H(4)	C(3)	H(5)	107.6	C(4)	C(5)	H(6)	117.6
C(6)	C(5)	H(6)	117.6	C(5)	C(6)	H(7)	118.6
C(7)	C(6)	H(7)	118.6	C(6)	C(7)	H(8)	108.3
C(6)	C(7)	H(9)	108.3	C(8)	C(7)	H(8)	108.3
C(8)	C(7)	H(9)	108.3	H(8)	C(7)	H(9)	107.4
C(7)	C(8)	H(10)	107.9	C(7)	C(8)	H(11)	107.9
C(9)	C(8)	H(10)	107.9	C(9)	C(8)	H(11)	107.9
H(10)	C(8)	H(11)	107.2	C(8)	C(9)	H(12)	108.1
C(8)	C(9)	H(13)	108.1	C(10)	C(9)	H(12)	108.2
C(10)	C(9)	H(13)	108.1	H(12)	C(9)	H(13)	107.3
O(3)	C(10)	H(14)	109.6	C(9)	C(10)	H(14)	109.6
C(11)	C(10)	H(14)	109.6	C(10)	C(11)	H(15)	109.5
C(10)	C(11)	H(16)	109.5	C(10)	C(11)	H(17)	109.5
H(15)	C(11)	H(16)	109.5	H(15)	C(11)	H(17)	109.5
H(16)	C(11)	H(17)	109.5	C(12)	C(13)	H(18)	119.0
C(14)	C(13)	H(18)	119.0	C(1)	C(14)	H(19)	118.7
C(13)	C(14)	H(19)	118.7	C(15)	C(16)	H(20)	109.5

C(15)	C(16)	H(21)	109.5	C(15)	C(16)	H(22)	109.5
H(20)	C(16)	H(21)	109.5	H(20)	C(16)	H(22)	109.5
H(21)	C(16)	H(22)	109.5	C(15)	C(17)	H(23)	109.5
C(15)	C(17)	H(24)	109.5	C(15)	C(17)	H(25)	109.5
H(23)	C(17)	H(24)	109.5	H(23)	C(17)	H(25)	109.5
H(24)	C(17)	H(25)	109.5	C(15)	C(18)	H(26)	109.5
C(15)	C(18)	H(27)	109.5	C(15)	C(18)	H(28)	109.5
H(26)	C(18)	H(27)	109.5	H(26)	C(18)	H(28)	109.5
H(27)	C(18)	H(28)	109.5	C(19)	C(20)	H(29)	119.5
C(21)	C(20)	H(29)	119.5	C(20)	C(21)	H(30)	119.6
C(22)	C(21)	H(30)	119.6	C(21)	C(22)	H(31)	120.4
C(23)	C(22)	H(31)	120.5	C(22)	C(23)	H(32)	120.1
C(24)	C(23)	H(32)	120.1	C(19)	C(24)	H(33)	118.7
C(23)	C(24)	H(33)	118.7	C(25)	C(26)	H(34)	119.3
C(27)	C(26)	H(34)	119.3	C(26)	C(27)	H(35)	120.1
C(28)	C(27)	H(35)	120.1	C(27)	C(28)	H(36)	119.7
C(29)	C(28)	H(36)	119.7	C(28)	C(29)	H(37)	119.9
C(30)	C(29)	H(37)	119.9	C(25)	C(30)	H(38)	119.4
C(29)	C(30)	H(38)	119.4	O(5)	C(31)	H(39)	109.6
C(32)	C(31)	H(39)	109.6	C(44)	C(31)	H(39)	109.6
C(31)	C(32)	H(40)	109.0	C(31)	C(32)	H(41)	109.0
C(33)	C(32)	H(40)	109.0	C(33)	C(32)	H(41)	109.0
H(40)	C(32)	H(41)	107.8	C(32)	C(33)	H(42)	108.6
C(32)	C(33)	H(43)	108.6	C(34)	C(33)	H(42)	108.6
C(34)	C(33)	H(43)	108.6	H(42)	C(33)	H(43)	107.6
C(34)	C(35)	H(44)	117.6	C(36)	C(35)	H(44)	117.6
C(35)	C(36)	H(45)	117.3	C(37)	C(36)	H(45)	117.3
C(36)	C(37)	H(46)	108.1	C(36)	C(37)	H(47)	108.1
C(38)	C(37)	H(46)	108.1	C(38)	C(37)	H(47)	108.1
H(46)	C(37)	H(47)	107.3	C(37)	C(38)	H(48)	107.3
C(37)	C(38)	H(49)	107.3	C(39)	C(38)	H(48)	107.3
C(39)	C(38)	H(49)	107.3	H(48)	C(38)	H(49)	106.9
C(38)	C(39)	H(50)	104.9	C(38)	C(39)	H(51)	104.9
C(40)	C(39)	H(50)	104.9	C(40)	C(39)	H(51)	104.9
H(50)	C(39)	H(51)	105.8	O(7)	C(40)	H(52)	107.4
C(39)	C(40)	H(52)	107.4	C(41)	C(40)	H(52)	107.4
C(40)	C(41)	H(53)	109.5	C(40)	C(41)	H(54)	109.5
C(40)	C(41)	H(55)	109.5	H(53)	C(41)	H(54)	109.5
H(53)	C(41)	H(55)	109.5	H(54)	C(41)	H(55)	109.5
C(42)	C(43)	H(56)	119.0	C(44)	C(43)	H(56)	119.0
C(31)	C(44)	H(57)	118.3	C(43)	C(44)	H(57)	118.3
C(45)	C(46)	H(58)	109.5	C(45)	C(46)	H(59)	109.5
C(45)	C(46)	H(60)	109.5	H(58)	C(46)	H(59)	109.5
H(58)	C(46)	H(60)	109.5	H(59)	C(46)	H(60)	109.5
C(45)	C(47)	H(61)	109.5	C(45)	C(47)	H(62)	109.5
C(45)	C(47)	H(63)	109.5	H(61)	C(47)	H(62)	109.5

H(61)	C(47)	H(63)	109.5	H(62)	C(47)	H(63)	109.5
C(45)	C(48)	H(64)	109.5	C(45)	C(48)	H(65)	109.5
C(45)	C(48)	H(66)	109.5	H(64)	C(48)	H(65)	109.5
H(64)	C(48)	H(66)	109.5	H(65)	C(48)	H(66)	109.5
C(49)	C(50)	H(67)	119.2	C(51)	C(50)	H(67)	119.2
C(50)	C(51)	H(68)	119.5	C(52)	C(51)	H(68)	119.5
C(51)	C(52)	H(69)	120.6	C(53)	C(52)	H(69)	120.6
C(52)	C(53)	H(70)	119.3	C(54)	C(53)	H(70)	119.3
C(49)	C(54)	H(71)	119.4	C(53)	C(54)	H(71)	119.4
C(55)	C(56)	H(72)	119.7	C(57)	C(56)	H(72)	119.7
C(56)	C(57)	H(73)	119.6	C(58)	C(57)	H(73)	119.6
C(57)	C(58)	H(74)	120.1	C(59)	C(58)	H(74)	120.1
C(58)	C(59)	H(75)	120.5	C(60)	C(59)	H(75)	120.5
C(55)	C(60)	H(76)	119.2	C(59)	C(60)	H(76)	119.3

Table 8. Torsion Angles(o)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
O(1)	Si(1)	C(15)	C(16)	70.4(3)	O(1)	Si(1)	C(15)	C(17)	-48.6(4)
O(1)	Si(1)	C(15)	C(18)	-170.3(3)	C(15)	Si(1)	O(1)	C(1)	66.0(4)
O(1)	Si(1)	C(19)	C(20)	71.4(4)	O(1)	Si(1)	C(19)	C(24)	-107.3(4)
C(19)	Si(1)	O(1)	C(1)	-59.6(4)	O(1)	Si(1)	C(25)	C(26)	-173.2(4)
O(1)	Si(1)	C(25)	C(30)	6.5(5)	C(25)	Si(1)	O(1)	C(1)	-175.2(3)
C(15)	Si(1)	C(19)	C(20)	-53.5(4)	C(15)	Si(1)	C(19)	C(24)	127.9(4)
C(19)	Si(1)	C(15)	C(16)	-165.0(3)	C(19)	Si(1)	C(15)	C(17)	75.9(4)
C(19)	Si(1)	C(15)	C(18)	-45.8(4)	C(15)	Si(1)	C(25)	C(26)	-54.6(4)
C(15)	Si(1)	C(25)	C(30)	125.1(5)	C(25)	Si(1)	C(15)	C(16)	-42.6(3)
C(25)	Si(1)	C(15)	C(17)	-161.6(3)	C(25)	Si(1)	C(15)	C(18)	76.7(3)
C(19)	Si(1)	C(25)	C(26)	69.9(4)	C(19)	Si(1)	C(25)	C(30)	-110.4(5)
C(25)	Si(1)	C(19)	C(20)	-177.4(4)	C(25)	Si(1)	C(19)	C(24)	3.9(4)
O(5)	Si(2)	C(45)	C(46)	-52.6(4)	O(5)	Si(2)	C(45)	C(47)	-176.2(3)
O(5)	Si(2)	C(45)	C(48)	64.3(3)	C(45)	Si(2)	O(5)	C(31)	99.4(4)
O(5)	Si(2)	C(49)	C(50)	-79.7(4)	O(5)	Si(2)	C(49)	C(54)	100.9(4)
C(49)	Si(2)	O(5)	C(31)	-27.1(4)	O(5)	Si(2)	C(55)	C(56)	173.4(4)
O(5)	Si(2)	C(55)	C(60)	-8.7(4)	C(55)	Si(2)	O(5)	C(31)	-143.9(3)
C(45)	Si(2)	C(49)	C(50)	156.7(3)	C(45)	Si(2)	C(49)	C(54)	-22.7(4)
C(49)	Si(2)	C(45)	C(46)	72.5(4)	C(49)	Si(2)	C(45)	C(47)	-51.1(4)
C(49)	Si(2)	C(45)	C(48)	-170.6(3)	C(45)	Si(2)	C(55)	C(56)	-70.5(4)
C(45)	Si(2)	C(55)	C(60)	107.4(4)	C(55)	Si(2)	C(45)	C(46)	-165.6(3)
C(55)	Si(2)	C(45)	C(47)	70.8(4)	C(55)	Si(2)	C(45)	C(48)	-48.7(3)
C(49)	Si(2)	C(55)	C(56)	54.5(4)	C(49)	Si(2)	C(55)	C(60)	-127.6(4)
C(55)	Si(2)	C(49)	C(50)	34.3(4)	C(55)	Si(2)	C(49)	C(54)	-145.1(3)
Si(1)	O(1)	C(1)	C(2)	109.8(4)	Si(1)	O(1)	C(1)	C(14)	-128.7(4)
C(10)	O(3)	C(12)	O(4)	4.3(8)	C(10)	O(3)	C(12)	C(13)	-175.5(4)
C(12)	O(3)	C(10)	C(9)	-146.4(5)	C(12)	O(3)	C(10)	C(11)	92.0(5)
Si(2)	O(5)	C(31)	C(32)	125.9(4)	Si(2)	O(5)	C(31)	C(44)	-113.3(4)

C(40)	O(7)	C(42)	O(8)	0.2(6)	C(40)	O(7)	C(42)	C(43)	178.8(4)
C(42)	O(7)	C(40)	C(39)	-84.5(8)	C(42)	O(7)	C(40)	C(41)	149.1(8)
O(1)	C(1)	C(2)	C(3)	175.2(4)	O(1)	C(1)	C(14)	C(13)	143.5(5)
C(2)	C(1)	C(14)	C(13)	-98.3(6)	C(14)	C(1)	C(2)	C(3)	58.3(7)
C(1)	C(2)	C(3)	C(4)	-76.2(6)	C(2)	C(3)	C(4)	O(2)	-36.6(7)
C(2)	C(3)	C(4)	C(5)	145.7(5)	O(2)	C(4)	C(5)	C(6)	16.8(8)
C(3)	C(4)	C(5)	C(6)	-165.6(5)	C(4)	C(5)	C(6)	C(7)	179.0(5)
C(5)	C(6)	C(7)	C(8)	-146.8(5)	C(6)	C(7)	C(8)	C(9)	85.4(7)
C(7)	C(8)	C(9)	C(10)	-82.0(7)	C(8)	C(9)	C(10)	O(3)	72.9(6)
C(8)	C(9)	C(10)	C(11)	-169.3(5)	O(3)	C(12)	C(13)	C(14)	-163.8(5)
O(4)	C(12)	C(13)	C(14)	16.4(9)	C(12)	C(13)	C(14)	C(1)	173.2(4)
Si(1)	C(19)	C(20)	C(21)	179.2(4)	Si(1)	C(19)	C(24)	C(23)	-177.9(4)
C(20)	C(19)	C(24)	C(23)	3.4(8)	C(24)	C(19)	C(20)	C(21)	-2.1(8)
C(19)	C(20)	C(21)	C(22)	-0.5(8)	C(20)	C(21)	C(22)	C(23)	2.0(9)
C(21)	C(22)	C(23)	C(24)	-0.7(9)	C(22)	C(23)	C(24)	C(19)	-2.0(9)
Si(1)	C(25)	C(26)	C(27)	179.8(3)	Si(1)	C(25)	C(30)	C(29)	-179.2(6)
C(26)	C(25)	C(30)	C(29)	0.5(10)	C(30)	C(25)	C(26)	C(27)	0.1(6)
C(25)	C(26)	C(27)	C(28)	-1.2(9)	C(26)	C(27)	C(28)	C(29)	1.8(12)
C(27)	C(28)	C(29)	C(30)	-1.2(14)	C(28)	C(29)	C(30)	C(25)	0.0(13)
O(5)	C(31)	C(32)	C(33)	-177.7(4)	O(5)	C(31)	C(44)	C(43)	143.7(4)
C(32)	C(31)	C(44)	C(43)	-97.8(6)	C(44)	C(31)	C(32)	C(33)	62.8(6)
C(31)	C(32)	C(33)	C(34)	-81.6(6)	C(32)	C(33)	C(34)	O(6)	-39.0(7)
C(32)	C(33)	C(34)	C(35)	138.6(5)	O(6)	C(34)	C(35)	C(36)	8.5(10)
C(33)	C(34)	C(35)	C(36)	-169.0(6)	C(34)	C(35)	C(36)	C(37)	176.8(6)
C(35)	C(36)	C(37)	C(38)	-145.9(7)	C(36)	C(37)	C(38)	C(39)	42.5(12)
C(37)	C(38)	C(39)	C(40)	61.4(16)	C(38)	C(39)	C(40)	O(7)	-65.3(14)
C(38)	C(39)	C(40)	C(41)	58.5(15)	O(7)	C(42)	C(43)	C(44)	-171.6(4)
O(8)	C(42)	C(43)	C(44)	6.9(8)	C(42)	C(43)	C(44)	C(31)	174.1(4)
Si(2)	C(49)	C(50)	C(51)	-179.3(4)	Si(2)	C(49)	C(54)	C(53)	179.1(3)
C(50)	C(49)	C(54)	C(53)	-0.3(5)	C(54)	C(49)	C(50)	C(51)	0.1(5)
C(49)	C(50)	C(51)	C(52)	0.4(6)	C(50)	C(51)	C(52)	C(53)	-0.6(8)
C(51)	C(52)	C(53)	C(54)	0.4(7)	C(52)	C(53)	C(54)	C(49)	0.0(7)
Si(2)	C(55)	C(56)	C(57)	176.3(4)	Si(2)	C(55)	C(60)	C(59)	-176.3(4)
C(56)	C(55)	C(60)	C(59)	1.7(8)	C(60)	C(55)	C(56)	C(57)	-1.7(8)
C(55)	C(56)	C(57)	C(58)	1.2(9)	C(56)	C(57)	C(58)	C(59)	-0.6(10)
C(57)	C(58)	C(59)	C(60)	0.6(9)	C(58)	C(59)	C(60)	C(55)	-1.2(9)

The sign is positive if when looking from atom 2 to atom 3 a clock-wise motion of atom 1 would superimpose it on atom 4.

Table 9. Distances beyond the asymmetric unit out to 3.60 Å

atom	atom	distance	atom	atom	distance
O(2)	C(33)	3.306(8)	O(2)	C(35)	3.309(9)
O(4)	C(47)[1)]	3.586(7)	O(6)	C(3)[2)]	3.577(8)
O(6)	C(5)[2)]	3.197(8)	O(8)	C(20)[2)]	3.571(7)
C(3)	O(6)[3)]	3.577(8)	C(4)	C(59)[4)]	3.562(7)
C(5)	O(6)[3)]	3.197(8)	C(5)	C(59)[4)]	3.415(8)
C(11)	C(22)[1)]	3.520(8)	C(20)	O(8)[3)]	3.571(7)
C(22)	C(11)[5)]	3.520(8)	C(33)	O(2)	3.306(8)
C(35)	O(2)	3.309(9)	C(47)	O(4)[5)]	3.586(7)
C(59)	C(4)[6)]	3.562(7)	C(59)	C(5)[6)]	3.415(8)

Symmetry Operators:

(1) X,Y-1,Z	(2) X-1,Y,Z
(3) X+1,Y,Z	(4) -X+1,Y+1/2-1,-Z+1
(5) X,Y+1,Z	(6) -X+1,Y+1/2,-Z+1

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
O(1)	H(56)	3.575	O(2)	H(39)	3.429
O(2)	H(42)	2.344	O(2)	H(44)	2.542
O(2)	H(56)	2.791	O(3)	H(35)[1)]	3.522
O(3)	H(45)[2)]	3.533	O(3)	H(49)[2)]	2.835
O(3)	H(51)[2)]	3.496	O(4)	H(35)[1)]	3.050
O(4)	H(47)	2.737	O(4)	H(55)	3.102
O(4)	H(61)[3)]	3.082	O(4)	H(63)[3)]	3.387
O(5)	H(69)[4)]	3.527	O(6)	H(4)[4)]	2.972
O(6)	H(5)[4)]	3.483	O(6)	H(6)[4)]	2.289
O(6)	H(11)[5)]	3.048	O(6)	H(68)[6)]	3.027
O(6)	H(75)[7)]	3.153	O(7)	H(19)	2.945
O(7)	H(38)	2.954	O(8)	H(1)[4)]	2.754
O(8)	H(4)[4)]	3.052	O(8)	H(18)[4)]	3.403
O(8)	H(23)[4)]	3.250	O(8)	H(25)[4)]	3.175
O(8)	H(29)[4)]	2.686	O(8)	H(37)	2.986
O(8)	H(38)	3.450	C(2)	H(39)	3.577
C(2)	H(56)	3.114	C(2)	H(70)	3.502
C(2)	H(71)	3.430	C(4)	H(42)	3.375
C(4)	H(75)[6)]	3.228	C(5)	H(45)[2)]	3.195
C(5)	H(75)[6)]	2.901	C(6)	H(75)[6)]	3.578

C(6)	H(76)[6)]	3.508	C(7)	H(40)[6)]	3.142
C(7)	H(41)[6)]	3.451	C(7)	H(68)[8)]	3.450
C(7)	H(69)[8)]	3.553	C(8)	H(40)[6)]	3.110
C(8)	H(41)[6)]	3.397	C(8)	H(43)[6)]	3.329
C(8)	H(72)[3)]	3.516	C(8)	H(75)[9)]	3.447
C(10)	H(49)[2)]	3.384	C(10)	H(63)[3)]	3.147
C(11)	H(31)[3)]	3.101	C(11)	H(32)[3)]	3.595
C(11)	H(49)[2)]	3.197	C(11)	H(63)[3)]	3.126
C(11)	H(65)[9)]	3.588	C(12)	H(35)[1)]	2.937
C(13)	H(35)[1)]	3.093	C(13)	H(51)[2)]	3.043
C(14)	H(55)	3.296	C(14)	H(56)	3.599
C(16)	H(32)[1)]	3.383	C(16)	H(33)[1)]	2.952
C(16)	H(59)[1)]	3.531	C(16)	H(62)[1)]	3.415
C(17)	H(52)[2)]	2.988	C(18)	H(36)[2)]	3.537
C(18)	H(48)[10)]	3.480	C(18)	H(54)[10)]	2.938
C(18)	H(62)[1)]	3.476	C(18)	H(65)[1)]	3.512
C(19)	H(54)[10)]	3.561	C(20)	H(37)[2)]	2.978
C(21)	H(37)[2)]	3.219	C(21)	H(59)[2)]	3.310
C(21)	H(64)[2)]	3.017	C(21)	H(70)	3.519
C(22)	H(15)[11)]	3.281	C(22)	H(16)[11)]	3.226
C(22)	H(17)[11)]	3.472	C(22)	H(27)[12)]	3.464
C(22)	H(64)[2)]	3.293	C(23)	H(15)[11)]	3.205
C(23)	H(22)[10)]	3.248	C(23)	H(53)[10)]	3.031
C(24)	H(21)[10)]	3.588	C(24)	H(22)[10)]	3.341
C(24)	H(53)[10)]	3.012	C(24)	H(54)[10)]	3.392
C(24)	H(58)	3.303	C(26)	H(20)[10)]	3.166
C(26)	H(32)[1)]	3.577	C(26)	H(54)[10)]	3.552
C(26)	H(55)[10)]	3.491	C(27)	H(20)[10)]	2.947
C(27)	H(24)[10)]	3.427	C(27)	H(32)[1)]	3.534
C(28)	H(20)[10)]	3.494	C(28)	H(24)[10)]	3.261
C(29)	H(23)[4)]	3.365	C(29)	H(29)[4)]	3.344
C(29)	H(30)[4)]	3.434	C(30)	H(53)	3.517
C(31)	H(2)	3.576	C(32)	H(8)[5)]	3.136
C(32)	H(10)[5)]	3.254	C(32)	H(11)[5)]	3.257
C(32)	H(73)[6)]	3.483	C(33)	H(10)[5)]	3.405
C(33)	H(11)[5)]	3.375	C(33)	H(67)[6)]	3.246
C(34)	H(6)[4)]	3.252	C(34)	H(11)[5)]	3.561
C(34)	H(67)[6)]	3.387	C(34)	H(68)[6)]	3.032
C(35)	H(67)[6)]	3.568	C(35)	H(68)[6)]	3.334
C(35)	H(73)[3)]	3.491	C(36)	H(6)[4)]	3.336
C(36)	H(9)[4)]	3.302	C(37)	H(12)[4)]	3.596
C(37)	H(61)[3)]	3.373	C(37)	H(73)[3)]	3.493
C(38)	H(17)[4)]	3.304	C(38)	H(61)[3)]	3.327
C(38)	H(65)[3)]	3.341	C(39)	H(18)[4)]	2.988
C(40)	H(23)[4)]	3.558	C(41)	H(19)	3.340
C(41)	H(28)[1)]	3.197	C(41)	H(34)[1)]	3.042

C(42)	H(37)	3.344	C(42)	H(38)	2.932
C(43)	H(2)	3.008	C(43)	H(38)	3.181
C(46)	H(21)[10)]	3.174	C(46)	H(30)[4)]	3.138
C(47)	H(14)[11)]	3.170	C(47)	H(15)[11)]	3.212
C(47)	H(21)[10)]	3.598	C(47)	H(26)[10)]	3.264
C(47)	H(48)[11)]	3.449	C(48)	H(16)[13)]	3.318
C(48)	H(17)[13)]	3.477	C(48)	H(30)[4)]	3.226
C(48)	H(31)[4)]	3.428	C(48)	H(48)[11)]	3.543
C(50)	H(43)[5)]	3.103	C(50)	H(74)[6)]	3.197
C(51)	H(8)[14)]	3.585	C(51)	H(13)[11)]	3.212
C(51)	H(43)[5)]	3.536	C(51)	H(74)[6)]	2.749
C(52)	H(5)	3.230	C(52)	H(8)[14)]	3.366
C(52)	H(13)[11)]	2.950	C(52)	H(74)[6)]	3.265
C(52)	H(76)[2)]	3.345	C(53)	H(3)	2.922
C(53)	H(5)	3.015	C(53)	H(13)[11)]	3.134
C(53)	H(16)[11)]	2.995	C(54)	H(2)	3.562
C(54)	H(3)	2.914	C(54)	H(5)	3.489
C(54)	H(13)[11)]	3.546	C(54)	H(16)[11)]	3.213
C(55)	H(43)[5)]	3.540	C(56)	H(10)[11)]	3.275
C(56)	H(43)[5)]	2.952	C(56)	H(46)[11)]	2.952
C(57)	H(41)[5)]	3.080	C(57)	H(42)[5)]	3.507
C(57)	H(43)[5)]	3.091	C(57)	H(46)[11)]	2.814
C(58)	H(5)[5)]	3.572	C(58)	H(9)[13)]	3.558
C(58)	H(42)[5)]	3.594	C(58)	H(46)[11)]	3.407
C(58)	H(68)[5)]	3.485	C(59)	H(6)[5)]	3.454
C(59)	H(11)[13)]	3.216	C(59)	H(12)[13)]	3.342
C(59)	H(13)[13)]	3.554	C(60)	H(69)[4)]	3.392
H(1)	O(8)[2)]	2.754	H(1)	H(51)[2)]	3.442
H(2)	C(31)	3.576	H(2)	C(43)	3.008
H(2)	C(54)	3.562	H(2)	H(39)	2.748
H(2)	H(56)	2.165	H(2)	H(71)	3.350
H(3)	C(53)	2.922	H(3)	C(54)	2.914
H(3)	H(70)	2.660	H(3)	H(71)	2.647
H(4)	O(6)[2)]	2.972	H(4)	O(8)[2)]	3.052
H(4)	H(45)[2)]	3.406	H(4)	H(57)[2)]	3.304
H(5)	O(6)[2)]	3.483	H(5)	C(52)	3.230
H(5)	C(53)	3.015	H(5)	C(54)	3.489
H(5)	C(58)[6)]	3.572	H(5)	H(69)	3.446
H(5)	H(70)	3.089	H(5)	H(74)[6)]	3.058
H(5)	H(75)[6)]	3.239	H(6)	O(6)[2)]	2.289
H(6)	C(34)[2)]	3.252	H(6)	C(36)[2)]	3.336
H(6)	C(59)[6)]	3.454	H(6)	H(45)[2)]	2.521
H(6)	H(68)[8)]	3.414	H(6)	H(75)[6)]	2.724
H(7)	H(41)[6)]	3.350	H(7)	H(44)	3.182
H(7)	H(73)[3)]	3.227	H(8)	C(32)[6)]	3.136

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(8)	C(51)[8)]	3.585	H(8)	C(52)[8)]	3.366
H(8)	H(40)[6)]	2.414	H(8)	H(41)[6)]	2.970
H(8)	H(68)[8)]	3.091	H(8)	H(69)[8)]	2.660
H(8)	H(76)[6)]	2.888	H(9)	C(36)[2)]	3.302
H(9)	C(58)[9)]	3.558	H(9)	H(45)[2)]	2.790
H(9)	H(68)[8)]	2.885	H(9)	H(74)[9)]	3.227
H(10)	C(32)[6)]	3.254	H(10)	C(33)[6)]	3.405
H(10)	C(56)[3)]	3.275	H(10)	H(40)[6)]	3.031
H(10)	H(41)[6)]	2.872	H(10)	H(43)[6)]	2.741
H(10)	H(72)[3)]	2.581	H(10)	H(73)[3)]	3.316
H(11)	O(6)[6)]	3.048	H(11)	C(32)[6)]	3.257
H(11)	C(33)[6)]	3.375	H(11)	C(34)[6)]	3.561
H(11)	C(59)[9)]	3.216	H(11)	H(40)[6)]	2.645
H(11)	H(41)[6)]	3.329	H(11)	H(43)[6)]	2.978
H(11)	H(75)[9)]	2.648	H(12)	C(37)[2)]	3.596
H(12)	C(59)[9)]	3.342	H(12)	H(45)[2)]	3.559
H(12)	H(46)[2)]	3.008	H(12)	H(49)[2)]	2.895
H(12)	H(66)[9)]	3.153	H(12)	H(75)[9)]	3.458
H(13)	C(51)[3)]	3.212	H(13)	C(52)[3)]	2.950
H(13)	C(53)[3)]	3.134	H(13)	C(54)[3)]	3.546
H(13)	C(59)[9)]	3.554	H(13)	H(66)[9)]	3.397
H(13)	H(69)[3)]	3.215	H(13)	H(70)[3)]	3.498
H(13)	H(75)[9)]	3.374	H(14)	C(47)[3)]	3.170
H(14)	H(61)[3)]	3.150	H(14)	H(63)[3)]	2.392
H(14)	H(72)[3)]	2.688	H(15)	C(22)[3)]	3.281
H(15)	C(23)[3)]	3.205	H(15)	C(47)[3)]	3.212
H(15)	H(31)[3)]	3.012	H(15)	H(32)[3)]	2.858
H(15)	H(35)[1)]	3.236	H(15)	H(62)[3)]	3.067
H(15)	H(63)[3)]	2.559	H(15)	H(71)[3)]	3.528
H(16)	C(22)[3)]	3.226	H(16)	C(48)[9)]	3.318
H(16)	C(53)[3)]	2.995	H(16)	C(54)[3)]	3.213
H(16)	H(31)[3)]	2.945	H(16)	H(63)[3)]	3.285
H(16)	H(64)[9)]	3.029	H(16)	H(65)[9)]	3.271
H(16)	H(66)[9)]	3.098	H(16)	H(70)[3)]	2.865
H(16)	H(71)[3)]	3.238	H(17)	C(22)[3)]	3.472
H(17)	C(38)[2)]	3.304	H(17)	C(48)[9)]	3.477
H(17)	H(31)[3)]	2.820	H(17)	H(35)[1)]	3.354

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(17)	H(48)[2)]	3.486	H(17)	H(49)[2)]	2.378
H(17)	H(50)[2)]	3.553	H(17)	H(64)[9)]	3.526
H(17)	H(65)[9)]	2.992	H(17)	H(66)[9)]	3.362
H(18)	O(8)[2)]	3.403	H(18)	C(39)[2)]	2.988
H(18)	H(35)[1)]	3.351	H(18)	H(45)[2)]	3.135
H(18)	H(49)[2)]	3.302	H(18)	H(50)[2)]	3.030
H(18)	H(51)[2)]	2.130	H(19)	O(7)	2.945
H(19)	C(41)	3.340	H(19)	H(47)	3.411
H(19)	H(55)	2.494	H(19)	H(56)	3.104
H(20)	C(26)[1)]	3.166	H(20)	C(27)[1)]	2.947
H(20)	C(28)[1)]	3.494	H(20)	H(33)[1)]	2.811
H(20)	H(34)[1)]	3.341	H(20)	H(35)[1)]	2.975
H(20)	H(53)	3.333	H(20)	H(55)	3.482
H(21)	C(24)[1)]	3.588	H(21)	C(46)[1)]	3.174
H(21)	C(47)[1)]	3.598	H(21)	H(32)[1)]	3.442
H(21)	H(33)[1)]	2.781	H(21)	H(58)[1)]	2.989
H(21)	H(59)[1)]	2.560	H(21)	H(62)[1)]	2.710
H(22)	C(23)[1)]	3.248	H(22)	C(24)[1)]	3.341
H(22)	H(32)[1)]	2.580	H(22)	H(33)[1)]	2.759
H(22)	H(53)	3.144	H(22)	H(62)[1)]	3.320
H(23)	O(8)[2)]	3.250	H(23)	C(29)[2)]	3.365
H(23)	C(40)[2)]	3.558	H(23)	H(36)[2)]	3.579
H(23)	H(37)[2)]	2.713	H(23)	H(52)[2)]	2.564
H(24)	C(27)[1)]	3.427	H(24)	C(28)[1)]	3.261
H(24)	H(35)[1)]	3.321	H(24)	H(36)[1)]	3.044
H(24)	H(50)[2)]	3.291	H(24)	H(52)[2)]	2.997
H(25)	O(8)[2)]	3.175	H(25)	H(35)[1)]	3.566
H(25)	H(50)[2)]	3.391	H(25)	H(51)[2)]	3.226
H(25)	H(52)[2)]	2.910	H(26)	C(47)[1)]	3.264
H(26)	H(48)[10)]	2.975	H(26)	H(54)[10)]	2.823
H(26)	H(61)[1)]	3.141	H(26)	H(62)[1)]	2.578
H(26)	H(65)[1)]	3.048	H(27)	C(22)[15)]	3.464
H(27)	H(31)[15)]	2.685	H(27)	H(36)[2)]	3.161
H(27)	H(48)[10)]	3.527	H(27)	H(54)[10)]	3.493
H(27)	H(65)[1)]	3.064	H(28)	C(41)[10)]	3.197
H(28)	H(36)[2)]	2.998	H(28)	H(48)[10)]	3.392
H(28)	H(50)[10)]	3.305	H(28)	H(53)[10)]	3.555

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(28)	H(54)[10)]	2.227	H(29)	O(8)[2)]	2.686
H(29)	C(29)[2)]	3.344	H(29)	H(37)[2)]	2.432
H(30)	C(29)[2)]	3.434	H(30)	C(46)[2)]	3.138
H(30)	C(48)[2)]	3.226	H(30)	H(37)[2)]	2.909
H(30)	H(59)[2)]	2.400	H(30)	H(60)[2)]	3.110
H(30)	H(64)[2)]	2.397	H(30)	H(65)[2)]	3.353
H(30)	H(70)	3.591	H(31)	C(11)[11)]	3.101
H(31)	C(48)[2)]	3.428	H(31)	H(15)[11)]	3.012
H(31)	H(16)[11)]	2.945	H(31)	H(17)[11)]	2.820
H(31)	H(27)[12)]	2.685	H(31)	H(64)[2)]	2.971
H(31)	H(65)[2)]	2.978	H(32)	C(11)[11)]	3.595
H(32)	C(16)[10)]	3.383	H(32)	C(26)[10)]	3.577
H(32)	C(27)[10)]	3.534	H(32)	H(15)[11)]	2.858
H(32)	H(21)[10)]	3.442	H(32)	H(22)[10)]	2.580
H(32)	H(53)[10)]	3.001	H(32)	H(62)	3.470
H(33)	C(16)[10)]	2.952	H(33)	H(20)[10)]	2.811
H(33)	H(21)[10)]	2.781	H(33)	H(22)[10)]	2.759
H(33)	H(53)[10)]	2.987	H(33)	H(54)[10)]	3.476
H(33)	H(58)	2.925	H(34)	C(41)[10)]	3.042
H(34)	H(20)[10)]	3.341	H(34)	H(53)[10)]	3.262
H(34)	H(54)[10)]	2.682	H(34)	H(55)[10)]	2.711
H(35)	O(3)[10)]	3.522	H(35)	O(4)[10)]	3.050
H(35)	C(12)[10)]	2.937	H(35)	C(13)[10)]	3.093
H(35)	H(15)[10)]	3.236	H(35)	H(17)[10)]	3.354
H(35)	H(18)[10)]	3.351	H(35)	H(20)[10)]	2.975
H(35)	H(24)[10)]	3.321	H(35)	H(25)[10)]	3.566
H(35)	H(50)[16)]	3.482	H(36)	C(18)[4)]	3.537
H(36)	H(23)[4)]	3.579	H(36)	H(24)[10)]	3.044
H(36)	H(27)[4)]	3.161	H(36)	H(28)[4)]	2.998
H(36)	H(50)[16)]	2.810	H(37)	O(8)	2.986
H(37)	C(20)[4)]	2.978	H(37)	C(21)[4)]	3.219
H(37)	C(42)	3.344	H(37)	H(23)[4)]	2.713
H(37)	H(29)[4)]	2.432	H(37)	H(30)[4)]	2.909
H(37)	H(52)	3.202	H(38)	O(7)	2.954
H(38)	O(8)	3.450	H(38)	C(42)	2.932
H(38)	C(43)	3.181	H(38)	H(52)	3.449
H(38)	H(53)	3.339	H(38)	H(56)	3.205

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(38)	H(58)	3.581	H(38)	H(60)	3.309
H(39)	O(2)	3.429	H(39)	C(2)	3.577
H(39)	H(2)	2.748	H(40)	C(7)[5)]	3.142
H(40)	C(8)[5)]	3.110	H(40)	H(8)[5)]	2.414
H(40)	H(10)[5)]	3.031	H(40)	H(11)[5)]	2.645
H(40)	H(69)[4)]	3.185	H(41)	C(7)[5)]	3.451
H(41)	C(8)[5)]	3.397	H(41)	C(57)[6)]	3.080
H(41)	H(7)[5)]	3.350	H(41)	H(8)[5)]	2.970
H(41)	H(10)[5)]	2.872	H(41)	H(11)[5)]	3.329
H(41)	H(73)[6)]	2.610	H(42)	O(2)	2.344
H(42)	C(4)	3.375	H(42)	C(57)[6)]	3.507
H(42)	C(58)[6)]	3.594	H(42)	H(67)[6)]	3.451
H(43)	C(8)[5)]	3.329	H(43)	C(50)[6)]	3.103
H(43)	C(51)[6)]	3.536	H(43)	C(55)[6)]	3.540
H(43)	C(56)[6)]	2.952	H(43)	C(57)[6)]	3.091
H(43)	H(10)[5)]	2.741	H(43)	H(11)[5)]	2.978
H(43)	H(67)[6)]	2.523	H(43)	H(68)[6)]	3.350
H(43)	H(72)[6)]	3.008	H(43)	H(73)[6)]	3.247
H(44)	O(2)	2.542	H(44)	H(7)	3.182
H(44)	H(67)[6)]	3.380	H(44)	H(73)[3)]	3.090
H(45)	O(3)[4)]	3.533	H(45)	C(5)[4)]	3.195
H(45)	H(4)[4)]	3.406	H(45)	H(6)[4)]	2.521
H(45)	H(9)[4)]	2.790	H(45)	H(12)[4)]	3.559
H(45)	H(18)[4)]	3.135	H(46)	C(56)[3)]	2.952
H(46)	C(57)[3)]	2.814	H(46)	C(58)[3)]	3.407
H(46)	H(12)[4)]	3.008	H(46)	H(61)[3)]	3.046
H(46)	H(66)[3)]	3.272	H(46)	H(72)[3)]	3.121
H(46)	H(73)[3)]	2.884	H(47)	O(4)	2.737
H(47)	H(19)	3.411	H(47)	H(61)[3)]	3.165
H(47)	H(72)[3)]	3.593	H(47)	H(73)[3)]	3.464
H(48)	C(18)[1)]	3.480	H(48)	C(47)[3)]	3.449
H(48)	C(48)[3)]	3.543	H(48)	H(17)[4)]	3.486
H(48)	H(26)[1)]	2.975	H(48)	H(27)[1)]	3.527
H(48)	H(28)[1)]	3.392	H(48)	H(61)[3)]	2.572
H(48)	H(62)[3)]	3.587	H(48)	H(65)[3)]	2.756
H(48)	H(66)[3)]	3.575	H(49)	O(3)[4)]	2.835
H(49)	C(10)[4)]	3.384	H(49)	C(11)[4)]	3.197

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(49)	H(12)[4)]	2.895	H(49)	H(17)[4)]	2.378
H(49)	H(18)[4)]	3.302	H(49)	H(65)[3)]	3.029
H(49)	H(66)[3)]	3.433	H(50)	H(17)[4)]	3.553
H(50)	H(18)[4)]	3.030	H(50)	H(24)[4)]	3.291
H(50)	H(25)[4)]	3.391	H(50)	H(28)[1)]	3.305
H(50)	H(35)[17)]	3.482	H(50)	H(36)[17)]	2.810
H(51)	O(3)[4)]	3.496	H(51)	C(13)[4)]	3.043
H(51)	H(1)[4)]	3.442	H(51)	H(18)[4)]	2.130
H(51)	H(25)[4)]	3.226	H(52)	C(17)[4)]	2.988
H(52)	H(23)[4)]	2.564	H(52)	H(24)[4)]	2.997
H(52)	H(25)[4)]	2.910	H(52)	H(37)	3.202
H(52)	H(38)	3.449	H(53)	C(23)[1)]	3.031
H(53)	C(24)[1)]	3.012	H(53)	C(30)	3.517
H(53)	H(20)	3.333	H(53)	H(22)	3.144
H(53)	H(28)[1)]	3.555	H(53)	H(32)[1)]	3.001
H(53)	H(33)[1)]	2.987	H(53)	H(34)[1)]	3.262
H(53)	H(38)	3.339	H(54)	C(18)[1)]	2.938
H(54)	C(19)[1)]	3.561	H(54)	C(24)[1)]	3.392
H(54)	C(26)[1)]	3.552	H(54)	H(26)[1)]	2.823
H(54)	H(27)[1)]	3.493	H(54)	H(28)[1)]	2.227
H(54)	H(33)[1)]	3.476	H(54)	H(34)[1)]	2.682
H(55)	O(4)	3.102	H(55)	C(14)	3.296
H(55)	C(26)[1)]	3.491	H(55)	H(19)	2.494
H(55)	H(20)	3.482	H(55)	H(34)[1)]	2.711
H(56)	O(1)	3.575	H(56)	O(2)	2.791
H(56)	C(2)	3.114	H(56)	C(14)	3.599
H(56)	H(2)	2.165	H(56)	H(19)	3.104
H(56)	H(38)	3.205	H(57)	H(4)[4)]	3.304
H(57)	H(70)[4)]	3.376	H(58)	C(24)	3.303
H(58)	H(21)[10)]	2.989	H(58)	H(33)	2.925
H(58)	H(38)	3.581	H(59)	C(16)[10)]	3.531
H(59)	C(21)[4)]	3.310	H(59)	H(21)[10)]	2.560
H(59)	H(30)[4)]	2.400	H(60)	H(30)[4)]	3.110
H(60)	H(38)	3.309	H(61)	O(4)[11)]	3.082
H(61)	C(37)[11)]	3.373	H(61)	C(38)[11)]	3.327
H(61)	H(14)[11)]	3.150	H(61)	H(26)[10)]	3.141
H(61)	H(46)[11)]	3.046	H(61)	H(47)[11)]	3.165

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(61)	H(48)$^{11)}$	2.572	H(62)	C(16)$^{10)}$	3.415
H(62)	C(18)$^{10)}$	3.476	H(62)	H(15)$^{11)}$	3.067
H(62)	H(21)$^{10)}$	2.710	H(62)	H(22)$^{10)}$	3.320
H(62)	H(26)$^{10)}$	2.578	H(62)	H(32)	3.470
H(62)	H(48)$^{11)}$	3.587	H(63)	O(4)$^{11)}$	3.387
H(63)	C(10)$^{11)}$	3.147	H(63)	C(11)$^{11)}$	3.126
H(63)	H(14)$^{11)}$	2.392	H(63)	H(15)$^{11)}$	2.559
H(63)	H(16)$^{11)}$	3.285	H(64)	C(21)$^{4)}$	3.017
H(64)	C(22)$^{4)}$	3.293	H(64)	H(16)$^{13)}$	3.029
H(64)	H(17)$^{13)}$	3.526	H(64)	H(30)$^{4)}$	2.397
H(64)	H(31)$^{4)}$	2.971	H(64)	H(70)$^{4)}$	3.227
H(65)	C(11)$^{13)}$	3.588	H(65)	C(18)$^{10)}$	3.512
H(65)	C(38)$^{11)}$	3.341	H(65)	H(16)$^{13)}$	3.271
H(65)	H(17)$^{13)}$	2.992	H(65)	H(26)$^{10)}$	3.048
H(65)	H(27)$^{10)}$	3.064	H(65)	H(30)$^{4)}$	3.353
H(65)	H(31)$^{4)}$	2.978	H(65)	H(48)$^{11)}$	2.756
H(65)	H(49)$^{11)}$	3.029	H(66)	H(12)$^{13)}$	3.153
H(66)	H(13)$^{13)}$	3.397	H(66)	H(16)$^{13)}$	3.098
H(66)	H(17)$^{13)}$	3.362	H(66)	H(46)$^{11)}$	3.272
H(66)	H(48)$^{11)}$	3.575	H(66)	H(49)$^{11)}$	3.433
H(67)	C(33)$^{5)}$	3.246	H(67)	C(34)$^{5)}$	3.387
H(67)	C(35)$^{5)}$	3.568	H(67)	H(42)$^{5)}$	3.451
H(67)	H(43)$^{5)}$	2.523	H(67)	H(44)$^{5)}$	3.380
H(67)	H(73)$^{6)}$	3.260	H(67)	H(74)$^{6)}$	3.404
H(68)	O(6)$^{5)}$	3.027	H(68)	C(7)$^{14)}$	3.450
H(68)	C(34)$^{5)}$	3.032	H(68)	C(35)$^{5)}$	3.334
H(68)	C(58)$^{6)}$	3.485	H(68)	H(6)$^{14)}$	3.414
H(68)	H(8)$^{14)}$	3.091	H(68)	H(9)$^{14)}$	2.885
H(68)	H(43)$^{5)}$	3.350	H(68)	H(74)$^{6)}$	2.666
H(69)	O(5)$^{2)}$	3.527	H(69)	C(7)$^{14)}$	3.553
H(69)	C(60)$^{2)}$	3.392	H(69)	H(5)	3.446
H(69)	H(8)$^{14)}$	2.660	H(69)	H(13)$^{11)}$	3.215
H(69)	H(40)$^{2)}$	3.185	H(69)	H(74)$^{6)}$	3.525
H(69)	H(76)$^{2)}$	2.554	H(70)	C(2)	3.502
H(70)	C(21)	3.519	H(70)	H(3)	2.660
H(70)	H(5)	3.089	H(70)	H(13)$^{11)}$	3.498
H(70)	H(16)$^{11)}$	2.865	H(70)	H(30)	3.591

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(70)	H(57)[2)]	3.376	H(70)	H(64)[2)]	3.227
H(71)	C(2)	3.430	H(71)	H(2)	3.350
H(71)	H(3)	2.647	H(71)	H(15)[11)]	3.528
H(71)	H(16)[11)]	3.238	H(72)	C(8)[11)]	3.516
H(72)	H(10)[11)]	2.581	H(72)	H(14)[11)]	2.688
H(72)	H(43)[5)]	3.008	H(72)	H(46)[11)]	3.121
H(72)	H(47)[11)]	3.593	H(73)	C(32)[5)]	3.483
H(73)	C(35)[11)]	3.491	H(73)	C(37)[11)]	3.493
H(73)	H(7)[11)]	3.227	H(73)	H(10)[11)]	3.316
H(73)	H(41)[5)]	2.610	H(73)	H(43)[5)]	3.247
H(73)	H(44)[11)]	3.090	H(73)	H(46)[11)]	2.884
H(73)	H(47)[11)]	3.464	H(73)	H(67)[5)]	3.260
H(74)	C(50)[5)]	3.197	H(74)	C(51)[5)]	2.749
H(74)	C(52)[5)]	3.265	H(74)	H(5)[5)]	3.058
H(74)	H(9)[13)]	3.227	H(74)	H(67)[5)]	3.404
H(74)	H(68)[5)]	2.666	H(74)	H(69)[5)]	3.525
H(75)	O(6)[18)]	3.153	H(75)	C(4)[5)]	3.228
H(75)	C(5)[5)]	2.901	H(75)	C(6)[5)]	3.578
H(75)	C(8)[13)]	3.447	H(75)	H(5)[5)]	3.239
H(75)	H(6)[5)]	2.724	H(75)	H(11)[13)]	2.648
H(75)	H(12)[13)]	3.458	H(75)	H(13)[13)]	3.374
H(76)	C(6)[5)]	3.508	H(76)	C(52)[4)]	3.345
H(76)	H(8)[5)]	2.888	H(76)	H(69)[4)]	2.554

Symmetry Operators:

(1) -X+1,Y+1/2-1,-Z
(2) X+1,Y,Z
(3) X,Y-1,Z
(4) X-1,Y,Z
(5) -X+1,Y+1/2,-Z+1
(6) -X+1,Y+1/2-1,-Z+1
(7) -X,Y+1/2-1,-Z+1
(8) -X+2,Y+1/2-1,-Z+1
(9) X+1,Y-1,Z
(10) -X+1,Y+1/2,-Z
(11) X,Y+1,Z
(12) -X+2,Y+1/2,-Z
(13) X-1,Y+1,Z
(14) -X+2,Y+1/2,-Z+1
(15) -X+2,Y+1/2-1,-Z
(16) -X,Y+1/2,-Z
(17) -X,Y+1/2-1,-Z
(18) -X,Y+1/2,-Z+1

Compound **6**

December 16, 2009

Experimental

Data Collection

A colorless block crystal of $O_4C_{14}H_{20}$ having approximate dimensions of 0.20 x 0.10 x 0.08 mm was mounted in a loop. All measurements were made on a Rigaku Mercury2 CCD area detector with filtered Mo-Ka radiation.

Indexing was performed from 6 images that were exposed for 20.0 seconds. The crystal-to-detector distance was 49.90 mm.

Cell constants and an orientation matrix for data collection corresponded to a primitive orthorhombic cell with dimensions:

a = 7.2838(7) Å
b = 9.5550(9) Å
c = 19.3652(19) Å
V = 1347.8(2) $Å^3$

For Z = 4 and F.W. = 252.31, the calculated density is 1.243 g/cm^3. The systematic absences of:

h00: h ± 2n
0k0: k ± 2n
00l: l ± 2n

uniquely determine the space group to be:

$P2_12_12_1$ (#19)

The data were collected at a temperature of -50 ± 1°C to a maximum 2q value of 55.0°. A total of 540 oscillation images were collected. A sweep of data was done using w scans from -120.0 to 60.0° in 1.0° step, at c=54.0° and f = 0.0°. The exposure rate was 60.0 [sec./°]. The detector swing angle was -28.40°. A second sweep was performed using w scans from -120.0 to 60.0° in 1.0° step, at c=54.0° and f = 120.0°. The exposure rate was 60.0 [sec./°]. The detector swing angle was -28.40°. Another sweep was performed using w scans from -120.0 to 60.0° in 1.0° step, at c=54.0° and f = 240.0°. The exposure rate was 60.0 [sec./°]. The detector swing angle was -28.40°. The crystal-to-detector distance was 49.90 mm. Readout was performed in the 0.146 mm pixel mode.

Data Reduction

Of the 13934 reflections that were collected, 3088 were unique (R_{int} = 0.066). Data were collected and processed using CrystalClear (Rigaku). Net intensities and sigmas were derived as follows:

$$F^2 = [S(P_i - mB_{ave})] \cdot Lp^{-1}$$

where P_i is the value in counts of the i^{th} pixel
m is the number of pixels in the integration area
B_{ave} is the background average
Lp is the Lorentz and polarization factor

$$B_{ave} = S(B_j)/n$$

where n is the number of pixels in the background area
B_j is the value of the j^{th} pixel in counts

$$s^2(F^2_{hkl}) = [(SP_i) + m((S(B_{ave} - B_j)^2)/(n-1))] \cdot Lp \cdot errmul + (erradd \cdot F^2)^2$$

where erradd = 0.00
errmul = 1.00

The linear absorption coefficient, m, for Mo-Ka radiation is 0.899 cm^{-1}. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.738 to 0.993. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods[2] and expanded using Fourier techniques[3]. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement[4] on F^2 was based on 3088 observed reflections and 168 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = S \; ||Fo| - |Fc|| \; / \; S \; |Fo| = 0.0606$$

$$wR2 = [S (w (Fo^2 - Fc^2)^2)/ S \; w(Fo^2)^2]^{1/2} = 0.1412$$

The standard deviation of an observation of unit weight[5] was 1.08. Unit weights were used. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.19 and -0.18 $e^-/Å^3$, respectively. The absolute structure was deduced based on Flack parameter, -1.8(16), using 1293 Friedel pairs.[6]

Neutral atom scattering factors were taken from Cromer and Waber[7]. Anomalous dispersion effects were included in Fcalc[8]; the values for Df' and Df'' were those of Creagh and McAuley[9]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[10]. All calculations were performed using the CrystalStructure[11] crystallographic software package except for refinement, which was performed using SHELXL-97[12].

References

(1) CrystalClear: Rigaku Corporation, 1999. CrystalClear Software User's Guide, Molecular Structure Corporation, (c) 2000.J.W.Pflugrath (1999) Acta Cryst. D55, 1718-1725.

(2) SHELX97: Sheldrick, G.M. (1997).

(3) DIRDIF99: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M.(1999). The DIRDIF-99 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(4) Least Squares function minimized: (SHELXL97)

$$\Sigma w(F_o^2-F_c^2)^2$$ where w = Least Squares weights.

(5) Standard deviation of an observation of unit weight:

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

where: N_o = number of observations
N_v = number of variables

(6) Flack, H. D. (1983), Acta Cryst. A39, 876-881.

(7) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(8) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(9) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(10) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(11) CrystalStructure 3.8: Crystal Structure Analysis Package, Rigaku and Rigaku Americas (2000-2007). 9009 New Trails Dr. The Woodlands TX 77381 USA.

(12) SHELX97: Sheldrick, G.M. (1997).

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$O_4C_{14}H_{20}$
Formula Weight	252.31
Crystal Color, Habit	colorless, block
Crystal Dimensions	0.20 X 0.10 X 0.08 mm
Crystal System	orthorhombic
Lattice Type	Primitive
Indexing Images	6 images @ 20.0 seconds
Detector Position	49.90 mm
Pixel Size	0.146 mm
Lattice Parameters	a = 7.2838(7) Å b = 9.5550(9) Å c = 19.3652(19) Å V = 1347.8(2) Å^3
Space Group	$P2_12_12_1$ (#19)

Z value	4
D_{calc}	1.243 g/cm^3
F_{000}	544.00
m(MoKa)	0.899 cm^{-1}

B. Intensity Measurements

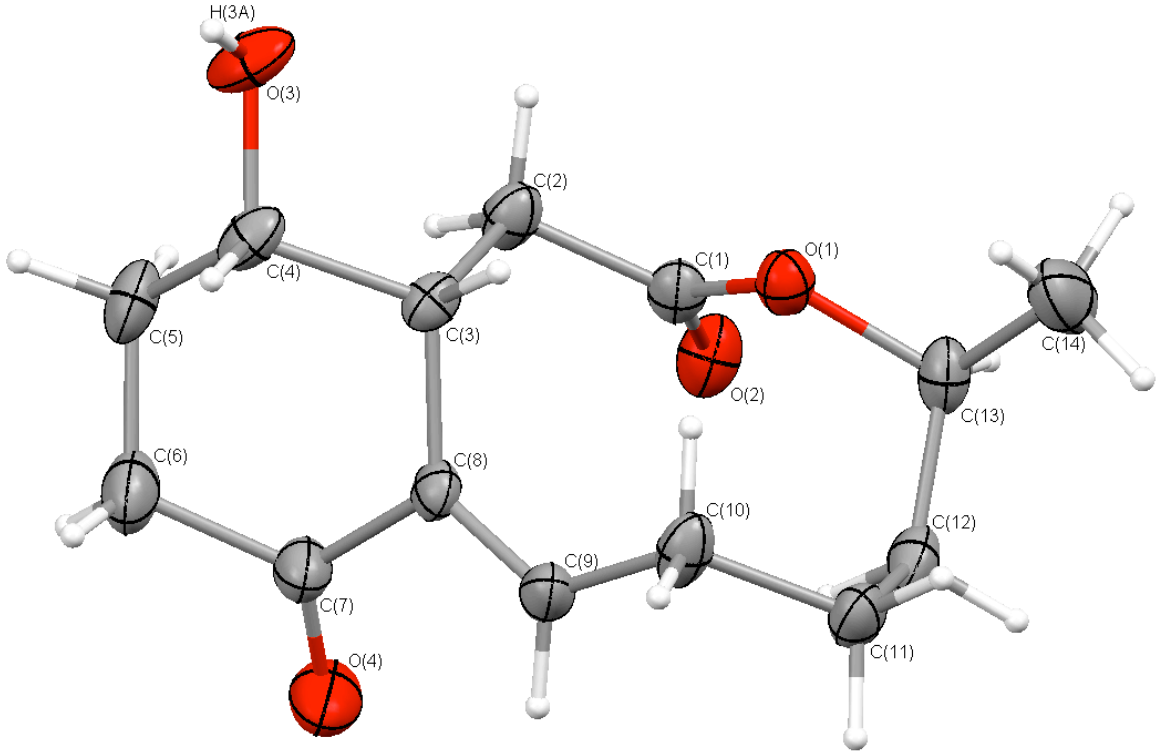
Diffractometer	Rigaku SCXmini
Radiation	MoKa (l = 0.71075 Å)
Detector Aperture	75 mm round
Data Images	540 exposures
w oscillation Range (c=54.0, f=0.0)	-120.0 - 60.0^o
Exposure Rate	60.0 sec./^{o}
Detector Swing Angle	-28.40^o
w oscillation Range (c=54.0, f=120.0)	-120.0 - 60.0^o
Exposure Rate	60.0 sec./^{o}
Detector Swing Angle	-28.40^o
w oscillation Range (c=54.0, f=240.0)	-120.0 - 60.0^o
Exposure Rate	60.0 sec./^{o}
Detector Swing Angle	-28.40^o
Detector Position	49.90 mm
Pixel Size	0.146 mm
$2q_{max}$	55.0^o

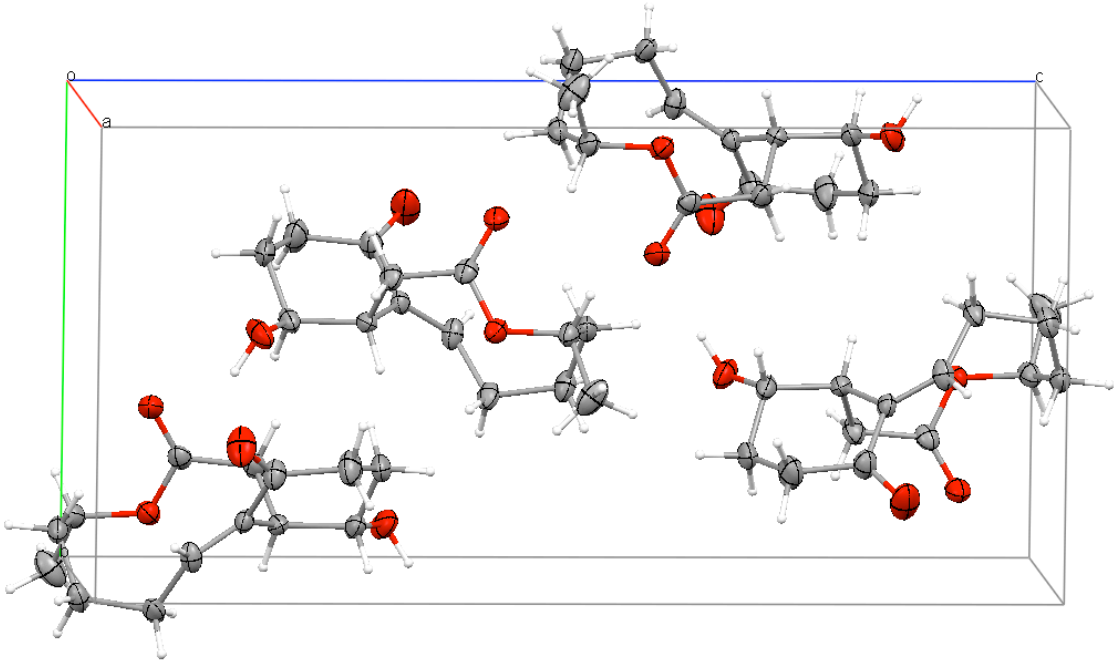
No. of Reflections Measured	Total: 13934 Unique: 3088 (R_{int} = 0.066) Friedel pairs: 1293
Corrections	Lorentz-polarization Absorption (trans. factors: 0.738 - 0.993)

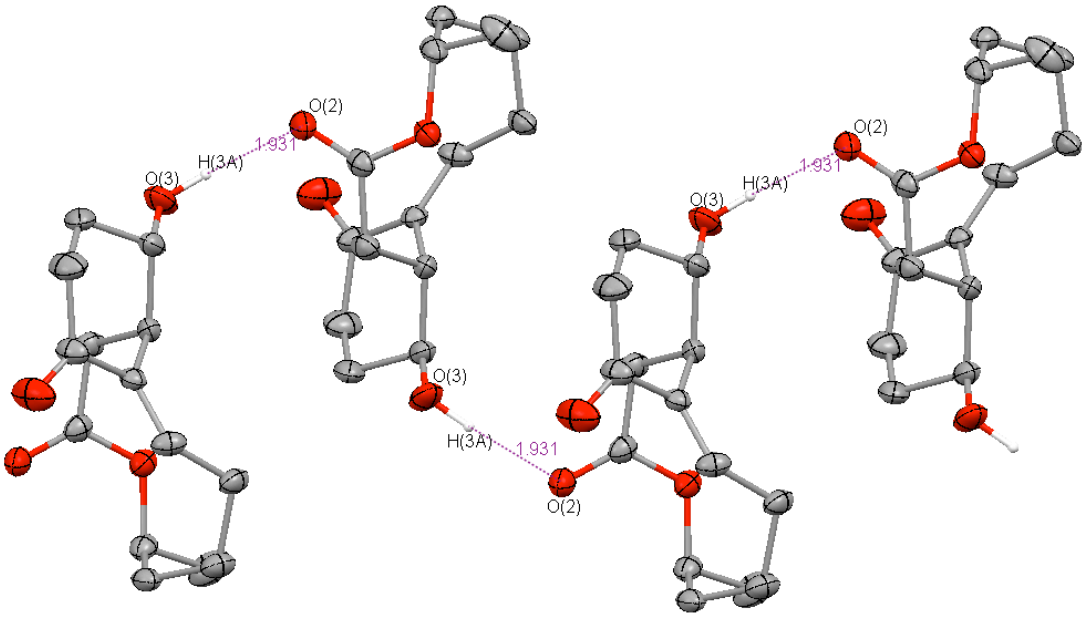
C. Structure Solution and Refinement

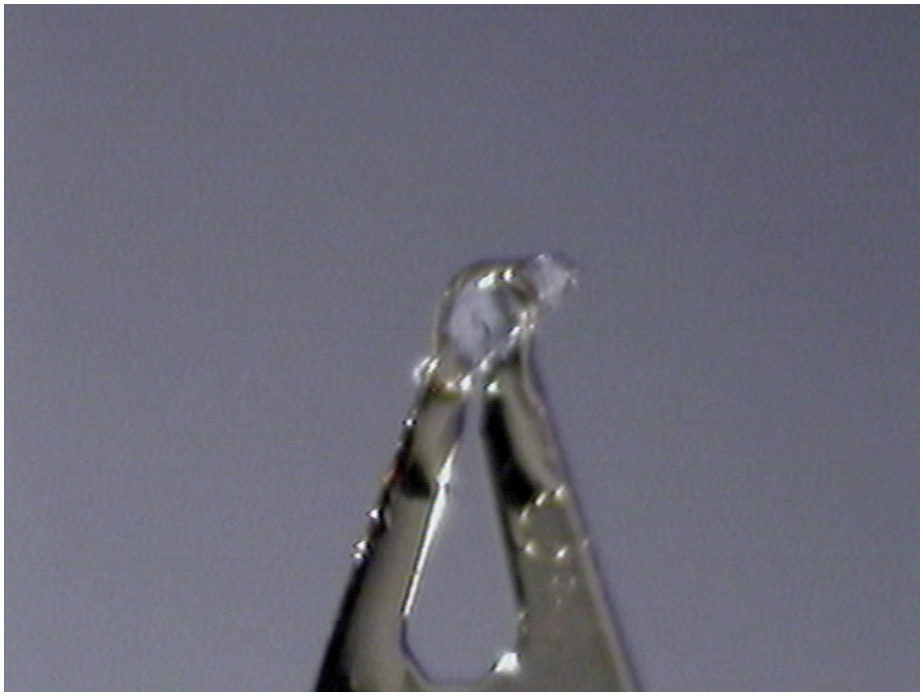
Structure Solution	Direct Methods (SHELX97)
Refinement	Full-matrix least-squares on F^2
Function Minimized	$S\ w\ (Fo^2 - Fc^2)^2$
Least Squares Weights	$w = 1/ [\ s^2(Fo^2) + (0.0611 \cdot P)^2 + 0.1034 \cdot P\ ]$ where $P = (Max(Fo^2,0) + 2Fc^2)/3$
$2q_{max}$ cutoff	55.0^o
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	3088
No. Variables	168
Reflection/Parameter Ratio	18.38
Residuals: R1 (I>2.00s(I))	0.0606
Residuals: R (All reflections)	0.0919
Residuals: wR2 (All reflections)	0.1412
Goodness of Fit Indicator	1.076
Flack Parameter	-1.8(16)
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	0.19 $e^-/Å^3$

Minimum peak in Final Diff. Map -0.18 $e^{-}/Å^3$

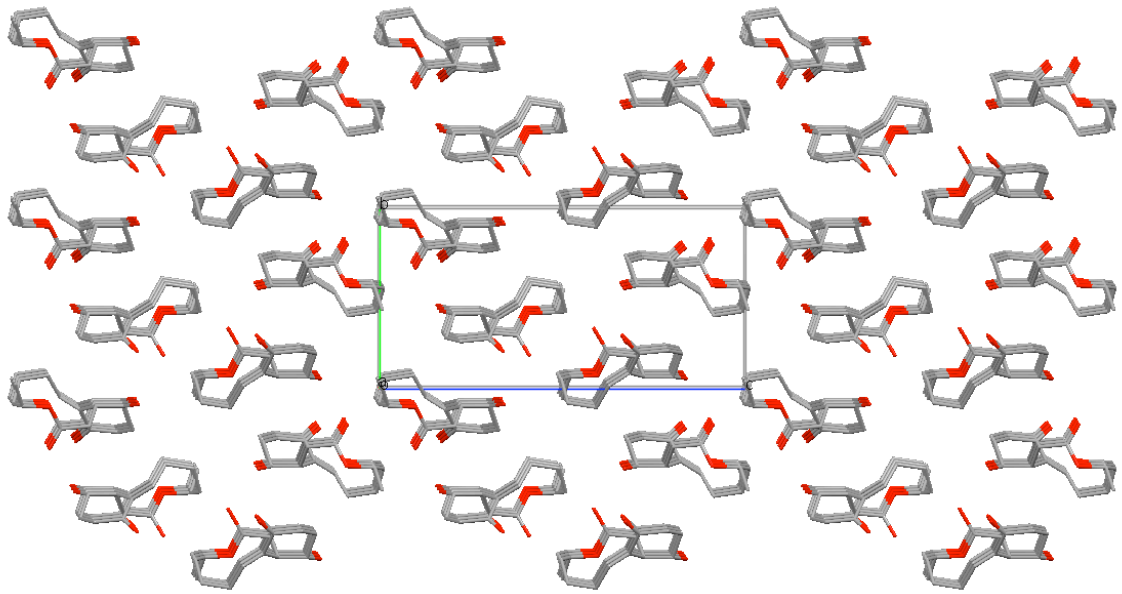




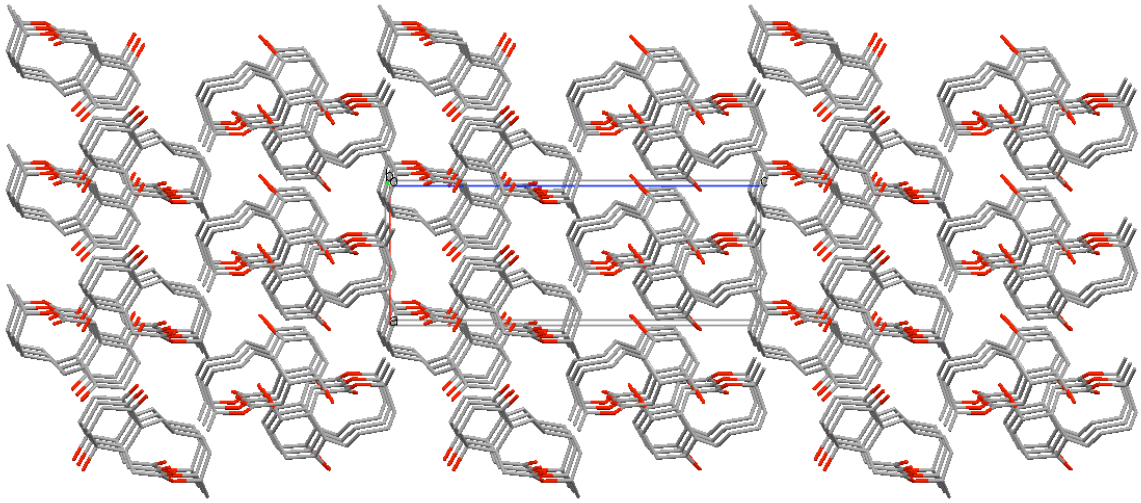

O(2)
H(3A)
1.931
O(3)
O(3)
H(3A)
1.931
O(2)
O(2)
H(3A)
1.931
O(3)



Packing diagram – View down the *a*-axis



Packing diagram – View down the *b*-axis



Packing diagram – View down the *c*-axis

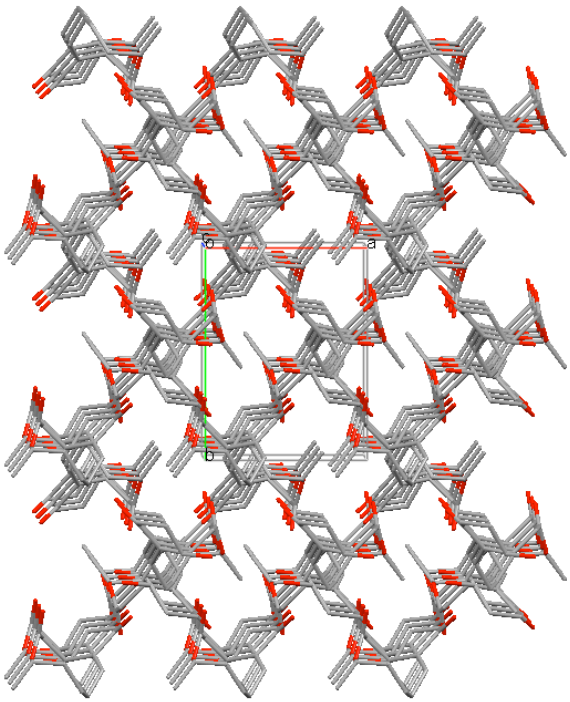


Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
O(1)	0.4064(2)	0.58237(17)	0.90649(8)	3.43(3)
O(2)	0.4752(2)	0.81168(18)	0.90785(9)	4.24(3)
O(3)	0.4874(3)	0.5673(2)	0.66579(9)	5.01(4)
O(4)	1.0204(3)	0.7820(2)	0.83365(11)	6.50(6)
C(1)	0.4516(3)	0.7032(2)	0.87639(13)	3.34(4)
C(2)	0.4723(3)	0.6883(2)	0.79992(12)	3.69(5)
C(3)	0.6197(3)	0.5789(2)	0.78060(11)	2.79(4)
C(4)	0.6568(3)	0.5793(2)	0.70208(11)	3.58(5)
C(5)	0.7582(4)	0.7117(2)	0.67970(12)	4.29(6)
C(6)	0.9390(4)	0.7289(3)	0.71800(14)	5.48(7)
C(7)	0.9226(3)	0.7139(3)	0.79542(13)	4.02(5)
C(8)	0.7944(3)	0.6036(2)	0.82144(11)	2.86(4)
C(9)	0.8451(3)	0.5327(3)	0.87776(12)	3.66(5)
C(10)	0.7516(4)	0.4118(2)	0.91195(12)	3.89(5)
C(11)	0.7156(4)	0.4356(2)	0.98955(12)	3.94(5)
C(12)	0.6072(3)	0.5667(2)	1.00736(12)	3.72(5)
C(13)	0.4107(3)	0.5757(2)	0.98268(12)	3.60(4)
C(14)	0.2923(4)	0.4542(3)	1.00244(16)	5.87(7)

$B_{eq} = 8/3\ p^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos g + 2U_{13}(aa^*cc^*)\cos b + 2U_{23}(bb^*cc^*)\cos a)$

Table 2. Atomic coordinates and B_{iso} involving hydrogens/B_{eq}

atom	x	y	z	B_{eq}
H(1)	0.5061	0.7791	0.7801	4.42
H(2)	0.3543	0.6603	0.7799	4.42
H(3)	0.5719	0.4854	0.7932	3.35
H(4)	0.7340	0.4973	0.6906	4.30
H(5)	0.7821	0.7075	0.6300	5.15
H(6)	0.6803	0.7934	0.6886	5.15
H(7)	1.0260	0.6588	0.7009	6.58
H(8)	0.9896	0.8215	0.7075	6.58
H(9)	0.9545	0.5628	0.8988	4.39
H(10)	0.8277	0.3280	0.9063	4.67
H(11)	0.6342	0.3945	0.8887	4.67
H(12)	0.6492	0.3543	1.0076	4.73
H(13)	0.8341	0.4399	1.0134	4.73
H(14)	0.6068	0.5766	1.0577	4.47
H(15)	0.6737	0.6473	0.9886	4.47
H(16)	0.3550	0.6623	1.0013	4.32
H(17)	0.1664	0.4855	1.0079	7.05
H(18)	0.3357	0.4148	1.0456	7.05
H(19)	0.2978	0.3835	0.9666	7.05
H(3A)	0.496(5)	0.487(3)	0.6406(18)	6.9(8)

$B_{eq} = 8/3\ p^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos g + 2U_{13}(aa^*cc^*)\cos b + 2U_{23}(bb^*cc^*)\cos a)$

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	0.0454(9)	0.0423(9)	0.0425(9)	0.0037(8)	0.0039(7)	0.0055(7)
O(2)	0.0760(13)	0.0393(9)	0.0459(9)	0.0098(10)	0.0080(9)	0.0026(8)
O(3)	0.0810(14)	0.0621(13)	0.0471(10)	0.0122(11)	-0.0261(11)	-0.0179(10)
O(4)	0.0799(15)	0.1082(18)	0.0590(13)	-0.0501(15)	-0.0014(12)	-0.0052(12)
C(1)	0.0415(14)	0.0444(14)	0.0409(13)	0.0131(11)	0.0040(11)	0.0069(12)
C(2)	0.0535(16)	0.0490(15)	0.0375(13)	0.0137(13)	-0.0016(12)	0.0052(11)
C(3)	0.0461(13)	0.0285(11)	0.0314(11)	0.0005(11)	-0.0036(10)	-0.0014(9)
C(4)	0.0616(16)	0.0423(14)	0.0321(12)	0.0105(14)	-0.0082(12)	-0.0068(11)
C(5)	0.083(2)	0.0515(15)	0.0283(11)	-0.0014(15)	0.0079(13)	0.0035(12)
C(6)	0.082(2)	0.084(2)	0.0424(15)	-0.0221(19)	0.0131(15)	0.0044(15)
C(7)	0.0505(16)	0.0643(18)	0.0378(13)	-0.0129(14)	0.0040(13)	-0.0027(13)
C(8)	0.0417(12)	0.0412(13)	0.0257(10)	-0.0000(11)	0.0047(10)	-0.0046(10)

C(9)	0.0374(13)	0.0672(17)	0.0344(12)	0.0063(12)	0.0022(10)	0.0051(12)
C(10)	0.0619(17)	0.0497(14)	0.0363(12)	0.0207(14)	0.0040(12)	0.0098(11)
C(11)	0.0497(15)	0.0654(17)	0.0347(12)	0.0104(14)	0.0003(11)	0.0112(13)
C(12)	0.0628(17)	0.0506(15)	0.0280(11)	-0.0098(14)	0.0034(11)	-0.0015(12)
C(13)	0.0577(16)	0.0422(14)	0.0370(12)	0.0086(13)	0.0153(12)	0.0053(11)
C(14)	0.0523(17)	0.095(2)	0.076(2)	-0.0057(17)	0.0062(16)	0.033(2)

The general temperature factor expression: $\exp(-2p^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
O(1)	C(1)	1.335(3)	O(1)	C(13)	1.477(2)
O(2)	C(1)	1.214(3)	O(3)	C(4)	1.425(3)
O(4)	C(7)	1.216(3)	C(1)	C(2)	1.495(3)
C(2)	C(3)	1.544(3)	C(3)	C(4)	1.544(3)
C(3)	C(8)	1.516(3)	C(4)	C(5)	1.528(3)
C(5)	C(6)	1.520(4)	C(6)	C(7)	1.511(3)
C(7)	C(8)	1.496(3)	C(8)	C(9)	1.336(3)
C(9)	C(10)	1.495(3)	C(10)	C(11)	1.542(3)
C(11)	C(12)	1.521(3)	C(12)	C(13)	1.511(3)
C(13)	C(14)	1.496(4)			

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
O(3)	H(3A)	0.92(3)	C(2)	H(1)	0.980
C(2)	H(2)	0.980	C(3)	H(3)	0.990
C(4)	H(4)	0.990	C(5)	H(5)	0.980
C(5)	H(6)	0.980	C(6)	H(7)	0.980
C(6)	H(8)	0.980	C(9)	H(9)	0.940
C(10)	H(10)	0.980	C(10)	H(11)	0.980
C(11)	H(12)	0.980	C(11)	H(13)	0.980
C(12)	H(14)	0.980	C(12)	H(15)	0.980
C(13)	H(16)	0.990	C(14)	H(17)	0.970
C(14)	H(18)	0.970	C(14)	H(19)	0.970

Table 6. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
C(1)	O(1)	C(13)	117.94(18)	O(1)	C(1)	O(2)	123.7(2)

O(1)	C(1)	C(2)	112.0(2)	O(2)	C(1)	C(2)	124.3(2)
C(1)	C(2)	C(3)	112.0(2)	C(2)	C(3)	C(4)	111.02(18)
C(2)	C(3)	C(8)	110.59(18)	C(4)	C(3)	C(8)	111.49(19)
O(3)	C(4)	C(3)	109.5(2)	O(3)	C(4)	C(5)	110.2(2)
C(3)	C(4)	C(5)	111.45(19)	C(4)	C(5)	C(6)	111.7(2)
C(5)	C(6)	C(7)	113.9(2)	O(4)	C(7)	C(6)	120.5(2)
O(4)	C(7)	C(8)	122.5(2)	C(6)	C(7)	C(8)	116.8(2)
C(3)	C(8)	C(7)	117.24(19)	C(3)	C(8)	C(9)	125.4(2)
C(7)	C(8)	C(9)	117.4(2)	C(8)	C(9)	C(10)	128.9(2)
C(9)	C(10)	C(11)	113.3(2)	C(10)	C(11)	C(12)	115.5(2)
C(11)	C(12)	C(13)	117.8(2)	O(1)	C(13)	C(12)	109.77(19)
O(1)	C(13)	C(14)	106.1(2)	C(12)	C(13)	C(14)	114.9(2)

Table 7. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
C(4)	O(3)	H(3A)	106(2)	C(1)	C(2)	H(1)	109.2
C(1)	C(2)	H(2)	109.2	C(3)	C(2)	H(1)	109.2
C(3)	C(2)	H(2)	109.2	H(1)	C(2)	H(2)	107.9
C(2)	C(3)	H(3)	107.9	C(4)	C(3)	H(3)	107.9
C(8)	C(3)	H(3)	107.9	O(3)	C(4)	H(4)	108.5
C(3)	C(4)	H(4)	108.5	C(5)	C(4)	H(4)	108.5
C(4)	C(5)	H(5)	109.3	C(4)	C(5)	H(6)	109.3
C(6)	C(5)	H(5)	109.3	C(6)	C(5)	H(6)	109.3
H(5)	C(5)	H(6)	107.9	C(5)	C(6)	H(7)	108.8
C(5)	C(6)	H(8)	108.8	C(7)	C(6)	H(7)	108.8
C(7)	C(6)	H(8)	108.8	H(7)	C(6)	H(8)	107.7
C(8)	C(9)	H(9)	115.6	C(10)	C(9)	H(9)	115.6
C(9)	C(10)	H(10)	108.9	C(9)	C(10)	H(11)	108.9
C(11)	C(10)	H(10)	108.9	C(11)	C(10)	H(11)	108.9
H(10)	C(10)	H(11)	107.7	C(10)	C(11)	H(12)	108.4
C(10)	C(11)	H(13)	108.4	C(12)	C(11)	H(12)	108.4
C(12)	C(11)	H(13)	108.4	H(12)	C(11)	H(13)	107.5
C(11)	C(12)	H(14)	107.9	C(11)	C(12)	H(15)	107.9
C(13)	C(12)	H(14)	107.9	C(13)	C(12)	H(15)	107.9
H(14)	C(12)	H(15)	107.2	O(1)	C(13)	H(16)	108.6
C(12)	C(13)	H(16)	108.6	C(14)	C(13)	H(16)	108.6
C(13)	C(14)	H(17)	109.5	C(13)	C(14)	H(18)	109.5
C(13)	C(14)	H(19)	109.5	H(17)	C(14)	H(18)	109.5
H(17)	C(14)	H(19)	109.5	H(18)	C(14)	H(19)	109.5

Table 8. Torsion Angles(°)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C(1)	O(1)	C(13)	C(12)	-75.7(2)	C(1)	O(1)	C(13)	C(14)	159.6(2)

C(13)	O(1)	C(1)	O(2)	-8.8(3)	C(13)	O(1)	C(1)	C(2)	170.3(2)
O(1)	C(1)	C(2)	C(3)	-58.5(2)	O(2)	C(1)	C(2)	C(3)	120.6(2)
C(1)	C(2)	C(3)	C(4)	-172.5(2)	C(1)	C(2)	C(3)	C(8)	-48.2(2)
C(2)	C(3)	C(4)	O(3)	-51.5(2)	C(2)	C(3)	C(4)	C(5)	70.7(2)
C(2)	C(3)	C(8)	C(7)	-81.6(2)	C(2)	C(3)	C(8)	C(9)	100.1(2)
C(4)	C(3)	C(8)	C(7)	42.5(2)	C(4)	C(3)	C(8)	C(9)	-135.8(2)
C(8)	C(3)	C(4)	O(3)	-175.27(19)	C(8)	C(3)	C(4)	C(5)	-53.1(2)
O(3)	C(4)	C(5)	C(6)	179.4(2)	C(3)	C(4)	C(5)	C(6)	57.6(2)
C(4)	C(5)	C(6)	C(7)	-49.8(3)	C(5)	C(6)	C(7)	O(4)	-146.5(2)
C(5)	C(6)	C(7)	C(8)	38.9(3)	O(4)	C(7)	C(8)	C(3)	149.6(2)
O(4)	C(7)	C(8)	C(9)	-32.0(3)	C(6)	C(7)	C(8)	C(3)	-35.9(3)
C(6)	C(7)	C(8)	C(9)	142.5(2)	C(3)	C(8)	C(9)	C(10)	2.8(4)
C(7)	C(8)	C(9)	C(10)	-175.5(2)	C(8)	C(9)	C(10)	C(11)	-126.5(2)
C(9)	C(10)	C(11)	C(12)	55.5(3)	C(10)	C(11)	C(12)	C(13)	64.8(3)
C(11)	C(12)	C(13)	O(1)	-66.1(2)	C(11)	C(12)	C(13)	C(14)	53.4(3)

The sign is positive if when looking from atom 2 to atom 3 a clock-wise motion of atom 1 would superimpose it on atom 4.

Table 9. Distances beyond the asymmetric unit out to 3.60 Å

atom	atom	distance	atom	atom	distance
O(2)	O(3)[1)]	2.841(2)	O(2)	C(4)[1)]	3.464(3)
O(2)	C(12)[2)]	3.351(3)	O(3)	O(2)[3)]	2.841(2)
O(4)	C(1)[4)]	3.334(3)	O(4)	C(2)[4)]	3.473(3)
O(4)	C(12)[5)]	3.460(3)	C(1)	O(4)[6)]	3.334(3)
C(2)	O(4)[6)]	3.473(3)	C(4)	O(2)[3)]	3.464(3)
C(12)	O(2)[5)]	3.351(3)	C(12)	O(4)[2)]	3.460(3)

Symmetry Operators:

(1) -X+1,Y+1/2,-Z+1/2+1	(2) X+1/2-1,-Y+1/2+1,-Z+2
(3) -X+1,Y+1/2-1,-Z+1/2+1	(4) X+1,Y,Z
(5) X+1/2,-Y+1/2+1,-Z+2	(6) X-1,Y,Z

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
O(1)	H(6)[1)]	3.378	O(1)	H(9)[2)]	3.300
O(2)	H(4)[3)]	3.017	O(2)	H(13)[4)]	3.002
O(2)	H(14)[4)]	2.964	O(2)	H(15)[4)]	3.000
O(2)	H(16)[5)]	3.288	O(2)	H(17)[5)]	2.890
O(2)	H(3A)[3)]	1.93(3)	O(3)	H(1)[1)]	2.947
O(3)	H(7)[2)]	3.538	O(3)	H(11)[3)]	3.417
O(3)	H(13)[6)]	3.226	O(3)	H(17)[7)]	3.294

O(3)	H(18)[7)]	3.314	O(4)	H(2)[8)]	2.890
O(4)	H(4)[9)]	2.766	O(4)	H(14)[5)]	2.578
O(4)	H(16)[5)]	3.456	C(1)	H(4)[3)]	3.377
C(1)	H(14)[4)]	3.516	C(1)	H(3A)[3)]	2.75(3)
C(2)	H(3)[3)]	3.378	C(2)	H(4)[3)]	3.318
C(2)	H(3A)[3)]	3.08(3)	C(3)	H(1)[1)]	3.230
C(3)	H(6)[1)]	3.546	C(4)	H(1)[1)]	3.124
C(5)	H(3)[3)]	3.591	C(5)	H(12)[6)]	3.457
C(5)	H(13)[6)]	3.595	C(5)	H(19)[3)]	3.299
C(6)	H(2)[8)]	3.320	C(6)	H(10)[9)]	3.096
C(7)	H(2)[8)]	3.200	C(8)	H(8)[10)]	3.171
C(9)	H(8)[10)]	2.871	C(9)	H(17)[8)]	3.470
C(10)	H(8)[10)]	3.106	C(10)	H(18)[11)]	3.285
C(11)	H(5)[12)]	3.043	C(11)	H(17)[8)]	3.337
C(11)	H(18)[11)]	3.527	C(11)	H(19)[11)]	3.221
C(12)	H(16)[5)]	3.161	C(13)	H(15)[4)]	3.208
C(14)	H(5)[1)]	3.525	C(14)	H(9)[2)]	3.341
C(14)	H(10)[13)]	3.234	C(14)	H(12)[13)]	3.132
C(14)	H(13)[2)]	3.346	C(14)	H(3A)[14)]	3.45(3)
H(1)	O(3)[3)]	2.947	H(1)	C(3)[3)]	3.230
H(1)	C(4)[3)]	3.124	H(1)	H(3)[3)]	2.495
H(1)	H(4)[3)]	2.780	H(1)	H(11)[3)]	3.598
H(1)	H(3A)[3)]	2.507	H(2)	O(4)[2)]	2.890
H(2)	C(6)[2)]	3.320	H(2)	C(7)[2)]	3.200
H(2)	H(3)[3)]	3.456	H(2)	H(4)[3)]	3.333
H(2)	H(6)[1)]	3.567	H(2)	H(7)[2)]	2.839
H(2)	H(8)[2)]	3.376	H(3)	C(2)[1)]	3.378
H(3)	C(5)[1)]	3.591	H(3)	H(1)[1)]	2.495
H(3)	H(2)[1)]	3.456	H(3)	H(6)[1)]	2.620
H(3)	H(8)[10)]	3.557	H(4)	O(2)[1)]	3.017
H(4)	O(4)[10)]	2.766	H(4)	C(1)[1)]	3.377
H(4)	C(2)[1)]	3.318	H(4)	H(1)[1)]	2.780
H(4)	H(2)[1)]	3.333	H(4)	H(8)[10)]	3.281
H(4)	H(13)[6)]	3.520	H(4)	H(14)[6)]	2.910
H(5)	C(11)[6)]	3.043	H(5)	C(14)[3)]	3.525
H(5)	H(10)[9)]	3.146	H(5)	H(11)[3)]	3.538
H(5)	H(12)[6)]	2.492	H(5)	H(13)[6)]	2.792
H(5)	H(14)[6)]	3.159	H(5)	H(18)[6)]	3.432
H(5)	H(19)[3)]	2.581	H(6)	O(1)[3)]	3.378
H(6)	C(3)[3)]	3.546	H(6)	H(2)[3)]	3.567
H(6)	H(3)[3)]	2.620	H(6)	H(11)[3)]	2.902
H(6)	H(19)[3)]	3.129	H(7)	O(3)[8)]	3.538
H(7)	H(2)[8)]	2.839	H(7)	H(10)[9)]	2.839
H(7)	H(18)[6)]	3.248	H(8)	C(8)[9)]	3.171
H(8)	C(9)[9)]	2.871	H(8)	C(10)[9)]	3.106
H(8)	H(2)[8)]	3.376	H(8)	H(3)[9)]	3.557

H(8)	H(4)[9)]	3.281	H(8)	H(9)[9)]	3.117
H(8)	H(10)[9)]	2.576	H(8)	H(11)[9)]	3.386
H(9)	O(1)[8)]	3.300	H(9)	C(14)[8)]	3.341
H(9)	H(8)[10)]	3.117	H(9)	H(16)[5)]	3.342
H(9)	H(17)[8)]	2.720	H(9)	H(19)[8)]	3.304
H(10)	C(6)[10)]	3.096	H(10)	C(14)[11)]	3.234
H(10)	H(5)[10)]	3.146	H(10)	H(7)[10)]	2.839
H(10)	H(8)[10)]	2.576	H(10)	H(12)[11)]	3.360
H(10)	H(17)[8)]	3.496	H(10)	H(18)[11)]	2.501
H(10)	H(19)[11)]	3.192	H(11)	O(3)[1)]	3.417
H(11)	H(1)[1)]	3.598	H(11)	H(5)[1)]	3.538
H(11)	H(6)[1)]	2.902	H(11)	H(8)[10)]	3.386
H(11)	H(18)[11)]	3.537	H(12)	C(5)[12)]	3.457
H(12)	C(14)[11)]	3.132	H(12)	H(5)[12)]	2.492
H(12)	H(10)[13)]	3.360	H(12)	H(17)[11)]	3.263
H(12)	H(18)[11)]	3.086	H(12)	H(19)[11)]	2.566
H(13)	O(2)[5)]	3.002	H(13)	O(3)[12)]	3.226
H(13)	C(5)[12)]	3.595	H(13)	C(14)[8)]	3.346
H(13)	H(4)[12)]	3.520	H(13)	H(5)[12)]	2.792
H(13)	H(17)[8)]	2.462	H(13)	H(18)[11)]	3.577
H(13)	H(19)[8)]	3.538	H(13)	H(19)[11)]	3.126
H(13)	H(3A)[12)]	2.846	H(14)	O(2)[5)]	2.964
H(14)	O(4)[4)]	2.578	H(14)	C(1)[5)]	3.516
H(14)	H(4)[12)]	2.910	H(14)	H(5)[12)]	3.159
H(14)	H(16)[5)]	3.286	H(14)	H(3A)[12)]	3.366
H(15)	O(2)[5)]	3.000	H(15)	C(13)[5)]	3.208
H(15)	H(16)[5)]	2.256	H(15)	H(17)[5)]	3.509
H(16)	O(2)[4)]	3.288	H(16)	O(4)[4)]	3.456
H(16)	C(12)[4)]	3.161	H(16)	H(9)[4)]	3.342
H(16)	H(14)[4)]	3.286	H(16)	H(15)[4)]	2.256
H(17)	O(2)[4)]	2.890	H(17)	O(3)[14)]	3.294
H(17)	C(9)[2)]	3.470	H(17)	C(11)[2)]	3.337
H(17)	H(9)[2)]	2.720	H(17)	H(10)[2)]	3.496
H(17)	H(12)[13)]	3.263	H(17)	H(13)[2)]	2.462
H(17)	H(15)[4)]	3.509	H(17)	H(3A)[14)]	2.840
H(18)	O(3)[14)]	3.314	H(18)	C(10)[13)]	3.285
H(18)	C(11)[13)]	3.527	H(18)	H(5)[12)]	3.432
H(18)	H(7)[12)]	3.248	H(18)	H(10)[13)]	2.501
H(18)	H(11)[13)]	3.537	H(18)	H(12)[13)]	3.086
H(18)	H(13)[13)]	3.577	H(18)	H(3A)[14)]	3.178
H(19)	C(5)[1)]	3.299	H(19)	C(11)[13)]	3.221
H(19)	H(5)[1)]	2.581	H(19)	H(6)[1)]	3.129
H(19)	H(9)[2)]	3.304	H(19)	H(10)[13)]	3.192
H(19)	H(12)[13)]	2.566	H(19)	H(13)[2)]	3.538
H(19)	H(13)[13)]	3.126	H(3A)	O(2)[1)]	1.93(3)
H(3A)	C(1)[1)]	2.75(3)	H(3A)	C(2)[1)]	3.08(3)

H(3A)	C(14)[7)]	3.45(3)	H(3A)	H(1)[1)]	2.507
H(3A)	H(13)[6)]	2.846	H(3A)	H(14)[6)]	3.366
H(3A)	H(17)[7)]	2.840	H(3A)	H(18)[7)]	3.178

Symmetry Operators:

(1) -X+1,Y+1/2-1,-Z+1/2+1	(2) X-1,Y,Z
(3) -X+1,Y+1/2,-Z+1/2+1	(4) X+1/2-1,-Y+1/2+1,-Z+2
(5) X+1/2,-Y+1/2+1,-Z+2	(6) -X+1/2+1,-Y+1,Z+1/2-1
(7) -X+1/2,-Y+1,Z+1/2-1	(8) X+1,Y,Z
(9) -X+2,Y+1/2,-Z+1/2+1	(10) -X+2,Y+1/2-1,-Z+1/2+1
(11) X+1/2,-Y+1/2,-Z+2	(12) -X+1/2+1,-Y+1,Z+1/2
(13) X+1/2-1,-Y+1/2,-Z+2	(14) -X+1/2,-Y+1,Z+1/2

Compound **26**

September 1, 2010

Experimental

Data Collection

A colorless needle crystal of $C_{14}H_{22}O_4$ having approximate dimensions of 0.08 x 0.08 x 0.07 mm was mounted in a loop. All measurements were made on a Rigaku Saturn944 CCD diffractometer using graphite monochromated Cu-Ka radiation.

The crystal-to-detector distance was 50.00 mm.

Cell constants and an orientation matrix for data collection corresponded to a primitive orthorhombic cell with dimensions:

a = 6.16325(11) Å
b = 11.2731(2) Å
c = 18.7642(13) Å
V = 1303.72(10) $Å^3$

For Z = 4 and F.W. = 254.33, the calculated density is 1.296 g/cm^3. The reflection conditions of:

h00: h = 2n
0k0: k = 2n
00l: l = 2n

uniquely determine the space group to be:

$P2_12_12_1$ (#19)

The data were collected at a temperature of -180 ± 1°C to a maximum 2q value of 131.8°. A total of 335 oscillation images were collected. A sweep of data was done using w scans from -48.0 to 132.0° in 2.0° step, at c=45.0° and f = 90.0°. The exposure rate was 7.5 [sec./°]. The detector swing angle was 42.00°. A second sweep was performed using w scans from 0.0 to 180.0° in 2.0° step, at c=45.0° and f = 45.0°. The exposure rate was 15.0 [sec./o]. The detector swing angle was 90.00°. Another sweep was performed using w scans from 0.0 to 150.0° in 2.0° step, at c=30.0° and f = 90.0°. The exposure rate was 15.0 [sec./o]. The detector swing angle was 90.00o. Another sweep was performed using w scans from 0.0 to 100.0° in 2.0° step, at c=45.0° and f = 270.0°. The exposure rate was 15.0 [sec./o]. The detector swing angle was 90.00o. Another sweep was performed using w scans from 10.0 to 40.0° in 2.0° step, at c=15.0° and f = 180.0°. The exposure rate was 15.0 [sec./o]. The detector swing angle was 90.00o. Another sweep was performed using w scans from 40.0 to 70.0° in 2.0° step, at c=30.0° and f = 45.0°. The exposure rate was 15.0 [sec./o]. The detector swing angle was 90.00o. The crystal-to-detector distance was 50.00 mm. Readout was performed in the 0.090 mm pixel mode.

Data Reduction

Of the 5898 reflections that were collected, 2202 were unique (R_{int} = 0.0376). Data were collected and processed using CrystalClear (Rigaku).

The linear absorption coefficient, m, for Cu-Ka radiation is 7.657 cm^{-1}. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.697 to 0.948. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods[2] and expanded using Fourier techniques. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement[3] on F^2 was based on 2194 observed reflections and 167 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = S \, ||Fo| - |Fc|| \, / \, S \, |Fo| = 0.0336$$

$$wR2 = [S (w (Fo^2 - Fc^2)^2)/ S w(Fo^2)^2]^{1/2} = 0.0832$$

The standard deviation of an observation of unit weight[4] was 1.05. Unit weights were used. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.14 and -0.17 $e^-/Å^3$, respectively. The absolute structure was deduced based on Flack parameter, 0.22(20), using 872 Friedel pairs.[5]

Neutral atom scattering factors were taken from Cromer and Waber[6]. Anomalous dispersion effects were included in Fcalc[7]; the values for Df' and Df'' were those of Creagh and McAuley[8]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[9]. All calculations were performed using the CrystalStructure[10] crystallographic software package except for refinement, which was performed using SHELXL-97[11].

References

(1) CrystalClear: Rigaku Corporation, 1999. CrystalClear Software User's Guide, Molecular Structure Corporation, (c) 2000.J.W.Pflugrath (1999) Acta Cryst. D55, 1718-1725.

(2) SIR2004: M.C. Burla, R. Caliandro, M. Camalli, B. Carrozzini, G.L. Cascarano, L. De Caro, C. Giacovazzo, G. Polidori, R. Spagna (2005)

(3) Least Squares function minimized: (SHELXL97)

$Sw(F_o^2-F_c^2)^2$ where w = Least Squares weights.

(4) Standard deviation of an observation of unit weight:

$[Sw(F_o^2-F_c^2)^2/(N_o-N_v)]^{1/2}$

where N_o = number of observations
N_v = number of variables

(5) Flack, H. D. (1983), Acta Cryst. A39, 876-881.

(6) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(7) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(8) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(9) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(10) CrystalStructure 4.0: Crystal Structure Analysis Package, Rigaku and Rigaku Americas (2000-2010). 9009 New Trails Dr. The Woodlands TX 77381 USA.

(11) SHELX97: Sheldrick, G.M. (1997).

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{14}H_{22}O_4$
Formula Weight	254.33
Crystal Color, Habit	colorless, needle
Crystal Dimensions	0.08 X 0.08 X 0.07 mm
Crystal System	orthorhombic
Lattice Type	Primitive
Lattice Parameters	a = 6.16325(11) Å b = 11.2731(2) Å c = 18.7642(13) Å V = 1303.72(10) Å^3
Space Group	$P2_12_12_1$ (#19)
Z value	4
D_{calc}	1.296 g/cm^3
F_{000}	552.00
m(CuKa)	7.657 cm^{-1}

B. Intensity Measurements

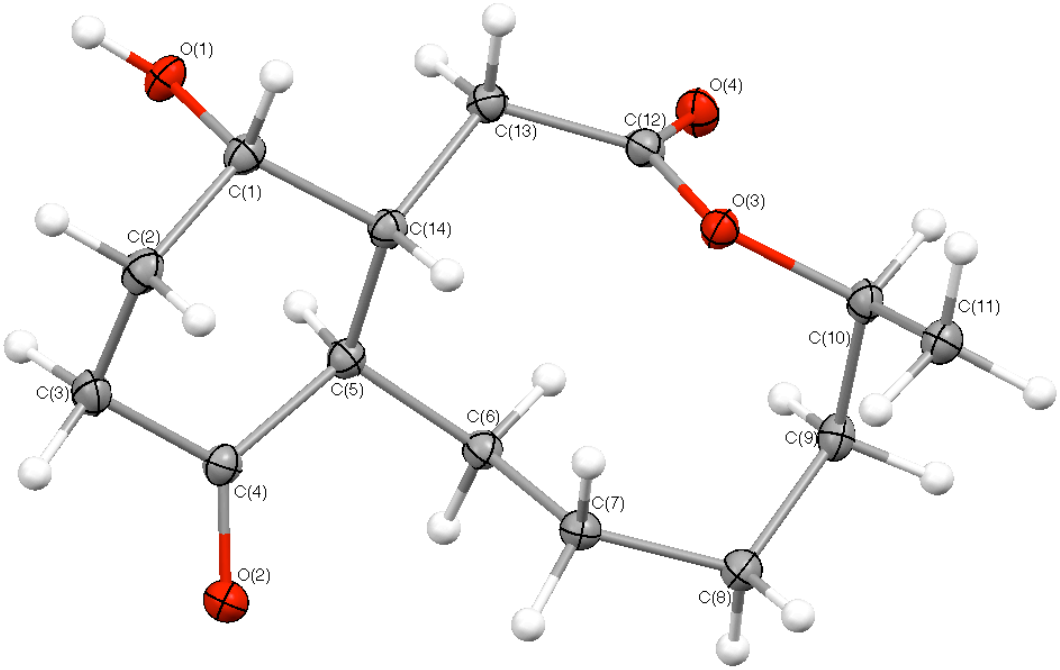
Diffractometer	Rigaku Saturn724 CCD
Radiation	CuKa (l = 1.54187 Å) graphite monochromated
Voltage, Current	50kV, 40mA
Temperature	-180.0°C
Detector Aperture	93 x 93 mm
Data Images	335 exposures
w oscillation Range (c=45.0, f=90.0)	-48.0 - 132.0°
Exposure Rate	7.5 sec./°
Detector Swing Angle	42.00°, 90.00 °
w oscillation Range (c=45.0, f=45.0)	0.0 - 180.0°
Exposure Rate	7.5 sec./°
Detector Swing Angle	42.00°
w oscillation Range (c=30.0, f=90.0)	0.0 - 150.0°
Exposure Rate	7.5 sec./°
Detector Swing Angle	42.00°
w oscillation Range (c=45.0, f=270.0)	0.0 - 100.0°
Exposure Rate	7.5 sec./°
Detector Swing Angle	42.00°
w oscillation Range (c=15.0, f=180.0)	10.0 - 40.0°
Exposure Rate	7.5 sec./°

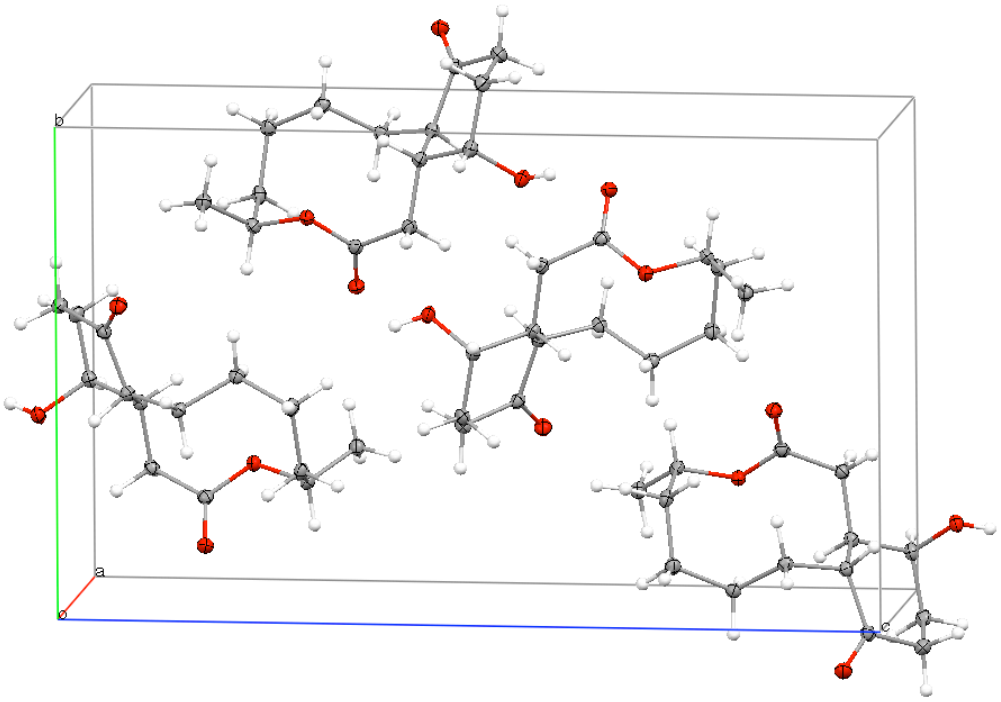
Detector Swing Angle	42.00°
w oscillation Range (c=30.0, f=45.0)	40.0 - 70.0°
Exposure Rate	7.5 sec./°
Detector Swing Angle	42.00°
Detector Position	50.00 mm
Pixel Size	0.090 mm
$2q_{max}$	131.8°
No. of Reflections Measured	Total: 5898 Unique: 2194 (R_{int} = 0.0376) Friedel pairs: 872
Corrections	Lorentz-polarization Absorption (trans. factors: 0.697 - 0.948)

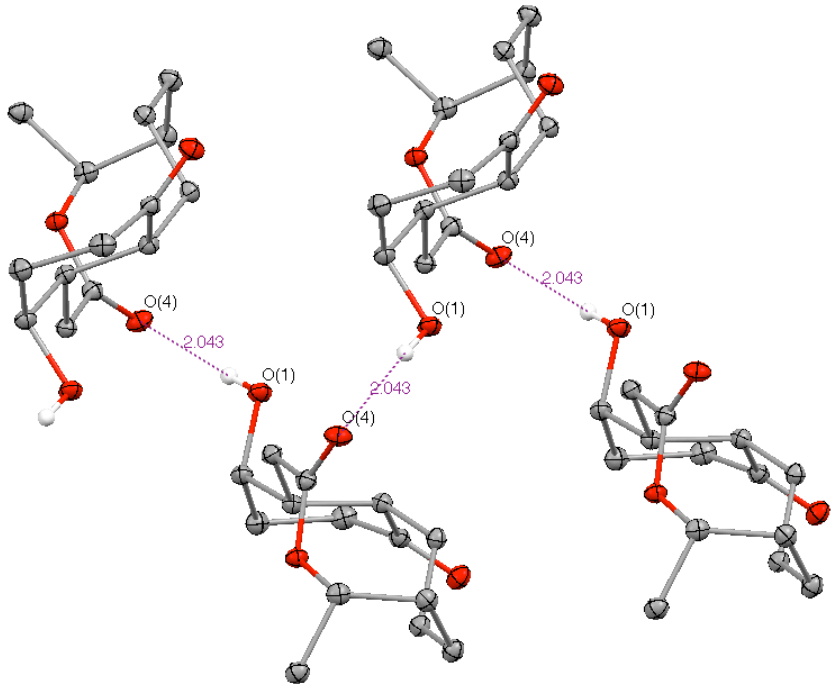
C. Structure Solution and Refinement

Structure Solution	Direct Methods
Refinement	Full-matrix least-squares on F^2
Function Minimized	$S\ w\ (Fo^2 - Fc^2)^2$
Least Squares Weights	$w = 1/ [\ s^2(Fo^2) + (0.0388 \cdot P)^2 + 0.2996 \cdot P\ ]$ where $P = (Max(Fo^2,0) + 2Fc^2)/3$
$2q_{max}$ cutoff	131.8°
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	2194
No. Variables	167

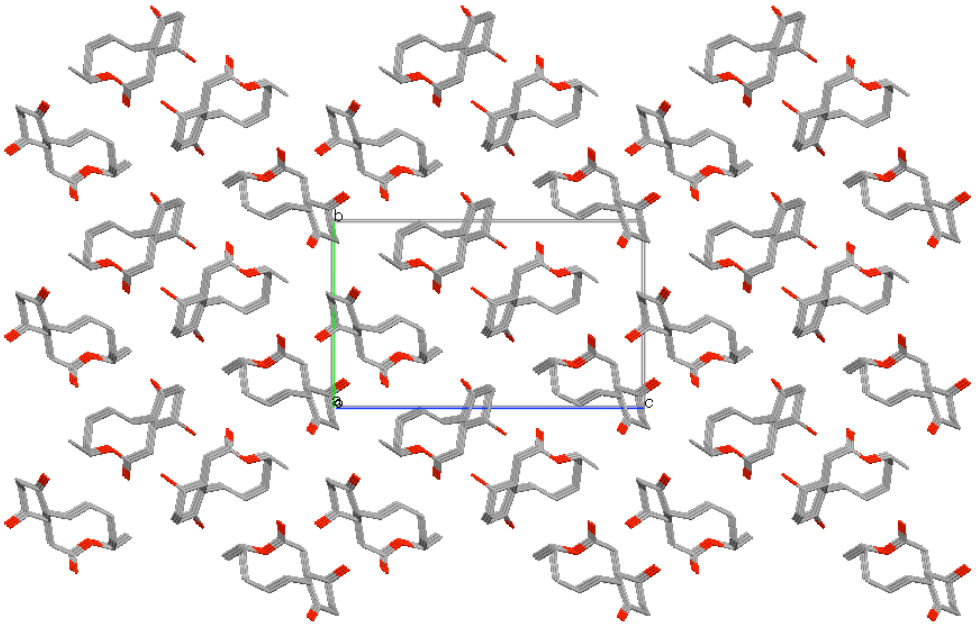
Reflection/Parameter Ratio	13.14
Residuals: R1 (I>2.00s(I))	0.0336
Residuals: R (All reflections)	0.0353
Residuals: wR2 (All reflections)	0.0832
Goodness of Fit Indicator	1.055
Flack Parameter	0.22(20)
Max Shift/Error in Final Cycle	0.001
Maximum peak in Final Diff. Map	0.14 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.17 $e^-/Å^3$



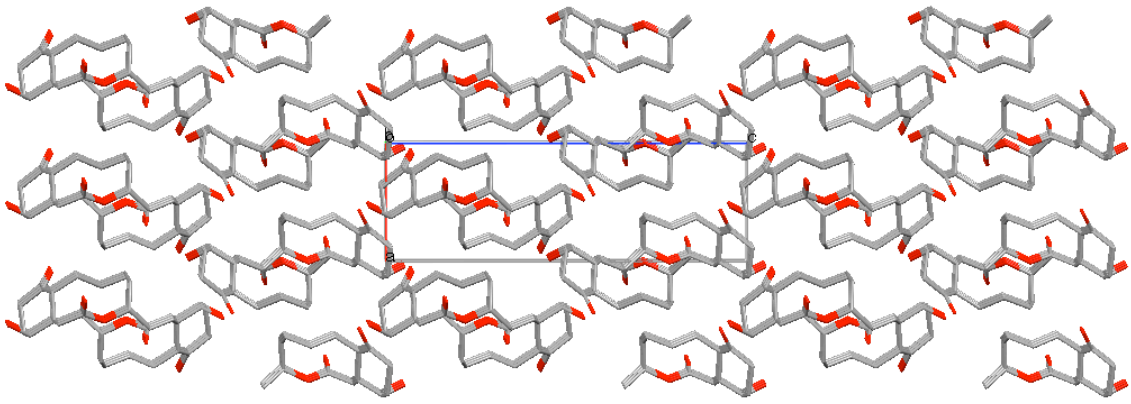

b
a
c


O(4)
2.043
O(1)
O(1)
2.043
O(4)
O(4)
2.043
O(1)

Packing diagram along the crystallographic *a*-axis



Packing diagram along the crystallographic *b*-axis



Packing diagram along the crystallographic *c*-axis

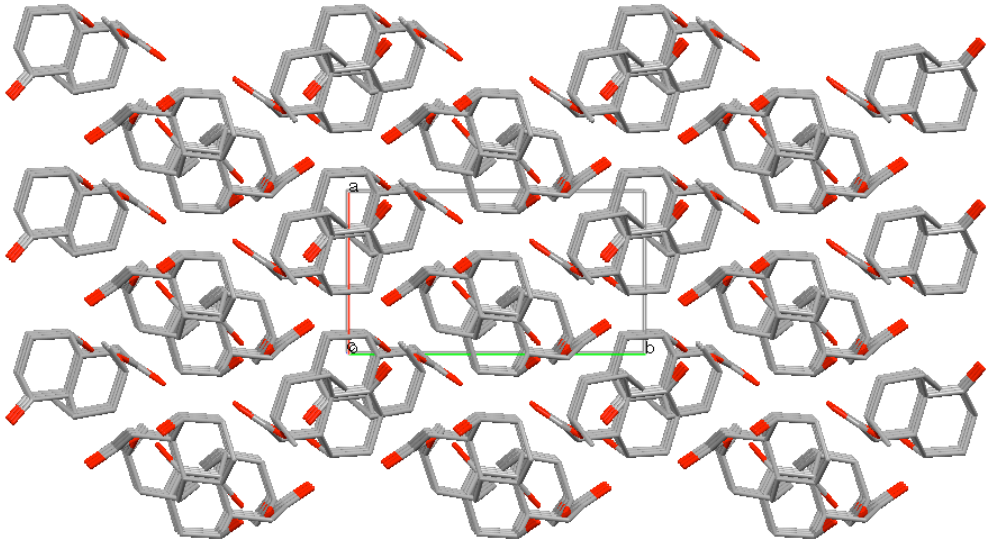


Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
O(1)	1.0271(2)	0.13254(11)	1.04621(6)	2.02(2)
O(2)	0.5816(2)	-0.13137(10)	0.92788(6)	2.23(2)
O(3)	0.99509(19)	0.22063(10)	0.78303(5)	1.75(2)
O(4)	0.8218(2)	0.37404(11)	0.83481(6)	2.18(2)
C(1)	1.1147(3)	0.06541(15)	0.98772(8)	1.82(3)
C(2)	1.1000(3)	-0.06708(15)	1.00199(9)	2.06(3)
C(3)	0.8654(3)	-0.10543(16)	1.01197(9)	2.13(3)
C(4)	0.7214(3)	-0.06653(15)	0.95177(8)	1.75(3)
C(5)	0.7479(3)	0.06075(14)	0.92483(8)	1.67(3)
C(6)	0.6153(3)	0.08082(15)	0.85680(8)	1.71(3)
C(7)	0.7007(3)	0.01775(14)	0.78973(8)	1.77(3)
C(8)	0.6189(3)	0.07247(16)	0.71994(9)	2.02(3)
C(9)	0.6620(3)	0.20567(15)	0.71025(9)	1.92(3)
C(10)	0.8962(3)	0.24754(14)	0.71310(9)	1.81(3)
C(11)	1.0442(3)	0.18842(16)	0.65941(9)	2.10(3)
C(12)	0.9319(3)	0.28469(15)	0.83907(8)	1.77(3)
C(13)	1.0164(3)	0.23445(14)	0.90822(8)	1.76(3)
C(14)	0.9891(3)	0.09992(13)	0.92040(8)	1.63(3)

$B_{eq} = 8/3\ p^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos g + 2U_{13}(aa^*cc^*)\cos b + 2U_{23}(bb^*cc^*)\cos a)$

Table 2. Atomic coordinates and B_{iso} involving hydrogen atoms

atom	x	y	z	B_{iso}
H(1)	1.2706	0.0875	0.9812	2.18
H(2A)	1.1841	-0.0867	1.0454	2.47
H(2B)	1.1644	-0.1111	0.9615	2.47
H(3A)	0.8597	-0.1930	1.0159	2.56
H(3B)	0.8097	-0.0717	1.0571	2.56
H(5)	0.6801	0.1124	0.9620	2.00
H(6A)	0.6100	0.1671	0.8471	2.06
H(6B)	0.4648	0.0539	0.8656	2.06
H(7A)	0.8612	0.0203	0.7901	2.12
H(7B)	0.6562	-0.0666	0.7914	2.12
H(8A)	0.4604	0.0590	0.7168	2.43
H(8B)	0.6872	0.0296	0.6798	2.43
H(9A)	0.6009	0.2298	0.6636	2.30
H(9B)	0.5801	0.2487	0.7475	2.30
H(10)	0.8999	0.3353	0.7053	2.17
H(11A)	1.0513	0.1031	0.6693	2.52
H(11B)	1.1899	0.2227	0.6630	2.52
H(11C)	0.9872	0.2011	0.6112	2.52
H(13A)	0.9430	0.2764	0.9478	2.12
H(13B)	1.1729	0.2533	0.9115	2.12
H(14)	1.0569	0.0578	0.8791	1.96
H(1A)	1.104(4)	0.116(2)	1.0833(14)	4.6(6)

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	0.0291(7)	0.0287(6)	0.0190(6)	0.0026(6)	-0.0016(5)	-0.0040(5)
O(2)	0.0306(7)	0.0271(6)	0.0271(6)	-0.0066(5)	-0.0008(5)	0.0008(5)
O(3)	0.0258(6)	0.0218(5)	0.0188(5)	0.0007(5)	-0.0008(5)	-0.0003(4)
O(4)	0.0371(7)	0.0213(6)	0.0245(6)	0.0036(6)	0.0033(5)	0.0001(5)
C(1)	0.0231(8)	0.0270(9)	0.0192(8)	0.0008(7)	0.0004(7)	-0.0026(7)
C(2)	0.0290(9)	0.0258(9)	0.0234(8)	0.0045(8)	-0.0047(8)	0.0012(7)
C(3)	0.0352(10)	0.0229(9)	0.0228(8)	-0.0006(8)	-0.0009(8)	0.0034(7)
C(4)	0.0235(9)	0.0235(9)	0.0195(8)	-0.0008(8)	0.0030(7)	-0.0022(6)
C(5)	0.0215(8)	0.0210(8)	0.0209(8)	0.0003(7)	0.0009(7)	-0.0003(7)
C(6)	0.0207(8)	0.0225(8)	0.0219(8)	-0.0013(7)	-0.0002(7)	0.0014(7)
C(7)	0.0221(8)	0.0225(8)	0.0224(8)	-0.0017(7)	-0.0006(7)	0.0003(7)
C(8)	0.0264(9)	0.0285(9)	0.0220(8)	-0.0037(8)	-0.0013(7)	0.0006(7)
C(9)	0.0252(9)	0.0283(9)	0.0194(8)	0.0031(7)	-0.0014(7)	0.0033(7)
C(10)	0.0286(9)	0.0211(9)	0.0191(8)	0.0019(7)	-0.0017(7)	0.0035(6)
C(11)	0.0303(10)	0.0279(9)	0.0215(8)	-0.0018(7)	0.0004(7)	-0.0004(7)
C(12)	0.0238(9)	0.0198(8)	0.0237(8)	-0.0029(7)	0.0029(7)	-0.0010(7)
C(13)	0.0235(9)	0.0222(8)	0.0214(8)	-0.0021(7)	0.0001(7)	-0.0020(6)
C(14)	0.0220(8)	0.0211(8)	0.0190(7)	0.0005(7)	0.0008(7)	-0.0015(6)

The general temperature factor expression: $\exp(-2p^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
O(1)	C(1)	1.4384(20)	O(2)	C(4)	1.215(2)
O(3)	C(10)	1.4784(20)	O(3)	C(12)	1.3338(19)
O(4)	C(12)	1.217(2)	C(1)	C(2)	1.520(2)
C(1)	C(14)	1.532(2)	C(2)	C(3)	1.521(3)
C(3)	C(4)	1.502(2)	C(4)	C(5)	1.530(2)
C(5)	C(6)	1.532(2)	C(5)	C(14)	1.553(2)
C(6)	C(7)	1.538(2)	C(7)	C(8)	1.533(2)
C(8)	C(9)	1.536(2)	C(9)	C(10)	1.519(2)
C(10)	C(11)	1.514(2)	C(12)	C(13)	1.508(2)
C(13)	C(14)	1.543(2)			

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
O(1)	H(1A)	0.86(3)	C(1)	H(1)	1.000
C(2)	H(2A)	0.990	C(2)	H(2B)	0.990
C(3)	H(3A)	0.990	C(3)	H(3B)	0.990
C(5)	H(5)	1.000	C(6)	H(6A)	0.990
C(6)	H(6B)	0.990	C(7)	H(7A)	0.990
C(7)	H(7B)	0.990	C(8)	H(8A)	0.990
C(8)	H(8B)	0.990	C(9)	H(9A)	0.990
C(9)	H(9B)	0.990	C(10)	H(10)	1.000
C(11)	H(11A)	0.980	C(11)	H(11B)	0.980
C(11)	H(11C)	0.980	C(13)	H(13A)	0.990
C(13)	H(13B)	0.990	C(14)	H(14)	1.000

Table 6. Bond angles (º)

atom	atom	atom	angle	atom	atom	atom	angle
C(10)	O(3)	C(12)	117.92(12)	O(1)	C(1)	C(2)	111.11(13)
O(1)	C(1)	C(14)	107.81(13)	C(2)	C(1)	C(14)	111.39(13)
C(1)	C(2)	C(3)	110.95(15)	C(2)	C(3)	C(4)	112.73(14)
O(2)	C(4)	C(3)	121.38(15)	O(2)	C(4)	C(5)	121.19(14)
C(3)	C(4)	C(5)	117.32(14)	C(4)	C(5)	C(6)	110.89(13)
C(4)	C(5)	C(14)	112.75(13)	C(6)	C(5)	C(14)	115.08(13)
C(5)	C(6)	C(7)	115.53(14)	C(6)	C(7)	C(8)	113.61(14)
C(7)	C(8)	C(9)	115.95(14)	C(8)	C(9)	C(10)	117.66(14)
O(3)	C(10)	C(9)	111.05(13)	O(3)	C(10)	C(11)	104.58(13)
C(9)	C(10)	C(11)	114.35(14)	O(3)	C(12)	O(4)	123.99(14)
O(3)	C(12)	C(13)	111.97(14)	O(4)	C(12)	C(13)	124.03(14)
C(12)	C(13)	C(14)	117.31(13)	C(1)	C(14)	C(5)	111.56(13)
C(1)	C(14)	C(13)	108.46(13)	C(5)	C(14)	C(13)	113.07(13)

Table 7. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
C(1)	O(1)	H(1A)	107.1(18)	O(1)	C(1)	H(1)	109
C(2)	C(1)	H(1)	109	C(14)	C(1)	H(1)	109
C(1)	C(2)	H(2A)	109	C(1)	C(2)	H(2B)	109
C(3)	C(2)	H(2A)	109	C(3)	C(2)	H(2B)	109
H(2A)	C(2)	H(2B)	108	C(2)	C(3)	H(3A)	109
C(2)	C(3)	H(3B)	109	C(4)	C(3)	H(3A)	109
C(4)	C(3)	H(3B)	109	H(3A)	C(3)	H(3B)	108
C(4)	C(5)	H(5)	106	C(6)	C(5)	H(5)	106
C(14)	C(5)	H(5)	106	C(5)	C(6)	H(6A)	108
C(5)	C(6)	H(6B)	108	C(7)	C(6)	H(6A)	108
C(7)	C(6)	H(6B)	108	H(6A)	C(6)	H(6B)	107
C(6)	C(7)	H(7A)	109	C(6)	C(7)	H(7B)	109
C(8)	C(7)	H(7A)	109	C(8)	C(7)	H(7B)	109
H(7A)	C(7)	H(7B)	108	C(7)	C(8)	H(8A)	108
C(7)	C(8)	H(8B)	108	C(9)	C(8)	H(8A)	108
C(9)	C(8)	H(8B)	108	H(8A)	C(8)	H(8B)	107
C(8)	C(9)	H(9A)	108	C(8)	C(9)	H(9B)	108
C(10)	C(9)	H(9A)	108	C(10)	C(9)	H(9B)	108
H(9A)	C(9)	H(9B)	107	O(3)	C(10)	H(10)	109
C(9)	C(10)	H(10)	109	C(11)	C(10)	H(10)	109
C(10)	C(11)	H(11A)	109	C(10)	C(11)	H(11B)	109
C(10)	C(11)	H(11C)	109	H(11A)	C(11)	H(11B)	109
H(11A)	C(11)	H(11C)	109	H(11B)	C(11)	H(11C)	109
C(12)	C(13)	H(13A)	108	C(12)	C(13)	H(13B)	108
C(14)	C(13)	H(13A)	108	C(14)	C(13)	H(13B)	108
H(13A)	C(13)	H(13B)	107	C(1)	C(14)	H(14)	108
C(5)	C(14)	H(14)	108	C(13)	C(14)	H(14)	108

Table 8. Torsion Angles(°)
(Those having bond angles > 160 degrees are excluded.)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C(10)	O(3)	C(12)	O(4)	-10.7(2)	C(12)	O(3)	C(10)	C(9)	-72.38(16)
O(1)	C(1)	C(2)	C(3)	60.41(16)	O(1)	C(1)	C(14)	C(5)	-65.80(15)
O(1)	C(1)	C(14)	C(13)	59.38(15)	C(2)	C(1)	C(14)	C(5)	56.35(16)
C(14)	C(1)	C(2)	C(3)	-59.81(16)	C(1)	C(2)	C(3)	C(4)	52.67(17)
C(2)	C(3)	C(4)	O(2)	139.43(15)	C(2)	C(3)	C(4)	C(5)	-44.32(19)
O(2)	C(4)	C(5)	C(6)	-12.2(2)	O(2)	C(4)	C(5)	C(14)	-142.92(14)
C(3)	C(4)	C(5)	C(14)	40.82(18)	C(4)	C(5)	C(6)	C(7)	-70.58(16)
C(4)	C(5)	C(14)	C(1)	-45.73(15)	C(6)	C(5)	C(14)	C(13)	63.14(16)
C(14)	C(5)	C(6)	C(7)	58.89(17)	C(5)	C(6)	C(7)	C(8)	-159.37(12)
C(6)	C(7)	C(8)	C(9)	55.12(19)	C(7)	C(8)	C(9)	C(10)	58.12(19)
C(8)	C(9)	C(10)	O(3)	-60.98(18)	C(8)	C(9)	C(10)	C(11)	57.07(18)
O(3)	C(12)	C(13)	C(14)	-47.64(19)	O(4)	C(12)	C(13)	C(14)	133.36(16)
C(12)	C(13)	C(14)	C(5)	-64.08(17)					

Table 9. Possible hydrogen bonds

Donor	H	Acceptor	D...A	D-H	H...A	D-H...A
O(1)	H(1A)	O(4)[1]	2.8791(17)	0.86	2.04(3)	163(2)

Symmetry Operators:

(1) X+1/2,-Y+1/2,-Z+2

Table 10. Intramolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
O(1)	C(3)	2.933(2)	O(1)	C(4)	3.424(2)
O(1)	C(5)	2.9670(20)	O(1)	C(13)	2.8335(19)
O(2)	C(2)	3.559(2)	O(2)	C(6)	2.7466(20)
O(2)	C(7)	3.1755(19)	O(3)	C(5)	3.5565(19)
O(3)	C(6)	3.1430(20)	O(3)	C(7)	2.9223(20)
O(3)	C(8)	3.093(2)	O(3)	C(14)	2.9150(18)
O(4)	C(6)	3.566(2)	O(4)	C(9)	3.168(2)
O(4)	C(10)	2.731(2)	C(1)	C(4)	2.923(2)
C(2)	C(5)	2.980(2)	C(3)	C(14)	2.982(2)
C(4)	C(7)	3.188(2)	C(5)	C(12)	3.201(2)
C(6)	C(9)	3.103(2)	C(6)	C(12)	3.033(2)
C(6)	C(13)	3.169(2)	C(7)	C(10)	3.198(2)
C(7)	C(12)	3.456(2)	C(7)	C(14)	3.167(2)

C(8)	C(11)	3.142(3)	C(9)	C(12)	3.066(2)

Table 11. Intramolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
O(1)	H(2A)	2.654	O(1)	H(2B)	3.284
O(1)	H(3B)	2.672	O(1)	H(5)	2.669
O(1)	H(13A)	2.511	O(1)	H(13B)	3.009
O(1)	H(14)	3.251	O(2)	H(3A)	2.480
O(2)	H(3B)	2.882	O(2)	H(5)	2.887
O(2)	H(6B)	2.500	O(2)	H(7A)	3.546
O(2)	H(7B)	2.702	O(3)	H(6A)	2.728
O(3)	H(7A)	2.408	O(3)	H(8B)	3.463
O(3)	H(9A)	3.306	O(3)	H(9B)	2.662
O(3)	H(11A)	2.536	O(3)	H(11B)	2.553
O(3)	H(11C)	3.231	O(3)	H(13A)	3.172
O(3)	H(13B)	2.673	O(3)	H(14)	2.601
O(4)	H(6A)	2.683	O(4)	H(9B)	2.626
O(4)	H(10)	2.516	O(4)	H(13A)	2.503
O(4)	H(13B)	2.933	C(1)	H(3A)	3.352
C(1)	H(3B)	2.760	C(1)	H(5)	2.773
C(1)	H(13A)	2.709	C(1)	H(13B)	2.581
C(2)	H(5)	3.370	C(2)	H(14)	2.714
C(2)	H(1A)	2.56(3)	C(3)	H(1)	3.361
C(3)	H(5)	2.867	C(3)	H(14)	3.315
C(3)	H(1A)	3.19(3)	C(4)	H(2A)	3.357
C(4)	H(2B)	2.782	C(4)	H(6A)	3.356
C(4)	H(6B)	2.638	C(4)	H(7A)	3.302
C(4)	H(7B)	3.035	C(4)	H(14)	2.846
C(5)	H(1)	3.404	C(5)	H(2B)	3.288
C(5)	H(3A)	3.402	C(5)	H(3B)	2.922
C(5)	H(7A)	2.662	C(5)	H(7B)	2.940
C(5)	H(13A)	2.746	C(5)	H(13B)	3.411
C(6)	H(8A)	2.806	C(6)	H(8B)	3.401
C(6)	H(9B)	2.799	C(6)	H(13A)	3.443
C(6)	H(14)	2.766	C(7)	H(5)	3.406
C(7)	H(9A)	3.419	C(7)	H(9B)	2.821
C(7)	H(11A)	3.272	C(7)	H(14)	2.800
C(8)	H(6A)	2.615	C(8)	H(6B)	2.901
C(8)	H(10)	3.443	C(8)	H(11A)	2.851
C(8)	H(11C)	3.379	C(9)	H(6A)	2.624
C(9)	H(6B)	3.592	C(9)	H(7A)	2.850

Table 11. Intramolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C(9)	H(7B)	3.427	C(9)	H(11A)	2.772
C(9)	H(11B)	3.378	C(9)	H(11C)	2.733
C(10)	H(6A)	3.203	C(10)	H(7A)	2.949
C(10)	H(8A)	3.426	C(10)	H(8B)	2.844
C(11)	H(7A)	3.298	C(11)	H(8B)	2.863
C(11)	H(9A)	2.773	C(11)	H(9B)	3.373
C(12)	H(5)	3.391	C(12)	H(6A)	2.391
C(12)	H(7A)	3.150	C(12)	H(9B)	2.796
C(12)	H(10)	2.582	C(12)	H(14)	2.775
C(13)	H(1)	2.661	C(13)	H(5)	2.684
C(13)	H(6A)	2.857	C(13)	H(7A)	3.414
C(13)	H(1A)	3.59(3)	C(14)	H(2A)	3.372
C(14)	H(2B)	2.724	C(14)	H(3B)	3.398
C(14)	H(6A)	2.815	C(14)	H(6B)	3.430
C(14)	H(7A)	2.721	C(14)	H(1A)	3.14(3)
H(1)	H(2A)	2.363	H(1)	H(2B)	2.361
H(1)	H(13A)	3.001	H(1)	H(13B)	2.361
H(1)	H(14)	2.349	H(1)	H(1A)	2.196
H(2A)	H(3A)	2.395	H(2A)	H(3B)	2.324
H(2A)	H(1A)	2.441	H(2B)	H(3A)	2.328
H(2B)	H(3B)	2.863	H(2B)	H(14)	2.540
H(2B)	H(1A)	3.450	H(3B)	H(5)	2.853
H(3B)	H(1A)	2.827	H(5)	H(6A)	2.283
H(5)	H(6B)	2.338	H(5)	H(7A)	3.568
H(5)	H(13A)	2.472	H(5)	H(13B)	3.556
H(5)	H(14)	2.861	H(5)	H(1A)	3.464
H(6A)	H(7A)	2.507	H(6A)	H(7B)	2.848
H(6A)	H(8A)	2.884	H(6A)	H(8B)	3.535
H(6A)	H(9A)	3.515	H(6A)	H(9B)	2.091
H(6A)	H(13A)	3.050	H(6A)	H(14)	3.077
H(6B)	H(7A)	2.850	H(6B)	H(7B)	2.275
H(6B)	H(8A)	2.793	H(6B)	H(9B)	3.199
H(7A)	H(8A)	2.861	H(7A)	H(8B)	2.334
H(7A)	H(9B)	3.205	H(7A)	H(11A)	2.718
H(7A)	H(14)	2.104	H(7B)	H(8A)	2.329
H(7B)	H(8B)	2.367	H(7B)	H(14)	3.282
H(8A)	H(9A)	2.335	H(8A)	H(9B)	2.335

Table 11. Intramolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(8B)	H(9A)	2.339	H(8B)	H(9B)	2.856
H(8B)	H(11A)	2.401	H(8B)	H(11C)	2.969
H(9A)	H(10)	2.328	H(9A)	H(11A)	3.123
H(9A)	H(11C)	2.596	H(9B)	H(10)	2.338
H(10)	H(11A)	2.859	H(10)	H(11B)	2.331
H(10)	H(11C)	2.385	H(13A)	H(14)	2.868
H(13A)	H(1A)	3.274	H(13B)	H(14)	2.396

Table 12. Intermolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
O(1)	O(4)[1]	2.8791(17)	O(1)	C(12)[1]	3.425(2)
O(1)	C(13)[2]	3.590(2)	O(1)	C(13)[1]	3.475(2)
O(2)	C(2)[3]	3.357(2)	O(2)	C(3)[4]	3.443(2)
O(2)	C(9)[5]	3.514(2)	O(2)	C(11)[6]	3.482(2)
O(4)	O(1)[2]	2.8791(17)	C(2)	O(2)[7]	3.357(2)
C(3)	O(2)[8]	3.443(2)	C(9)	O(2)[9]	3.514(2)
C(11)	O(2)[10]	3.482(2)	C(12)	O(1)[2]	3.425(2)
C(13)	O(1)[2]	3.475(2)	C(13)	O(1)[1]	3.590(2)

Symmetry Operators:

(1) X+1/2,-Y+1/2,-Z+2
(2) X+1/2-1,-Y+1/2,-Z+2
(3) X-1,Y,Z
(4) X+1/2-1,-Y+1/2-1,-Z+2
(5) -X+1,Y+1/2-1,-Z+1/2+1
(6) -X+2,Y+1/2-1,-Z+1/2+1
(7) X+1,Y,Z
(8) X+1/2,-Y+1/2-1,-Z+2
(9) -X+1,Y+1/2,-Z+1/2+1
(10) -X+2,Y+1/2,-Z+1/2+1

Table 13. Intermolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
O(1)	H(1)[1]	3.568	O(1)	H(5)[2]	3.029
O(1)	H(6A)[2]	3.061	O(1)	H(8B)[3]	3.371
O(1)	H(13A)[2]	2.763	O(1)	H(13B)[1]	2.655
O(2)	H(1)[4]	3.281	O(2)	H(2A)[4]	3.334
O(2)	H(2A)[5]	3.279	O(2)	H(2B)[4]	2.658
O(2)	H(3A)[5]	2.628	O(2)	H(9A)[6]	2.582
O(2)	H(11B)[7]	2.756	O(2)	H(11C)[7]	3.341
O(2)	H(11C)[3]	3.555	O(3)	H(7B)[8]	3.511

O(4)	H(1)[1]	3.493	O(4)	H(2A)[1]	3.394
O(4)	H(7A)[8]	3.468	O(4)	H(8A)[9]	2.883
O(4)	H(8B)[8]	3.508	O(4)	H(11A)[8]	2.700
O(4)	H(1A)[1]	2.04(3)	C(1)	H(5)[10]	3.558
C(1)	H(6B)[10]	3.150	C(1)	H(13A)[2]	2.956
C(2)	H(3A)[11]	3.161	C(2)	H(11C)[7]	3.411
C(2)	H(11C)[12]	3.600	C(3)	H(2B)[5]	3.464
C(3)	H(8B)[3]	3.279	C(3)	H(9A)[3]	3.179
C(3)	H(11C)[7]	3.305	C(3)	H(11C)[3]	3.059
C(4)	H(1)[4]	3.322	C(4)	H(2B)[4]	3.474
C(4)	H(3A)[5]	3.562	C(4)	H(11B)[7]	3.252
C(4)	H(11C)[7]	3.389	C(4)	H(11C)[3]	3.593
C(5)	H(1)[4]	3.141	C(5)	H(13A)[1]	3.551
C(6)	H(1)[4]	3.158	C(6)	H(13B)[4]	3.503
C(6)	H(14)[4]	3.477	C(7)	H(9B)[6]	3.561
C(7)	H(10)[7]	3.210	C(7)	H(11B)[7]	3.508
C(8)	H(3B)[13]	3.087	C(8)	H(11B)[4]	3.317
C(9)	H(3B)[13]	3.251	C(9)	H(7B)[9]	3.231
C(9)	H(11B)[4]	3.048	C(10)	H(7A)[8]	3.419
C(10)	H(7B)[8]	3.465	C(11)	H(2A)[14]	2.949
C(11)	H(2B)[8]	3.451	C(11)	H(3B)[13]	3.189
C(11)	H(7B)[8]	3.448	C(11)	H(8A)[10]	3.141
C(11)	H(9A)[10]	3.464	C(12)	H(11A)[8]	3.595
C(12)	H(1A)[1]	2.73(3)	C(13)	H(1)[1]	3.260
C(13)	H(5)[2]	3.151	C(13)	H(6B)[10]	3.524
C(13)	H(1A)[1]	3.06(3)	C(14)	H(6B)[10]	3.150
H(1)	O(1)[2]	3.568	H(1)	O(2)[10]	3.281
H(1)	O(4)[2]	3.493	H(1)	C(4)[10]	3.322
H(1)	C(5)[10]	3.141	H(1)	C(6)[10]	3.158
H(1)	C(13)[2]	3.260	H(1)	H(5)[10]	2.565
H(1)	H(5)[2]	3.590	H(1)	H(6A)[10]	3.393
H(1)	H(6B)[10]	2.507	H(1)	H(13A)[2]	2.292
H(2A)	O(2)[10]	3.334	H(2A)	O(2)[11]	3.279
H(2A)	O(4)[2]	3.394	H(2A)	C(11)[12]	2.949
H(2A)	H(3A)[11]	2.944	H(2A)	H(8A)[3]	3.352
H(2A)	H(8B)[3]	3.466	H(2A)	H(9A)[3]	3.258
H(2A)	H(11A)[12]	2.845	H(2A)	H(11B)[12]	2.797
H(2A)	H(11C)[12]	2.701	H(2B)	O(2)[10]	2.658
H(2B)	C(3)[11]	3.464	H(2B)	C(4)[10]	3.474
H(2B)	C(11)[7]	3.451	H(2B)	H(3A)[11]	2.552
H(2B)	H(6B)[10]	3.182	H(2B)	H(9A)[7]	3.290
H(2B)	H(10)[7]	3.212	H(2B)	H(11C)[7]	2.687
H(3A)	O(2)[11]	2.628	H(3A)	C(2)[5]	3.161
H(3A)	C(4)[11]	3.562	H(3A)	H(2A)[5]	2.944
H(3A)	H(2B)[5]	2.552	H(3A)	H(3A)[5]	3.392
H(3A)	H(3A)[11]	3.392	H(3A)	H(8B)[3]	3.595

H(3A)	H(9A)^{3}	2.813	H(3A)	H(11B)^{7}	3.502
H(3A)	H(11C)^{7}	2.830	H(3A)	H(11C)^{3}	2.789
H(3B)	C(8)^{3}	3.087	H(3B)	C(9)^{3}	3.251
H(3B)	C(11)^{3}	3.189	H(3B)	H(8A)^{3}	3.317
H(3B)	H(8B)^{3}	2.350	H(3B)	H(9A)^{3}	2.734
H(3B)	H(11A)^{3}	3.083	H(3B)	H(11C)^{3}	2.551
H(5)	O(1)^{1}	3.029	H(5)	C(1)^{4}	3.558
H(5)	C(13)^{1}	3.151	H(5)	H(1)^{4}	2.565
H(5)	H(1)^{1}	3.590	H(5)	H(13A)^{1}	2.563
H(5)	H(13B)^{1}	2.816	H(5)	H(1A)^{1}	3.214
H(6A)	O(1)^{1}	3.061	H(6A)	H(1)^{4}	3.393
H(6A)	H(13B)^{4}	3.108	H(6A)	H(1A)^{1}	2.775
H(6B)	C(1)^{4}	3.150	H(6B)	C(13)^{4}	3.524
H(6B)	C(14)^{4}	3.150	H(6B)	H(1)^{4}	2.507
H(6B)	H(2B)^{4}	3.182	H(6B)	H(10)^{6}	3.591
H(6B)	H(13B)^{4}	3.005	H(6B)	H(14)^{4}	2.527
H(7A)	O(4)^{7}	3.468	H(7A)	C(10)^{7}	3.419
H(7A)	H(10)^{7}	2.554	H(7A)	H(11B)^{7}	3.482
H(7B)	O(3)^{7}	3.511	H(7B)	C(9)^{6}	3.231
H(7B)	C(10)^{7}	3.465	H(7B)	C(11)^{7}	3.448
H(7B)	H(9A)^{6}	2.914	H(7B)	H(9B)^{6}	2.644
H(7B)	H(10)^{7}	2.952	H(7B)	H(11B)^{7}	2.697
H(8A)	O(4)^{6}	2.883	H(8A)	C(11)^{4}	3.141
H(8A)	H(2A)^{13}	3.352	H(8A)	H(3B)^{13}	3.317
H(8A)	H(9B)^{6}	3.570	H(8A)	H(11A)^{4}	2.720
H(8A)	H(11B)^{4}	2.684	H(8A)	H(1A)^{13}	3.211
H(8B)	O(1)^{13}	3.371	H(8B)	O(4)^{7}	3.508
H(8B)	C(3)^{13}	3.279	H(8B)	H(2A)^{13}	3.466
H(8B)	H(3A)^{13}	3.595	H(8B)	H(3B)^{13}	2.350
H(8B)	H(1A)^{13}	3.030	H(9A)	O(2)^{9}	2.582
H(9A)	C(3)^{13}	3.179	H(9A)	C(11)^{4}	3.464
H(9A)	H(2A)^{13}	3.258	H(9A)	H(2B)^{8}	3.290
H(9A)	H(3A)^{13}	2.813	H(9A)	H(3B)^{13}	2.734
H(9A)	H(7B)^{9}	2.914	H(9A)	H(11B)^{4}	2.534
H(9B)	C(7)^{9}	3.561	H(9B)	H(7B)^{9}	2.644
H(9B)	H(8A)^{9}	3.570	H(9B)	H(11B)^{4}	2.896
H(9B)	H(1A)^{1}	3.526	H(10)	C(7)^{8}	3.210
H(10)	H(2B)^{8}	3.212	H(10)	H(6B)^{9}	3.591
H(10)	H(7A)^{8}	2.554	H(10)	H(7B)^{8}	2.952
H(10)	H(14)^{8}	2.979	H(11A)	O(4)^{7}	2.700
H(11A)	C(12)^{7}	3.595	H(11A)	H(2A)^{14}	2.845
H(11A)	H(3B)^{13}	3.083	H(11A)	H(8A)^{10}	2.720
H(11B)	O(2)^{8}	2.756	H(11B)	C(4)^{8}	3.252
H(11B)	C(7)^{8}	3.508	H(11B)	C(8)^{10}	3.317
H(11B)	C(9)^{10}	3.048	H(11B)	H(2A)^{14}	2.797
H(11B)	H(3A)^{8}	3.502	H(11B)	H(7A)^{8}	3.482

H(11B)	H(7B)[8]	2.697	H(11B)	H(8A)[10]	2.684
H(11B)	H(9A)[10]	2.534	H(11B)	H(9B)[10]	2.896
H(11C)	O(2)[8]	3.341	H(11C)	O(2)[13]	3.555
H(11C)	C(2)[8]	3.411	H(11C)	C(2)[14]	3.600
H(11C)	C(3)[8]	3.305	H(11C)	C(3)[13]	3.059
H(11C)	C(4)[8]	3.389	H(11C)	C(4)[13]	3.593
H(11C)	H(2A)[14]	2.701	H(11C)	H(2B)[8]	2.687
H(11C)	H(3A)[8]	2.830	H(11C)	H(3A)[13]	2.789
H(11C)	H(3B)[13]	2.551	H(13A)	O(1)[1]	2.763
H(13A)	C(1)[1]	2.956	H(13A)	C(5)[2]	3.551
H(13A)	H(1)[1]	2.292	H(13A)	H(5)[2]	2.563
H(13A)	H(13B)[1]	3.139	H(13A)	H(1A)[1]	2.488
H(13B)	O(1)[2]	2.655	H(13B)	C(6)[10]	3.503
H(13B)	H(5)[2]	2.816	H(13B)	H(6A)[10]	3.108
H(13B)	H(6B)[10]	3.005	H(13B)	H(13A)[2]	3.139
H(13B)	H(1A)[2]	3.040	H(14)	C(6)[10]	3.477
H(14)	H(6B)[10]	2.527	H(14)	H(10)[7]	2.979
H(1A)	O(4)[2]	2.04(3)	H(1A)	C(12)[2]	2.73(3)
H(1A)	C(13)[2]	3.06(3)	H(1A)	H(5)[2]	3.214
H(1A)	H(6A)[2]	2.775	H(1A)	H(8A)[3]	3.211
H(1A)	H(8B)[3]	3.030	H(1A)	H(9B)[2]	3.526
H(1A)	H(13A)[2]	2.488	H(1A)	H(13B)[1]	3.040

Symmetry Operators:

(1) X+1/2-1,-Y+1/2,-Z+2
(2) X+1/2,-Y+1/2,-Z+2
(3) -X+1/2+1,-Y,Z+1/2
(4) X-1,Y,Z
(5) X+1/2-1,-Y+1/2-1,-Z+2
(6) -X+1,Y+1/2-1,-Z+1/2+1
(7) -X+2,Y+1/2-1,-Z+1/2+1
(8) -X+2,Y+1/2,-Z+1/2+1
(9) -X+1,Y+1/2,-Z+1/2+1
(10) X+1,Y,Z
(11) X+1/2,-Y+1/2-1,-Z+2
(12) -X+1/2+2,-Y,Z+1/2
(13) -X+1/2+1,-Y,Z+1/2-1
(14) -X+1/2+2,-Y,Z+1/2-1

Sch-642305

June 8, 2010

Experimental

Data Collection

A colorless plate crystal of $O_4C_{14}H_{20}$ having approximate dimensions of 0.35 x 0.22 x 0.10 mm was mounted in a loop. All measurements were made on a Rigaku RAXIS RAPID

imaging plate area detector with graphite monochromated Cu-Ka radiation.

Indexing was performed from 4 oscillations that were exposed for 180 seconds. The crystal-to-detector distance was 127.40 mm.

Cell constants and an orientation matrix for data collection corresponded to a primitive orthorhombic cell with dimensions:

a = 6.14187(12) Å
b = 11.3932(2) Å
c = 18.2633(4) Å
V = 1277.98(4) $Å^3$

For Z = 4 and F.W. = 252.31, the calculated density is 1.311 g/cm^3. The systematic absences of:

h00: h ± 2n
0k0: k ± 2n
00l: l ± 2n

uniquely determine the space group to be:

$P2_12_12_1$ (#19)

The data were collected at a temperature of -180 ± 1°C to a maximum 2q value of 136.4°. A total of 95 oscillation images were collected. A sweep of data was done using w scans from 20.0 to 200.0° in 5.0° step, at c=54.0° and f = 0.0°. The exposure rate was 36.0 [sec./°]. A second sweep was performed using w scans from 20.0 to 155.0° in 5.0° step, at c=0.0° and f = 180.0°. The exposure rate was 36.0 [sec./°]. Another sweep was performed using w scans from 24.0 to 184.0° in 5.0° step, at c=54.0° and f = 180.0°. The exposure rate was 36.0 [sec./°]. The crystal-to-detector distance was 127.40 mm. Readout was performed in the 0.100 mm pixel mode.

Data Reduction

Of the 7709 reflections that were collected, 2228 were unique (R_{int} = 0.043).

The linear absorption coefficient, m, for Cu-Ka radiation is 7.807 cm^{-1}. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.770 to 0.925. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods[1] and expanded using Fourier techniques[2]. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement[3] on F^2 was based on 2219 observed reflections and 168 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = S \, ||Fo| - |Fc|| \, / \, S \, |Fo| = 0.0378$$

$$wR2 = [S (w (Fo^2 - Fc^2)^2)/ S \, w(Fo^2)^2]^{1/2} = 0.0987$$

The standard deviation of an observation of unit weight[4] was 1.09. Unit weights were used. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.27 and -0.24 $e^-/Å^3$, respectively. The absolute structure was deduced based on Flack parameter, -0.2(2), using 857 Friedel pairs.[5]

Neutral atom scattering factors were taken from Cromer and Waber[6]. Anomalous dispersion effects were included in Fcalc[7]; the values for Df' and Df'' were those of Creagh and McAuley[8]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[9]. All calculations were performed using the CrystalStructure[10] crystallographic software package except for refinement, which was performed using SHELXL-97[11].

References

(1) SHELX97: Sheldrick, G.M. (1997).

(2) DIRDIF99: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M.(1999). The DIRDIF-99 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(3) Least Squares function minimized: (SHELXL97)

$$Sw(F_o^2-F_c^2)^2$$ where w = Least Squares weights.

(4) Standard deviation of an observation of unit weight:

$$[Sw(F_o^2-F_c^2)^2/(N_o-N_v)]^{1/2}$$

where N_o = number of observations

N_v = number of variables

(5) Flack, H. D. (1983), Acta Cryst. A39, 876-881.

(6) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(7) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(8) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(9) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(10) CrystalStructure 3.8: Crystal Structure Analysis Package, Rigaku and Rigaku Americas (2000-2007). 9009 New Trails Dr. The Woodlands TX 77381 USA.

(11) SHELX97: Sheldrick, G.M. (1997).

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$O_4C_{14}H_{20}$
Formula Weight	252.31
Crystal Color, Habit	colorless, plate
Crystal Dimensions	0.35 X 0.22 X 0.10 mm
Crystal System	orthorhombic
Lattice Type	Primitive
Indexing Images	4 oscillations @ 180.0 seconds
Detector Position	127.40 mm
Pixel Size	0.100 mm
Lattice Parameters	a = 6.14187(12) Å b = 11.3932(2) Å

	c = 18.2633(4) Å V = 1277.98(4) Å^3
Space Group	$P2_12_12_1$ (#19)
Z value	4
D_{calc}	1.311 g/cm^3
F_{000}	544.00
m(CuKa)	7.807 cm^{-1}

B. Intensity Measurements

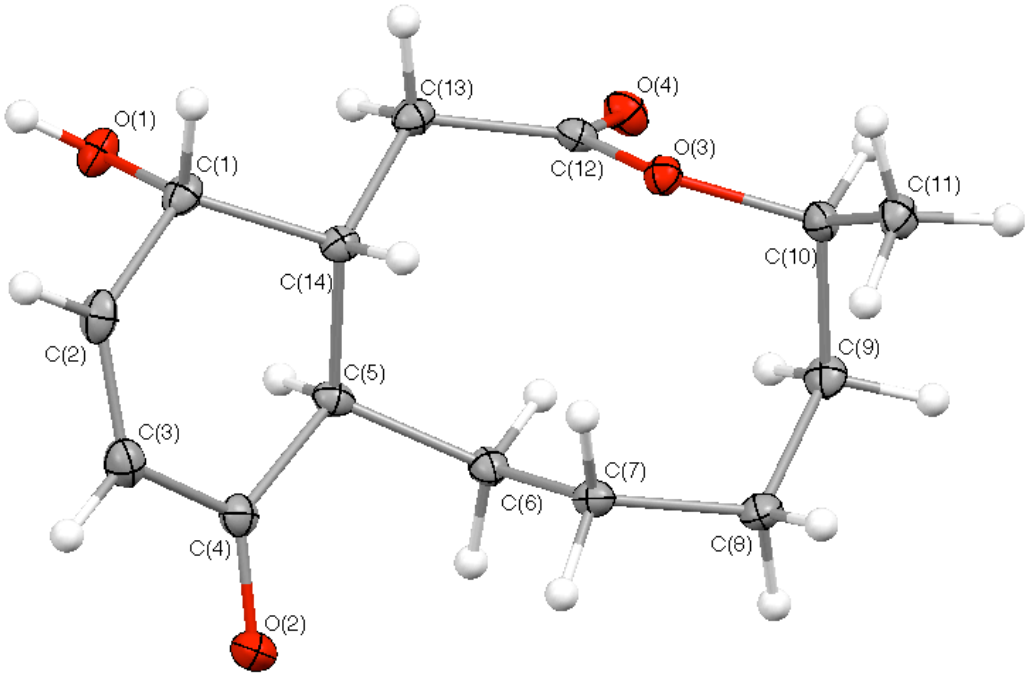
Diffractometer	Rigaku RAXIS-RAPID
Radiation	CuKa (l = 1.54187 Å) graphite monochromated
Detector Aperture	280 mm x 256 mm
Data Images	95 exposures
w oscillation Range (c=54.0, f=0.0)	20.0 - 200.0^o
Exposure Rate	36.0 sec./^{o}
w oscillation Range (c=0.0, f=180.0)	20.0 - 155.0^o
Exposure Rate	36.0 sec./^{o}
w oscillation Range (c=54.0, f=180.0)	24.0 - 184.0^o
Exposure Rate	36.0 sec./^{o}
Detector Position	127.40 mm
Pixel Size	0.100 mm
$2q_{max}$	136.4^o

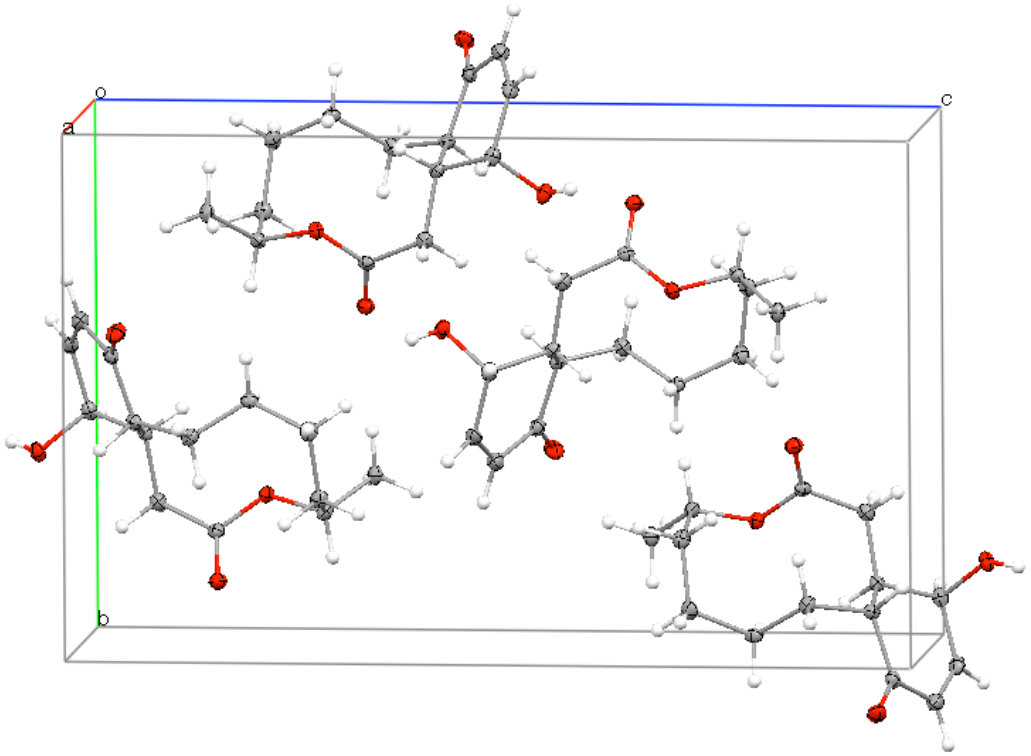
No. of Reflections Measured	Total: 7709 Unique: 2219 (R_{int} = 0.043) Friedel pairs: 857
Corrections	Lorentz-polarization Absorption (trans. factors: 0.770 - 0.925)

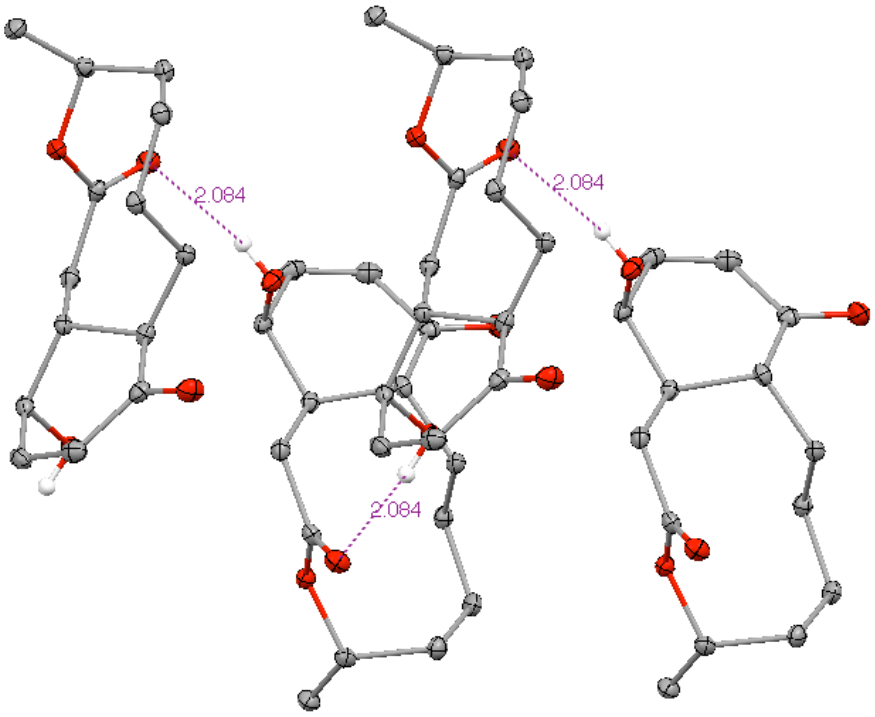
C. Structure Solution and Refinement

Structure Solution	Direct Methods (SHELX97)
Refinement	Full-matrix least-squares on F^2
Function Minimized	$S\ w\ (Fo^2 - Fc^2)^2$
Least Squares Weights	$w = 1/ [\ s^2(Fo^2) + (0.0332 \cdot P)^2 + 0.5629 \cdot P\ ]$ where $P = (Max(Fo^2,0) + 2Fc^2)/3$
$2q_{max}$ cutoff	136.4°
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	2219
No. Variables	168
Reflection/Parameter Ratio	13.21
Residuals: R1 (I>2.00s(I))	0.0378
Residuals: R (All reflections)	0.0499
Residuals: wR2 (All reflections)	0.0987
Goodness of Fit Indicator	1.086
Flack Parameter	-0.2(2)
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	0.27 $e^-/Å^3$

Minimum peak in Final Diff. Map -0.24 $e^{-}/Å^3$



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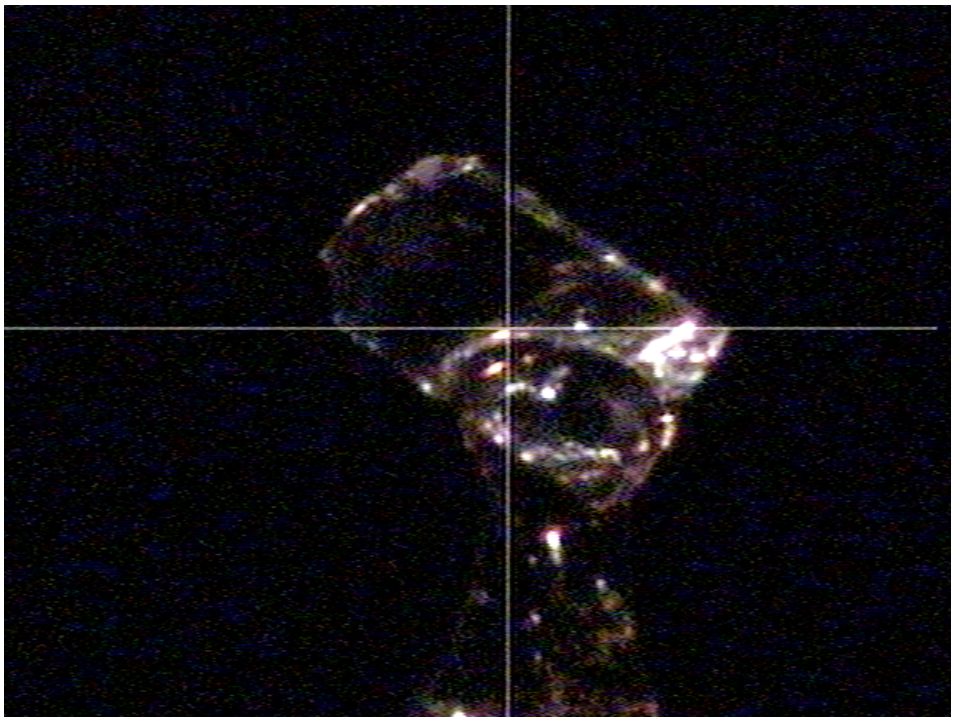
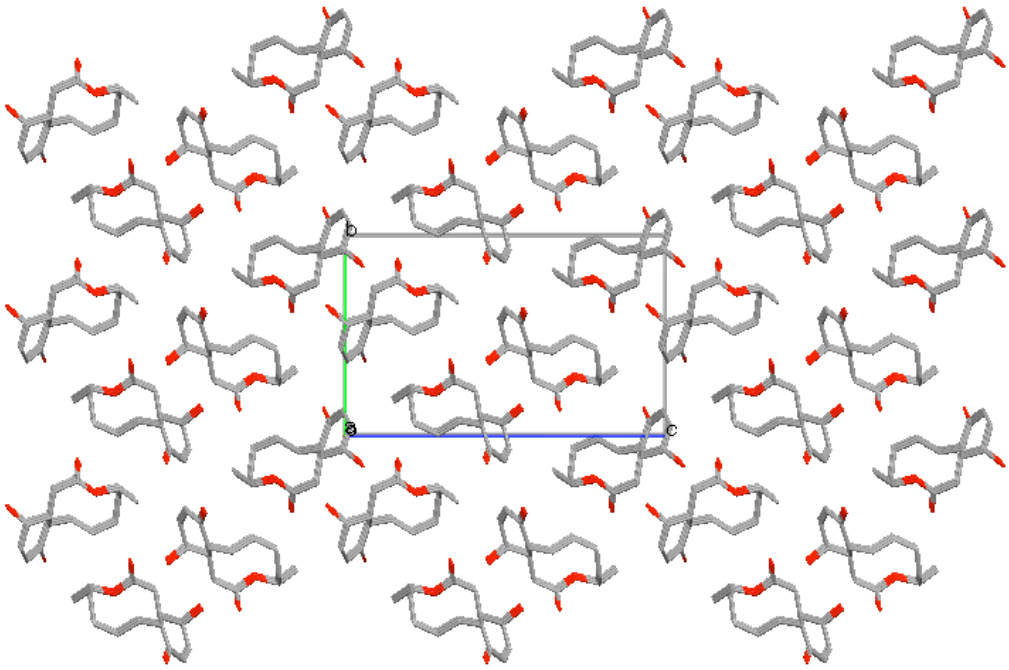
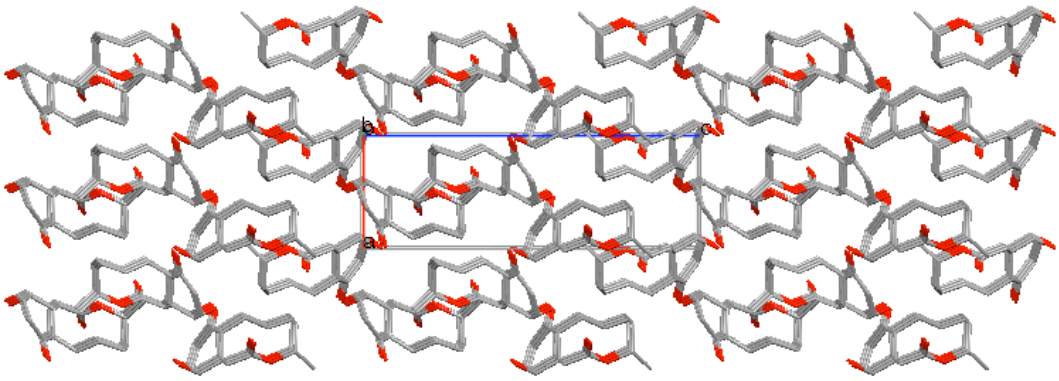


Packing diagram along the crystallographic *a*-axis



Packing diagram along the crystallographic *b*-axis



Packing diagram along the crystallographic *c*-axis

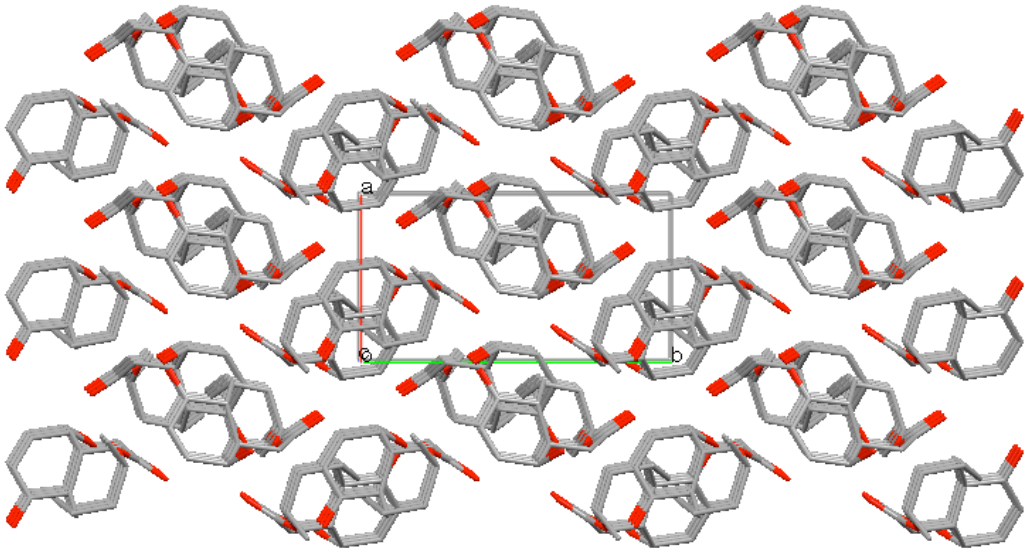


Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
O(1)	0.4866(2)	0.63721(16)	-0.05079(9)	2.04(3)
O(2)	0.9658(2)	0.37861(15)	0.06134(9)	2.46(3)
O(3)	0.5109(2)	0.71260(13)	0.22021(8)	1.63(2)
O(4)	0.6829(2)	0.86625(15)	0.16901(9)	2.09(3)
C(1)	0.4088(3)	0.5628(2)	0.00741(12)	1.75(4)
C(2)	0.4432(4)	0.4366(2)	-0.01290(12)	2.07(4)
C(3)	0.6228(4)	0.3786(2)	0.00563(13)	2.24(4)
C(4)	0.7976(3)	0.4325(2)	0.04875(13)	1.80(4)
C(5)	0.7697(3)	0.5588(2)	0.07461(12)	1.54(4)
C(6)	0.8995(3)	0.5791(2)	0.14542(12)	1.70(4)
C(7)	0.8128(3)	0.5142(2)	0.21271(13)	1.68(4)
C(8)	0.8887(3)	0.5668(2)	0.28562(13)	1.97(4)
C(9)	0.8435(3)	0.6983(2)	0.29578(14)	1.87(4)
C(10)	0.6057(3)	0.7382(2)	0.29266(13)	1.72(4)
C(11)	0.4583(3)	0.6770(2)	0.34678(13)	1.96(4)
C(12)	0.5757(3)	0.7776(2)	0.16312(12)	1.55(3)
C(13)	0.4950(3)	0.7287(2)	0.09153(12)	1.73(4)
C(14)	0.5267(3)	0.59686(19)	0.07758(12)	1.46(3)

$B_{eq} = 8/3\ p^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos g + 2U_{13}(aa^*cc^*)\cos b + 2U_{23}(bb^*cc^*)\cos a)$

Table 2. Atomic coordinates and B_{iso} involving hydrogens/B_{eq}

atom	x	y	z	B_{eq}
H(1)	0.2494	0.5768	0.0142	2.10
H(1A)	0.410(4)	0.623(2)	-0.0845(15)	2.9(6)
H(2)	0.3332	0.3968	-0.0398	2.49
H(3)	0.6385	0.2993	-0.0096	2.69
H(4)	0.8397	0.6092	0.0364	1.85
H(5)	1.0521	0.5545	0.1370	2.04
H(6)	0.9007	0.6643	0.1561	2.04
H(7)	0.8604	0.4313	0.2103	2.02
H(8)	0.6517	0.5151	0.2113	2.02
H(9)	1.0475	0.5536	0.2903	2.37
H(10)	0.8168	0.5235	0.3259	2.37
H(11)	0.9039	0.7223	0.3437	2.24
H(12)	0.9250	0.7415	0.2576	2.24
H(13)	0.5992	0.8247	0.3015	2.07
H(14)	0.5682	0.7720	0.0515	2.07
H(15)	0.3374	0.7460	0.0879	2.07
H(16)	0.4564	0.5535	0.1189	1.76
H(17)	0.4842	0.5922	0.3448	2.36
H(18)	0.3060	0.6934	0.3344	2.36
H(19)	0.4891	0.7058	0.3963	2.36

$B_{eq} = 8/3\ p^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos g + 2U_{13}(aa^*cc^*)\cos b + 2U_{23}(bb^*cc^*)\cos a)$

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	0.0245(8)	0.0330(9)	0.0200(8)	-0.0050(8)	-0.0039(7)	0.0041(7)
O(2)	0.0321(9)	0.0302(9)	0.0313(9)	0.0112(9)	-0.0027(7)	-0.0054(7)
O(3)	0.0186(7)	0.0233(8)	0.0202(8)	-0.0011(7)	-0.0010(6)	-0.0008(6)
O(4)	0.0298(8)	0.0212(8)	0.0283(9)	-0.0012(8)	0.0044(7)	-0.0011(7)
C(1)	0.0162(10)	0.0297(13)	0.0206(12)	-0.0034(10)	-0.0015(9)	0.0027(11)
C(2)	0.0289(12)	0.0283(13)	0.0215(12)	-0.0107(12)	-0.0021(10)	-0.0009(11)
C(3)	0.0347(13)	0.0259(13)	0.0246(13)	-0.0018(12)	0.0009(10)	-0.0027(12)
C(4)	0.0242(11)	0.0254(13)	0.0190(11)	0.0009(11)	0.0042(10)	0.0023(10)
C(5)	0.0177(10)	0.0181(12)	0.0226(12)	0.0008(10)	0.0037(9)	0.0018(10)
C(6)	0.0173(10)	0.0247(12)	0.0225(12)	0.0000(10)	0.0002(9)	-0.0034(10)
C(7)	0.0194(10)	0.0195(12)	0.0250(12)	0.0016(9)	-0.0013(10)	0.0007(10)
C(8)	0.0209(11)	0.0297(13)	0.0243(12)	0.0051(10)	-0.0039(10)	-0.0002(11)

C(9)	0.0181(11)	0.0280(14)	0.0249(13)	-0.0036(10)	-0.0009(9)	-0.0012(11)
C(10)	0.0229(11)	0.0211(12)	0.0215(12)	0.0013(10)	-0.0011(10)	-0.0040(10)
C(11)	0.0235(12)	0.0306(13)	0.0205(12)	-0.0006(11)	0.0036(10)	0.0006(11)
C(12)	0.0174(10)	0.0186(11)	0.0230(12)	0.0026(10)	0.0036(9)	-0.0003(10)
C(13)	0.0178(11)	0.0244(12)	0.0235(11)	0.0008(11)	-0.0002(9)	0.0025(10)
C(14)	0.0160(10)	0.0199(11)	0.0197(11)	-0.0010(9)	0.0006(8)	0.0021(9)

The general temperature factor expression: $\exp(-2p^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
O(1)	C(1)	1.441(2)	O(2)	C(4)	1.224(2)
O(3)	C(10)	1.475(2)	O(3)	C(12)	1.339(2)
O(4)	C(12)	1.210(2)	C(1)	C(2)	1.500(3)
C(1)	C(14)	1.522(3)	C(2)	C(3)	1.330(3)
C(3)	C(4)	1.466(3)	C(4)	C(5)	1.524(3)
C(5)	C(6)	1.537(3)	C(5)	C(14)	1.555(3)
C(6)	C(7)	1.530(3)	C(7)	C(8)	1.532(3)
C(8)	C(9)	1.535(3)	C(9)	C(10)	1.530(3)
C(10)	C(11)	1.511(3)	C(12)	C(13)	1.505(3)
C(13)	C(14)	1.536(3)			

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
O(1)	H(1A)	0.79(2)	C(1)	H(1)	1.000
C(2)	H(2)	0.950	C(3)	H(3)	0.950
C(5)	H(4)	1.000	C(6)	H(5)	0.990
C(6)	H(6)	0.990	C(7)	H(7)	0.990
C(7)	H(8)	0.990	C(8)	H(9)	0.990
C(8)	H(10)	0.990	C(9)	H(11)	0.990
C(9)	H(12)	0.990	C(10)	H(13)	1.000
C(11)	H(17)	0.980	C(11)	H(18)	0.980
C(11)	H(19)	0.980	C(13)	H(14)	0.990
C(13)	H(15)	0.990	C(14)	H(16)	1.000

Table 6. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
C(10)	O(3)	C(12)	118.16(16)	O(1)	C(1)	C(2)	109.58(18)
O(1)	C(1)	C(14)	108.27(18)	C(2)	C(1)	C(14)	112.68(18)
C(1)	C(2)	C(3)	122.0(2)	C(2)	C(3)	C(4)	122.4(2)
O(2)	C(4)	C(3)	120.6(2)	O(2)	C(4)	C(5)	120.7(2)
C(3)	C(4)	C(5)	118.7(2)	C(4)	C(5)	C(6)	110.16(18)
C(4)	C(5)	C(14)	112.45(17)	C(6)	C(5)	C(14)	115.25(17)
C(5)	C(6)	C(7)	115.00(18)	C(6)	C(7)	C(8)	113.79(18)
C(7)	C(8)	C(9)	115.54(19)	C(8)	C(9)	C(10)	117.27(19)
O(3)	C(10)	C(9)	110.58(18)	O(3)	C(10)	C(11)	105.03(17)
C(9)	C(10)	C(11)	114.2(2)	O(3)	C(12)	O(4)	123.6(2)
O(3)	C(12)	C(13)	111.94(18)	O(4)	C(12)	C(13)	124.4(2)
C(12)	C(13)	C(14)	117.65(18)	C(1)	C(14)	C(5)	110.86(17)
C(1)	C(14)	C(13)	109.18(17)	C(5)	C(14)	C(13)	113.59(17)

Table 7. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
C(1)	O(1)	H(1A)	105(2)	O(1)	C(1)	H(1)	108.7
C(2)	C(1)	H(1)	108.7	C(14)	C(1)	H(1)	108.7
C(1)	C(2)	H(2)	119.0	C(3)	C(2)	H(2)	119.0
C(2)	C(3)	H(3)	118.8	C(4)	C(3)	H(3)	118.8
C(4)	C(5)	H(4)	106.1	C(6)	C(5)	H(4)	106.1
C(14)	C(5)	H(4)	106.1	C(5)	C(6)	H(5)	108.5
C(5)	C(6)	H(6)	108.5	C(7)	C(6)	H(5)	108.5
C(7)	C(6)	H(6)	108.5	H(5)	C(6)	H(6)	107.5
C(6)	C(7)	H(7)	108.8	C(6)	C(7)	H(8)	108.8
C(8)	C(7)	H(7)	108.8	C(8)	C(7)	H(8)	108.8
H(7)	C(7)	H(8)	107.7	C(7)	C(8)	H(9)	108.4
C(7)	C(8)	H(10)	108.4	C(9)	C(8)	H(9)	108.4
C(9)	C(8)	H(10)	108.4	H(9)	C(8)	H(10)	107.5
C(8)	C(9)	H(11)	108.0	C(8)	C(9)	H(12)	108.0
C(10)	C(9)	H(11)	108.0	C(10)	C(9)	H(12)	108.0
H(11)	C(9)	H(12)	107.2	O(3)	C(10)	H(13)	108.9
C(9)	C(10)	H(13)	108.9	C(11)	C(10)	H(13)	108.9
C(10)	C(11)	H(17)	109.5	C(10)	C(11)	H(18)	109.5
C(10)	C(11)	H(19)	109.5	H(17)	C(11)	H(18)	109.5
H(17)	C(11)	H(19)	109.5	H(18)	C(11)	H(19)	109.5
C(12)	C(13)	H(14)	107.9	C(12)	C(13)	H(15)	107.9
C(14)	C(13)	H(14)	107.9	C(14)	C(13)	H(15)	107.9
H(14)	C(13)	H(15)	107.2	C(1)	C(14)	H(16)	107.7

C(5)	C(14)	H(16)	107.7		C(13)	C(14)	H(16)	107.7	

Table 8. Torsion Angles(°)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C(10)	O(3)	C(12)	O(4)	-11.0(3)	C(10)	O(3)	C(12)	C(13)	170.36(17)
C(12)	O(3)	C(10)	C(9)	-71.9(2)	C(12)	O(3)	C(10)	C(11)	164.39(18)
O(1)	C(1)	C(2)	C(3)	92.0(2)	O(1)	C(1)	C(14)	C(5)	-70.8(2)
O(1)	C(1)	C(14)	C(13)	55.0(2)	C(2)	C(1)	C(14)	C(5)	50.5(2)
C(2)	C(1)	C(14)	C(13)	176.40(18)	C(14)	C(1)	C(2)	C(3)	-28.6(3)
C(1)	C(2)	C(3)	C(4)	1.6(3)	C(2)	C(3)	C(4)	O(2)	-175.9(2)
C(2)	C(3)	C(4)	C(5)	1.4(3)	O(2)	C(4)	C(5)	C(6)	-30.4(2)
O(2)	C(4)	C(5)	C(14)	-160.4(2)	C(3)	C(4)	C(5)	C(6)	152.4(2)
C(3)	C(4)	C(5)	C(14)	22.3(2)	C(4)	C(5)	C(6)	C(7)	-68.3(2)
C(4)	C(5)	C(14)	C(1)	-47.5(2)	C(4)	C(5)	C(14)	C(13)	-170.82(18)
C(6)	C(5)	C(14)	C(1)	-174.83(18)	C(6)	C(5)	C(14)	C(13)	61.8(2)
C(14)	C(5)	C(6)	C(7)	60.2(2)	C(5)	C(6)	C(7)	C(8)	-159.60(19)
C(6)	C(7)	C(8)	C(9)	54.0(2)	C(7)	C(8)	C(9)	C(10)	59.7(2)
C(8)	C(9)	C(10)	O(3)	-62.0(2)	C(8)	C(9)	C(10)	C(11)	56.2(2)
O(3)	C(12)	C(13)	C(14)	-48.2(2)	O(4)	C(12)	C(13)	C(14)	133.1(2)
C(12)	C(13)	C(14)	C(1)	172.71(18)	C(12)	C(13)	C(14)	C(5)	-63.0(2)

The sign is positive if when looking from atom 2 to atom 3 a clock-wise motion of atom 1 would superimpose it on atom 4.

Table 9. Distances beyond the asymmetric unit out to 3.60 Å

atom	atom	distance	atom	atom	distance
O(1)	O(4)[1)]	2.854(2)	O(1)	C(12)[1)]	3.394(2)
O(1)	C(13)[1)]	3.465(2)	O(1)	C(13)[2)]	3.555(2)
O(2)	C(1)[3)]	3.575(2)	O(2)	C(2)[3)]	3.297(2)
O(2)	C(3)[4)]	3.319(3)	O(2)	C(9)[5)]	3.522(3)
O(4)	O(1)[2)]	2.854(2)	O(4)	C(8)[6)]	3.582(2)
C(1)	O(2)[7)]	3.575(2)	C(2)	O(2)[7)]	3.297(2)
C(3)	O(2)[8)]	3.319(3)	C(3)	C(11)[9)]	3.576(3)
C(8)	O(4)[5)]	3.582(2)	C(9)	O(2)[6)]	3.522(3)
C(11)	C(3)[10)]	3.576(3)	C(12)	O(1)[2)]	3.394(2)
C(13)	O(1)[1)]	3.555(2)	C(13)	O(1)[2)]	3.465(2)

Symmetry Operators:

(1) X+1/2-1,-Y+1/2+1,-Z (2) X+1/2,-Y+1/2+1,-Z
(3) X+1,Y,Z (4) X+1/2,-Y+1/2,-Z

(5) -X+2,Y+1/2-1,-Z+1/2 (6) -X+2,Y+1/2,-Z+1/2
(7) X-1,Y,Z (8) X+1/2-1,-Y+1/2,-Z
(9) -X+1,Y+1/2-1,-Z+1/2 (10) -X+1,Y+1/2,-Z+1/2

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
O(1)	H(4)[1)]	3.039	O(1)	H(6)[1)]	3.015
O(1)	H(10)[2)]	3.143	O(1)	H(14)[1)]	2.770
O(1)	H(15)[3)]	2.621	O(2)	H(1)[4)]	2.979
O(2)	H(2)[4)]	2.924	O(2)	H(2)[5)]	3.266
O(2)	H(3)[5)]	2.475	O(2)	H(11)[6)]	2.611
O(2)	H(18)[7)]	3.297	O(2)	H(19)[7)]	3.505
O(2)	H(19)[2)]	3.177	O(4)	H(1)[3)]	3.432
O(4)	H(1A)[3)]	2.09(2)	O(4)	H(8)[8)]	3.446
O(4)	H(9)[9)]	2.802	O(4)	H(10)[8)]	3.555
O(4)	H(10)[9)]	3.558	O(4)	H(17)[8)]	2.783
C(1)	H(4)[10)]	3.575	C(1)	H(5)[10)]	3.227
C(1)	H(14)[1)]	3.012	C(2)	H(3)[11)]	3.301
C(2)	H(9)[2)]	3.596	C(2)	H(10)[2)]	3.324
C(2)	H(11)[2)]	3.318	C(2)	H(18)[12)]	3.509
C(2)	H(19)[7)]	3.409	C(2)	H(19)[12)]	3.526
C(3)	H(2)[5)]	3.451	C(3)	H(10)[2)]	3.486
C(3)	H(11)[2)]	3.176	C(3)	H(19)[7)]	2.749
C(3)	H(19)[2)]	3.255	C(4)	H(1)[4)]	3.287
C(4)	H(3)[5)]	3.446	C(4)	H(11)[6)]	3.599
C(4)	H(18)[7)]	3.519	C(4)	H(19)[7)]	3.283
C(4)	H(19)[2)]	3.458	C(5)	H(1)[4)]	3.153
C(5)	H(14)[3)]	3.519	C(6)	H(1)[4)]	3.219
C(6)	H(1A)[3)]	3.57(2)	C(6)	H(15)[4)]	3.457
C(6)	H(16)[4)]	3.467	C(7)	H(12)[6)]	3.542
C(7)	H(13)[7)]	3.337	C(8)	H(1A)[13)]	3.44(2)
C(8)	H(18)[4)]	3.073	C(9)	H(3)[13)]	3.556
C(9)	H(7)[9)]	3.220	C(9)	H(18)[4)]	2.928
C(10)	H(8)[8)]	3.530	C(11)	H(2)[14)]	2.864
C(11)	H(3)[8)]	3.338	C(11)	H(9)[10)]	3.067
C(11)	H(11)[10)]	3.444	C(12)	H(1A)[3)]	2.75(2)
C(13)	H(1)[3)]	3.328	C(13)	H(1A)[3)]	3.06(2)
C(13)	H(4)[1)]	3.128	C(13)	H(5)[10)]	3.468
C(14)	H(5)[10)]	3.148	H(1)	O(2)[10)]	2.979
H(1)	O(4)[1)]	3.432	H(1)	C(4)[10)]	3.287
H(1)	C(5)[10)]	3.153	H(1)	C(6)[10)]	3.219
H(1)	C(13)[1)]	3.328	H(1)	H(4)[10)]	2.575
H(1)	H(5)[10)]	2.562	H(1)	H(6)[10)]	3.507

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(1)	H(14)[1)]	2.375	H(1A)	O(4)[1)]	2.09(2)
H(1A)	C(6)[1)]	3.57(2)	H(1A)	C(8)[2)]	3.44(2)
H(1A)	C(12)[1)]	2.75(2)	H(1A)	C(13)[1)]	3.06(2)
H(1A)	H(4)[1)]	3.206	H(1A)	H(6)[1)]	2.756
H(1A)	H(9)[2)]	3.056	H(1A)	H(10)[2)]	2.875
H(1A)	H(12)[1)]	3.520	H(1A)	H(14)[1)]	2.493
H(1A)	H(15)[3)]	3.019	H(2)	O(2)[10)]	2.924
H(2)	O(2)[11)]	3.266	H(2)	C(3)[11)]	3.451
H(2)	C(11)[12)]	2.864	H(2)	H(3)[11)]	2.691
H(2)	H(9)[2)]	3.238	H(2)	H(10)[2)]	3.385
H(2)	H(11)[2)]	2.995	H(2)	H(17)[12)]	2.874
H(2)	H(18)[12)]	2.658	H(2)	H(19)[7)]	3.578
H(2)	H(19)[12)]	2.579	H(3)	O(2)[11)]	2.475
H(3)	C(2)[5)]	3.301	H(3)	C(4)[11)]	3.446
H(3)	C(9)[2)]	3.556	H(3)	C(11)[7)]	3.338
H(3)	H(2)[5)]	2.691	H(3)	H(3)[11)]	3.289
H(3)	H(3)[5)]	3.289	H(3)	H(11)[2)]	2.702
H(3)	H(18)[7)]	3.437	H(3)	H(19)[7)]	2.457
H(3)	H(19)[2)]	2.862	H(4)	O(1)[3)]	3.039
H(4)	C(1)[4)]	3.575	H(4)	C(13)[3)]	3.128
H(4)	H(1)[4)]	2.575	H(4)	H(1A)[3)]	3.206
H(4)	H(14)[3)]	2.526	H(4)	H(15)[4)]	3.558
H(4)	H(15)[3)]	2.807	H(5)	C(1)[4)]	3.227
H(5)	C(13)[4)]	3.468	H(5)	C(14)[4)]	3.148
H(5)	H(1)[4)]	2.562	H(5)	H(13)[6)]	3.564
H(5)	H(15)[4)]	2.938	H(5)	H(16)[4)]	2.505
H(6)	O(1)[3)]	3.015	H(6)	H(1)[4)]	3.507
H(6)	H(1A)[3)]	2.756	H(6)	H(15)[4)]	3.101
H(7)	C(9)[6)]	3.220	H(7)	H(11)[6)]	2.956
H(7)	H(12)[6)]	2.600	H(7)	H(13)[7)]	3.081
H(7)	H(13)[6)]	3.541	H(7)	H(18)[7)]	3.010
H(8)	O(4)[7)]	3.446	H(8)	C(10)[7)]	3.530
H(8)	H(13)[7)]	2.672	H(9)	O(4)[6)]	2.802
H(9)	C(2)[13)]	3.596	H(9)	C(11)[4)]	3.067
H(9)	H(1A)[13)]	3.056	H(9)	H(2)[13)]	3.238
H(9)	H(17)[4)]	2.894	H(9)	H(18)[4)]	2.389
H(10)	O(1)[13)]	3.143	H(10)	O(4)[7)]	3.555

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(10)	O(4)[6)]	3.558	H(10)	C(2)[13)]	3.324
H(10)	C(3)[13)]	3.486	H(10)	H(1A)[13)]	2.875
H(10)	H(2)[13)]	3.385	H(10)	H(18)[4)]	3.578
H(11)	O(2)[9)]	2.611	H(11)	C(2)[13)]	3.318
H(11)	C(3)[13)]	3.176	H(11)	C(4)[9)]	3.599
H(11)	C(11)[4)]	3.444	H(11)	H(2)[13)]	2.995
H(11)	H(3)[13)]	2.702	H(11)	H(7)[9)]	2.956
H(11)	H(18)[4)]	2.498	H(12)	C(7)[9)]	3.542
H(12)	H(1A)[3)]	3.520	H(12)	H(7)[9)]	2.600
H(12)	H(18)[4)]	2.783	H(13)	C(7)[8)]	3.337
H(13)	H(5)[9)]	3.564	H(13)	H(7)[8)]	3.081
H(13)	H(7)[9)]	3.541	H(13)	H(8)[8)]	2.672
H(13)	H(16)[8)]	3.004	H(14)	O(1)[3)]	2.770
H(14)	C(1)[3)]	3.012	H(14)	C(5)[1)]	3.519
H(14)	H(1)[3)]	2.375	H(14)	H(1A)[3)]	2.493
H(14)	H(4)[1)]	2.526	H(14)	H(15)[3)]	3.042
H(15)	O(1)[1)]	2.621	H(15)	C(6)[10)]	3.457
H(15)	H(1A)[1)]	3.019	H(15)	H(4)[10)]	3.558
H(15)	H(4)[1)]	2.807	H(15)	H(5)[10)]	2.938
H(15)	H(6)[10)]	3.101	H(15)	H(14)[1)]	3.042
H(16)	C(6)[10)]	3.467	H(16)	H(5)[10)]	2.505
H(16)	H(13)[7)]	3.004	H(17)	O(4)[7)]	2.783
H(17)	H(2)[14)]	2.874	H(17)	H(9)[10)]	2.894
H(18)	O(2)[8)]	3.297	H(18)	C(2)[14)]	3.509
H(18)	C(4)[8)]	3.519	H(18)	C(8)[10)]	3.073
H(18)	C(9)[10)]	2.928	H(18)	H(2)[14)]	2.658
H(18)	H(3)[8)]	3.437	H(18)	H(7)[8)]	3.010
H(18)	H(9)[10)]	2.389	H(18)	H(10)[10)]	3.578
H(18)	H(11)[10)]	2.498	H(18)	H(12)[10)]	2.783
H(19)	O(2)[8)]	3.505	H(19)	O(2)[13)]	3.177
H(19)	C(2)[8)]	3.409	H(19)	C(2)[14)]	3.526
H(19)	C(3)[8)]	2.749	H(19)	C(3)[13)]	3.255
H(19)	C(4)[8)]	3.283	H(19)	C(4)[13)]	3.458
H(19)	H(2)[8)]	3.578	H(19)	H(2)[14)]	2.579
H(19)	H(3)[8)]	2.457	H(19)	H(3)[13)]	2.862

Symmetry Operators:

(1) X+1/2-1,-Y+1/2+1,-Z (2) -X+1/2+1,-Y+1,Z+1/2-1
(3) X+1/2,-Y+1/2+1,-Z (4) X+1,Y,Z

(5) X+1/2,-Y+1/2,-Z
(6) -X+2,Y+1/2-1,-Z+1/2
(7) -X+1,Y+1/2-1,-Z+1/2
(8) -X+1,Y+1/2,-Z+1/2
(9) -X+2,Y+1/2,-Z+1/2
(10) X-1,Y,Z
(11) X+1/2-1,-Y+1/2,-Z
(12) -X+1/2,-Y+1,Z+1/2-1
(13) -X+1/2+1,-Y+1,Z+1/2
(14) -X+1/2,-Y+1,Z+1/2

Compound **24**

February 23, 2009

Experimental

Data Collection

A colorless platelet crystal of $O_3C_{13}H_{18}$ having approximate dimensions of 0.25 x 0.20 x 0.10 mm was mounted on a glass fiber. All measurements were made on a Rigaku Mercury2 CCD area detector with graphite monochromated Mo-Ka radiation.

Indexing was performed from 1 images that were exposed for -6442450560 seconds. The crystal-to-detector distance was 49.90 mm.

Cell constants and an orientation matrix for data collection corresponded to a primitive monoclinic cell with dimensions:

a = 12.956(2) Å
b = 8.4492(15) Å b = 90.405(4)°
c = 11.2977(20) Å
V = 1236.7(4) Å^3

For Z = 4 and F.W. = 222.28, the calculated density is 1.194 g/cm^3. The systematic absences of:

h0l: l ± 2n
0k0: k ± 2n

uniquely determine the space group to be:

$P2_1/c$ (#14)

The data were collected at a temperature of -50 ± 1°C to a maximum 2q value of 55.0°. A total of 344 oscillation images were collected. A sweep of data was done using w scans from -120.0 to 60.0° in 1.0° step, at c=54.0° and f = 0.0°. The exposure rate was 80.0 [sec./°]. The detector swing angle was -28.40°. A second sweep was performed using w scans from -120.0 to 45.0° in 1.0° step, at c=54.0° and f = 120.0°. The exposure rate was 80.0 [sec./°]. The detector swing angle was -28.40°. The crystal-to-detector distance was 49.90 mm. Readout was performed in the 0.146 mm pixel mode.

Data Reduction

Of the 7859 reflections that were collected, 2820 were unique (R_{int} = 0.050). Data were collected and processed using CrystalClear (Rigaku). Net intensities and sigmas were derived as follows:

$$F^2 = [S(P_i - mB_{ave})] \cdot Lp^{-1}$$

where P_i is the value in counts of the i^{th} pixel
m is the number of pixels in the integration area
B_{ave} is the background average
Lp is the Lorentz and polarization factor

$$B_{ave} = S(B_j)/n$$

where n is the number of pixels in the background area
B_j is the value of the j^{th} pixel in counts

$$s^2(F^2_{hkl}) = [(SP_i) + m((S(B_{ave} - B_j)^2)/(n-1))] \cdot Lp \cdot errmul + (erradd \cdot F^2)^2$$

where erradd = 0.00
errmul = 1.00

The linear absorption coefficient, m, for Mo-Ka radiation is 0.834 cm^{-1}. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods[2] and expanded using Fourier techniques[3]. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement[4] on F^2 was based on 2812 observed reflections and 146 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = S ||Fo| - |Fc|| / S |Fo| = 0.0665$$

$$wR2 = [S (w (Fo^2 - Fc^2)^2)/ S w(Fo^2)^2]^{1/2} = 0.1613$$

The standard deviation of an observation of unit weight[5] was 1.07. Unit weights were used. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.15 and -0.13 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[6]. Anomalous dispersion effects were included in Fcalc[7]; the values for Df' and Df'' were those of Creagh and McAuley[8]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[9]. All calculations were performed using the CrystalStructure[10] crystallographic software package except for refinement, which was performed using SHELXL-97[11].

References

(1) CrystalClear: Rigaku Corporation, 1999. CrystalClear Software User's Guide, Molecular Structure Corporation, (c) 2000.J.W.Pflugrath (1999) Acta Cryst. D55, 1718-1725.

(2) SHELX97: Sheldrick, G.M. (1997).

(3) DIRDIF99: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M.(1999). The DIRDIF-99 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(4) Least Squares function minimized: (SHELXL97)

$Sw(F_o^2-F_c^2)^2$ where w = Least Squares weights.

(5) Standard deviation of an observation of unit weight:

$[Sw(F_o^2-F_c^2)^2/(N_o-N_v)]^{1/2}$

where: N_o = number of observations
N_v = number of variables

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(10) CrystalStructure 3.8: Crystal Structure Analysis Package, Rigaku and Rigaku Americas (2000-2007). 9009 New Trails Dr. The Woodlands TX 77381 USA.

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EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$O_3C_{13}H_{18}$
Formula Weight	222.28
Crystal Color, Habit	colorless, platelet
Crystal Dimensions	0.25 X 0.20 X 0.10 mm
Crystal System	monoclinic
Lattice Type	Primitive
Indexing Images	1 images @ -6442450560.0 seconds
Detector Position	49.90 mm
Pixel Size	0.146 mm
Lattice Parameters	a = 12.956(2) Å b = 8.4492(15) Å c = 11.2977(20) Å b = 90.405(4) ° V = 1236.7(4) $Å^3$
Space Group	$P2_1/c$ (#14)
Z value	4
D_{calc}	1.194 g/cm^3
F_{000}	480.00
m(MoKa)	0.834 cm^{-1}

B. Intensity Measurements

Diffractometer	Rigaku SCXmini
Radiation	MoKa (l = 0.71075 Å) graphite monochromated
Detector Aperture	75 mm round
Data Images	344 exposures
w oscillation Range (c=54.0, f=0.0)	-120.0 - 60.0^{o}
Exposure Rate	80.0 sec./^{o}
Detector Swing Angle	-28.40^{o}
w oscillation Range (c=54.0, f=120.0)	-120.0 - 45.0^{o}
Exposure Rate	80.0 sec./^{o}
Detector Swing Angle	-28.40^{o}
Detector Position	49.90 mm
Pixel Size	0.146 mm
$2q_{max}$	55.0^{o}
No. of Reflections Measured	Total: 7859 Unique: 2812 (R_{int} = 0.050)
Corrections	Lorentz-polarization

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SHELX97)
Refinement	Full-matrix least-squares on F^2
Function Minimized	$S\ w\ (Fo^2 - Fc^2)^2$
Least Squares Weights	$w = 1/ [\ s^2(Fo^2) + (0.0478 \cdot P)^2 + 0.4198 \cdot P\ ]$ where $P = (Max(Fo^2,0) + 2Fc^2)/3$
$2q_{max}$ cutoff	55.0^o
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	2812
No. Variables	146
Reflection/Parameter Ratio	19.26
Residuals: R1 (I>2.00s(I))	0.0665
Residuals: R (All reflections)	0.1167
Residuals: wR2 (All reflections)	0.1613
Goodness of Fit Indicator	1.066
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	$0.15\ e^-/Å^3$
Minimum peak in Final Diff. Map	$-0.13\ e^-/Å^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
O(1)	0.65639(13)	0.6625(2)	0.30315(14)	4.95(3)
O(2)	0.62587(17)	0.7118(2)	0.11068(17)	6.78(4)
O(3)	0.82337(18)	0.3443(2)	-0.05998(17)	7.06(5)
C(1)	0.62518(18)	0.6221(3)	0.1937(2)	4.59(4)
C(2)	0.59663(17)	0.4550(3)	0.1894(2)	4.58(4)
C(3)	0.58952(17)	0.3740(3)	0.0904(2)	4.78(5)
C(4)	0.58014(19)	0.1985(3)	0.0822(2)	5.38(5)
C(5)	0.67781(19)	0.1236(3)	0.0327(2)	4.94(5)
C(6)	0.77589(17)	0.1760(2)	0.0973(2)	4.20(4)
C(7)	0.82623(18)	0.3213(2)	0.0459(2)	4.32(4)
C(8)	0.88439(17)	0.4335(2)	0.1212(2)	4.58(4)
C(9)	0.88545(17)	0.4402(2)	0.2361(2)	4.55(4)
C(10)	0.94528(19)	0.5585(3)	0.3093(2)	5.45(5)
C(11)	0.8749(2)	0.6743(3)	0.3735(2)	5.32(5)
C(12)	0.8208(2)	0.7933(3)	0.2927(2)	5.19(5)
C(13)	0.7085(2)	0.8135(3)	0.3181(2)	5.36(5)

$B_{eq} = 8/3\ p^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos g + 2U_{13}(aa^*cc^*)\cos b + 2U_{23}(bb^*cc^*)\cos a)$

Table 2. Atomic coordinates and B_{iso} involving hydrogens/B_{eq}

atom	x	y	z	B_{eq}
H(1)	0.5827	0.4028	0.2610	5.49
H(2)	0.5904	0.4313	0.0191	5.73
H(3)	0.5214	0.1715	0.0309	6.45
H(4)	0.5667	0.1551	0.1610	6.45
H(5)	0.6836	0.1513	-0.0512	5.92
H(6)	0.6719	0.0082	0.0382	5.92
H(7)	0.7591	0.1971	0.1803	5.04
H(8)	0.8257	0.0887	0.0960	5.04
H(9)	0.9252	0.5084	0.0816	5.50
H(10)	0.8457	0.3652	0.2769	5.46
H(11)	0.9918	0.6178	0.2576	6.54
H(12)	0.9877	0.5018	0.3676	6.54
H(13)	0.9161	0.7325	0.4322	6.38
H(14)	0.8225	0.6140	0.4163	6.38
H(15)	0.8550	0.8962	0.3009	6.23
H(16)	0.8285	0.7588	0.2105	6.23
H(17)	0.6996	0.8516	0.3993	6.43

H(18)	0.6786	0.8919	0.2639	6.43

$B_{eq} = 8/3\ p^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos g + 2U_{13}(aa^*cc^*)\cos b + 2U_{23}(bb^*cc^*)\cos a)$

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	0.0624(10)	0.0710(11)	0.0549(10)	-0.0030(8)	0.0049(7)	0.0013(8)
O(2)	0.1217(17)	0.0684(12)	0.0673(12)	0.0119(11)	-0.0139(10)	0.0111(9)
O(3)	0.1263(18)	0.0834(14)	0.0586(12)	-0.0173(12)	0.0140(10)	-0.0013(9)
C(1)	0.0550(14)	0.0646(16)	0.0548(15)	0.0144(11)	0.0019(11)	0.0032(12)
C(2)	0.0463(13)	0.0697(16)	0.0578(15)	0.0031(11)	-0.0019(10)	0.0065(12)
C(3)	0.0443(13)	0.0732(17)	0.0640(16)	0.0090(11)	-0.0093(10)	0.0040(13)
C(4)	0.0520(14)	0.0741(18)	0.0780(18)	-0.0066(12)	-0.0111(11)	-0.0051(13)
C(5)	0.0614(15)	0.0568(14)	0.0693(16)	-0.0036(11)	-0.0084(11)	-0.0083(12)
C(6)	0.0517(13)	0.0471(13)	0.0608(14)	0.0055(10)	-0.0049(10)	-0.0006(10)
C(7)	0.0571(14)	0.0515(13)	0.0555(15)	0.0047(10)	0.0059(10)	-0.0039(11)
C(8)	0.0524(14)	0.0519(14)	0.0700(17)	-0.0029(10)	0.0123(11)	-0.0010(11)
C(9)	0.0453(13)	0.0532(14)	0.0743(18)	-0.0017(10)	-0.0002(10)	-0.0004(11)
C(10)	0.0521(14)	0.0682(17)	0.0866(19)	-0.0083(12)	-0.0114(12)	-0.0122(13)
C(11)	0.0676(16)	0.0667(17)	0.0677(17)	-0.0127(13)	-0.0027(12)	-0.0097(13)
C(12)	0.0748(17)	0.0515(14)	0.0711(17)	-0.0092(12)	0.0146(12)	-0.0062(12)
C(13)	0.0832(18)	0.0549(15)	0.0658(17)	0.0028(13)	0.0092(13)	-0.0103(12)

The general temperature factor expression: $\exp(-2p^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
O(1)	C(1)	1.343(2)	O(1)	C(13)	1.453(3)
O(2)	C(1)	1.206(3)	O(3)	C(7)	1.212(3)
C(1)	C(2)	1.460(3)	C(2)	C(3)	1.314(3)
C(3)	C(4)	1.491(3)	C(4)	C(5)	1.524(3)
C(5)	C(6)	1.527(3)	C(6)	C(7)	1.508(3)
C(7)	C(8)	1.477(3)	C(8)	C(9)	1.299(3)
C(9)	C(10)	1.508(3)	C(10)	C(11)	1.524(3)
C(11)	C(12)	1.526(3)	C(12)	C(13)	1.494(3)

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C(2)	H(1)	0.940	C(3)	H(2)	0.940
C(4)	H(3)	0.980	C(4)	H(4)	0.980
C(5)	H(5)	0.980	C(5)	H(6)	0.980
C(6)	H(7)	0.980	C(6)	H(8)	0.980
C(8)	H(9)	0.940	C(9)	H(10)	0.940
C(10)	H(11)	0.980	C(10)	H(12)	0.980
C(11)	H(13)	0.980	C(11)	H(14)	0.980
C(12)	H(15)	0.980	C(12)	H(16)	0.980
C(13)	H(17)	0.980	C(13)	H(18)	0.980

Table 6. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
C(1)	O(1)	C(13)	117.84(19)	O(1)	C(1)	O(2)	123.6(2)
O(1)	C(1)	C(2)	110.5(2)	O(2)	C(1)	C(2)	125.8(2)
C(1)	C(2)	C(3)	123.2(2)	C(2)	C(3)	C(4)	125.2(2)
C(3)	C(4)	C(5)	111.6(2)	C(4)	C(5)	C(6)	113.2(2)
C(5)	C(6)	C(7)	114.40(19)	O(3)	C(7)	C(6)	120.0(2)
O(3)	C(7)	C(8)	118.5(2)	C(6)	C(7)	C(8)	121.4(2)
C(7)	C(8)	C(9)	127.3(2)	C(8)	C(9)	C(10)	125.3(2)
C(9)	C(10)	C(11)	112.3(2)	C(10)	C(11)	C(12)	114.3(2)
C(11)	C(12)	C(13)	113.9(2)	O(1)	C(13)	C(12)	109.3(2)

Table 7. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
C(1)	C(2)	H(1)	118.4	C(3)	C(2)	H(1)	118.4
C(2)	C(3)	H(2)	117.4	C(4)	C(3)	H(2)	117.4
C(3)	C(4)	H(3)	109.3	C(3)	C(4)	H(4)	109.3
C(5)	C(4)	H(3)	109.3	C(5)	C(4)	H(4)	109.3
H(3)	C(4)	H(4)	108.0	C(4)	C(5)	H(5)	108.9
C(4)	C(5)	H(6)	108.9	C(6)	C(5)	H(5)	108.9
C(6)	C(5)	H(6)	108.9	H(5)	C(5)	H(6)	107.7
C(5)	C(6)	H(7)	108.7	C(5)	C(6)	H(8)	108.7
C(7)	C(6)	H(7)	108.7	C(7)	C(6)	H(8)	108.7
H(7)	C(6)	H(8)	107.6	C(7)	C(8)	H(9)	116.4
C(9)	C(8)	H(9)	116.4	C(8)	C(9)	H(10)	117.3
C(10)	C(9)	H(10)	117.3	C(9)	C(10)	H(11)	109.1
C(9)	C(10)	H(12)	109.1	C(11)	C(10)	H(11)	109.1
C(11)	C(10)	H(12)	109.1	H(11)	C(10)	H(12)	107.9
C(10)	C(11)	H(13)	108.7	C(10)	C(11)	H(14)	108.7

C(12)	C(11)	H(13)	108.7	C(12)	C(11)	H(14)	108.7
H(13)	C(11)	H(14)	107.6	C(11)	C(12)	H(15)	108.8
C(11)	C(12)	H(16)	108.8	C(13)	C(12)	H(15)	108.8
C(13)	C(12)	H(16)	108.8	H(15)	C(12)	H(16)	107.7
O(1)	C(13)	H(17)	109.8	O(1)	C(13)	H(18)	109.8
C(12)	C(13)	H(17)	109.8	C(12)	C(13)	H(18)	109.8
H(17)	C(13)	H(18)	108.3				

Table 8. Torsion Angles(o)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C(1)	O(1)	C(13)	C(12)	-85.0(2)	C(13)	O(1)	C(1)	O(2)	-10.0(3)
C(13)	O(1)	C(1)	C(2)	166.91(19)	O(1)	C(1)	C(2)	C(3)	-161.6(2)
O(2)	C(1)	C(2)	C(3)	15.2(3)	C(1)	C(2)	C(3)	C(4)	168.0(2)
C(2)	C(3)	C(4)	C(5)	-110.2(2)	C(3)	C(4)	C(5)	C(6)	51.6(2)
C(4)	C(5)	C(6)	C(7)	-90.2(2)	C(5)	C(6)	C(7)	O(3)	-34.3(3)
C(5)	C(6)	C(7)	C(8)	149.0(2)	O(3)	C(7)	C(8)	C(9)	170.6(2)
C(6)	C(7)	C(8)	C(9)	-12.7(3)	C(7)	C(8)	C(9)	C(10)	-179.2(2)
C(8)	C(9)	C(10)	C(11)	111.9(2)	C(9)	C(10)	C(11)	C(12)	-70.6(2)
C(10)	C(11)	C(12)	C(13)	134.5(2)	C(11)	C(12)	C(13)	O(1)	-60.0(2)

The sign is positive if when looking from atom 2 to atom 3 a clock-wise motion of atom 1 would superimpose it on atom 4.

Table 9. Distances beyond the asymmetric unit out to 3.60 Å

atom	atom	distance	atom	atom	distance
O(1)	C(4)[1)]	3.349(3)	O(1)	C(5)[2)]	3.555(3)
O(2)	C(4)[3)]	3.516(3)	O(2)	C(13)[4)]	3.488(3)
O(3)	C(9)[5)]	3.429(3)	O(3)	C(12)[4)]	3.485(3)
O(3)	C(13)[4)]	3.528(3)	C(4)	O(1)[6)]	3.349(3)
C(4)	O(2)[3)]	3.516(3)	C(5)	O(1)[5)]	3.555(3)
C(9)	O(3)[2)]	3.429(3)	C(12)	O(3)[7)]	3.485(3)
C(13)	O(2)[7)]	3.488(3)	C(13)	O(3)[7)]	3.528(3)

Symmetry Operators:

(1) -X+1,Y+1/2,-Z+1/2
(2) X,-Y+1/2,Z+1/2
(3) -X+1,-Y+1,-Z
(4) X,-Y+1/2+1,Z+1/2-1
(5) X,-Y+1/2,Z+1/2-1
(6) -X+1,Y+1/2-1,-Z+1/2
(7) X,-Y+1/2+1,Z+1/2

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
O(1)	H(3)[1)]	2.981	O(1)	H(4)[1)]	2.923
O(1)	H(5)[2)]	3.139	O(1)	H(6)[2)]	3.027
O(2)	H(1)[1)]	3.473	O(2)	H(2)[3)]	3.377
O(2)	H(3)[3)]	2.670	O(2)	H(6)[4)]	2.703
O(2)	H(17)[5)]	2.633	O(3)	H(7)[6)]	3.065
O(3)	H(9)[7)]	3.498	O(3)	H(10)[6)]	2.572
O(3)	H(11)[7)]	3.301	O(3)	H(15)[5)]	2.731
O(3)	H(17)[5)]	3.062	O(3)	H(18)[5)]	3.521
C(1)	H(4)[1)]	3.002	C(1)	H(17)[5)]	3.476
C(2)	H(2)[3)]	3.501	C(2)	H(4)[1)]	3.201
C(2)	H(5)[2)]	3.259	C(3)	H(2)[3)]	3.103
C(3)	H(17)[5)]	3.480	C(4)	H(18)[8)]	3.538
C(5)	H(1)[6)]	3.308	C(5)	H(14)[6)]	3.051
C(5)	H(18)[8)]	3.264	C(6)	H(11)[9)]	3.453
C(6)	H(12)[9)]	3.419	C(6)	H(14)[6)]	3.251
C(6)	H(15)[8)]	3.450	C(6)	H(18)[8)]	3.306
C(7)	H(10)[6)]	3.434	C(7)	H(13)[9)]	3.429
C(8)	H(9)[7)]	3.415	C(8)	H(11)[9)]	3.396
C(8)	H(13)[9)]	3.155	C(8)	H(13)[5)]	3.565
C(8)	H(15)[9)]	3.497	C(9)	H(11)[9)]	3.155
C(9)	H(15)[9)]	3.411	C(10)	H(8)[10)]	3.158
C(10)	H(15)[9)]	3.189	C(11)	H(6)[2)]	3.582
C(11)	H(8)[2)]	3.419	C(12)	H(8)[4)]	3.343
C(12)	H(12)[10)]	3.550	C(13)	H(2)[11)]	3.492
C(13)	H(6)[4)]	3.593	H(1)	O(2)[12)]	3.473
H(1)	C(5)[2)]	3.308	H(1)	H(3)[1)]	3.541
H(1)	H(3)[2)]	3.219	H(1)	H(4)[1)]	3.015
H(1)	H(5)[2)]	2.526	H(1)	H(6)[2)]	3.415
H(1)	H(18)[12)]	3.395	H(2)	O(2)[3)]	3.377
H(2)	C(2)[3)]	3.501	H(2)	C(3)[3)]	3.103
H(2)	C(13)[5)]	3.492	H(2)	H(2)[3)]	2.647
H(2)	H(17)[5)]	2.687	H(2)	H(18)[5)]	3.448
H(3)	O(1)[12)]	2.981	H(3)	O(2)[3)]	2.670
H(3)	H(1)[12)]	3.541	H(3)	H(1)[6)]	3.219
H(3)	H(3)[13)]	3.032	H(3)	H(6)[13)]	3.026
H(3)	H(17)[12)]	3.341	H(4)	O(1)[12)]	2.923
H(4)	C(1)[12)]	3.002	H(4)	C(2)[12)]	3.201

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

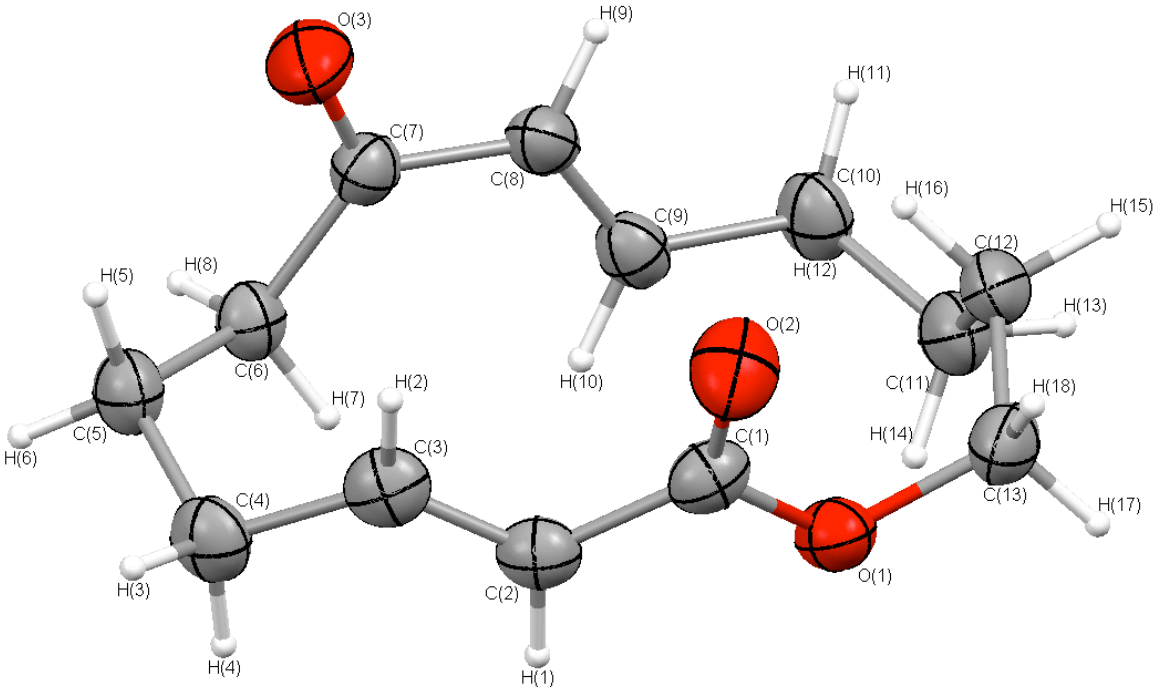
atom	atom	distance	atom	atom	distance
H(4)	H(1)$^{12)}$	3.015	H(4)	H(18)$^{8)}$	2.893
H(5)	O(1)$^{6)}$	3.139	H(5)	C(2)$^{6)}$	3.259
H(5)	H(1)$^{6)}$	2.526	H(5)	H(7)$^{6)}$	3.442
H(5)	H(10)$^{6)}$	2.874	H(5)	H(14)$^{6)}$	2.899
H(6)	O(1)$^{6)}$	3.027	H(6)	O(2)$^{8)}$	2.703
H(6)	C(11)$^{6)}$	3.582	H(6)	C(13)$^{8)}$	3.593
H(6)	H(1)$^{6)}$	3.415	H(6)	H(3)$^{13)}$	3.026
H(6)	H(14)$^{6)}$	2.609	H(6)	H(16)$^{8)}$	3.505
H(6)	H(17)$^{6)}$	3.441	H(6)	H(18)$^{8)}$	2.734
H(7)	O(3)$^{2)}$	3.065	H(7)	H(5)$^{2)}$	3.442
H(7)	H(11)$^{9)}$	3.366	H(7)	H(15)$^{8)}$	3.138
H(7)	H(18)$^{8)}$	2.941	H(8)	C(10)$^{9)}$	3.158
H(8)	C(11)$^{6)}$	3.419	H(8)	C(12)$^{8)}$	3.343
H(8)	H(11)$^{9)}$	2.887	H(8)	H(12)$^{9)}$	2.558
H(8)	H(12)$^{6)}$	3.424	H(8)	H(13)$^{9)}$	3.576
H(8)	H(13)$^{6)}$	3.491	H(8)	H(14)$^{6)}$	2.656
H(8)	H(15)$^{8)}$	2.853	H(8)	H(16)$^{8)}$	3.073
H(8)	H(18)$^{8)}$	3.171	H(9)	O(3)$^{7)}$	3.498
H(9)	C(8)$^{7)}$	3.415	H(9)	H(9)$^{7)}$	2.689
H(9)	H(13)$^{9)}$	3.114	H(9)	H(13)$^{5)}$	2.766
H(9)	H(15)$^{9)}$	3.273	H(9)	H(15)$^{5)}$	3.391
H(10)	O(3)$^{2)}$	2.573	H(10)	C(7)$^{2)}$	3.434
H(10)	H(5)$^{2)}$	2.874	H(10)	H(11)$^{9)}$	2.995
H(11)	O(3)$^{7)}$	3.301	H(11)	C(6)$^{10)}$	3.453
H(11)	C(8)$^{10)}$	3.396	H(11)	C(9)$^{10)}$	3.155
H(11)	H(7)$^{10)}$	3.366	H(11)	H(8)$^{10)}$	2.887
H(11)	H(10)$^{10)}$	2.995	H(11)	H(12)$^{10)}$	3.550
H(11)	H(15)$^{9)}$	2.810	H(12)	C(6)$^{10)}$	3.419
H(12)	C(12)$^{9)}$	3.550	H(12)	H(8)$^{10)}$	2.558
H(12)	H(8)$^{2)}$	3.424	H(12)	H(11)$^{9)}$	3.550
H(12)	H(12)$^{14)}$	3.007	H(12)	H(13)$^{14)}$	3.248
H(12)	H(14)$^{14)}$	3.589	H(12)	H(15)$^{9)}$	2.937
H(12)	H(16)$^{9)}$	3.270	H(13)	C(7)$^{10)}$	3.429
H(13)	C(8)$^{10)}$	3.155	H(13)	C(8)$^{11)}$	3.565
H(13)	H(8)$^{10)}$	3.576	H(13)	H(8)$^{2)}$	3.491
H(13)	H(9)$^{10)}$	3.114	H(13)	H(9)$^{11)}$	2.766
H(13)	H(12)$^{14)}$	3.248	H(13)	H(16)$^{11)}$	3.350

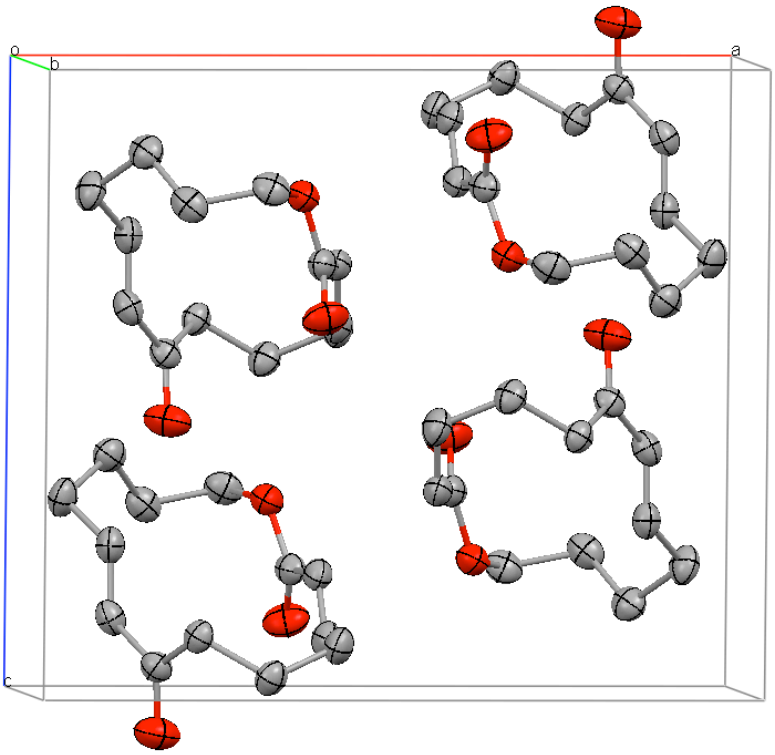
Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

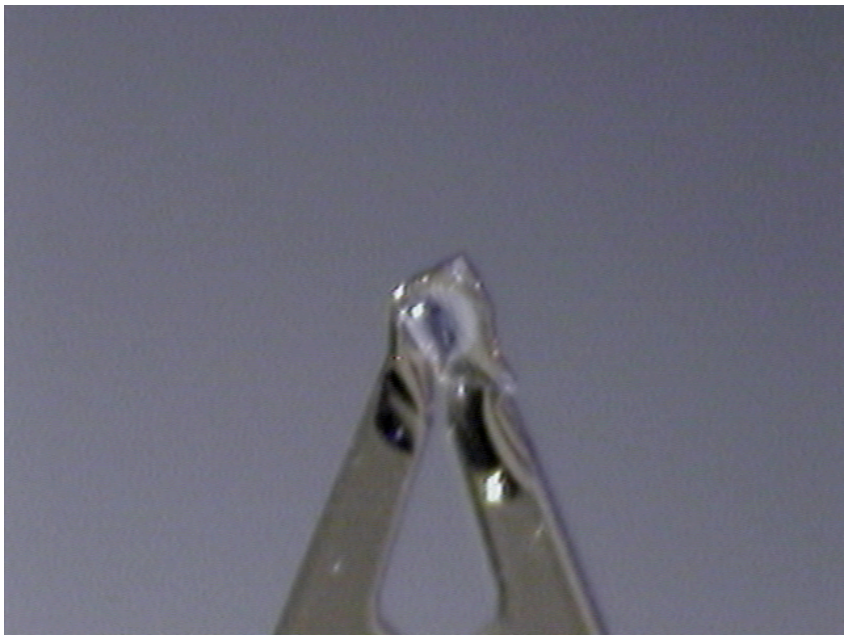
atom	atom	distance	atom	atom	distance
H(14)	C(5)[2)]	3.051	H(14)	C(6)[2)]	3.251
H(14)	H(5)[2)]	2.899	H(14)	H(6)[2)]	2.609
H(14)	H(8)[2)]	2.656	H(14)	H(12)[14)]	3.589
H(14)	H(16)[11)]	3.493	H(15)	O(3)[11)]	2.731
H(15)	C(6)[4)]	3.450	H(15)	C(8)[10)]	3.497
H(15)	C(9)[10)]	3.411	H(15)	C(10)[10)]	3.189
H(15)	H(7)[4)]	3.138	H(15)	H(8)[4)]	2.853
H(15)	H(9)[10)]	3.273	H(15)	H(9)[11)]	3.391
H(15)	H(11)[10)]	2.810	H(15)	H(12)[10)]	2.937
H(16)	H(6)[4)]	3.505	H(16)	H(8)[4)]	3.073
H(16)	H(12)[10)]	3.270	H(16)	H(13)[5)]	3.350
H(16)	H(14)[5)]	3.493	H(17)	O(2)[11)]	2.633
H(17)	O(3)[11)]	3.062	H(17)	C(1)[11)]	3.476
H(17)	C(3)[11)]	3.480	H(17)	H(2)[11)]	2.687
H(17)	H(3)[1)]	3.341	H(17)	H(6)[2)]	3.441
H(18)	O(3)[11)]	3.521	H(18)	C(4)[4)]	3.538
H(18)	C(5)[4)]	3.264	H(18)	C(6)[4)]	3.306
H(18)	H(1)[1)]	3.395	H(18)	H(2)[11)]	3.448
H(18)	H(4)[4)]	2.893	H(18)	H(6)[4)]	2.734
H(18)	H(7)[4)]	2.941	H(18)	H(8)[4)]	3.171

Symmetry Operators:

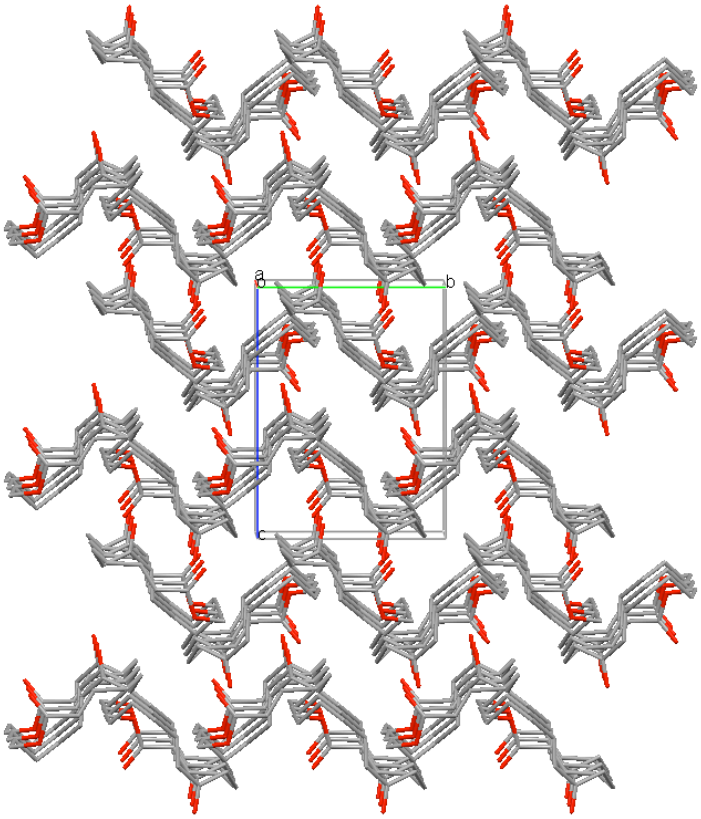
(1) -X+1,Y+1/2,-Z+1/2
(2) X,-Y+1/2,Z+1/2
(3) -X+1,-Y+1,-Z
(4) X,Y+1,Z
(5) X,-Y+1/2+1,Z+1/2-1
(6) X,-Y+1/2,Z+1/2-1
(7) -X+2,-Y+1,-Z
(8) X,Y-1,Z
(9) -X+2,Y+1/2-1,-Z+1/2
(10) -X+2,Y+1/2,-Z+1/2
(11) X,-Y+1/2+1,Z+1/2
(12) -X+1,Y+1/2-1,-Z+1/2
(13) -X+1,-Y,-Z
(14) -X+2,-Y+1,-Z+1


O(3)
H(9)
H(11)
C(7)
C(8)
C(10)
H(16)
H(15)
C(9)
C(12)
H(5)
H(8)
H(12)
H(13)
O(2)
C(6)
H(10)
C(11)
H(18)
H(7)
H(2)
C(5)
C(1)
H(14)
H(6)
C(3)
C(13)
C(4)
H(17)
O(1)
H(3)
C(2)
H(4)
H(1)

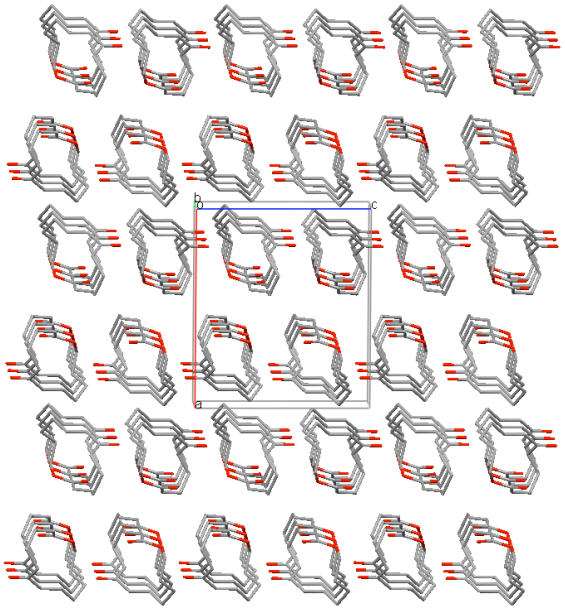

a
b
c



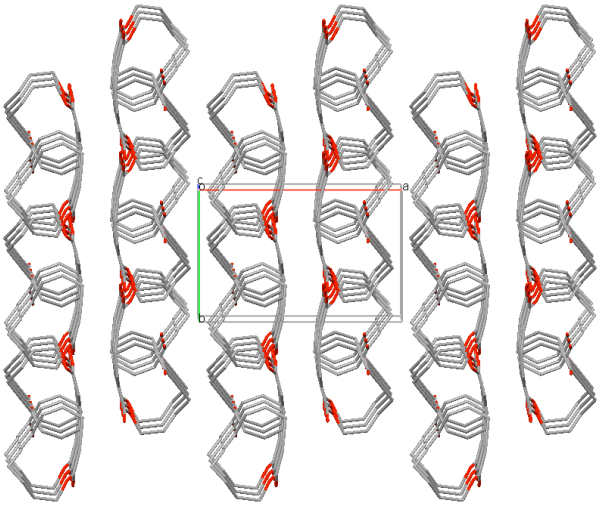
Packing diagram – View down the *a*-axis



Packing diagram – View down the *b*-axis



Packing diagram – View down the *c*-axis



IV. Biological Assay

Cell Culture – Antiviral activity and cellular toxicity were determined using the MTT colorimetric method.[14,15,16] MT-2 cells[17,18] at a concentration of 1 x 10^5 cells per millilitre were infected with wild-type HIV IIIB[19,20,21] at a multiplicity of infection (MOI) of 0.1. Infected and mock-infected cells were incubated in growth medium (RPMI 1640, 10% dFBS, kanamycin) for 5 days with varying concentrations of each compound being tested in triplicate in a 96-well plate. MTT (thiazolyl blue tetrazolium bromide), a cell-permeable tetrazolium dye was then added to each well. Active mitochondria reduce the yellow tetrazolium salt to a blue formazan precipitate. After 5 h, stop solution (86% isopropanol, 4% NP-40, 10% H_2O, and 0.3% concentrated HCl) was added to lyse the cells and stop the reaction. The plates were gently shaken gently overnight on a horizontal rotator, and quantitated the following morning. Cell viability was measured spectrophotometrically by quantifying the amount of purple precipitate via determining the absorbance at 595 nM using a Multiskan Plus microplate reader from Labsystems (Helsinki, Finland). The average of these triplicate samples was then plotted versus inhibitor concentration to generate dose–response curves. The 50% effective concentration (EC_{50}) and 50% cytotoxic concentration (CC_{50}) of the compounds were defined as the concentrations required to inhibit viral replication and to reduce the number of viable cells by 50%, respectively. Positive controls

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were done during each set of experiments using d4T and the appropriate parent NNRTI (HI-236 or TMC-derivative). Data were quantified using KaleidaGraph (Synergy Software).