Studies on the Synthesis of Gymnodimine.
Stereocontrolled Construction of the Tetrahydrofuran Subunit

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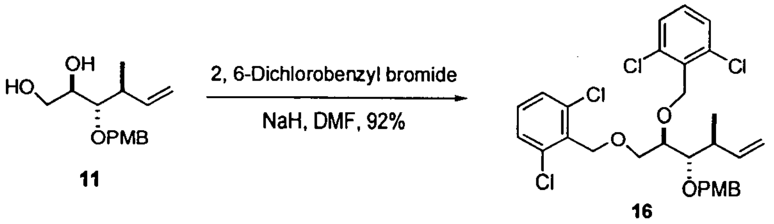
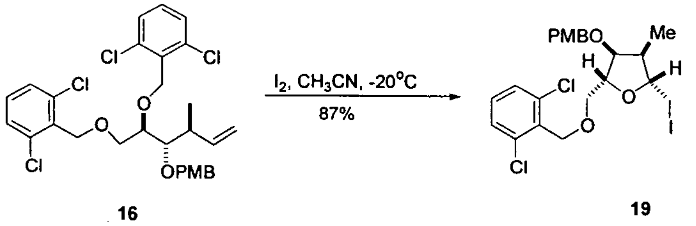
SUPPORTING INFORMATION



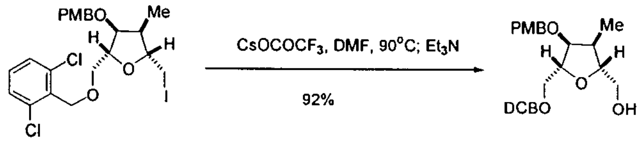
(3*S*,4*S*,5*R*)-5,6-Bis-(2,6-dichlorobenzyloxy)-4-(4-methoxybenzyloxy)-3-methylhex-1-ene (16).

To a suspension of NaH (1.15 g, 48 mmol) in 100 mL of dry DMF was added a solution of diol **11** (3.25 g, 12 mmol) in 10 mL of dry DMF at 0 °C. The mixture was stirred for 20 min at 0 °C, and a solution of 2,6-dichlorobenzyl bromide (11.5 g, 48 mmol) in 20 mL of dry DMF was added slowly, followed by Bu_4NI (17.7g, 48mmol) at 0 °C. The resulting mixture was stirred overnight at room temperature. The reaction was quenched by the addition of saturated NH_4Cl and the mixture was diluted with Et_2O. The phases were separated, and the organic layer was washed with water and brine. The solution was dried over Na_2SO_4, and concentrated under vacuum. Flash chromatography (hexane-ethyl acetate, 15:1) of the residue afforded 6.55 g (92%) of **16**: $[\alpha]_D^{23}$ +0.01 (c 0.77 , $CHCl_3$); IR (neat) 3073, 2959, 2878, 1613, 1583, 1564, 1514, 1436, 1249, 1197, 1098, 1037, 993, 916, 822, 767, 733 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.04 (d, *J* = 7.0 Hz, 1H), 2.67 (m, 1H), 3.53 (dd, *J* = 3.9, 7.7 Hz, 1H), 3.58 (m, 1H), 3.79 (s, 3H), 3.82 (dd, *J* = 3.5, 10.5 Hz, 1H), 4.01 (dd, *J* = 3.2, 10.5 Hz, 1H), 4.47 (d, *J* = 10.9 Hz, 1H), 4.59 (d, *J* = 10.9 Hz, 1H), 4.80 – 5.0 (m, 6H), 5.85 (ddd, *J* = 8.5, 11.1, 16.6 Hz, 1H), 7.81 (m, 2H), 7.12 – 7.33 (m, 8H); ^{13}C NMR (100 Mz, $CDCl_3$) δ 18.6, 40.0, 55.7, 66.4, 68.1, 69.5, 74.7, 79.9, 82.1, 114.0, 115.5, 129.7, 130.2, 131.6, 134.1, 134.2, 137.4, 137.4, 140.7, 159.2; HRMS (FAB) *m/z* 583.0996 ($[M+H]^+$; calcd for $C_{29}H_{31}O_4{}^{35}Cl_4$: 583.0977).



(2*R*,3*S*,4*R*,5*R*)-2-(2,6-Dichlorobenzyloxymethyl)-5-iodomethyl-3-(4-methoxybenzyloxy)-4-

methyltetrahydrofuran (19). A solution of alkene **16** (2.60 g, 4.40 mmol) in dry CH_3CN (450 mL) was cooled to –20 °C and a solution of iodine in dry CH_3CN (34 mL of a 0.2 M solution, 6.8 mmol) was slowly added via syringe. The dark-brown mixture was stirred for 3 h at –20°C and was diluted with Et_2O and saturated $Na_2S_2O_3$ (50 mL). The phases were separated and the aqueous phase was extracted with Et_2O (200 mL x 2). The combined organic layer was washed with brine and dried over Na_2SO_4, filtered, and concentrated. Flash chromatography (hexane-ethyl acetate, 10:1) of the residue afforded 2.10 g (87%) of **19**: $[\alpha]_D^{23}$ –35.8 (c 0.34, $CHCl_3$); IR (neat) 2961, 2921, 2852, 1558, 1508, 1457, 1247, 1104, 1035, 812, 767 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 1.02 (d, *J* = 7.0 Hz, 3H), 1.99 (m, 1H), 3.20 (dd, *J* = 5.0, 10.6 Hz, 1H), 3.43 (dd, *J* = 3.7, 10.6 Hz, 1H), 3.48 (m, 1H), 3.49 (dd, *J* = 7.0, 9.9 Hz, 1H), 3.60 (dd, *J* = 4.5, 9.9 Hz, 1H), 3.81 (s, 3H), 3.87 (dd, *J* = 2.0, 5.8 Hz, 1H), 4.15 (ddd, *J* = 2.1, 4.5, 6.7 Hz, 1H), 4.28 (d, *J* = 11.6 Hz, 1H), 4.47 (d, *J* = 11.6 Hz, 1H), 4.78 (d, *J* = 10.8 Hz, 1H), 4.83 (d, *J* = 10.8 Hz, 1H), 6.82 (m, 2H), 7.15-7.35 (m, 5H); ^{13}C NMR (100 Mz, $CDCl_3$) δ 10.5, 42.8, 55.7, 68.1, 71.4, 71.6, 82.6, 82.8, 82.9, 114.1, 128.8, 129.7, 130.3, 133.8, 137.3, 160.0; HRMS (FAB) *m/z* 550.0168 ($[M]^+$; calcd for $C_{22}H_{25}O_4{}^{35}Cl_2I$: 550.0175).

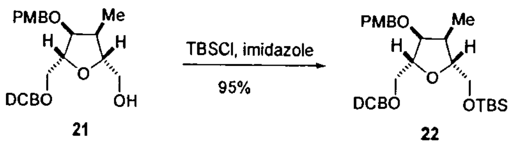


19

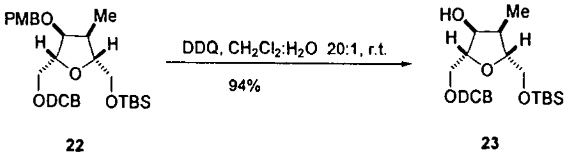
21

(2*R*,3*R*,4*S*,5*R*)-[5-(2,6-Dichlorobenzyloxymethyl)-4-(4-methoxyphenoxymethyl)-3-methyltetrahydrofuran-2-yl]methanol (21). To a solution of iodide **19** (9.40 g, 17 mmol) in 150 mL of DMF was added cesium trifloroacetate (20.9 g, 85 mmol) at room temperature. The mixture was stirred for 36 h at 90 °C and then cooled to room temperature. Diethylamine (10 mL) was added, and the resulting solution was stirred for 3 h at room temperature. The mixture was diluted with Et_2O and washed with water and brine, and the organic layer was dried over Na_2SO_4, filtered, and concentrated. Flash chromatography (hexane-ethyl acetate, 2:1) of the residue afforded 6.80 g (92%) of **21**: $[\alpha]_D^{23}$ +2.4

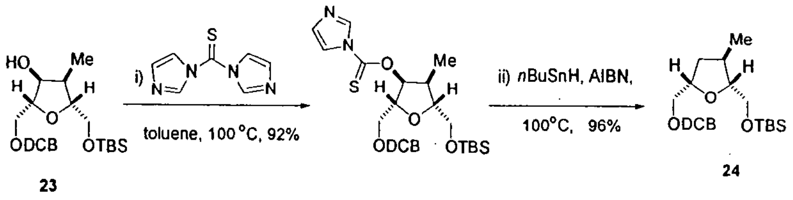
(c 0.70, $CHCl_3$); IR (neat) 3454, 2926, 2874, 1612, 1583, 1563, 1513, 1437, 1247, 1105, 1035, 822, 780, 768, 783 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 1.02 (d, J = 7.0 Hz, 3H), 2.28 (m, 1H), 2.31 (brs, 1H), 3.47 (m, 1H), 3.54 (dd, J = 3.6, 9.8 Hz, 1H), 3.62 (dd, J = 4.9, 9.8 Hz, 1H), 3.80(s, 3H), 3.81 (m, 2H), 3.95 (dd, J = 3.0, 6.1 Hz, 1H), 4.12 (m, 1H), 4.39 (d, J = 11.5 Hz, 1H), 4.47 (d, J = 11.5 Hz, 1H), 4.80(s, 2H), 6.81 (d, J = 8.6 Hz, 2H), 7.27 (m, 3H), 7.34 (d, J =8.0 Hz, 1H); ^{13}C NMR (100 Mz, $CDCl_3$) δ 13.6, 37.3, 55.5, 63.3, 67.8, 71.2, 71.6, 82.1, 82.4, 85.6, 114.0, 128.6, 129.5, 130.3, 130.6, 133.2, 137.1, 159.4; HRMS (CI) *m/z* 440.1161 ($[M]^+$; calcd for $C_{22}H_{26}O_5{}^{35}Cl_2$: 440.1157).



(2*R*,3*R*,4*S*,5*R*)-*tert*-Butyl-[5-(2,6-dichlorobenzyloxymethyl)-4-(4-methoxybenzyloxy)-3-methyltetrahydrofuran-2-yl-methoxy]dimethylsilane (22). To a solution of alcohol **21** (6.8 g, 15 mmol) in 50 mL of DMF was added TBSCl (2.60 g, 17 mmol) and imidazole (1.60 g, 20 mmol) at 0 °C. The mixture was stirred overnight at room temperature and DMF was removed under high vacuum. The residue was diluted with Et_2O and water, the phases were separated, and the aqueous phase was extracted with Et_2O (200 mL x 2). The combined organic layer was washed with brine and dried over Na_2SO_4, filtered and concentrated. Flash chromatography (hexane-ethyl acetate, 6:1) of the residue afforded 8.20 g (95%) of **22**: $[\alpha]_D^{23}$ –21.4 (c 1.51, $CHCl_3$); IR (neat) 2954, 2928, 2857, 1614, 1583, 1563, 1513, 1461, 1248, 1109, 1004, 837, 778, 768 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 0.05 (s, 6H), 0.89 (s, 9H), (d, J = 7.0 Hz, 3H), 2.18 (m, 1H), 3.48 (m, 1H), 3.60 (m, 2H), 3.72 (m, 2H), 3.81(s, 3H), 3.81 (m, 1H), 4.12 (m, 1H), 4.37 (d, J = 11.5 Hz, 1H), 4.49 (d, J = 11.5 Hz, 1H), 4.77(d, J = 10.8 Hz, 1H), 4.52 (d, J = 10.8 Hz, 1H), 6.81 (d, J = 8.6 Hz, 2H), 7.19 (m, 3H), 7.34 (d, J = 8.0 Hz, 1H); ^{13}C NMR (100 Mz, $CDCl_3$) δ -5.1, -5.2, 10.8, 26.2, 37.8, 55.4, 64.3, 67.9, 71.1, 81.9, 82.6, 85.1, 113.8, 128.6, 129.5, 130.0, 130.8, 133.7, 137.1, 159.2; HRMS (CI) *m/z* 553.1958 ($[M\text{-}H]^+$; calcd for $C_{28}H_{39}O_5{}^{35}Cl_2Si$: 553.1944).

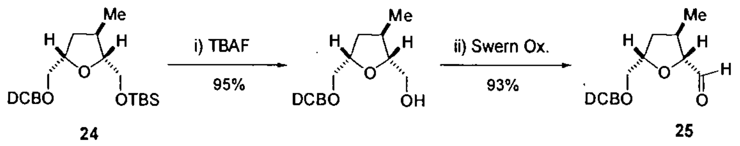


(2*R*,3*R*,4*S*,5*R*)-5-(*tert*-Butyldimethylsilanyloxymethyl)-2-(2,6-dichlorobenzyloxymethyl)-4-methyltetrahydrofuran-3-ol (23). To a solution of ether **22** (0.67 g, 1.20 mmol) in a mixture of CH_2Cl_2 (10 mL) and buffer (pH = 7, 0.5 mL) was added 2,3–dichloro-4, 5–dicyano–1,4–benzoquinone (0.32 g, 1.40 mmol) at 0 °C, and the mixture was stirred for 3 h at room temperature. The reaction was quenched by the addition of saturated aqueous $NaHCO_3$, and the mixture was diluted with CH_2Cl_2. The phases were separated and the aqueous phase was extracted with CH_2Cl_2. The combined organic phase was washed with brine, dried over Na_2SO_4, and concentrated under vacuum. Flash chromatography (hexane-ethyl acetate, 4:1) of the residue afforded 0.48 g (94%) of **23**: $[\alpha]_D^{23}$ −20.3(c 2.15, $CHCl_3$); IR (neat) 3439, 2954, 2928, 2883, 2857, 1582, 1564, 1471, 1462, 1437, 1252, 1197, 1108, 1003, 837, 777 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 0.04 (s, 6H), 0.9 (s, 9H), 1.02 (d, *J* = 7.1 Hz, 3H), 1.87 (d, *J* = 3.6 Hz, 1H), 2.12 (m, 1H), 3.43 (dd, *J* = 7.4, 9.8 Hz, 1H), 3.65 – 3.70 (m, 4H), 4.00 (m, 1H), 4.12 (m, 1H), 4.75 (d, *J* = 10.8 Hz, 2H), 4.81 (d, *J* = 10.9 Hz, 1H), 7.17 (dd, *J* = 7.4, 8.9 Hz, 1H), 7.31 (d, *J* = 8.1 Hz, 2H); ^{13}C NMR (100 Mz, $CDCl_3$) δ -5.0, 10.7, 18.8, 26.4, 38.8, 64.7, 68.1, 72.0, 76.9, 84.5, 85.1, 128.8, 130.3, 133.7, 137.3.



(2*R*,3*R*,4*S*,5*R*)-*tert*-Butyl-[5-(2,6-dichlorobenzyloxymethyl)-3-methyltetrahydrofuran-2-ylmethoxy]dimethylsilane (24). To a solution of alcohol **23** (0.42 g, 0.96 mmol) in 10 mL of dry toluene was added thiocarbonyldiimidazole (0.51 g, 2.9 mmol) at room temperature under argon. The mixture was heated at 100 °C for 3 h, then was cooled to room temperature and the solvent was removed under vacuum. Flash chromatography (hexane-ethyl acetate, 1:1) of the residue afforded 0.48 g (92%) of the thiocarbonylimidazolide: $[\alpha]_D^{23}$ −24.2 (c 0.73, $CHCl_3$); IR (neat) 3128, 2953, 2928, 2882, 2856, 1582, 1564, 1471, 1462, 1437, 1390, 1337, 1285, 1230, 1101, 1038, 976, 838, 778 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 0.08 (s, 6H), 0.90 (s, 9H), 1.08 (d, *J* = 7.0 Hz, 1H), 2.41 (m, 1H), 3.68 (dd, *J* = 4.2, 10.2 Hz, 1H), 3.73 –3.83 (m, 4H), 4.25 (td, *J* = 1.6, 4.0 Hz, 1H), 4.82 (s, 2H), 5.84 (dd, *J* = 1.6, 4.5 Hz, 1H), 7.03 (s, 1H), 7.19 (dd, *J* = 7.2, 8.8 Hz, 1H), 7.32 (dd, *J* = 1.0, 7.6 Hz, 2H), 7.59 (s, 1H), 8.32 (s, 1H); ^{13}C NMR (100 Mz, $CDCl_3$) δ -5.0, 10.9, 18.8, 26.4, 39.6, 64.8, 68.4, 71.3, 83.1, 85.8, 88.7, 118.2, 128.8, 130.4, 131.3, 133.3, 137.3, 183.8; HRMS (CI) *m/z* 545.1460 ($[M]^+$; calcd for $C_{24}H_{35}O_4N_2Si\,^{35}Cl_2S$: 545.1464).

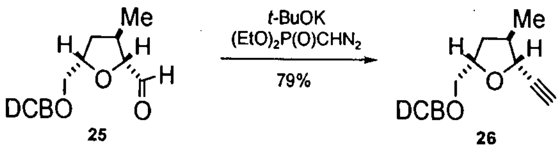
To a solution of *n*-Bu_3SnH (0.21 mL, 0.79 mmol) and a catalytic amount of AIBN in 30 mL of dry, deoxygenated toluene was slowly added a solution of the imidazolide obtained above (0.22 g, 0.39 mmol) in 10 mL of toluene at 100 °C via syringe during 30 min. The mixture was stirred for 5 min at 100 °C, then was cooled to room temperature and concentrated under vacuum. Flash chromatography (hexane-ethyl acetate, 10:1) of the residue afforded 0.16 g (96%) of **24** as a colorless oil: $[\alpha]_D^{23}$ −10.6 (c 0.32, $CHCl_3$); IR (neat) 2955, 2928, 2856, 1564, 1471, 1461, 1437, 1254, 1103, 836, 776 cm^{-1}; 1H NMR (300 MHz, $CDCl_3$) δ 0.05 (s, 6H), 0.89 (s, 9H), 1.02 (d, *J* = 6.7 Hz, 3H), 1.60 (m, 1H), 1.95 (m, 1H), 2.08 (m, 1H), 3.40-3.65 (m, 5H), 4.18 (m, 1H), 4.79 (d, *J* = 11.8 Hz, 1 H), 4.82 (d, *J* = 11.8 Hz, 1 H), 7.18 (dd, *J* = 7.4, 8.6 Hz, 1 H), 7.31 (d, *J* = 7.9 Hz, 2 H); ^{13}C NMR (100 Mz, $CDCl_3$) δ -4.9, 18.4, 18.8, 26.4, 35.0, 37.3, 65.5, 68.1, 74.0, 77.4, 87.2, 128.7, 130.2, 133.9, 137.3; HRMS (CI) *m/z* 417.1422 ($[M]^+$; calcd for $C_{20}H_{31}O_3\,^{35}Cl_2Si$: 417.1420).



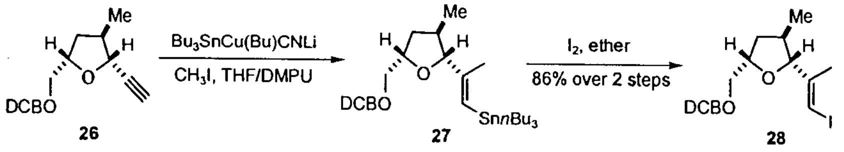
(2*R*,3*R*,5*R*)-5-(2,6-Dichlorobenzyloxymethyl)-3-methyltetrahydrofuran-2-carbaldehyde (25). To a solution of TBS ether **24** (1.10 g, 2.60 mmol) in 30 mL of THF was added a solution of TBAF in

THF (1 M, 3.7 mL) at 0 °C under argon, and the mixture was stirred for 2 h at room temperature. The reaction was quenched by addition of saturated aqueous NH_4Cl, and the mixture was diluted with EtOAc. The phases were separated and the aqueous phase was extracted with EtOAc (2 x). The organic layer was washed with brine, dried over Na_2SO_4, and concentrated under vacuum. Flash chromatography (hexane-ethyl acetate, 2:1) of the residue afforded 0.76 g (95%) of the alcohol as a colorless oil: $[\alpha]_D^{23}$ –6.1 (c 0.72, $CHCl_3$); IR (neat) 3443, 2956, 2924, 1872, 1582, 1564, 1456, 1437, 1379, 1198, 1102, 779, 767 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 1.05 (d, *J* = 6.7 Hz, 3H), 1.64 (ddd, *J* = 7.8, 12.4, 15.6 Hz, 1H), 2.03 (ddd, *J* = 3.6, 9.8 Hz, 1H), 3.55 (m, 1H), 3.62 (dd, *J* = 4.1, 9.8 Hz, 1H), 3.75 (m, 1H), 4.23 (ddd, *J* = 4.6, 9.4, 12.5 Hz, 1H), 4.80(s, 2H), 7.15 – 7.35 (m, 3H); ^{13}C NMR (100 Mz, $CDCl_3$) δ 18.1, 34.1, 37.3, 63.9, 68.0, 73.6, 77.4, 87.4, 128.8, 130.4, 135.5, 137.3; HRMS (CI) *m/z* 305.0713 ($[M]^+$; calcd. for $C_{14}H_{19}O_3{}^{35}Cl_2$: 305.0711).

To a solution of oxalyl chloride (0.41 mL, 4.6 mmol) in CH_2Cl_2 (4 mL) was added a solution of DMSO (0.65 mL, 9.24 mmol) in CH_2Cl_2 (2 mL) at –78 °C. The mixture was stirred for 5 min, and a solution of the alcohol obtained above (0.71 g, 2.3 mmol) in CH_2Cl_2 (14 mL) was added. The mixture was stirred for 15 min at –78 °C, Et_3N (1.3 mL, 9.2 mmol) was added, and the resulting mixture was allowed to warm to room temperature slowly. The reaction was quenched by addition of saturated aqueous $NaHCO_3$, and the mixture was diluted with CH_2Cl_2. The phases were separated, the aqueous phase was extracted with CH_2Cl_2 (2 x), and the combined organic layer was washed with brine, dried over Na_2SO_4, and concentrated under vacuum. Flash chromatography (hexane-ethyl acetate, 4:1) of the residue afforded 0.66 mg (93%) of **25**: $[\alpha]_D^{23}$ +22.8 (c 0.24, $CHCl_3$); IR (neat) 2958, 2923, 1852, 1731, 1461, 1436, 1277, 1101, 767, 728 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 1.70 (m, 1H), 1.95 (m, 1H), 2.35 (m, 1H), 3.59 (dd, *J* = 1.7, 4.76 Hz, 2H), 3.75 (dd, *J* = 2.5, 7.5 Hz, 1H), 4.38 (m, 1H), 4.82 (d, *J* = 1.8 Hz, 2H), 7.15-7.35 (m, 3H), 9.62 (d, *J* = 2.5 Hz, 1 H); ^{13}C NMR (100 Mz, $CDCl_3$) δ 17.9, 36.4, 36.9, 68.1, 73.5, 79.4, 90.2, 128.8, 130.4, 133.6, 137.3, 203.6; HRMS (FAB) *m/z* 303.0561 ($[M]^+$; calcd for $C_{14}H_{17}O_3{}^{35}Cl_2$: 303.0555).

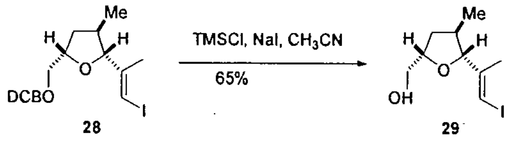


(2*R*,3*R*,5*R*)-5-(2,6-Dichlorobenzyloxymethyl)-2-ethynyl-3-methyltetrahydrofuran (26). To a solution of potassium *tert*-butoxide (1M in *i*-PrOH, 4.3 mL, 4.3 mmol) in THF (10 mL) was added a solution of diethyl (diazomethyl)phosphonate (0.77 g, 4.3 mmol) in THF (2 mL) at –78 °C. The resulting mixture was stirred for 5 min, and aldehyde **25** (0.66 g, 2.2 mmol) was added slowly. The mixture was allowed to slowly warm to room temperature, after which the reaction was quenched by addition of water (5 mL). The solvent was removed and the residue was partitioned between EtOAc and water. The phases were separated, the aqueous phase was extracted with EtOAc (2 x), and the combined organic layer was washed with brine, dried over Na_2SO_4, and concentrated under vacuum. Flash chromatography (hexane-ethyl acetate, 15:1) of the residue afforded 0.51 g (79%) of **26**: $[\alpha]^{23}_{D}$ –20.7 (c 1.04, $CHCl_3$); IR (neat) 3302, 2961, 2931, 2874, 1735, 1563, 1437, 1197, 1102, 779, 767 cm^{-1}; 1H NMR (300 MHz, $CDCl_3$) δ 1.13 (d, *J* = 6.7 Hz, 3H), 1.70 (m, 1H), 2.09 (m, 1H), 2.38 (m, 1H), 2.48 (d, *J* = 1.9 Hz, 1H), 3.55 (dd, *J* = 5.8, 9.9 Hz, 1H), 3.70 (dd, *J* = 6.0, 10.0 Hz, 1H), 4.07 (dd, *J* = 2.2, 7.5 Hz, 1H), 4.25 (m, 1H), 4.85 (d, *J* = 10.7 Hz, 1H), 4.90 (d, *J* = 10.7 Hz, 1H), 7.21 (dd, *J* = 7.7, 8.4 Hz, 1H), 7.35 (d, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 Mz, $CDCl_3$) δ 17.1, 36.8, 41.1, 66.3, 68.2, 73.5, 75.5, 83.5, 128.8, 130.3, 135.7, 137.3; HRMS (CI) *m/z* 298.0524($[M]^+$; calcd for $C_{15}H_{16}O_2Cl_2$: 298.0527).



(2*R*,3*R*,4*S*,5*R*)-5-(2,6-Dichlorobenzyloxymethyl)-2-(2-iodo-1-methylvinyl)-3-methyltetrahydrofuran (28). To flame-dried CuCN (33 mg, 0.37 mmol) was added THF (1.5 mL)under argon, and the mixture was cooled to –78 °C. *n*-BuLi (2.58 M, 0.29 mL, 0.74 mmol) was added slowly, and the resulting mixture was stirred for 10 min at –65 °C. The suspension gradually became a homogenous, colorless solution which was recooled to –78 °C. To this solution was added *n*-Bu_3SnH (0.13 mL, 0.74 mmol), and the resulting mixture was stirred for 10 min, during which time the solution became yellow. A solution of alkyne **26** (32 mg, 0.11 mmol) in THF (0.5 mL) was slowly added to the reaction mixture which was stirred for 30 min at –78 °C. MeI (50 μL) and DMPU (80 μL) were added, and the mixture was allowed to warm slowly to room temperature. The reaction was quenched by addition of a solution of saturated $NH_4Cl/NH_3 \cdot H_2O$ (9/1), and the mixture was diluted with EtOAc. The phases were separated and the aqueous phase was extracted with EtOAc (2 x). The organic layer was washed with brine, dried over Na_2SO_4, and concentrated under vacuum to give the crude stannane **27**.

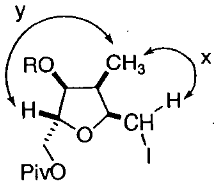
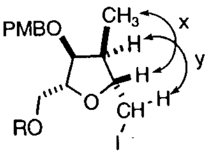
To a solution of the crude stannane **27** in THF (1 mL) was added I_2 (28 mg, 0.11 mmol) at 0 °C, and the mixture was stirred for 30 min at room temperature. The mixture was diluted with water and extracted with EtOAc, and the organic layer was washed with brine, dried over Na_2SO_4, and concentrated under vacuum. Flash chromatography (hexane-ethyl acetate, 40:1) of the residue afforded 40 mg (86%) of **28**: $[\alpha]_D^{23}$ –13.8 (c 0.16, $CHCl_3$); IR (neat) 2957, 2928, 1563, 1437, 1197, 1102, 1044, 777, 768 cm^{-1}; 1H NMR (300 MHz, $CDCl_3$) δ 1.01 (d, *J* = 6.4 Hz, 3H), 1.66 (m, 1H), 1.82 (s, 3H), 2.05 (m, 2H), 3.60 (m, 2H), 3.90 (d, *J* = 8.7 Hz, 1H), 4.25 (m, 1H), 4.81 (d, *J* = 10.8 Hz, 1H), 4.87 (d, *J* = 10.7 Hz, 1H), 6.29 (s, 1H), 7.21 (m, 1H), 7.36 (d, *J* = 8.1 Hz, 2H); ^{13}C NMR (100 Mz, $CDCl_3$) δ 17.4, 20.1, 36.9, 37.2, 68.0, 73.7, 77.8, 79.1, 91.1, 128.8, 130.3, 133.8, 137.3, 147.6; HRMS (CI) *m/z* 298.0524 ($[M]^+$; calcd for $C_{15}H_{16}O_2Cl_2$: 298.0527).



(2*S*,4*R*,5*R*)-[5-(2-Iodo-1-methylvinyl)-4-methyltetrahydrofuran-2-yl]methanol (29). To a solution of iodide **28** (9.0 mg, 0.020 mmol) in dry CH_3CN (0.2 mL) was added NaI (9.0 mg, 0.064

mmol) and TMSCl (8 μL, 0.063 mmol) at 0 °C under argon. The resulting mixture was stirred for 3 h at room temperature, after which the reaction was quenched by addition of saturated aqueous $NaHCO_3$ and the mixture was diluted with EtOAc. The phases were separated, the aqueous phase was extracted with EtOAc (2 x), and the combined organic layer was washed with brine, dried over Na_2SO_4, and concentrated under vacuum. Flash chromatography (hexane-ethyl acetate, 4:1) of the residue returned 1.2 mg of **28** and afforded 3.7 mg (65%) of **29**: $[\alpha]_D^{23}$ −10.0 (c 0.33, $CHCl_3$); IR (neat) 3420, 2956, 2923, 2853, 1456, 1378, 1282, 1104, 1044, 777 cm^{-1}; 1H NMR (300 MHz, $CDCl_3$) δ 1.01 (d, *J* = 6.7 Hz, 3H), 1.65 (m, 1H), 1.84 (s, 3H), 1.95 (m, 1H), 2.05 (m, 1H), 3.55 (m, 1H), 3.70 (m, 1H), 3.90 (d, *J* = 8.6 Hz, 1H), 4.15 (m, 1H), 6.29 (s, 1H); ^{13}C NMR (100 Mz, $CDCl_3$) δ 17.4, 20.0, 36.0, 37.5, 66.0, 78.7, 79.6, 91.2, 147.6; HRMS (CI) *m/z* 283.0197 ($[M]^+$; calcd for $C_{15}H_{16}O_2Cl_2$: 283.0195).

Table 2. Nuclear Overhauser Enhancements (nOe) of Proton Signals in the NMR Spectra of Tetrahydrofurans.

compound	R	nOe (%)	
		x	y
	H	2.8	1.8
	PMB	4.0	–
	Piv	3.0	–
	DCB	2.5	1.6

X-Ray Crystallographic Analysis of 19 (Sample gw121701)

Experimental details for gw121701.

The crystalline sample used was that supplied by Mr. Guoqiang Wang. Determination of the crystallographic parameters, data collection and structure solution and refinement was done as described elsewhere,[1] with the following details:

From a mass of crystals, a well shaped crystal of dimensions 0.3x 0.02 x 0.02 mm^3 was selected and mounted on the tip of a thin glass fiber using epoxy glue. An automated routine was used to find and center 12 reflections with $3° < \theta < 12.5°$, with which the crystal was indexed. The reflection list was then expanded to include 68 reflections with $6.605° < \theta < 39.513°$, and the lattice parameters refined against this list. All unique data, including a small set of redundant reflections, were collected (-5 – 5,-17 – 17,-38 – 38). Monitoring of three strong reflections as intensity standards during data collection showed no decay. Correction for the effects of absorption anisotropy was carried out by means of multiscans[2] as programmed in WinGX v1.64.[3]

The structure was solved using direct methods as programmed in SHELXS-90,[4] which revealed the positions of the three ring systems in the molecule. The solution was refined using the program SHELXL-97,[5] followed by Fourier synthesis, which revealed the positions of the remaining atoms. It became apparent that at least the iodine atoms were disordered, and a physically reasonable model included inclusion of several split atoms (O2, C6, C7 and I were split into components 1 and 2) which required the use of 22 restraints (geometric and displacement parameter restraints). The refined occupancy for the disordered groups was about 80%-20%. Hydrogen atoms were placed in geometrically idealized positions. The hydrogen atoms were given a displacement parameter equal to 1.5 times (methyl group) or 1.2 times (all other hydrogens) the equivalent isotropic displacement parameter of the atom it is attached to. During the final cycle of least squares refinement, all non-hydrogen atoms were refined with anisotropic displacement parameters. The refined value of the absolute structure parameter (Flack parameter)[6] of 0.000(16) indicates that the model obtained accurately depicts the absolute structure of the molecule.

An ORTEP[7] of the final model is given in Figure 1, with displacement ellipsoids drawn at the 20% probability level. An illustration of the model employed to fit the disordered groups is given in Figure 2.

All publications arising from this report must either include Dr. Yokochi as a coauthor or acknowledge Dr. Yokochi and the Molecular Structure Facility Core of the Marine/Freshwater Biomedical Sciences Center at Oregon State University, and include an acknowledgement for funding of this research facility using a variant of the

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[1] Blakemore, P. R., Kim, S.-K., Schulze, V. K., White, J. D., Yokochi, A. F. T. *J. Chem. Soc. Perkin Trans. 1* 2001, 1831-1847.

[2] Blessing, R. H. *Acta Crystallogr., Sect A* **1995**, *51*, 33-38.

[3] Farrugia, L. J. *J. Appl. Crystallogr.*, **1999**, *32*, 837-838.

[4] Sheldrick, G. M. *Acta Crystallogr.* **1990**, *A46*, 467.

[5] Sheldrick, G. M. In *Crystallographic Computing 6*; Flack, H. D., Parkanyi, L., Simon, K., Eds.; Oxford University Press: Oxford, 1993.

[6] Flack, H. D. *Acta Crystallogr.* **1983**, *A39*, 876-881.

[7] ORTEP-III - Burnett, M. N.; Johnson, C.K., Report ORNL-6895. Oak Ridge National Laboratory, Oak Ridge, Tennessee, 1996. ORTEP3 for Windows - Farrugia, L. J. *J. Appl. Crystallogr.* **1997**, *30*, 565.

following sentence: "AY wishes to acknowledge the Marine/Freshwater Biomedical Sciences Center at Oregon State University for partial funding under grant No. P30-ES03850". Drafts must be supplied to Dr. Yokochi prior to journal submission. In addition, if Dr. Yokochi is not included as a co-author in the publication, this will be considered tacit permission for publication of the details of the structural determination in an appropriate section of *Acta Crystallographica*.. A CIF (Crystallographic Information File), SFT (Structure Factor Table) and PDB (Cartesian Coordinates) corresponding to this work can also be provided.

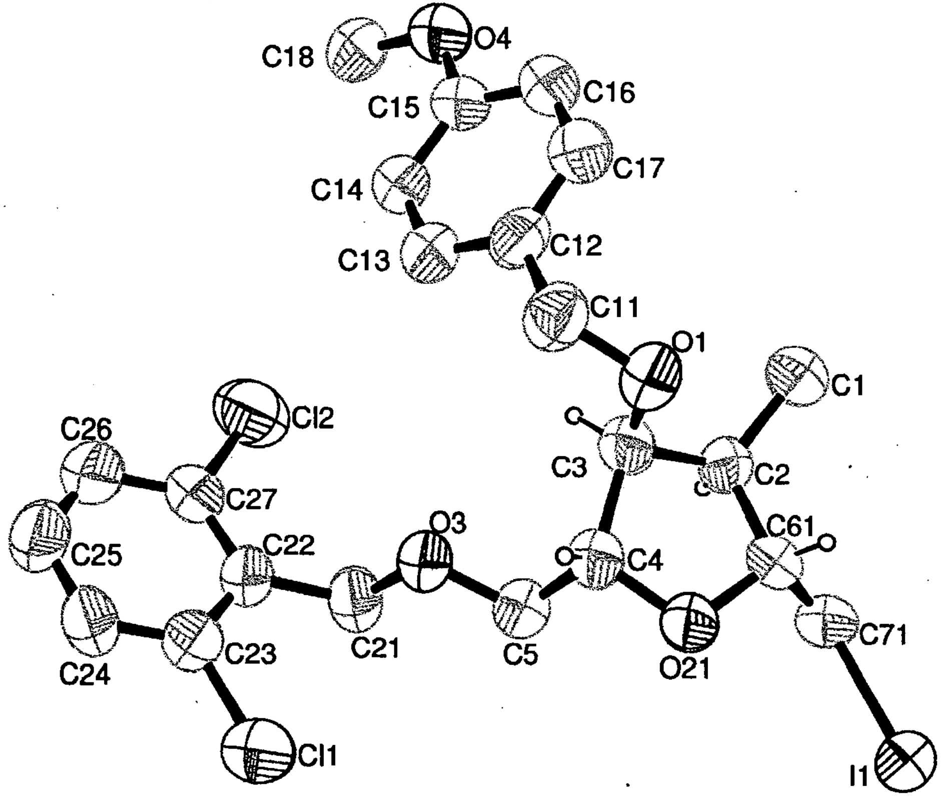

Figure 1: ORTEP with 20% displacement ellipsoids.

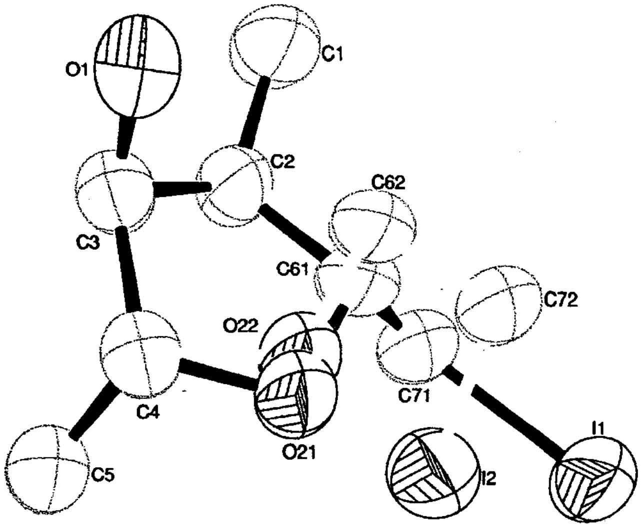

Figure 2. Illustration of the model used to fit the disorder problem.

Table 1. Crystal data and structure refinement for gw121701.

Identification code	gw121701	
Empirical formula	$C_{22}H_{25}Cl_2IO_4$	
Formula weight	551.22	
Temperature	293(2) K	
Wavelength	1.54170 Å	
Crystal system	Orthorhombic	
Space group	$P2_12_12_1$ (#18)	
Unit cell dimensions	a = 4.987(1) Å	α= 90°.
	b = 14.790(1) Å	β= 90°.
	c = 32.050(3) Å	γ = 90°.
Volume	2363.9(5) $Å^3$	
Z	4	
Density (calculated)	1.549 Mg/m^3	
Absorption coefficient	12.926 mm^{-1}	
F(000)	1104	
Crystal size	0.3 x 0.02 x 0.02 mm^3	
Theta range for data collection	2.76 to 67.69°.	
Index ranges	-5<=h<=5, -17<=k<=17, -38<=l<=38	
Reflections collected	4406	
Independent reflections	3681 [R(int) = 0.0317]	
Completeness to theta = 67.69°	94.7 %	
Absorption correction	Semi-empirical (SORTAV)	
Max. and min. transmission	0.3434 and 0.0139	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3681 / 22 / 301	
Goodness-of-fit on F^2	0.952	
Final R indices [I>2sigma(I)]	R1 = 0.0488, wR2 = 0.1297	
R indices (all data)	R1 = 0.1207, wR2 = 0.1918	
Absolute structure parameter	0.000(16)	
Extinction coefficient	0.00067(15)	
Largest diff. peak and hole	0.141 and -0.190 e.$Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for gw121701. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Cl(1)	173(11)	4542(3)	7825(1)	229(2)
Cl(2)	-6081(11)	7256(3)	7253(1)	248(2)
C(1)	-2980(30)	5118(7)	5208(3)	201(5)
C(2)	-2640(20)	4747(6)	5641(3)	148(3)
C(3)	-1280(30)	5302(8)	5965(3)	152(4)
C(4)	-470(30)	4582(8)	6296(3)	176(4)
C(5)	-2370(30)	4507(7)	6650(3)	171(4)
O(21)	-360(150)	3725(10)	6098(4)	193(5)
C(61)	-1340(40)	3814(10)	5676(4)	189(8)
C(71)	-3470(50)	3103(11)	5582(5)	210(9)
I(1)	-1997(4)	1744(1)	5598(1)	236(1)
O(22)	-300(700)	3850(40)	6010(20)	193(6)
C(62)	-600(130)	3990(40)	5570(20)	189(8)
C(72)	-1160(180)	3010(40)	5440(20)	209(9)
I(2)	-4412(16)	2414(5)	5806(2)	239(4)
C(11)	1890(30)	6499(8)	5993(4)	178(4)
C(12)	0(30)	7284(8)	5951(5)	168(4)
C(13)	-1480(30)	7593(9)	6283(4)	182(5)
C(14)	-3260(30)	8319(8)	6243(4)	165(4)
C(15)	-3470(30)	8706(9)	5862(4)	170(5)
C(16)	-2180(30)	8399(9)	5523(4)	180(5)
C(17)	-410(30)	7696(10)	5567(4)	185(5)
C(18)	-6760(30)	9758(8)	6111(4)	197(5)
C(21)	-3940(30)	5293(9)	7235(3)	188(5)
C(22)	-2950(40)	5927(9)	7568(3)	172(4)
C(23)	-1000(30)	5642(9)	7859(5)	184(5)
C(24)	-160(50)	6196(13)	8163(4)	232(8)
C(25)	-980(50)	7062(12)	8193(5)	232(9)
C(26)	-2930(40)	7382(11)	7916(5)	215(7)
C(27)	-3760(30)	6806(11)	7602(4)	177(4)
O(1)	950(20)	5696(5)	5776(3)	175(3)

O(3)	-2007(19)	5280(5)	6901(2)	174(3)
O(4)	-5130(20)	9428(7)	5779(3)	195(3)

Table 3. Bond lengths [Å] and angles [°] for gw121701.

Cl(1)-C(23)	1.733(14)	C(25)-C(26)	1.40(2)
Cl(2)-C(27)	1.740(13)	C(26)-C(27)	1.383(19)
C(1)-C(2)	1.503(11)		
C(2)-C(3)	1.488(13)	C(3)-C(2)-C(1)	119.7(9)
C(2)-C(61)	1.529(15)	C(3)-C(2)-C(61)	104.6(10)
C(2)-C(62)	1.529(16)	C(1)-C(2)-C(61)	116.5(9)
C(3)-O(1)	1.392(13)	C(3)-C(2)-C(62)	102(3)
C(3)-C(4)	1.555(14)	C(1)-C(2)-C(62)	101(3)
C(4)-O(21)	1.420(12)	C(61)-C(2)-C(62)	22(3)
C(4)-O(22)	1.420(14)	O(1)-C(3)-C(2)	106.9(9)
C(4)-C(5)	1.483(15)	O(1)-C(3)-C(4)	112.2(11)
C(5)-O(3)	1.409(11)	C(2)-C(3)-C(4)	102.5(9)
O(21)-C(61)	1.44(2)	O(21)-C(4)-O(22)	14(5)
C(61)-C(71)	1.53(2)	O(21)-C(4)-C(5)	107(3)
C(71)-I(1)	2.141(15)	O(22)-C(4)-C(5)	118(10)
O(22)-C(62)	1.44(2)	O(21)-C(4)-C(3)	108.4(11)
C(62)-C(72)	1.53(2)	O(22)-C(4)-C(3)	95(4)
C(72)-I(2)	2.18(8)	C(5)-C(4)-C(3)	114.1(11)
C(11)-O(1)	1.456(12)	O(3)-C(5)-C(4)	107.0(10)
C(11)-C(12)	1.503(16)	C(4)-O(21)-C(61)	108.9(16)
C(12)-C(13)	1.373(17)	O(21)-C(61)-C(71)	111(2)
C(12)-C(17)	1.387(16)	O(21)-C(61)-C(2)	107.2(14)
C(13)-C(14)	1.398(16)	C(71)-C(61)-C(2)	108.1(15)
C(14)-C(15)	1.352(14)	C(61)-C(71)-I(1)	113.8(13)
C(15)-C(16)	1.340(16)	C(4)-O(22)-C(62)	121(5)
C(15)-O(4)	1.377(14)	O(22)-C(62)-C(72)	98(6)
C(16)-C(17)	1.372(16)	O(22)-C(62)-C(2)	91(10)
C(18)-O(4)	1.424(13)	C(72)-C(62)-C(2)	128(6)
C(21)-O(3)	1.440(13)	C(62)-C(72)-I(2)	113(5)
C(21)-C(22)	1.502(15)	O(1)-C(11)-C(12)	112.5(10)
C(22)-C(27)	1.366(16)	C(13)-C(12)-C(17)	117.5(14)
C(22)-C(23)	1.412(18)	C(13)-C(12)-C(11)	121.7(14)
C(23)-C(24)	1.340(17)	C(17)-C(12)-C(11)	120.7(15)
C(24)-C(25)	1.35(2)	C(12)-C(13)-C(14)	121.6(12)

C(15)-C(14)-C(13)	117.3(12)	C(24)-C(23)-Cl(1)	120.9(16)
C(16)-C(15)-C(14)	123.3(14)	C(22)-C(23)-Cl(1)	118.2(12)
C(16)-C(15)-O(4)	113.3(13)	C(23)-C(24)-C(25)	123(2)
C(14)-C(15)-O(4)	123.3(14)	C(24)-C(25)-C(26)	119.1(19)
C(15)-C(16)-C(17)	118.9(13)	C(27)-C(26)-C(25)	117.6(16)
C(16)-C(17)-C(12)	121.2(14)	C(22)-C(27)-C(26)	123.7(14)
O(3)-C(21)-C(22)	108.6(11)	C(22)-C(27)-Cl(2)	120.6(13)
C(27)-C(22)-C(23)	115.8(12)	C(26)-C(27)-Cl(2)	115.6(14)
C(27)-C(22)-C(21)	123.7(16)	C(3)-O(1)-C(11)	113.0(9)
C(23)-C(22)-C(21)	120.5(14)	C(5)-O(3)-C(21)	110.4(9)
C(24)-C(23)-C(22)	120.9(15)	C(15)-O(4)-C(18)	117.6(11)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2$x 10^3)for gw121701. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Cl(1)	323(5)	176(3)	187(3)	26(2)	13(3)	28(3)
Cl(2)	300(5)	240(4)	206(3)	45(3)	29(3)	54(4)
C(1)	303(16)	163(9)	137(7)	19(6)	-36(10)	10(12)
C(2)	191(9)	128(6)	124(6)	-7(5)	-27(8)	3(7)
C(3)	192(11)	138(8)	125(7)	2(6)	11(8)	30(8)
C(4)	259(13)	141(8)	127(7)	-7(7)	12(9)	35(10)
C(5)	230(11)	139(7)	144(7)	-8(6)	-10(10)	7(10)
O(21)	307(11)	145(7)	127(8)	-3(7)	-18(19)	61(14)
C(61)	320(20)	137(11)	105(12)	17(7)	10(12)	5(13)
C(71)	330(30)	145(11)	152(12)	26(11)	3(17)	51(18)
I(1)	412(2)	139(1)	158(1)	-7(1)	-8(1)	39(1)
O(22)	306(11)	144(9)	129(9)	-2(8)	-20(20)	61(15)
C(62)	320(20)	138(12)	104(13)	18(9)	8(13)	5(14)
C(72)	330(30)	145(12)	152(13)	25(12)	4(18)	51(19)
I(2)	309(7)	190(6)	217(5)	1(4)	33(5)	-41(5)
C(11)	188(11)	163(10)	184(10)	-22(8)	-18(9)	-10(10)
C(12)	203(12)	124(8)	177(11)	-16(8)	-18(11)	-4(9)
C(13)	259(15)	134(8)	154(9)	-8(7)	-7(10)	11(11)
C(14)	213(12)	133(8)	149(8)	-7(7)	16(8)	-3(10)
C(15)	231(14)	132(8)	147(9)	16(7)	11(10)	-2(10)
C(16)	233(14)	160(10)	148(9)	5(8)	23(11)	7(11)
C(17)	217(13)	169(11)	169(11)	-2(10)	32(11)	4(11)
C(18)	247(15)	147(9)	198(11)	-19(8)	10(11)	26(11)
C(21)	251(14)	181(10)	131(7)	-23(8)	16(9)	-27(11)
C(22)	255(14)	139(9)	123(7)	-14(6)	14(10)	-21(11)
C(23)	255(15)	152(10)	147(9)	-2(8)	23(10)	-23(11)
C(24)	360(20)	212(15)	124(8)	-10(10)	-9(12)	-63(19)
C(25)	350(30)	176(15)	166(13)	-37(11)	50(15)	-53(17)
C(26)	300(20)	168(12)	174(12)	-8(11)	25(15)	3(15)
C(27)	238(13)	161(10)	131(8)	13(8)	8(8)	0(12)
O(1)	209(8)	147(6)	169(6)	-26(5)	10(6)	2(6)

O(3)	249(8)	141(5)	133(5)	-16(4)	18(6)	-5(7)
O(4)	258(9)	157(6)	169(7)	14(6)	22(7)	8(7)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for gw121701.

	x	y	z	U(eq)
H(1A)	-4040	4706	5045	302
H(1B)	-1257	5193	5080	302
H(1C)	-3876	5693	5222	302
H(2)	-4470	4664	5746	177
H(3)	-2481	5759	6083	182
H(4)	1313	4731	6405	211
H(5A)	-4201	4480	6548	205
H(5B)	-2009	3963	6810	205
H(61)	149	3763	5478	226
H(71A)	-4215	3221	5307	252
H(71B)	-4912	3162	5783	252
H(62)	1059	4223	5439	227
H(72A)	459	2659	5485	251
H(72B)	-1614	2990	5150	251
H(11A)	3629	6671	5882	214
H(11B)	2124	6359	6286	214
H(13)	-1297	7313	6541	219
H(14)	-4252	8528	6469	198
H(16)	-2483	8658	5263	216
H(17)	538	7492	5336	222
H(18A)	-7851	10246	6012	296
H(18B)	-5628	9970	6333	296
H(18C)	-7883	9280	6212	296
H(21A)	-4148	4690	7349	226
H(21B)	-5662	5494	7131	226
H(24)	1036	5975	8360	279
H(25)	-264	7442	8395	279
H(26)	-3640	7961	7942	259

Table 6. Torsion angles [°] for gw121701.

C(1)-C(2)-C(3)-O(1)	-42.8(14)	C(1)-C(2)-C(62)-O(22)	172(12)
C(61)-C(2)-C(3)-O(1)	89.9(11)	C(61)-C(2)-C(62)-O(22)	-52(14)
C(62)-C(2)-C(3)-O(1)	68(3)	C(3)-C(2)-C(62)-C(72)	148(6)
C(1)-C(2)-C(3)-C(4)	-161.0(12)	C(1)-C(2)-C(62)-C(72)	-88(7)
C(61)-C(2)-C(3)-C(4)	-28.3(13)	C(61)-C(2)-C(62)-C(72)	49(8)
C(62)-C(2)-C(3)-C(4)	-50(3)	O(22)-C(62)-C(72)-I(2)	52(14)
O(1)-C(3)-C(4)-O(21)	-92(4)	C(2)-C(62)-C(72)-I(2)	-45(8)
C(2)-C(3)-C(4)-O(21)	23(4)	O(1)-C(11)-C(12)-C(13)	109.9(14)
O(1)-C(3)-C(4)-O(22)	-87(14)	O(1)-C(11)-C(12)-C(17)	-67.2(16)
C(2)-C(3)-C(4)-O(22)	27(14)	C(17)-C(12)-C(13)-C(14)	-2.4(19)
O(1)-C(3)-C(4)-C(5)	148.5(11)	C(11)-C(12)-C(13)-C(14)	-179.6(12)
C(2)-C(3)-C(4)-C(5)	-97.2(12)	C(12)-C(13)-C(14)-C(15)	0.2(19)
O(21)-C(4)-C(5)-O(3)	167(2)	C(13)-C(14)-C(15)-C(16)	4(2)
O(22)-C(4)-C(5)-O(3)	176(10)	C(13)-C(14)-C(15)-O(4)	-179.6(11)
C(3)-C(4)-C(5)-O(3)	-73.2(14)	C(14)-C(15)-C(16)-C(17)	-5(2)
O(22)-C(4)-O(21)-C(61)	-26(65)	O(4)-C(15)-C(16)-C(17)	178.2(11)
C(5)-C(4)-O(21)-C(61)	117(4)	C(15)-C(16)-C(17)-C(12)	2(2)
C(3)-C(4)-O(21)-C(61)	-7(5)	C(13)-C(12)-C(17)-C(16)	1(2)
C(4)-O(21)-C(61)-C(71)	-129(4)	C(11)-C(12)-C(17)-C(16)	178.5(12)
C(4)-O(21)-C(61)-C(2)	-12(5)	O(3)-C(21)-C(22)-C(27)	96.8(16)
C(3)-C(2)-C(61)-O(21)	26(3)	O(3)-C(21)-C(22)-C(23)	-80.5(14)
C(1)-C(2)-C(61)-O(21)	160(3)	C(27)-C(22)-C(23)-C(24)	4(2)
C(62)-C(2)-C(61)-O(21)	111(9)	C(21)-C(22)-C(23)-C(24)	-178.4(14)
C(3)-C(2)-C(61)-C(71)	145.6(12)	C(27)-C(22)-C(23)-Cl(1)	-178.5(10)
C(1)-C(2)-C(61)-C(71)	-79.8(16)	C(21)-C(22)-C(23)-Cl(1)	-1.0(17)
C(62)-C(2)-C(61)-C(71)	-129(9)	C(22)-C(23)-C(24)-C(25)	-4(3)
O(21)-C(61)-C(71)-I(1)	-64(3)	Cl(1)-C(23)-C(24)-C(25)	178.4(16)
C(2)-C(61)-C(71)-I(1)	178.4(8)	C(23)-C(24)-C(25)-C(26)	5(3)
O(21)-C(4)-O(22)-C(62)	168(93)	C(24)-C(25)-C(26)-C(27)	-5(3)
C(5)-C(4)-O(22)-C(62)	127(20)	C(23)-C(22)-C(27)-C(26)	-5(2)
C(3)-C(4)-O(22)-C(62)	6(26)	C(21)-C(22)-C(27)-C(26)	177.8(14)
C(4)-O(22)-C(62)-C(72)	-163(23)	C(23)-C(22)-C(27)-Cl(2)	178.0(9)
C(4)-O(22)-C(62)-C(2)	-35(25)	C(21)-C(22)-C(27)-Cl(2)	1(2)
C(3)-C(2)-C(62)-O(22)	48(12)	C(25)-C(26)-C(27)-C(22)	5(2)

C(25)-C(26)-C(27)-Cl(2)	-177.3(13)	C(4)-C(5)-O(3)-C(21)	175.5(10)
C(2)-C(3)-O(1)-C(11)	158.3(9)	C(22)-C(21)-O(3)-C(5)	161.5(11)
C(4)-C(3)-O(1)-C(11)	-90.0(11)	C(16)-C(15)-O(4)-C(18)	175.1(12)
C(12)-C(11)-O(1)-C(3)	-70.1(13)	C(14)-C(15)-O(4)-C(18)	-2.1(19)

Symmetry transformations used to generate equivalent atoms: