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2,3,4,6-Tetra-*O*-acetyl-β-D-galacto-pyranosyl 2,3,4,6-tetra-*O*-acetyl-β-D-glucopyranosyl disulfide tetrahydrofuran solvate

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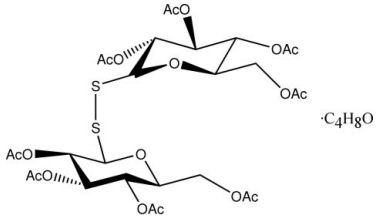
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.010 Å; disorder in main residue; R factor = 0.054; wR factor = 0.151; data-to-parameter ratio = 14.9.

The asymmetric unit of title compound, $C_{28}H_{38}O_{18}S_2\cdot C_4H_8O$, comprises one disulfide-bridged sugar molecule and one solvent molecule. No significant differences in structural parameters are found between the present structure and the previously determined unsolvated form [Brito, López-Rodríguez, Bényei & Szilagyi (2006). *Carbohydr. Res.* **341**, 2967–2972]. The compounds are characterized by a compact structure with spatial proximity of the two pyranosyl rings. One of the carbonyl atoms is disordered over two sites [site occupancy = 0.69 (7) for major component] and the displacement parameters for the THF species are unsually large.

Related literature

For analysis of conformation, see: Cremer & Pople (1975). For the synthesis, see: Szilágyi *et al.* (2001). For background to disulfide linkage and diglycosyl disulfides, see: André *et al.* (2006); Chakka *et al.* (2005); Pérez *et al.* (1978); Szilágyi & Varela (2006). For the structure of the unsolvated form, see: Brito *et al.* (2006).



Experimental

Crystal data

$C_{28}H_{38}O_{18}S_2\cdot C_4H_8O$
M_r = 798.81
Monoclinic, $P2_1$
a = 14.6499 (14) Å
b = 10.0096 (10) Å
c = 15.4029 (15) Å
β = 113.573 (2)°
V = 2070.2 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.20 mm^{-1}
T = 298 (2) K
0.40 × 0.30 × 0.20 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
T_{min} = 0.920, T_{max} = 0.960
13257 measured reflections
7410 independent reflections
4763 reflections with $I > 2\sigma(I)$
R_{int} = 0.046

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.151
S = 0.85
7410 reflections
497 parameters
45 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}
Absolute structure: Flack, (1983), 3438 Friedel pairs
Flack parameter: 0.02 (9)

Table 1
Selected torsion angle (°).

C1—S1—S2—C21	−80.25 (19)

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2332).

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supporting information

Acta Cryst. (2008). E**64**, o2472–o2473 [doi:10.1107/S1600536808039494]

2,3,4,6-Tetra-*O*-acetyl-*β*-D-galactopyranosyl 2,3,4,6-tetra-*O*-acetyl-*β*-D-gluco-pyranosyl disulfide tetrahydrofuran solvate

Iván Brito, Lászlo Szilágyi and Matías López-Rodríguez

S1. Comment

The disulfide linkage which plays an essential role in stabilizing the tertiary structure of proteins, was not known in carbohydrate chemistry until recently when it was introduced as a new interglycosidic connecting element (Szilágyi & Varela, 2006). Diglycosyl disulfides were proposed as novel carbohydrate scaffolds with potential biological activity (Szilágyi & Varela, 2006, Chakka *et al.*, 2005). This has recently been demonstrated in binding studies with galectins and in assays with tumor cells (André *et al.*, 2006). We report herein the crystal and molecular structure of the title compound, (I), Fig. 1. The averaged bond lengths [C—O 1.429 (6), O—C 1.341 (8), C=O 1.181 (8), C—C 1.478 (10) Å] and other parameters, Table 1, compare well with those for the previously determined unsolvated form (Brito *et al.*, 2006). The pyranosyl rings adopt chair conformations with Cremer & Pople (1975) puckering parameters: Q_T = 0.604 (6) (Glc) and 0.566 (5) Å (Gal), θ = 2.4 (5) (Glc) and 9.6 (6)°(Gal), φ = 15 (11) (Glc) and 9(4)° (Gal); these parameters are similar to those for the unsolvated form. The conformations of the acetyl groups are in agreement with the observation that in acetylated pyranoses, the carbonyl C=O bonds tend to align so that they nearly eclipse the axial-H atoms atoms on the common ring C atoms.

S2. Experimental

Compound (I) was synthesized as described by Szilágyi *et al.* (2001).

S3. Refinement

The H atoms were geometrically placed (C—H = 0.96–0.98 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl-C). The O29 atom was refined over two sites using a disorder model, with occupancies of 0.69 (7) and 0.31 (7). The THF molecule is disordered and was modelled with restraints for distances and anisotropic displacement parameters using a 'rigid-bond′ restraint to U^{ij}, implemented as the DELU instruction in *SHELXL97* (Sheldrick, 2008).

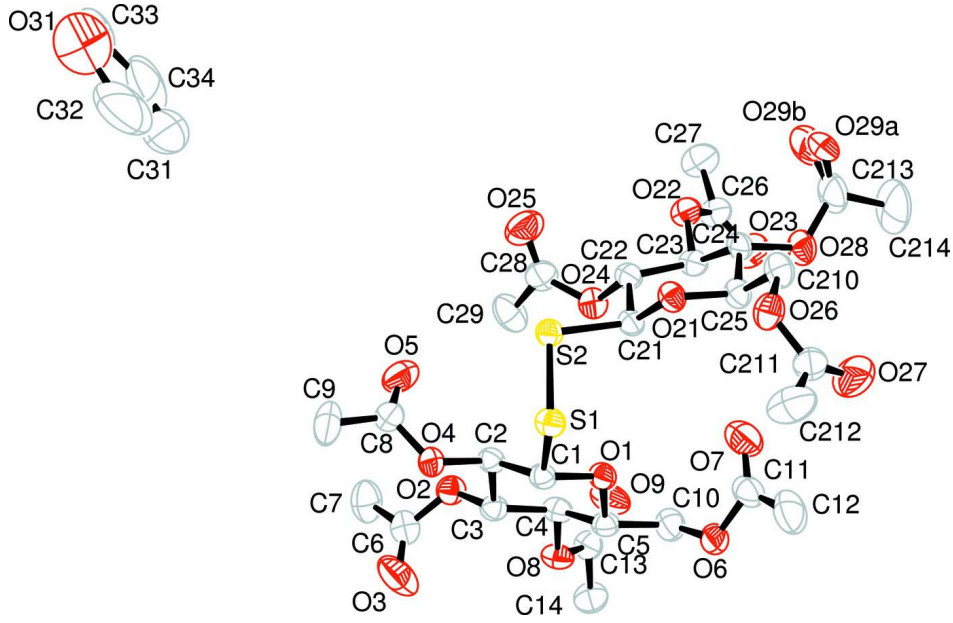

Figure 1

The molecular structure of (I), with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. All H atoms have been omitted for clarity.

2,3,4,6-Tetra-*O*-acetyl-*β*-D-galactopyranosyl 2,3,4,6-tetra-*O*-acetyl-*β*-D-glucopyranosyl disulfide tetrahydrofuran solvate

Crystal data

$C_{28}H_{38}O_{18}S_2 \cdot C_4H_8O$
$M_r = 798.81$
Monoclinic, $P2_1$
Hall symbol: P 2yb
$a = 14.6499\ (14)$ Å
$b = 10.0096\ (10)$ Å
$c = 15.4029\ (15)$ Å
$\beta = 113.573\ (2)°$
$V = 2070.2\ (4)$ Å^3
$Z = 2$
$F(000) = 844$
$D_x = 1.281$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4312 reflections
$\theta = 2.4–25.0°$
$\mu = 0.20$ mm^{-1}
$T = 298$ K
Prism, yellow
0.40 × 0.30 × 0.20 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ scans, and ω scans with κ offsets
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
$T_{min} = 0.920$, $T_{max} = 0.960$
13257 measured reflections
7410 independent reflections
4763 reflections with $I > 2\sigma(I)$
$R_{int} = 0.046$
$\theta_{max} = 25.2°$, $\theta_{min} = 2.5°$
$h = -17 \rightarrow 16$
$k = -12 \rightarrow 11$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.151$
$S = 0.85$
7410 reflections
497 parameters
45 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0875P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.042$
$\Delta\rho_{max} = 0.34$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ e Å^{-3}
Absolute structure: Flack, (1983), 3438 Friedel pairs
Absolute structure parameter: 0.02 (9)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of *F*^2^ against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on *F*^2^, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative *F*^2^. The threshold expression of *F*^2^ > σ(*F*^2^) is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on *F*^2^ are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
S1	0.94859 (8)	0.98543 (12)	0.93468 (8)	0.0609 (3)	
O1	0.8235 (2)	1.1214 (3)	0.98368 (19)	0.0587 (7)	
O2	0.5745 (2)	1.1063 (3)	0.7358 (2)	0.0692 (8)	
O3	0.5648 (4)	1.2681 (6)	0.6357 (3)	0.146 (2)	
O4	0.7645 (2)	1.0285 (3)	0.7398 (2)	0.0644 (8)	
O5	0.6752 (4)	0.8414 (4)	0.7054 (3)	0.1135 (14)	
O6	0.8497 (3)	1.2956 (4)	1.1307 (2)	0.0822 (9)	
O7	0.8713 (4)	1.0964 (5)	1.2001 (3)	0.1132 (14)	
O8	0.5850 (2)	1.2941 (3)	0.8764 (2)	0.0684 (8)	
O9	0.5145 (3)	1.1965 (4)	0.9648 (3)	0.1042 (13)	
C1	0.8424 (3)	1.0964 (4)	0.9008 (3)	0.0546 (10)	
H1	0.8612	1.1814	0.8809	0.066*	
C2	0.7471 (3)	1.0494 (4)	0.8225 (3)	0.0548 (10)	
H2	0.7253	0.9658	0.8412	0.066*	
C3	0.6682 (3)	1.1554 (4)	0.8047 (3)	0.0580 (11)	
H3	0.6874	1.2365	0.7806	0.07*	
C4	0.6549 (3)	1.1872 (5)	0.8953 (3)	0.0624 (11)	
H4	0.6301	1.1083	0.9166	0.075*	
C5	0.7552 (3)	1.2286 (4)	0.9704 (3)	0.0601 (11)	
H5	0.7793	1.3079	0.9487	0.072*	
C6	0.5307 (4)	1.1702 (7)	0.6531 (4)	0.0865 (16)	
C7	0.4395 (5)	1.1036 (8)	0.5895 (4)	0.116 (2)	

H7A	0.3886	1.1144	0.6135	0.174*	
H7B	0.4524	1.0102	0.5859	0.174*	
H7C	0.4175	1.1426	0.5275	0.174*	
C8	0.7260 (4)	0.9197 (6)	0.6875 (4)	0.0773 (14)	
C9	0.7533 (5)	0.9114 (8)	0.6047 (4)	0.116 (2)	
H9A	0.7772	0.8231	0.6009	0.175*	
H9B	0.8046	0.9754	0.6117	0.175*	
H9C	0.6959	0.9302	0.5478	0.175*	
C10	0.7520 (4)	1.2569 (6)	1.0644 (3)	0.0802 (15)	
H10A	0.7049	1.3282	1.058	0.096*	
H10B	0.7302	1.1779	1.0873	0.096*	
C11	0.9038 (5)	1.2052 (7)	1.1941 (4)	0.0896 (17)	
C12	1.0060 (5)	1.2548 (8)	1.2510 (4)	0.122 (2)	
H12A	1.0442	1.1855	1.2929	0.183*	
H12B	1.0025	1.3309	1.2875	0.183*	
H12C	1.0374	1.2801	1.2094	0.183*	
C13	0.5199 (3)	1.2900 (6)	0.9193 (3)	0.0704 (12)	
C14	0.4608 (4)	1.4138 (5)	0.9033 (4)	0.0890 (16)	
H14A	0.4028	1.3978	0.9161	0.133*	
H14B	0.4408	1.4417	0.8386	0.133*	
H14C	0.5005	1.4826	0.9447	0.133*	
S2	0.89370 (8)	0.80485 (11)	0.95029 (7)	0.0611 (3)	
O21	0.98609 (19)	0.7760 (3)	1.13641 (18)	0.0584 (7)	
O22	0.7552 (2)	0.5961 (3)	1.1697 (2)	0.0684 (8)	
O23	0.6851 (3)	0.7007 (4)	1.2553 (3)	0.0950 (11)	
O24	0.7182 (2)	0.7573 (3)	1.0062 (2)	0.0678 (8)	
O25	0.6962 (4)	0.5714 (5)	0.9230 (3)	0.1235 (16)	
O26	1.1652 (2)	0.8389 (4)	1.2729 (2)	0.0751 (9)	
O27	1.1690 (4)	0.9965 (5)	1.3749 (4)	0.1336 (17)	
O28	0.9183 (2)	0.7190 (4)	1.3348 (2)	0.0803 (9)	
O29A	0.988 (5)	0.519 (4)	1.3922 (18)	0.103 (15)	0.31 (7)
O29B	0.921 (4)	0.5063 (16)	1.3697 (18)	0.155 (9)	0.69 (7)
C21	0.8909 (3)	0.8131 (4)	1.0674 (3)	0.0531 (9)	
H21	0.8739	0.9037	1.0799	0.064*	
C22	0.8144 (3)	0.7149 (4)	1.0712 (3)	0.0576 (10)	
H22	0.8288	0.626	1.0531	0.069*	
C23	0.8154 (3)	0.7094 (5)	1.1694 (3)	0.0601 (11)	
H23	0.7853	0.791	1.1815	0.072*	
C24	0.9198 (3)	0.6941 (4)	1.2442 (3)	0.0593 (11)	
H24	0.943	0.6028	1.2424	0.071*	
C25	0.9914 (3)	0.7911 (5)	1.2309 (3)	0.0593 (10)	
H25	0.974	0.8827	1.2407	0.071*	
C26	0.6918 (4)	0.6043 (5)	1.2123 (4)	0.0740 (13)	
C27	0.6347 (5)	0.4806 (6)	1.2017 (5)	0.1028 (18)	
H27A	0.584	0.494	1.2256	0.154*	
H27B	0.6785	0.4103	1.2367	0.154*	
H27C	0.6042	0.4566	1.136	0.154*	
C28	0.6681 (4)	0.6785 (6)	0.9334 (4)	0.0798 (14)	

C29	0.5757 (4)	0.7449 (7)	0.8683 (4)	0.105 (2)
H29A	0.5918	0.8306	0.8502	0.157*
H29B	0.5313	0.7564	0.8997	0.157*
H29C	0.5441	0.6907	0.8129	0.157*
C210	1.0967 (3)	0.7613 (5)	1.2966 (3)	0.0708 (13)
H21A	1.1056	0.7808	1.3612	0.085*
H21B	1.1102	0.6671	1.2928	0.085*
C211	1.1968 (4)	0.9552 (5)	1.3177 (4)	0.0830 (15)
C212	1.2625 (6)	1.0235 (7)	1.2826 (6)	0.128 (2)
H21C	1.2737	1.1132	1.3067	0.191*
H21D	1.2324	1.0257	1.2146	0.191*
H21E	1.3249	0.9769	1.3032	0.191*
C213	0.9328 (8)	0.6160 (10)	1.3954 (5)	0.115 (3)
C214	0.9212 (7)	0.6644 (11)	1.4834 (5)	0.174 (4)
H21F	0.9701	0.6219	1.5381	0.261*
H21G	0.8557	0.6426	1.4792	0.261*
H21H	0.9304	0.7595	1.4888	0.261*
O31	0.7418 (11)	0.0601 (14)	0.3640 (9)	0.306 (5)
C31	0.6827 (15)	0.2535 (13)	0.4405 (8)	0.301 (9)
H31A	0.6559	0.3386	0.4113	0.361*
H31B	0.71	0.265	0.5087	0.361*
C32	0.7705 (12)	0.2047 (18)	0.4071 (10)	0.291 (7)
H32A	0.7751	0.2654	0.3599	0.349*
H32B	0.8343	0.203	0.4608	0.349*
C33	0.6337 (11)	0.0251 (15)	0.3603 (6)	0.235 (5)
H33A	0.5878	0.0138	0.2947	0.282*
H33B	0.637	−0.059	0.3927	0.282*
C34	0.5917 (12)	0.1382 (19)	0.4080 (8)	0.281 (6)
H34A	0.5818	0.1036	0.4624	0.337*
H34B	0.5293	0.1746	0.363	0.337*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0625 (6)	0.0635 (6)	0.0677 (7)	0.0038 (6)	0.0377 (5)	0.0038 (6)
O1	0.0704 (18)	0.0570 (17)	0.0581 (17)	0.0048 (13)	0.0358 (15)	0.0102 (15)
O2	0.0641 (19)	0.0688 (19)	0.071 (2)	−0.0002 (16)	0.0228 (16)	−0.0020 (16)
O3	0.132 (4)	0.165 (5)	0.098 (3)	0.056 (3)	0.001 (3)	−0.036 (4)
O4	0.081 (2)	0.0657 (19)	0.0530 (17)	−0.0031 (15)	0.0335 (15)	−0.0036 (16)
O5	0.157 (4)	0.073 (3)	0.121 (3)	−0.025 (2)	0.066 (3)	−0.029 (3)
O6	0.110 (3)	0.070 (2)	0.0621 (19)	0.0011 (18)	0.0296 (19)	0.023 (2)
O7	0.144 (4)	0.103 (3)	0.106 (3)	0.029 (3)	0.064 (3)	0.023 (3)
O8	0.0714 (19)	0.0543 (17)	0.089 (2)	0.0138 (16)	0.0425 (17)	0.0106 (16)
O9	0.117 (3)	0.098 (3)	0.135 (3)	0.041 (3)	0.090 (3)	0.031 (2)
C1	0.063 (3)	0.051 (2)	0.059 (2)	0.004 (2)	0.033 (2)	0.000 (2)
C2	0.061 (3)	0.053 (2)	0.055 (2)	0.0059 (19)	0.028 (2)	−0.002 (2)
C3	0.056 (3)	0.054 (2)	0.062 (3)	0.002 (2)	0.021 (2)	−0.002 (2)
C4	0.063 (3)	0.058 (3)	0.073 (3)	0.011 (2)	0.034 (2)	0.007 (2)

C5	0.075 (3)	0.048 (2)	0.066 (3)	0.004 (2)	0.037 (2)	0.008 (2)
C6	0.088 (4)	0.098 (4)	0.071 (4)	0.003 (3)	0.030 (3)	0.001 (3)
C7	0.092 (4)	0.161 (6)	0.077 (4)	−0.009 (4)	0.016 (3)	−0.015 (4)
C8	0.090 (4)	0.069 (3)	0.067 (3)	−0.005 (3)	0.025 (3)	0.003 (3)
C9	0.137 (6)	0.140 (6)	0.069 (4)	−0.033 (4)	0.039 (4)	0.000 (5)
C10	0.087 (4)	0.087 (4)	0.071 (3)	0.001 (3)	0.036 (3)	0.020 (3)
C11	0.125 (5)	0.082 (4)	0.076 (4)	0.014 (3)	0.054 (4)	0.030 (4)
C12	0.116 (5)	0.149 (7)	0.079 (4)	0.012 (4)	0.017 (4)	0.024 (5)
C13	0.069 (3)	0.075 (3)	0.074 (3)	0.000 (3)	0.036 (2)	0.006 (3)
C14	0.084 (4)	0.081 (4)	0.108 (4)	−0.003 (3)	0.046 (3)	0.013 (3)
S2	0.0789 (7)	0.0549 (6)	0.0570 (6)	0.0025 (5)	0.0350 (5)	0.0060 (6)
O21	0.0597 (17)	0.0695 (19)	0.0547 (16)	0.0044 (13)	0.0320 (14)	0.0082 (14)
O22	0.080 (2)	0.0643 (18)	0.080 (2)	−0.0035 (16)	0.0514 (18)	−0.0061 (17)
O23	0.105 (3)	0.095 (3)	0.118 (3)	−0.019 (2)	0.079 (2)	−0.014 (2)
O24	0.0570 (18)	0.079 (2)	0.0675 (18)	−0.0007 (16)	0.0246 (16)	0.0021 (16)
O25	0.129 (4)	0.086 (3)	0.124 (4)	−0.026 (3)	0.017 (3)	−0.016 (3)
O26	0.070 (2)	0.091 (3)	0.0701 (19)	−0.0123 (18)	0.0342 (16)	−0.0047 (18)
O27	0.186 (5)	0.107 (3)	0.133 (4)	−0.030 (3)	0.090 (4)	0.005 (4)
O28	0.095 (2)	0.097 (2)	0.0640 (19)	0.0000 (19)	0.0475 (18)	0.002 (2)
O29A	0.14 (3)	0.077 (17)	0.066 (10)	0.025 (8)	0.016 (13)	0.010 (13)
O29B	0.23 (3)	0.128 (9)	0.093 (8)	0.030 (6)	0.052 (14)	−0.045 (10)
C21	0.056 (2)	0.054 (2)	0.052 (2)	0.005 (2)	0.0246 (19)	0.012 (2)
C22	0.064 (3)	0.055 (2)	0.060 (2)	0.005 (2)	0.031 (2)	0.009 (2)
C23	0.067 (3)	0.058 (3)	0.068 (3)	0.005 (2)	0.041 (2)	0.010 (2)
C24	0.078 (3)	0.061 (3)	0.051 (2)	0.006 (2)	0.038 (2)	0.011 (2)
C25	0.062 (3)	0.069 (3)	0.054 (2)	0.002 (2)	0.031 (2)	0.006 (2)
C26	0.081 (3)	0.070 (3)	0.092 (3)	−0.006 (3)	0.055 (3)	−0.001 (3)
C27	0.124 (5)	0.090 (4)	0.130 (5)	−0.011 (4)	0.088 (4)	−0.024 (4)
C28	0.074 (4)	0.075 (4)	0.087 (4)	0.000 (3)	0.028 (3)	−0.024 (3)
C29	0.073 (3)	0.137 (5)	0.086 (4)	0.028 (4)	0.013 (3)	−0.014 (4)
C210	0.067 (3)	0.091 (4)	0.062 (3)	0.005 (2)	0.034 (2)	0.007 (3)
C211	0.094 (4)	0.072 (4)	0.082 (4)	−0.001 (3)	0.035 (3)	0.016 (3)
C212	0.145 (6)	0.086 (4)	0.176 (7)	−0.019 (4)	0.089 (6)	−0.021 (4)
C213	0.156 (8)	0.133 (7)	0.066 (4)	0.007 (5)	0.055 (4)	−0.037 (7)
C214	0.240 (10)	0.230 (11)	0.092 (5)	−0.013 (6)	0.109 (6)	−0.051 (8)
O31	0.397 (14)	0.285 (12)	0.252 (10)	0.024 (9)	0.148 (11)	0.016 (11)
C31	0.48 (2)	0.171 (9)	0.103 (7)	0.016 (6)	−0.034 (11)	0.121 (11)
C32	0.372 (14)	0.227 (12)	0.150 (11)	0.082 (9)	−0.024 (10)	0.003 (12)
C33	0.352 (13)	0.214 (10)	0.069 (5)	0.032 (5)	0.011 (7)	0.003 (10)
C34	0.376 (13)	0.332 (17)	0.094 (7)	0.074 (8)	0.050 (9)	0.126 (12)

Geometric parameters (Å, º)

S1—C1	1.810 (4)	O24—C22	1.429 (5)
S1—S2	2.0313 (16)	O25—C28	1.182 (7)
O1—C5	1.425 (5)	O26—C211	1.339 (6)
O1—C1	1.432 (4)	O26—C210	1.427 (5)
O2—C6	1.338 (7)	O27—C211	1.183 (6)

O2—C3	1.445 (5)	O28—C213	1.349 (8)
O3—C6	1.178 (7)	O28—C24	1.427 (5)
O4—C8	1.337 (6)	O29A—C213	1.27 (3)
O4—C2	1.410 (5)	O29B—C213	1.156 (17)
O5—C8	1.187 (6)	C21—C22	1.509 (6)
O6—C11	1.335 (6)	C21—H21	0.98
O6—C10	1.440 (6)	C22—C23	1.509 (6)
O7—C11	1.207 (7)	C22—H22	0.98
O8—C13	1.361 (5)	C23—C24	1.510 (6)
O8—C4	1.428 (5)	C23—H23	0.98
O9—C13	1.191 (6)	C24—C25	1.503 (6)
C1—C2	1.510 (6)	C24—H24	0.98
C1—H1	0.98	C25—C210	1.497 (6)
C2—C3	1.511 (6)	C25—H25	0.98
C2—H2	0.98	C26—C27	1.466 (7)
C3—C4	1.517 (6)	C27—H27A	0.96
C3—H3	0.98	C27—H27B	0.96
C4—C5	1.520 (6)	C27—H27C	0.96
C4—H4	0.98	C28—C29	1.481 (7)
C5—C10	1.495 (6)	C29—H29A	0.96
C5—H5	0.98	C29—H29B	0.96
C6—C7	1.463 (8)	C29—H29C	0.96
C7—H7A	0.96	C210—H21A	0.97
C7—H7B	0.96	C210—H21B	0.97
C7—H7C	0.96	C211—C212	1.450 (9)
C8—C9	1.484 (8)	C212—H21C	0.96
C9—H9A	0.96	C212—H21D	0.96
C9—H9B	0.96	C212—H21E	0.96
C9—H9C	0.96	C213—C214	1.513 (9)
C10—H10A	0.97	C214—H21F	0.96
C10—H10B	0.97	C214—H21G	0.96
C11—C12	1.486 (9)	C214—H21H	0.96
C12—H12A	0.96	O31—C32	1.578 (16)
C12—H12B	0.96	O31—C33	1.601 (14)
C12—H12C	0.96	C31—C32	1.640 (17)
C13—C14	1.475 (7)	C31—C34	1.68 (2)
C14—H14A	0.96	C31—H31A	0.97
C14—H14B	0.96	C31—H31B	0.97
C14—H14C	0.96	C32—H32A	0.97
S2—C21	1.823 (4)	C32—H32B	0.97
O21—C21	1.424 (4)	C33—C34	1.601 (15)
O21—C25	1.434 (4)	C33—H33A	0.97
O22—C26	1.337 (5)	C33—H33B	0.97
O22—C23	1.437 (5)	C34—H34A	0.97
O23—C26	1.196 (6)	C34—H34B	0.97
O24—C28	1.326 (6)		
C1—S1—S2	104.17 (14)	C21—C22—C23	110.7 (4)

C5—O1—C1	112.2 (3)	O24—C22—H22	109.5
C6—O2—C3	119.4 (4)	C21—C22—H22	109.5
C8—O4—C2	118.8 (4)	C23—C22—H22	109.5
C11—O6—C10	117.9 (5)	O22—C23—C22	105.8 (3)
C13—O8—C4	117.6 (3)	O22—C23—C24	110.7 (4)
O1—C1—C2	108.7 (3)	C22—C23—C24	111.6 (3)
O1—C1—S1	107.5 (3)	O22—C23—H23	109.6
C2—C1—S1	116.9 (3)	C22—C23—H23	109.6
O1—C1—H1	107.8	C24—C23—H23	109.6
C2—C1—H1	107.8	O28—C24—C25	107.8 (3)
S1—C1—H1	107.8	O28—C24—C23	108.8 (3)
O4—C2—C1	109.0 (3)	C25—C24—C23	112.4 (3)
O4—C2—C3	110.0 (3)	O28—C24—H24	109.3
C1—C2—C3	108.8 (3)	C25—C24—H24	109.3
O4—C2—H2	109.7	C23—C24—H24	109.3
C1—C2—H2	109.7	O21—C25—C210	106.7 (3)
C3—C2—H2	109.7	O21—C25—C24	108.2 (3)
O2—C3—C2	109.1 (3)	C210—C25—C24	111.5 (4)
O2—C3—C4	108.3 (3)	O21—C25—H25	110.1
C2—C3—C4	110.6 (3)	C210—C25—H25	110.1
O2—C3—H3	109.6	C24—C25—H25	110.1
C2—C3—H3	109.6	O23—C26—O22	123.2 (5)
C4—C3—H3	109.6	O23—C26—C27	125.0 (4)
O8—C4—C3	108.4 (3)	O22—C26—C27	111.8 (4)
O8—C4—C5	110.3 (3)	C26—C27—H27A	109.5
C3—C4—C5	108.4 (3)	C26—C27—H27B	109.5
O8—C4—H4	109.9	H27A—C27—H27B	109.5
C3—C4—H4	109.9	C26—C27—H27C	109.5
C5—C4—H4	109.9	H27A—C27—H27C	109.5
O1—C5—C10	107.2 (3)	H27B—C27—H27C	109.5
O1—C5—C4	108.3 (3)	O25—C28—O24	123.3 (5)
C10—C5—C4	113.1 (4)	O25—C28—C29	126.1 (6)
O1—C5—H5	109.4	O24—C28—C29	110.6 (5)
C10—C5—H5	109.4	C28—C29—H29A	109.5
C4—C5—H5	109.4	C28—C29—H29B	109.5
O3—C6—O2	121.9 (6)	H29A—C29—H29B	109.5
O3—C6—C7	125.7 (6)	C28—C29—H29C	109.5
O2—C6—C7	112.4 (6)	H29A—C29—H29C	109.5
C6—C7—H7A	109.5	H29B—C29—H29C	109.5
C6—C7—H7B	109.5	O26—C210—C25	111.0 (4)
H7A—C7—H7B	109.5	O26—C210—H21A	109.4
C6—C7—H7C	109.5	C25—C210—H21A	109.4
H7A—C7—H7C	109.5	O26—C210—H21B	109.4
H7B—C7—H7C	109.5	C25—C210—H21B	109.4
O5—C8—O4	123.3 (5)	H21A—C210—H21B	108
O5—C8—C9	124.9 (6)	O27—C211—O26	122.4 (6)
O4—C8—C9	111.8 (5)	O27—C211—C212	126.2 (6)
C8—C9—H9A	109.5	O26—C211—C212	111.2 (5)

C8—C9—H9B	109.5	C211—C212—H21C	109.5
H9A—C9—H9B	109.5	C211—C212—H21D	109.5
C8—C9—H9C	109.5	H21C—C212—H21D	109.5
H9A—C9—H9C	109.5	C211—C212—H21E	109.5
H9B—C9—H9C	109.5	H21C—C212—H21E	109.5
O6—C10—C5	109.3 (4)	H21D—C212—H21E	109.5
O6—C10—H10A	109.8	O29B—C213—O29A	43.8 (10)
C5—C10—H10A	109.8	O29B—C213—O28	121.9 (11)
O6—C10—H10B	109.8	O29A—C213—O28	118.7 (18)
C5—C10—H10B	109.8	O29B—C213—C214	123.7 (11)
H10A—C10—H10B	108.3	O29A—C213—C214	125.3 (12)
O7—C11—O6	122.0 (6)	O28—C213—C214	109.4 (8)
O7—C11—C12	126.3 (6)	C213—C214—H21F	109.5
O6—C11—C12	111.6 (6)	C213—C214—H21G	109.5
C11—C12—H12A	109.5	H21F—C214—H21G	109.5
C11—C12—H12B	109.5	C213—C214—H21H	109.5
H12A—C12—H12B	109.5	H21F—C214—H21H	109.5
C11—C12—H12C	109.5	H21G—C214—H21H	109.5
H12A—C12—H12C	109.5	C32—O31—C33	108.0 (14)
H12B—C12—H12C	109.5	C32—C31—C34	109.3 (9)
O9—C13—O8	122.3 (4)	C32—C31—H31A	109.8
O9—C13—C14	125.9 (4)	C34—C31—H31A	109.8
O8—C13—C14	111.8 (5)	C32—C31—H31B	109.8
C13—C14—H14A	109.5	C34—C31—H31B	109.8
C13—C14—H14B	109.5	H31A—C31—H31B	108.3
H14A—C14—H14B	109.5	O31—C32—C31	107.2 (12)
C13—C14—H14C	109.5	O31—C32—H32A	110.3
H14A—C14—H14C	109.5	C31—C32—H32A	110.3
H14B—C14—H14C	109.5	O31—C32—H32B	110.3
C21—S2—S1	104.10 (15)	C31—C32—H32B	110.3
C21—O21—C25	111.7 (3)	H32A—C32—H32B	108.5
C26—O22—C23	120.0 (4)	C34—C33—O31	112.7 (12)
C28—O24—C22	118.2 (4)	C34—C33—H33A	109.1
C211—O26—C210	118.4 (4)	O31—C33—H33A	109
C213—O28—C24	118.9 (5)	C34—C33—H33B	109.1
O21—C21—C22	108.8 (3)	O31—C33—H33B	109
O21—C21—S2	108.9 (2)	H33A—C33—H33B	107.8
C22—C21—S2	108.6 (3)	C33—C34—C31	102.6 (10)
O21—C21—H21	110.2	C33—C34—H34A	111.2
C22—C21—H21	110.2	C31—C34—H34A	111.3
S2—C21—H21	110.2	C33—C34—H34B	111.2
O24—C22—C21	108.6 (3)	C31—C34—H34B	111.3
O24—C22—C23	108.9 (3)	H34A—C34—H34B	109.2

C5—O1—C1—C2	−64.8 (4)	S1—S2—C21—C22	156.3 (2)
C5—O1—C1—S1	167.8 (3)	C28—O24—C22—C21	116.5 (4)
S2—S1—C1—O1	74.3 (3)	C28—O24—C22—C23	−122.8 (4)
S2—S1—C1—C2	−48.2 (3)	O21—C21—C22—O24	176.3 (3)

C8—O4—C2—C1	137.4 (4)	S2—C21—C22—O24	−65.2 (3)
C8—O4—C2—C3	−103.4 (4)	O21—C21—C22—C23	56.8 (4)
O1—C1—C2—O4	178.3 (3)	S2—C21—C22—C23	175.2 (3)
S1—C1—C2—O4	−59.9 (4)	C26—O22—C23—C22	−138.6 (4)
O1—C1—C2—C3	58.3 (4)	C26—O22—C23—C24	100.4 (4)
S1—C1—C2—C3	−179.9 (3)	O24—C22—C23—O22	71.7 (4)
C6—O2—C3—C2	−118.1 (4)	C21—C22—C23—O22	−169.0 (3)
C6—O2—C3—C4	121.5 (4)	O24—C22—C23—C24	−167.9 (4)
O4—C2—C3—O2	65.7 (4)	C21—C22—C23—C24	−48.5 (5)
C1—C2—C3—O2	−175.0 (3)	C213—O28—C24—C25	−128.2 (6)
O4—C2—C3—C4	−175.3 (3)	C213—O28—C24—C23	109.8 (6)
C1—C2—C3—C4	−56.0 (4)	O22—C23—C24—O28	−75.1 (4)
C13—O8—C4—C3	142.4 (4)	C22—C23—C24—O28	167.4 (4)
C13—O8—C4—C5	−99.0 (4)	O22—C23—C24—C25	165.7 (3)
O2—C3—C4—O8	−64.5 (4)	C22—C23—C24—C25	48.1 (5)
C2—C3—C4—O8	175.9 (3)	C21—O21—C25—C210	−175.0 (4)
O2—C3—C4—C5	175.6 (3)	C21—O21—C25—C24	64.9 (4)
C2—C3—C4—C5	56.1 (4)	O28—C24—C25—O21	−174.4 (3)
C1—O1—C5—C10	−172.6 (4)	C23—C24—C25—O21	−54.6 (4)
C1—O1—C5—C4	65.0 (4)	O28—C24—C25—C210	68.6 (4)
O8—C4—C5—O1	−177.4 (3)	C23—C24—C25—C210	−171.6 (4)
C3—C4—C5—O1	−58.8 (4)	C23—O22—C26—O23	−3.7 (8)
O8—C4—C5—C10	63.9 (5)	C23—O22—C26—C27	177.8 (4)
C3—C4—C5—C10	−177.6 (4)	C22—O24—C28—O25	5.0 (7)
C3—O2—C6—O3	−2.4 (8)	C22—O24—C28—C29	−173.7 (4)
C3—O2—C6—C7	176.9 (4)	C211—O26—C210—C25	93.8 (5)
C2—O4—C8—O5	2.4 (7)	O21—C25—C210—O26	52.3 (5)
C2—O4—C8—C9	−178.6 (4)	C24—C25—C210—O26	170.2 (4)
C11—O6—C10—C5	−102.8 (5)	C210—O26—C211—O27	−1.1 (8)
O1—C5—C10—O6	60.9 (5)	C210—O26—C211—C212	−177.6 (5)
C4—C5—C10—O6	−179.8 (4)	C24—O28—C213—O29B	−20 (3)
C10—O6—C11—O7	−3.9 (7)	C24—O28—C213—O29A	31 (3)
C10—O6—C11—C12	174.3 (4)	C24—O28—C213—C214	−176.2 (6)
C4—O8—C13—O9	−6.1 (7)	C33—O31—C32—C31	5.7 (13)
C4—O8—C13—C14	172.9 (4)	C34—C31—C32—O31	−4.8 (14)
C1—S1—S2—C21	−80.25 (19)	C32—O31—C33—C34	−4.8 (13)
C25—O21—C21—C22	−66.6 (4)	O31—C33—C34—C31	1.7 (12)
C25—O21—C21—S2	175.1 (3)	C32—C31—C34—C33	1.9 (12)
S1—S2—C21—O21	−85.4 (3)		