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Bis(3-deoxy-1,2-*O*-isopropylidene-α-D-glucofuranos-3-yl) disulfide

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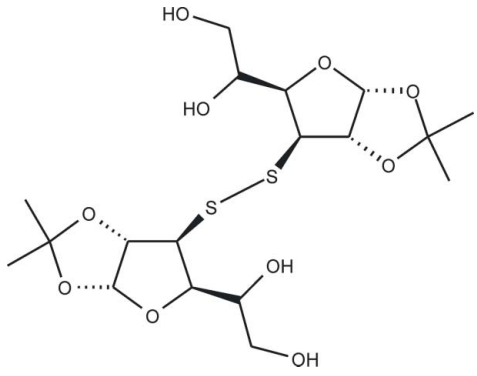
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.029; wR factor = 0.066; data-to-parameter ratio = 18.4.

The title compound, $C_{18}H_{30}O_{10}S_2$, was prepared as a part of a project aimed at the synthesis of potential gelators of organic solvents. In the crystalline state, it forms hydrogen-bonded chains.

Related literature

For low-molecular-mass organogelators see: Terech & Weiss (1997); Luboradzki *et al.* (2005). For related literature, see: Risbood *et al.* (1981).



Experimental

Crystal data

$C_{18}H_{30}O_{10}S_2$
M_r = 470.54
Orthorhombic, $P2_12_12_1$
a = 8.7266 (1) Å
b = 14.4778 (4) Å
c = 17.8606 (5) Å
V = 2256.54 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.29 mm^{-1}
T = 293 (2) K
0.4 × 0.2 × 0.1 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: none
22012 measured reflections
5134 independent reflections
4969 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.066
S = 1.09
5134 reflections
279 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}
Absolute structure: Flack (1983), 2211 Friedel pairs
Flack parameter: 0.02 (5)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4O···O10^{i}	0.82	1.94	2.7249 (18)	159
O5—H5O···O3^{i}	0.82	1.99	2.8055 (18)	176
O9—H9O···O5^{ii}	0.82	2.05	2.8338 (17)	159
O10—H10O···O9^{iii}	0.82	2.04	2.8305 (18)	163

Symmetry codes: (i) $x-1, y, z$; (ii) $x+1, y, z$; (iii) $x+\frac{1}{2}, -y+\frac{3}{2}, -z+1$.

Data collection: *COLLECT* (Nonius, 1997); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2343).

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supplementary materials

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Bis(3-deoxy-1,2-*O*-isopropylidene-*α*-D-glucofuranos-3-yl) disulfide

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Comment

The title compound, (I), was prepared as a part of a project aimed at the synthesis of potential gelators of organic solvents (Luboradzki *et al.*, 2005). The class of saccharide-based gelators having free OH groups and the glucofuranose fragment are able to form gels with huge spectrum of organic solvents. According to the driving forces responsible for molecular aggregation they may be classified as hydrogen-bond-based gelators (Terech & Weiss 1997).

The crystal structure of (I) (Fig. 1) contains one independent molecule of the title compound per asymmetric unit. The molecules are bound into one dimensional hydrogen bond-based chains (generated by the screw axis along the *a* axis) utilizing all hydroxy H atoms (Fig. 2). Each molecule is involved in four hydrogen bonds as proton donor and in four as acceptor.

Experimental

The title compound (I) was prepared from 1,2-*O*-Isopropylidene-3-thio-α-*D*-glucofuranose by oxidation in hot methanol involving oxygen from the air. Subsequently it was recrystallized by cooling hot methanol solution.

Refinement

All H atoms were geometrically constrained to ride on their parent atom, fixing the bond lengths at 0.82, 0.96, 0.97 and 0.98 Å for hydroxyl, methine, methylene and methyl H atoms, respectively, and with U_{iso}(H)= 1.2U_{eq}(1.5U_{eq} for methyl and hydroxyl) of the carrier atom. –OH hydrogen atoms were refined by use of a rotating group refinement (AFIX 147). The absolute configuration determined from anomalous scattering effects is as expected from the synthesis.

Figures

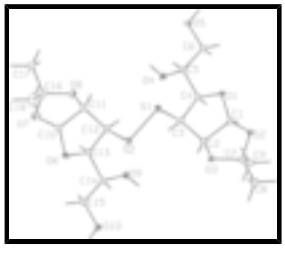

Fig. 1. The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level. H atoms are shown as circles of arbitrary radii.

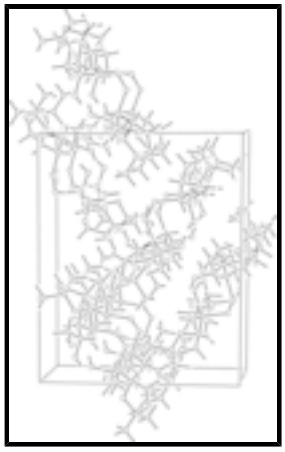

Fig. 2. The packing of (I), viewed down the *a* axis, with the *b* axis vertical.

Bis(3-deoxy-1,2-*O*-isopropylidene-α-*D*-glucofuranos-3-yl) disulfide

Crystal data

$C_{18}H_{30}O_{10}S_2$	F_{000} = 1000
M_r = 470.54	D_x = 1.385 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 17407 reflections
a = 8.7266 (1) Å	θ = 1.0–27.5º
b = 14.4778 (4) Å	μ = 0.29 mm^{-1}
c = 17.8606 (5) Å	T = 293 (2) K
V = 2256.54 (9) Å^3	Plate, colourless
Z = 4	0.4 × 0.2 × 0.1 mm

Data collection

Nonius KappaCCD diffractometer	5134 independent reflections
Radiation source: fine-focus sealed tube	4969 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.032
Detector resolution: 1242 (horizontal) x 1152 (vertical) pixels, CCD pixel size is 22.5 x 22.5 μm which gives at the imput a pixel of 110 x 110 μm (with 2 x 2 binning) pixels mm^{-1}	θ_{max} = 27.5º
T = 293(2) K	θ_{min} = 2.6º
189 frames via φ rotation (rotation angle 1.9°) and 2*287 s per frame and 13 frames via ω rotation (rotation angle 1.9°) and 2*287 s per frame scans	h = −11→11
Absorption correction: none	k = −18→18
22012 measured reflections	l = −23→23

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.029$	$w = 1/[\sigma^2(F_o^2) + (0.0134P)^2 + 1.4932P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.066$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.09$	$\Delta\rho_{max} = 0.21$ e Å^{-3}
5134 reflections	$\Delta\rho_{min} = -0.21$ e Å^{-3}
279 parameters	Extinction correction: none
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 2211 Freidel pairs
Secondary atom site location: difference Fourier map	Flack parameter: 0.02 (5)

Special details

Experimental. Synthesis of 1,2-*O*-Isopropylidene-3-thio-α-*D*-glucofuranose: To a suspension of $LiAlH_4$ (160 mg, 4.2 m*M*) in THF (25 ml) cooled in ice bath a solution of 5,6-di-*O*-acetyl-1,2-*O*-isopropylidene-3-thiocyanato-α-*D*-glucofuranose (Risbood *et al.*1981) (1, 629 mg, 1.8 m*M*) in THF (5 ml) was slowly added and stirred at room temp. for 3 h under an argon atmosphere. An excess of $LiAlH_4$ was decomposed by addition of ethyl acetate (1 ml), then the reaction mixture was cautiously acidified by addition of 5% aqueous NH_4Cl and solvents were evaporated to dryness. Column chromatography of the residue (hexane ? ethyl acetate, 5:1 than 1:1, then hexane ? ethyl acetate - methanol, 5:3:1) yielded 301 mg (70%) of the title compound as foam. $[\alpha]_D^2$ −9.7 (c 1/2, chloroform ? methanol, 1:1); ^{1}H NMR (CD_3OD) δ: 5.86 (d, 1H, $J_{1,2}$ 3.5 Hz, H-1), 4.66 (d, 1H, H-2), 4.18 (dd, 1H, $J_{4,3}$ 3.7, $J_{4,5}$ 9.4 Hz, H-4), 3.88 (m, 1H, H-5), 3.75 (dd, 1H, $J_{6,5}$ 2.8, $J_{6,6?}$ 11.6 Hz, H-6), 3.58 (dd, 1H, $J_{6?,5}$ 5.7, H-6?), 3.48 (d, 1H, H-3), 3.31 (m, 1H, SH), 1.46 (s, 3H, CH_3), 1.29 (s, 3H, CH_3). ^{13}C NMR (CD_3OD) δ: 112.9 (C), 106.0 (C-1), 88.7, 79.7, 71.8, 65.2 (C-6), 46.3 (C-3), 26.8 (CH_3), 26.4 (CH_3). HR—MS (ESI) calc. for $C_9H_{16}NaO_5S$ [*M*+Na]+: 259.0611. Found: 259.0615.

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 $F^2 > \sigma(F^2)$ 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}
S1	0.06709 (5)	0.57977 (3)	0.73252 (2)	0.01991 (9)
S2	0.25277 (5)	0.54599 (3)	0.66943 (2)	0.01858 (9)
O1	−0.05475 (13)	0.73533 (8)	0.84580 (6)	0.0182 (2)
O2	0.13077 (14)	0.82781 (9)	0.90160 (7)	0.0221 (3)
O3	0.29050 (13)	0.80305 (8)	0.80408 (7)	0.0193 (3)

O4	−0.15521 (16)	0.76064 (10)	0.65014 (7)	0.0264 (3)
H4O	−0.2082	0.7303	0.6213	0.040*
O5	−0.45500 (14)	0.75117 (9)	0.71783 (7)	0.0227 (3)
H5O	−0.5321	0.7658	0.7413	0.034*
O6	0.35095 (14)	0.49495 (8)	0.50471 (7)	0.0197 (3)
O7	0.15464 (15)	0.42656 (10)	0.43354 (7)	0.0266 (3)
O8	0.00365 (14)	0.54097 (9)	0.48132 (7)	0.0230 (3)
O9	0.41651 (15)	0.72871 (8)	0.57326 (7)	0.0203 (3)
H9O	0.4679	0.7450	0.6094	0.030*
O10	0.72117 (14)	0.67699 (10)	0.52755 (7)	0.0254 (3)
H10O	0.7829	0.6932	0.4955	0.038*
C1	0.09359 (19)	0.73702 (12)	0.87676 (9)	0.0181 (3)
H1	0.1045	0.6917	0.9171	0.022*
C2	0.20792 (19)	0.71817 (12)	0.81233 (9)	0.0168 (3)
H2	0.2750	0.6653	0.8222	0.020*
C3	0.10501 (19)	0.70371 (11)	0.74457 (9)	0.0174 (3)
H3	0.1510	0.7302	0.6994	0.021*
C4	−0.03968 (19)	0.75640 (11)	0.76711 (9)	0.0162 (3)
H4	−0.0204	0.8227	0.7616	0.019*
C5	−0.18473 (19)	0.73217 (12)	0.72500 (9)	0.0179 (3)
H5A	−0.2013	0.6652	0.7265	0.022*
C6	−0.32189 (19)	0.78143 (12)	0.75760 (10)	0.0188 (3)
H6A	−0.3319	0.7670	0.8104	0.023*
H6B	−0.3098	0.8477	0.7524	0.023*
C7	0.2787 (2)	0.85227 (12)	0.87392 (10)	0.0204 (4)
C8	0.4020 (2)	0.82149 (14)	0.92836 (11)	0.0265 (4)
H8A	0.3869	0.8516	0.9757	0.040*
H8B	0.5009	0.8376	0.9088	0.040*
H8C	0.3961	0.7558	0.9351	0.040*
C9	0.2824 (2)	0.95448 (13)	0.85718 (12)	0.0284 (4)
H9A	0.2020	0.9695	0.8226	0.043*
H9B	0.3796	0.9704	0.8356	0.043*
H9C	0.2679	0.9886	0.9027	0.043*
C10	0.21131 (19)	0.44449 (12)	0.50562 (10)	0.0195 (3)
H10A	0.2237	0.3868	0.5336	0.023*
C11	0.0902 (2)	0.50586 (12)	0.54282 (9)	0.0189 (3)
H11A	0.0274	0.4729	0.5796	0.023*
C12	0.18450 (19)	0.58362 (12)	0.57694 (9)	0.0165 (3)
H12	0.1264	0.6414	0.5799	0.020*
C13	0.31752 (19)	0.59043 (11)	0.52161 (9)	0.0161 (3)
H13	0.2827	0.6217	0.4760	0.019*
C14	0.46207 (19)	0.63771 (11)	0.55074 (9)	0.0166 (3)
H14	0.5000	0.6040	0.5945	0.020*
C15	0.5868 (2)	0.64160 (12)	0.49218 (10)	0.0203 (3)
H15A	0.6063	0.5804	0.4723	0.024*
H15B	0.5562	0.6816	0.4512	0.024*
C16	0.0177 (2)	0.47929 (12)	0.41967 (10)	0.0207 (4)
C17	−0.1173 (2)	0.41418 (14)	0.41582 (11)	0.0288 (4)
H17A	−0.1226	0.3787	0.4611	0.043*

H17B	−0.1051	0.3734	0.3739	0.043*
H17C	−0.2100	0.4491	0.4100	0.043*
C18	0.0392 (3)	0.53557 (16)	0.34954 (12)	0.0432 (6)
H18A	−0.0497	0.5734	0.3414	0.065*
H18B	0.0531	0.4950	0.3076	0.065*
H18C	0.1278	0.5743	0.3549	0.065*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0209 (2)	0.01871 (19)	0.02014 (19)	−0.00514 (17)	0.00455 (17)	−0.00471 (16)
S2	0.01921 (19)	0.01991 (18)	0.01661 (18)	0.00203 (17)	0.00090 (17)	−0.00052 (15)
O1	0.0146 (6)	0.0254 (6)	0.0145 (5)	−0.0016 (5)	−0.0011 (4)	−0.0011 (5)
O2	0.0169 (6)	0.0228 (6)	0.0266 (7)	−0.0005 (5)	−0.0004 (5)	−0.0102 (5)
O3	0.0157 (6)	0.0193 (6)	0.0229 (6)	−0.0036 (5)	−0.0008 (5)	−0.0047 (5)
O4	0.0241 (7)	0.0420 (8)	0.0130 (6)	−0.0132 (6)	−0.0020 (5)	0.0002 (5)
O5	0.0147 (6)	0.0289 (7)	0.0246 (6)	−0.0020 (5)	−0.0027 (5)	−0.0026 (5)
O6	0.0168 (6)	0.0146 (5)	0.0276 (6)	−0.0005 (5)	0.0041 (5)	−0.0045 (5)
O7	0.0240 (7)	0.0302 (7)	0.0257 (7)	0.0022 (6)	−0.0003 (5)	−0.0131 (6)
O8	0.0238 (6)	0.0235 (6)	0.0219 (6)	0.0034 (5)	−0.0048 (5)	−0.0076 (5)
O9	0.0229 (6)	0.0144 (5)	0.0237 (6)	0.0017 (5)	−0.0047 (5)	−0.0030 (5)
O10	0.0185 (7)	0.0374 (8)	0.0202 (6)	−0.0108 (6)	0.0027 (5)	−0.0039 (6)
C1	0.0169 (8)	0.0196 (8)	0.0178 (7)	0.0002 (6)	−0.0029 (6)	−0.0016 (6)
C2	0.0140 (8)	0.0170 (8)	0.0196 (8)	−0.0006 (6)	−0.0017 (6)	−0.0019 (6)
C3	0.0174 (8)	0.0172 (8)	0.0176 (8)	−0.0029 (6)	0.0008 (6)	−0.0027 (6)
C4	0.0171 (8)	0.0179 (7)	0.0136 (7)	−0.0022 (6)	−0.0009 (6)	−0.0006 (6)
C5	0.0170 (8)	0.0213 (8)	0.0155 (7)	−0.0042 (6)	−0.0012 (6)	0.0003 (6)
C6	0.0177 (8)	0.0194 (8)	0.0193 (8)	−0.0012 (7)	−0.0051 (7)	−0.0008 (6)
C7	0.0162 (8)	0.0220 (9)	0.0229 (8)	0.0007 (7)	−0.0026 (7)	−0.0070 (7)
C8	0.0200 (9)	0.0303 (10)	0.0290 (10)	0.0039 (8)	−0.0069 (7)	−0.0084 (8)
C9	0.0235 (9)	0.0212 (8)	0.0406 (11)	−0.0007 (8)	−0.0030 (8)	−0.0072 (8)
C10	0.0209 (9)	0.0158 (8)	0.0219 (8)	−0.0023 (6)	0.0004 (6)	−0.0028 (6)
C11	0.0179 (8)	0.0205 (8)	0.0181 (8)	−0.0014 (7)	0.0004 (7)	−0.0032 (6)
C12	0.0176 (8)	0.0166 (7)	0.0155 (7)	0.0021 (6)	−0.0003 (6)	−0.0014 (6)
C13	0.0174 (8)	0.0125 (7)	0.0183 (8)	0.0010 (6)	0.0006 (6)	−0.0010 (6)
C14	0.0173 (8)	0.0151 (7)	0.0173 (7)	−0.0002 (6)	−0.0016 (6)	0.0009 (6)
C15	0.0199 (9)	0.0216 (8)	0.0194 (8)	−0.0043 (7)	0.0000 (7)	−0.0020 (7)
C16	0.0272 (9)	0.0192 (8)	0.0156 (8)	−0.0010 (7)	−0.0001 (7)	−0.0014 (6)
C17	0.0290 (10)	0.0252 (9)	0.0323 (10)	−0.0045 (8)	−0.0071 (8)	−0.0029 (8)
C18	0.0697 (17)	0.0351 (12)	0.0247 (10)	−0.0063 (12)	0.0037 (10)	0.0069 (9)

Geometric parameters (Å, °)

S1—C3	1.8373 (17)	C5—H5A	0.9800
S1—S2	2.0335 (6)	C6—H6A	0.9700
S2—C12	1.8386 (17)	C6—H6B	0.9700
O1—C1	1.408 (2)	C7—C9	1.510 (3)
O1—C4	1.4442 (19)	C7—C8	1.517 (2)
O2—C1	1.425 (2)	C8—H8A	0.9600

O2—C7	1.427 (2)	C8—H8B	0.9600
O3—C2	1.432 (2)	C8—H8C	0.9600
O3—C7	1.440 (2)	C9—H9A	0.9600
O4—C5	1.423 (2)	C9—H9B	0.9600
O4—H4O	0.8200	C9—H9C	0.9600
O5—C6	1.430 (2)	C10—C11	1.533 (2)
O5—H5O	0.8200	C10—H10A	0.9800
O6—C10	1.421 (2)	C11—C12	1.522 (2)
O6—C13	1.4446 (19)	C11—H11A	0.9800
O7—C10	1.403 (2)	C12—C13	1.528 (2)
O7—C16	1.440 (2)	C12—H12	0.9800
O8—C16	1.423 (2)	C13—C14	1.527 (2)
O8—C11	1.426 (2)	C13—H13	0.9800
O9—C14	1.434 (2)	C14—C15	1.511 (2)
O9—H9O	0.8200	C14—H14	0.9800
O10—C15	1.427 (2)	C15—H15A	0.9700
O10—H10O	0.8200	C15—H15B	0.9700
C1—C2	1.547 (2)	C16—C18	1.506 (3)
C1—H1	0.9800	C16—C17	1.510 (3)
C2—C3	1.522 (2)	C17—H17A	0.9600
C2—H2	0.9800	C17—H17B	0.9600
C3—C4	1.529 (2)	C17—H17C	0.9600
C3—H3	0.9800	C18—H18A	0.9600
C4—C5	1.514 (2)	C18—H18B	0.9600
C4—H4	0.9800	C18—H18C	0.9600
C5—C6	1.510 (2)		
C3—S1—S2	98.99 (6)	C7—C9—H9A	109.5
C12—S2—S1	99.69 (6)	C7—C9—H9B	109.5
C1—O1—C4	107.15 (12)	H9A—C9—H9B	109.5
C1—O2—C7	109.10 (13)	C7—C9—H9C	109.5
C2—O3—C7	107.43 (13)	H9A—C9—H9C	109.5
C5—O4—H4O	109.5	H9B—C9—H9C	109.5
C6—O5—H5O	109.5	O7—C10—O6	112.76 (14)
C10—O6—C13	108.45 (12)	O7—C10—C11	105.18 (14)
C10—O7—C16	110.64 (13)	O6—C10—C11	107.37 (13)
C16—O8—C11	109.08 (13)	O7—C10—H10A	110.5
C14—O9—H9O	109.5	O6—C10—H10A	110.5
C15—O10—H10O	109.5	C11—C10—H10A	110.5
O1—C1—O2	110.35 (13)	O8—C11—C12	109.33 (14)
O1—C1—C2	107.32 (13)	O8—C11—C10	103.76 (13)
O2—C1—C2	104.33 (13)	C12—C11—C10	103.25 (13)
O1—C1—H1	111.5	O8—C11—H11A	113.2
O2—C1—H1	111.5	C12—C11—H11A	113.2
C2—C1—H1	111.5	C10—C11—H11A	113.2
O3—C2—C3	109.47 (13)	C11—C12—C13	101.52 (13)
O3—C2—C1	104.45 (13)	C11—C12—S2	108.41 (12)
C3—C2—C1	103.61 (13)	C13—C12—S2	110.74 (11)
O3—C2—H2	112.9	C11—C12—H12	111.9
C3—C2—H2	112.9	C13—C12—H12	111.9

C1—C2—H2	112.9	S2—C12—H12	111.9
C2—C3—C4	102.08 (13)	O6—C13—C14	109.48 (13)
C2—C3—S1	109.50 (12)	O6—C13—C12	103.11 (13)
C4—C3—S1	111.67 (11)	C14—C13—C12	115.88 (14)
C2—C3—H3	111.1	O6—C13—H13	109.4
C4—C3—H3	111.1	C14—C13—H13	109.4
S1—C3—H3	111.1	C12—C13—H13	109.4
O1—C4—C5	111.01 (13)	O9—C14—C15	111.09 (14)
O1—C4—C3	103.07 (13)	O9—C14—C13	106.17 (13)
C5—C4—C3	116.36 (13)	C15—C14—C13	112.10 (14)
O1—C4—H4	108.7	O9—C14—H14	109.1
C5—C4—H4	108.7	C15—C14—H14	109.1
C3—C4—H4	108.7	C13—C14—H14	109.1
O4—C5—C6	111.65 (14)	O10—C15—C14	107.40 (13)
O4—C5—C4	104.38 (13)	O10—C15—H15A	110.2
C6—C5—C4	111.21 (14)	C14—C15—H15A	110.2
O4—C5—H5A	109.8	O10—C15—H15B	110.2
C6—C5—H5A	109.8	C14—C15—H15B	110.2
C4—C5—H5A	109.8	H15A—C15—H15B	108.5
O5—C6—C5	107.91 (13)	O8—C16—O7	105.71 (13)
O5—C6—H6A	110.1	O8—C16—C18	108.36 (15)
C5—C6—H6A	110.1	O7—C16—C18	109.05 (17)
O5—C6—H6B	110.1	O8—C16—C17	111.09 (15)
C5—C6—H6B	110.1	O7—C16—C17	108.93 (15)
H6A—C6—H6B	108.4	C18—C16—C17	113.40 (17)
O2—C7—O3	104.00 (13)	C16—C17—H17A	109.5
O2—C7—C9	109.33 (14)	C16—C17—H17B	109.5
O3—C7—C9	108.17 (15)	H17A—C17—H17B	109.5
O2—C7—C8	110.29 (15)	C16—C17—H17C	109.5
O3—C7—C8	111.04 (14)	H17A—C17—H17C	109.5
C9—C7—C8	113.56 (15)	H17B—C17—H17C	109.5
C7—C8—H8A	109.5	C16—C18—H18A	109.5
C7—C8—H8B	109.5	C16—C18—H18B	109.5
H8A—C8—H8B	109.5	H18A—C18—H18B	109.5
C7—C8—H8C	109.5	C16—C18—H18C	109.5
H8A—C8—H8C	109.5	H18A—C18—H18C	109.5
H8B—C8—H8C	109.5	H18B—C18—H18C	109.5
C3—S1—S2—C12	−81.01 (8)	C16—O7—C10—O6	−110.42 (15)
C4—O1—C1—O2	89.04 (15)	C16—O7—C10—C11	6.27 (18)
C4—O1—C1—C2	−24.05 (17)	C13—O6—C10—O7	101.54 (16)
C7—O2—C1—O1	−131.43 (14)	C13—O6—C10—C11	−13.84 (17)
C7—O2—C1—C2	−16.44 (17)	C16—O8—C11—C12	133.98 (14)
C7—O3—C2—C3	133.13 (14)	C16—O8—C11—C10	24.34 (17)
C7—O3—C2—C1	22.72 (16)	O7—C10—C11—O8	−18.43 (17)
O1—C1—C2—O3	113.11 (14)	O6—C10—C11—O8	101.89 (15)
O2—C1—C2—O3	−3.99 (16)	O7—C10—C11—C12	−132.51 (14)
O1—C1—C2—C3	−1.50 (17)	O6—C10—C11—C12	−12.19 (17)
O2—C1—C2—C3	−118.60 (14)	O8—C11—C12—C13	−78.46 (16)
O3—C2—C3—C4	−86.56 (15)	C10—C11—C12—C13	31.52 (16)

C1—C2—C3—C4	24.41 (16)	O8—C11—C12—S2	164.88 (11)
O3—C2—C3—S1	154.98 (11)	C10—C11—C12—S2	−85.14 (13)
C1—C2—C3—S1	−94.05 (13)	S1—S2—C12—C11	−84.82 (11)
S2—S1—C3—C2	−87.01 (11)	S1—S2—C12—C13	164.62 (10)
S2—S1—C3—C4	160.68 (10)	C10—O6—C13—C14	158.11 (13)
C1—O1—C4—C5	165.20 (13)	C10—O6—C13—C12	34.22 (16)
C1—O1—C4—C3	39.93 (15)	C11—C12—C13—O6	−40.33 (16)
C2—C3—C4—O1	−39.16 (15)	S2—C12—C13—O6	74.62 (14)
S1—C3—C4—O1	77.74 (13)	C11—C12—C13—C14	−159.90 (14)
C2—C3—C4—C5	−160.87 (14)	S2—C12—C13—C14	−44.95 (17)
S1—C3—C4—C5	−43.97 (17)	O6—C13—C14—O9	−174.31 (12)
O1—C4—C5—O4	177.28 (13)	C12—C13—C14—O9	−58.27 (18)
C3—C4—C5—O4	−65.29 (18)	O6—C13—C14—C15	64.22 (17)
O1—C4—C5—C6	56.74 (18)	C12—C13—C14—C15	−179.75 (14)
C3—C4—C5—C6	174.17 (14)	O9—C14—C15—O10	67.98 (18)
O4—C5—C6—O5	67.08 (17)	C13—C14—C15—O10	−173.41 (14)
C4—C5—C6—O5	−176.77 (13)	C11—O8—C16—O7	−20.99 (18)
C1—O2—C7—O3	30.63 (17)	C11—O8—C16—C18	−137.78 (17)
C1—O2—C7—C9	145.97 (15)	C11—O8—C16—C17	97.02 (17)
C1—O2—C7—C8	−88.49 (17)	C10—O7—C16—O8	8.42 (18)
C2—O3—C7—O2	−33.02 (16)	C10—O7—C16—C18	124.73 (17)
C2—O3—C7—C9	−149.18 (14)	C10—O7—C16—C17	−111.03 (16)
C2—O3—C7—C8	85.58 (17)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4O···O10^{i}	0.82	1.94	2.7249 (18)	159
O5—H5O···O3^{i}	0.82	1.99	2.8055 (18)	176
O9—H9O···O5^{ii}	0.82	2.05	2.8338 (17)	159
O10—H10O···O9^{iii}	0.82	2.04	2.8305 (18)	163

Symmetry codes: (i) *x*−1, *y*, *z*; (ii) *x*+1, *y*, *z*; (iii) *x*+1/2, −*y*+3/2, −*z*+1.

Fig. 1

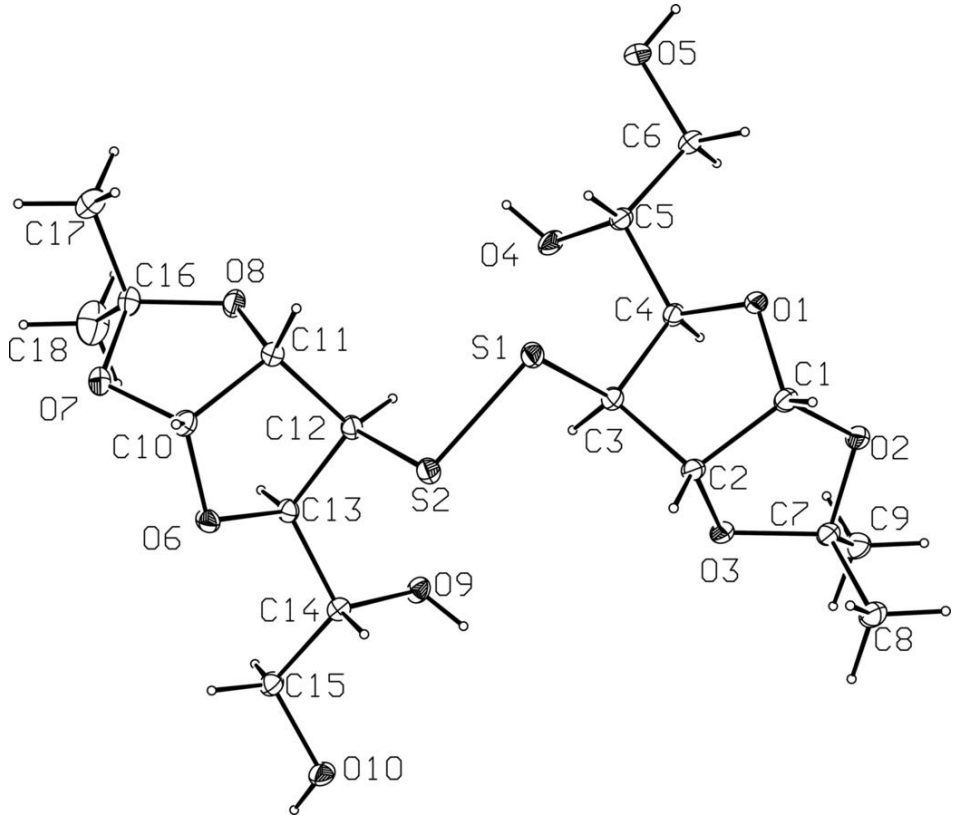
O5
C6
C5
O4
C4
O1
C1
O2
C2
C3
S1
S2
C12
C13
C11
O8
C16
C17
C18
O7
C10
O6
C14
O9
C15
O10
C7
C9
O3
C8

Fig. 2