research communications

Synthesis and crystal structure of topiramate azidosulfate at 90 K and 298 K

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The low (90 K) and room (298 K) temperature crystal structures of topiramate azidosulfate [systematic name 2,3:4,5-bis-O-(1-methylethylidene)-β-D-fructopyranose azidosulfate], $C_{12}H_{19}N_3O_8S$, an intermediate in the synthesis of the anti-convulsant drug topiramate, are described. Topiramate azidosulfate (**I**) finds use as a reference impurity standard for topiramate. A modified synthesis and some spectroscopic details are also presented.

1. Chemical context

Topiramate, sold under the brand name Topamax (amongst others), is a carbonic anhydrase inhibitor, used alone or with other medications, to treat epilepsy and to prevent migraines (Maryanoff *et al.*, 1987; 1998; Maryanoff, 2009). It is also prescribed for the treatment of bipolar disorder, post-traumatic stress disorder, mood instability disorder, binge-eating disorders, bulimia nervosa and obesity (Silberstein *et al.*, 2005). The vibrational and thermal properties of topiramate were investigated by Sena *et al.* (2008). Topiramate azidosulfate (a topiramate intermediate) is useful as a reference impurity standard. In view of the importance of topiramate and its derivatives, this paper reports the synthesis, crystal structure, and some spectroscopic data for topiramate azidosulfate, $C_{12}H_{19}N_3O_8S$, at low and room temperature (90 K and 298 K).
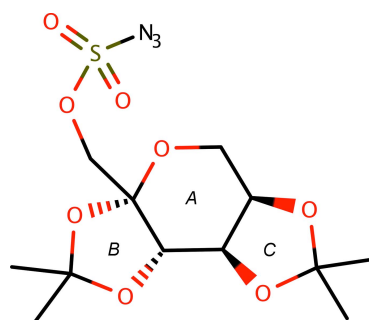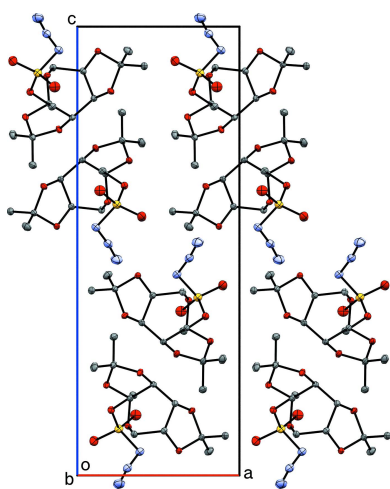
2. Structural commentary

The molecule of **I** (see scheme and Fig. 1) has a central core consisting of three fused rings: a pyran ring (labelled *A* in the scheme) with two fused dioxolane rings (labelled *B* and *C*).

Table 1
Cremer–Pople ring-puckering parameters (Å, °) for **I** at 90 K.

Pyran	Q	θ	φ
A: O1, C1, C2, C3, C4, C5	0.6368 (16)	100.85 (14)	142.37 (15)
Dioxolane	Q_2	φ_2	
B: O4, C2, C1, O5, C9	0.3076 (15)	4.5 (3)	
C: O2, C4, C3, O3, C6	0.3539 (16)	133.4 (3)	

Cremer–Pople ring-puckering parameters were calculated using *PLATON* (Spek, 2020). For six-membered rings, the θ angles for ideal 'boat', 'twist-boat', and 'screw-boat' configurations are $\theta = 90°$ (boat, twist-boat) and $\theta = 112.5°$ (screw-boat). The φ values, are quantified as either $(60k)°$ (boat) or $(60k + 30)$ (twist-boat, screw-boat), with the one having k closest to an integer giving the conformation (Boeyens, 1978). Thus, pyran ring A in **I** is between 'twist-boat' and 'screw boat', though marginally closer to the former. For five-membered rings, φ quantified as either $(36k)°$ ('envelope') or $(36k + 18)°$ ('half-chair') with k closest to an integer (Cremer & Pople, 1975), assigns dioxolane B as an 'envelope' configuration and dioxolane C as between 'envelope' and 'half-chair' conformations, though somewhat closer to the latter.

Table 2
Selected torsion angles (°) for **I** at 90 K.

N1—S1—O6—C12	61.39 (12)	S1—O6—C12—C1	−133.17 (11)
O6—S1—N1—N2	71.03 (13)	C2—C1—C12—O6	177.58 (12)

The points of fusion, atoms C1, C2, C3, C4 (Fig. 1), are contiguous chiral centres, the absolute configurations of which were confirmed unambiguously from the anomalous scattering by the sulfur to be 1*S*, 2*S*, 3*R*, 4*R* (see Flack, 1983; Hooft *et al.*, 2008; Parsons *et al.*, 2013). All three rings are non-planar, as indicated by their r.m.s. deviations from planarity (pyran *A*: 0.2597 Å; dioxolanes *B*, *C*: 0.1375, 0.1583 Å respectively) and by their Cremer–Pople (1975) ring-puckering parameters (Table 1). The distal carbon atoms of the dioxolane rings (*i.e.*, C6 and C9) each bear two methyl groups. The azidosulfonate group attaches to atom C1 *via* a methylene linker, with the position of the azide relative to the fused-ring system determined by torsions about four bonds (C1—C12, C12—O6, O6—S1, S1—N1), as summarized in Table 2. The structure was refined against both low-temperature (90 K) and room-
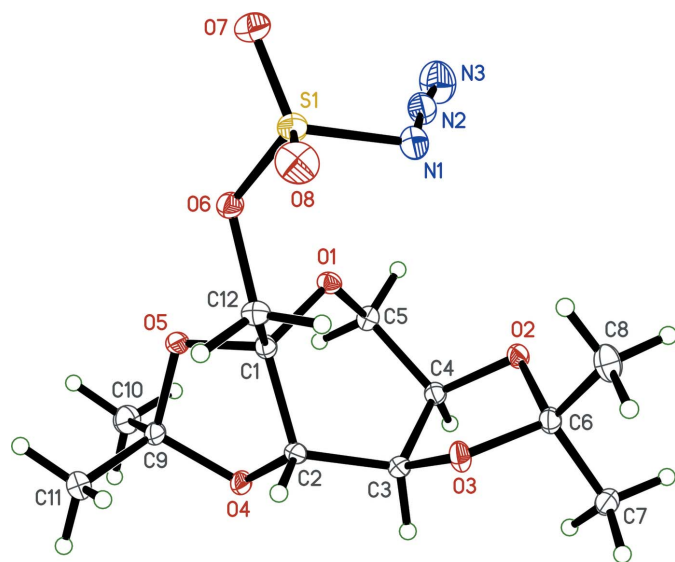
Figure 1
An ellipsoid plot of **I** (50% probability) for the structure at 90 K. The structure at 298 K is essentially unchanged, other than having much larger ellipsoids.

Table 3
Hydrogen bonds and short intermolecular contacts (Å, °) for **I** at 90 K.

| D—H···A	D—H	H···A	D···A	D—H···A
C7—H7A···O5[i]	0.98	2.50	3.456 (2)	164.8
C7—H7A···O1[i]	0.98	2.65	3.473 (2)	141.3
C5—H5B···O8[ii]	0.99	2.65	3.328 (2)	125.5
C12—H12A···O4[iii]	0.99	2.58	3.163 (2)	117.4

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

temperature (298 K) data in order to analyse the behaviour of methyl atom C7 (see Section 3: *Supramolecular features*). As there are no substantive differences, unless stated otherwise, numerical quantities quoted in the discussion pertain to the low-temperature structure.

3. Supramolecular features

There are no strong intermolecular interactions in crystals of **I**. The 'HTAB' instruction in *SHELXL* flags four 'potential hydrogen bonds' (Table 3), but two of these have very small C—H···O angles, such that the associated interaction energy would be negligible (Wood *et al.*, 2009). The remaining two involve contacts between the methyl group at C7 with O1[i] and O5[i] of an adjacent molecule [symmetry code: (i) $x - 1$, y, z], the latter being the stronger of the two. During structure analysis, the question arose of whether these contacts would be structurally significant, owing to the possibility of rapid methyl-group rotation at room temperature (Riddell & Rogerson, 1996; 1997). To answer this, the structure was also refined using room-temperature data. At low temperature (90 K) and room temperature (298 K), difference electron density for the three C7 methyl hydrogen atoms is very well resolved (Fig. 2), implying the absence of any disorder, rotational or static. Analysis of the Hirshfeld surface (Spackman & Jayatilaka, 2009) mapped over d_{norm} for **I** using *Crystal-Explorer* (Spackman *et al.*, 2021) reveals only two (equivalent) prominent red spots, corresponding to the C7—H7A···O5[i] interactions, in which the methyl group at C7 juts into a
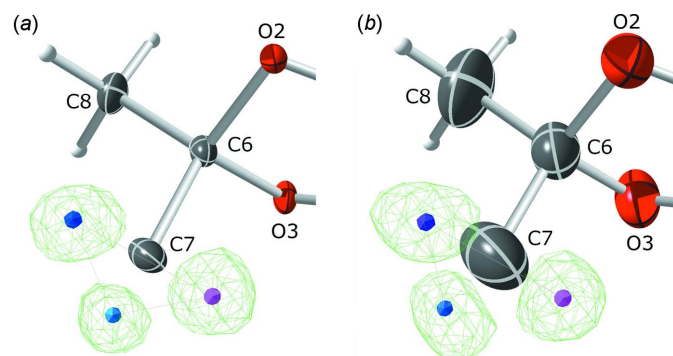
Figure 2
Difference-electron density showing the presence of well-ordered hydrogen atoms at both (*a*) 90 K and (*b*) 298 K for the methyl group at C7. Ellipsoids are drawn at the 50% probability level. Diagram generated using *ShelXle* (Hübschle *et al.*, 2011).
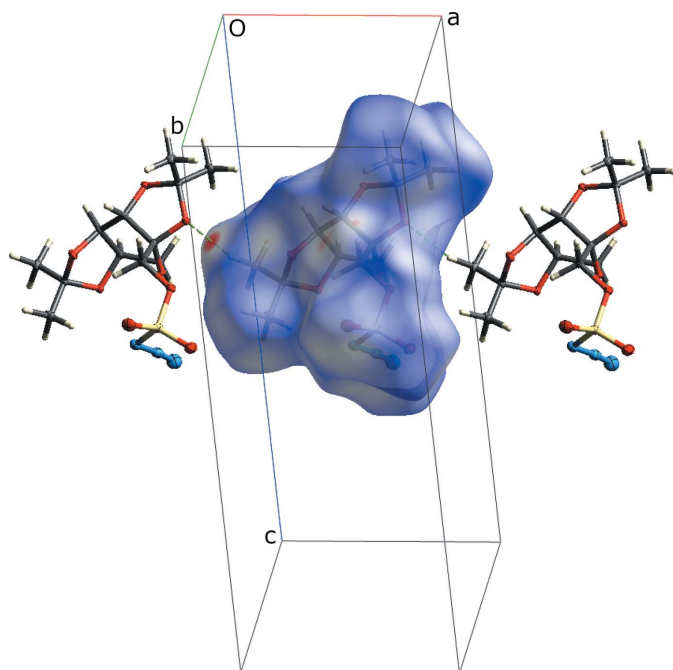
Figure 3
A plot of the Hirshfeld surface calculated over d_{norm} for **I** at 90 K, showing two adjacent molecules. Hydrogen bonds are drawn as green dashed lines. The red spot at the left corresponds to the C7—H7A···O5[i] [symmetry code: (i) $x - 1, y, z$] hydrogen bond (Table 3). The symmetry-equivalent red spot on the right side of the Hirshfeld surface is obscured from view.
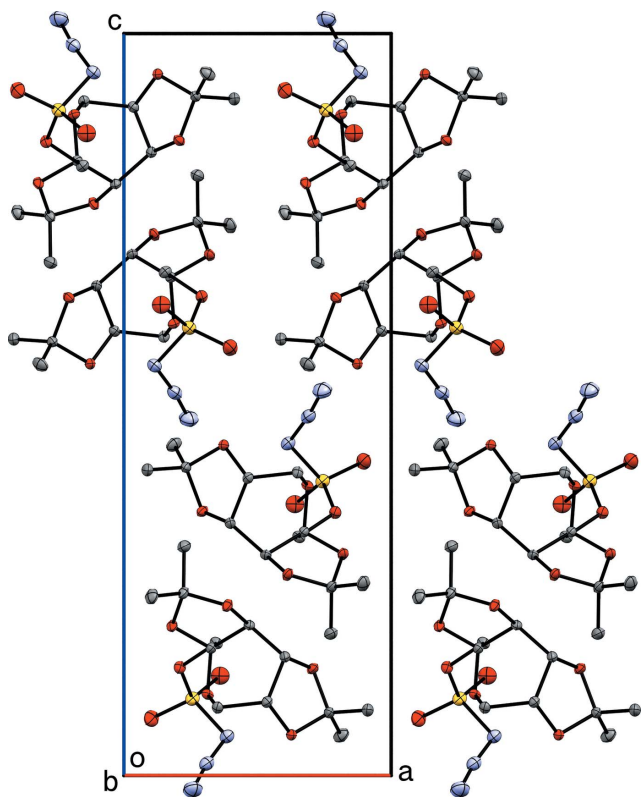
Figure 4
A packing plot of **I** viewed in projection down the *b*-axis, showing segregation of like groups, leading to the formation of double layers parallel to the *ab* plane. Diagram generated using *Mercury* (Macrae *et al.*, 2020).

concave recess of an adjacent molecule. These hydrogen bonds link the molecules into chains that extend along the *a*-axis direction (Fig. 3). There are no especially short contacts involving the azido group; N2 and N3 are 3.118 (2) and 3.166 (2) Å, respectively from a screw-related sulfonyl O7 (*via* $\frac{1}{2} + x, \frac{3}{2} - y, 1 - z$), but these are marginally greater than the sum of van der Waals radii of Bondi (1964). In spite of the lack of extensive intermolecular interactions, the overall packing exhibits segregation of like groups, leading to double layers that extend in the *ab* plane (Fig. 4). A summary of the various atom–atom contacts obtained using *CrystalExplorer* fingerprint plots is given in Fig. 5.

4. Database survey

A search of the Cambridge Structural Database (version 5.43 with updates through June 2022; Groom *et al.*, 2016) for the three-ring core of topiramate plus the four methyl groups, but
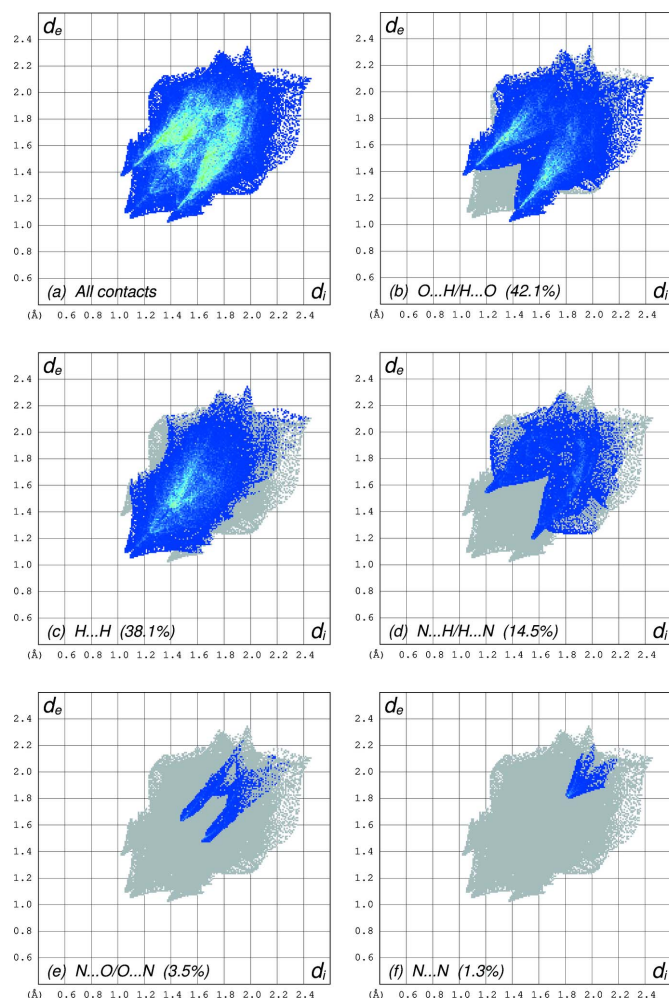
Figure 5
Fingerprint plots obtained from a Hirshfeld surface analysis for **I** at 90 K using *CrystalExplorer* (Spackman *et al.*, 2021). (*a*) All contacts, (*b*) O···H/H···O (42.1% coverage), (*c*) H···H (38.1%), (*d*) N···H/H···N (14.5%), (*e*) N···O/O···N (3.5%), (*f*) N···N (1.3%). All other contacts are negligible.

Table 4
Experimental details.

	I at 90 K	I at 298 K
Crystal data		
Chemical formula	$C_{12}H_{19}N_3O_8S$	$C_{12}H_{19}N_3O_8S$
M_r	365.36	365.36
Crystal system, space group	Orthorhombic, $P2_12_12_1$	Orthorhombic, $P2_12_12_1$
Temperature (K)	90	298
a, b, c (Å)	7.9857 (4), 9.0145 (4), 22.1621 (10)	8.0717 (8), 9.1135 (12), 22.506 (3)
V (Å^3)	1595.39 (13)	1655.6 (3)
Z	4	4
Radiation type	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.25	0.24
Crystal size (mm)	$0.30 \times 0.28 \times 0.20$	$0.24 \times 0.22 \times 0.14$
Data collection		
Diffractometer	Bruker D8 Venture dual source	Bruker D8 Venture dual source
Absorption correction	Multi-scan *SADABS* (Krause *et al.*, 2015)	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.845, 0.958	0.815, 0.959
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	22942, 3662, 3599	22913, 3786, 3523
R_{int}	0.031	0.067
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.649	0.650
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.023, 0.060, 1.07	0.036, 0.099, 1.04
No. of reflections	3662	3786
No. of parameters	221	221
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å^{-3})	0.27, −0.27	0.20, −0.27
Absolute structure	Flack x determined using 1497 quotients $[(I^+)−(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)	Flack x determined using 1388 quotients $[(I^+)−(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	−0.006 (18)	0.07 (5)

Computer programs: *APEX3* (Bruker, 2016), *SHELXT* (Sheldrick, 2015a), *SHELXL2019/2* (Sheldrick, 2015b), *XP* in *SHELXTL* (Sheldrick, 2008), *SHELXTL* (Sheldrick, 2008) and *publCIF* (Westrip, 2010).

disregarding stereochemistry yielded 239 hits. A search fragment also including –CH$_2$−Z (where Z is not H) attached to the equivalent of C1 in **I** returned 26 hits (21 excluding duplicates). A search using the keyword 'topiramate' gave only three hits, all being the structure of topiramate itself (with NH$_2$ in place of N$_3$ in **I**): SEQKAA (Maryanoff *et al.*, 1998) and duplicates SEQKAA01 (Kubicki *et al.*, 1999) and SEQKAA02 (Bolte, 2005). An amido derivative (with NHCHMePh in place of N$_3$) is present as entry ZARCEC (Xie *et al.*, 2012). These crystal structures all have the symmetry of $P2_12_12_1$, but pack differently from **I**. SEQKAA (and duplicates) form a tri-periodic hydrogen-bonded supramolecular assembly, while ZARCEC forms $C(4)$ chains (notation after Etter *et al.*, 1990).

5. Synthesis, crystallization and spectroscopic details

Topiramate azidosulfate was synthesized using a modification of procedures found in the literature (Maryanoff *et al.*, 1987; Kankan *et al.*, 2004; Arvai *et al.*, 2006; Koruyucu *et al.*, 2016). The synthesis involved three steps, *viz.*, (1) synthesis of 2,3:4,5 bis-*O*-(1-methylethylidene)-β-D-fructopyranose, (2) synthesis of 2,3:4,5-bis-*O*-(1-methylethylidene)-1-chlorosulfate-β-D-fructopyranose, and (3) synthesis of topiramate azidosulfate (**I**), as depicted in Fig. 6. X-ray quality crystals of **I** were obtained by crystallization from dichloromethane (m.p.: 358–359 K). Some spectroscopic details are as follows:
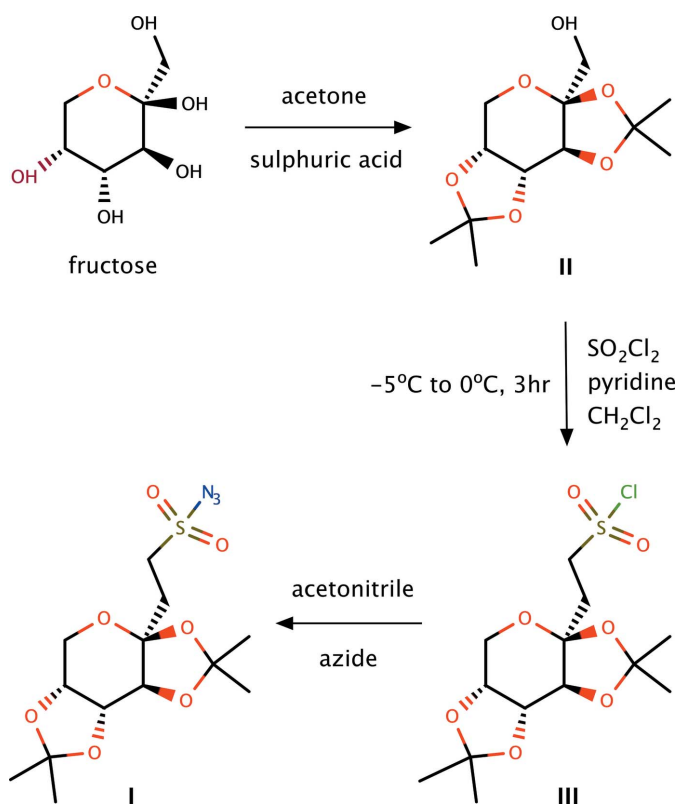
Figure 6
The reaction scheme for the synthesis of **I** starting from fructose.

IR (cm^{-1}): 2157 (N=N=N stretching); 1392 (S=O stretching); 1167 and 1081 (C—O stretching); ^{1}H NMR: CDCl$_3$ (400 MHz, δ ppm): 1.355 (3H, s, –CH$_3$); 1.422 (3H, s, –CH$_3$); 1.489 (3H, s, –CH$_3$); 1.566 (3H, s, –CH$_3$); 3.783–3.817 and 3.908–3.945 (2H, dd, –CH$_2$); 4.246–4.268 (1H, dd, –CH); 4.306–4.332 (2H, m, –CH$_2$); 4.398–4.424 (1H, dd, –CH); 4.622–4.649 (1H, dd, –CH). MS m/z: 364.03 $(M-H)^+$.

6. Refinement

Crystal data, data collection, and structure refinement details are summarized in Table 4. All H atoms were found in difference-Fourier maps, but subsequently included in the refinement using riding models, with constrained distances set to 0.98 Å (RCH$_3$), 0.99 Å (R_2CH$_2$) and 1.00 Å (R_3CH). U_{iso}(H) parameters were set to values of either $1.2U_{eq}$ or $1.5U_{eq}$ (RCH$_3$ only) of the attached atom. The absolute configuration was determined unambiguously from the anomalous scattering by sulfur using established methods (Flack, 1983; Hooft $et\ al.$, 2008; Parsons $et\ al.$, 2013).

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Synthesis and crystal structure of topiramate azidosulfate at 90 K and 298 K

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Computing details

For both structures, data collection: *APEX3* (Bruker, 2016); cell refinement: *APEX3* (Bruker, 2016); data reduction: *APEX3* (Bruker, 2016); program(s) used to solve structure: *SHELXT* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2019/2* (Sheldrick, 2015b); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008) and *publCIF* (Westrip, 2010).

2,3:4,5-Bis-*O*-(1-methylethylidene)-*β*-D-fructopyranose azidosulfate (I-90K)

Crystal data

$C_{12}H_{19}N_3O_8S$	$D_x = 1.521$ Mg m^{-3}
$M_r = 365.36$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Orthorhombic, $P2_12_12_1$	Cell parameters from 9985 reflections
$a = 7.9857$ (4) Å	$\theta = 3.4–27.5°$
$b = 9.0145$ (4) Å	$\mu = 0.25$ mm^{-1}
$c = 22.1621$ (10) Å	$T = 90$ K
$V = 1595.39$ (13) Å^3	Cut block, colourless
$Z = 4$	$0.30 \times 0.28 \times 0.20$ mm
$F(000) = 768$	

Data collection

Bruker D8 Venture dual source diffractometer	22942 measured reflections
Radiation source: microsource	3662 independent reflections
Detector resolution: 7.41 pixels mm^{-1}	3599 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.031$
Absorption correction: multi-scan *SADABS* (Krause *et al.*, 2015)	$\theta_{max} = 27.5°$, $\theta_{min} = 2.4°$
	$h = -10 \rightarrow 10$
	$k = -11 \rightarrow 11$
$T_{min} = 0.845$, $T_{max} = 0.958$	$l = -28 \rightarrow 28$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.023$	H-atom parameters constrained
$wR(F^2) = 0.060$	$w = 1/[\sigma^2(F_o^2) + (0.0311P)^2 + 0.3897P]$
$S = 1.07$	where $P = (F_o^2 + 2F_c^2)/3$
3662 reflections	$(\Delta/\sigma)_{max} = 0.001$
221 parameters	$\Delta\rho_{max} = 0.27$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.27$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	

Absolute structure: Flack *x* determined using 1497 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)

Absolute structure parameter: −0.006 (18)

Special details

Experimental. The crystal was mounted using polyisobutene oil on the tip of a fine glass fibre, which was fastened in a copper mounting pin with electrical solder. It was placed directly into the cold gas stream of a liquid-nitrogen based cryostat (Hope, 1994; Parkin & Hope, 1998).
Diffraction data were collected with the crystal at 90K, which is standard practice in this laboratory for the majority of flash-cooled crystals.

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

Refinement. Refinement progress was checked using *Platon* (Spek, 2020) and by an *R*-tensor (Parkin, 2000). The final model was further checked with the IUCr utility *checkCIF*.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.74373 (5)	0.79578 (4)	0.39659 (2)	0.01482 (10)
O1	0.68696 (14)	0.40140 (12)	0.39079 (5)	0.0110 (2)
O2	0.37338 (14)	0.30579 (13)	0.44523 (5)	0.0132 (2)
O3	0.29357 (14)	0.40358 (13)	0.35605 (5)	0.0140 (2)
O4	0.61310 (14)	0.21617 (13)	0.27084 (5)	0.0133 (2)
O5	0.82007 (14)	0.37553 (13)	0.29850 (5)	0.0129 (2)
O6	0.79097 (14)	0.66339 (13)	0.35441 (5)	0.0131 (2)
O7	0.89734 (17)	0.84031 (15)	0.42335 (6)	0.0227 (3)
O8	0.63978 (18)	0.89829 (14)	0.36559 (6)	0.0233 (3)
N1	0.61434 (19)	0.72167 (17)	0.44750 (6)	0.0176 (3)
N2	0.6901 (2)	0.63604 (18)	0.48404 (7)	0.0195 (3)
N3	0.7424 (3)	0.5607 (2)	0.51895 (7)	0.0303 (4)
C1	0.6742 (2)	0.42586 (18)	0.32847 (7)	0.0111 (3)
C2	0.5308 (2)	0.34038 (18)	0.29730 (7)	0.0112 (3)
H2	0.478553	0.403202	0.265228	0.013*
C3	0.39740 (19)	0.28231 (19)	0.33997 (7)	0.0121 (3)
H3	0.329322	0.204201	0.319356	0.015*
C4	0.46440 (19)	0.22228 (18)	0.40096 (7)	0.0115 (3)
H4	0.440093	0.113922	0.405033	0.014*
C5	0.6496 (2)	0.25192 (18)	0.40849 (7)	0.0115 (3)
H5A	0.682113	0.236671	0.451149	0.014*
H5B	0.714547	0.181756	0.383316	0.014*
C6	0.2319 (2)	0.36980 (19)	0.41541 (7)	0.0131 (3)
C7	0.0877 (2)	0.2609 (2)	0.41185 (7)	0.0172 (4)
H7A	−0.000562	0.301745	0.386016	0.026*
H7B	0.042898	0.243492	0.452406	0.026*
H7C	0.127531	0.166947	0.394831	0.026*
C8	0.1871 (2)	0.5135 (2)	0.44592 (8)	0.0196 (4)

H8A	0.285516	0.578470	0.446858	0.029*
H8B	0.149912	0.493503	0.487260	0.029*
H8C	0.096764	0.562297	0.423507	0.029*
C9	0.7769 (2)	0.26520 (18)	0.25392 (7)	0.0132 (3)
C10	0.8959 (2)	0.1363 (2)	0.25870 (8)	0.0190 (3)
H10A	0.865031	0.060356	0.229074	0.028*
H10B	0.889904	0.094120	0.299397	0.028*
H10C	1.010288	0.170461	0.250730	0.028*
C11	0.7748 (2)	0.3354 (2)	0.19133 (7)	0.0187 (3)
H11A	0.748270	0.259460	0.161161	0.028*
H11B	0.884938	0.378143	0.182579	0.028*
H11C	0.689687	0.413681	0.190035	0.028*
C12	0.6546 (2)	0.59209 (18)	0.32148 (7)	0.0130 (3)
H12A	0.659331	0.619732	0.278277	0.016*
H12B	0.545271	0.624352	0.337951	0.016*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.01728 (19)	0.01052 (18)	0.01667 (18)	−0.00154 (16)	0.00150 (16)	−0.00177 (14)
O1	0.0132 (5)	0.0106 (5)	0.0093 (5)	−0.0008 (4)	−0.0014 (4)	0.0007 (4)
O2	0.0101 (5)	0.0190 (6)	0.0104 (5)	0.0033 (5)	−0.0002 (4)	−0.0009 (4)
O3	0.0127 (5)	0.0181 (6)	0.0112 (5)	0.0047 (4)	0.0029 (4)	0.0018 (4)
O4	0.0109 (5)	0.0147 (5)	0.0142 (5)	−0.0016 (5)	0.0031 (4)	−0.0049 (5)
O5	0.0102 (5)	0.0154 (6)	0.0131 (5)	−0.0004 (5)	0.0011 (4)	−0.0052 (4)
O6	0.0126 (5)	0.0120 (5)	0.0148 (5)	−0.0008 (4)	0.0007 (4)	−0.0020 (4)
O7	0.0210 (7)	0.0223 (7)	0.0247 (6)	−0.0071 (6)	0.0008 (5)	−0.0065 (5)
O8	0.0302 (7)	0.0121 (6)	0.0277 (7)	0.0037 (5)	−0.0002 (6)	0.0007 (5)
N1	0.0176 (7)	0.0187 (7)	0.0164 (6)	0.0006 (6)	0.0027 (6)	−0.0004 (6)
N2	0.0209 (7)	0.0195 (7)	0.0180 (7)	−0.0028 (6)	0.0044 (6)	−0.0021 (6)
N3	0.0337 (9)	0.0329 (9)	0.0242 (8)	0.0009 (9)	0.0026 (8)	0.0072 (7)
C1	0.0104 (7)	0.0127 (8)	0.0102 (7)	0.0010 (6)	0.0006 (6)	0.0003 (6)
C2	0.0105 (7)	0.0133 (7)	0.0098 (6)	0.0000 (6)	−0.0005 (6)	−0.0023 (6)
C3	0.0095 (7)	0.0155 (8)	0.0114 (6)	−0.0002 (7)	−0.0003 (6)	−0.0017 (6)
C4	0.0117 (7)	0.0118 (7)	0.0111 (7)	0.0002 (6)	0.0008 (6)	−0.0005 (6)
C5	0.0112 (7)	0.0102 (7)	0.0131 (7)	0.0008 (6)	0.0002 (5)	0.0024 (6)
C6	0.0106 (7)	0.0187 (8)	0.0102 (6)	0.0028 (7)	0.0006 (6)	0.0012 (5)
C7	0.0114 (7)	0.0264 (9)	0.0140 (7)	−0.0016 (7)	0.0000 (6)	0.0015 (7)
C8	0.0210 (8)	0.0187 (9)	0.0190 (8)	0.0051 (7)	0.0052 (7)	−0.0005 (7)
C9	0.0114 (7)	0.0151 (7)	0.0130 (7)	−0.0026 (6)	0.0028 (6)	−0.0042 (6)
C10	0.0159 (8)	0.0192 (8)	0.0217 (8)	0.0028 (7)	0.0026 (7)	−0.0053 (7)
C11	0.0174 (8)	0.0256 (9)	0.0132 (7)	−0.0033 (7)	0.0025 (6)	−0.0014 (6)
C12	0.0138 (8)	0.0122 (7)	0.0130 (7)	−0.0005 (6)	−0.0021 (6)	0.0002 (6)

Geometric parameters (Å, º)

S1—O8	1.4195 (14)	C4—C5	1.512 (2)
S1—O7	1.4205 (14)	C4—H4	1.0000

S1—O6	1.5622 (12)	C5—H5A	0.9900
S1—N1	1.6694 (15)	C5—H5B	0.9900
O1—C1	1.4022 (18)	C6—C8	1.505 (2)
O1—C5	1.4347 (19)	C6—C7	1.515 (2)
O2—C6	1.4306 (19)	C7—H7A	0.9800
O2—C4	1.4345 (18)	C7—H7B	0.9800
O3—C3	1.4176 (19)	C7—H7C	0.9800
O3—C6	1.4373 (18)	C8—H8A	0.9800
O4—C2	1.4245 (19)	C8—H8B	0.9800
O4—C9	1.4306 (18)	C8—H8C	0.9800
O5—C1	1.4156 (19)	C9—C10	1.505 (2)
O5—C9	1.4436 (19)	C9—C11	1.525 (2)
O6—C12	1.4600 (19)	C10—H10A	0.9800
N1—N2	1.272 (2)	C10—H10B	0.9800
N2—N3	1.111 (2)	C10—H10C	0.9800
C1—C12	1.515 (2)	C11—H11A	0.9800
C1—C2	1.543 (2)	C11—H11B	0.9800
C2—C3	1.518 (2)	C11—H11C	0.9800
C2—H2	1.0000	C12—H12A	0.9900
C3—C4	1.551 (2)	C12—H12B	0.9900
C3—H3	1.0000		
O8—S1—O7	121.55 (8)	H5A—C5—H5B	108.2
O8—S1—O6	110.42 (7)	O2—C6—O3	103.74 (12)
O7—S1—O6	104.91 (7)	O2—C6—C8	109.11 (13)
O8—S1—N1	103.04 (8)	O3—C6—C8	108.07 (14)
O7—S1—N1	111.43 (8)	O2—C6—C7	111.28 (13)
O6—S1—N1	104.37 (7)	O3—C6—C7	110.49 (13)
C1—O1—C5	113.70 (12)	C8—C6—C7	113.64 (14)
C6—O2—C4	107.21 (11)	C6—C7—H7A	109.5
C3—O3—C6	105.50 (12)	C6—C7—H7B	109.5
C2—O4—C9	106.66 (12)	H7A—C7—H7B	109.5
C1—O5—C9	110.20 (12)	C6—C7—H7C	109.5
C12—O6—S1	117.09 (10)	H7A—C7—H7C	109.5
N2—N1—S1	112.27 (12)	H7B—C7—H7C	109.5
N3—N2—N1	173.36 (19)	C6—C8—H8A	109.5
O1—C1—O5	110.61 (13)	C6—C8—H8B	109.5
O1—C1—C12	105.31 (13)	H8A—C8—H8B	109.5
O5—C1—C12	110.74 (13)	C6—C8—H8C	109.5
O1—C1—C2	114.60 (13)	H8A—C8—H8C	109.5
O5—C1—C2	103.91 (12)	H8B—C8—H8C	109.5
C12—C1—C2	111.80 (13)	O4—C9—O5	104.59 (11)
O4—C2—C3	108.01 (13)	O4—C9—C10	108.69 (14)
O4—C2—C1	103.56 (12)	O5—C9—C10	109.45 (13)
C3—C2—C1	114.46 (13)	O4—C9—C11	110.88 (13)
O4—C2—H2	110.2	O5—C9—C11	109.83 (13)
C3—C2—H2	110.2	C10—C9—C11	113.04 (14)
C1—C2—H2	110.2	C9—C10—H10A	109.5

O3—C3—C2	107.54 (13)	C9—C10—H10B	109.5
O3—C3—C4	104.57 (12)	H10A—C10—H10B	109.5
C2—C3—C4	114.90 (13)	C9—C10—H10C	109.5
O3—C3—H3	109.9	H10A—C10—H10C	109.5
C2—C3—H3	109.9	H10B—C10—H10C	109.5
C4—C3—H3	109.9	C9—C11—H11A	109.5
O2—C4—C5	109.11 (13)	C9—C11—H11B	109.5
O2—C4—C3	103.78 (12)	H11A—C11—H11B	109.5
C5—C4—C3	111.83 (13)	C9—C11—H11C	109.5
O2—C4—H4	110.6	H11A—C11—H11C	109.5
C5—C4—H4	110.6	H11B—C11—H11C	109.5
C3—C4—H4	110.6	O6—C12—C1	107.89 (13)
O1—C5—C4	109.82 (13)	O6—C12—H12A	110.1
O1—C5—H5A	109.7	C1—C12—H12A	110.1
C4—C5—H5A	109.7	O6—C12—H12B	110.1
O1—C5—H5B	109.7	C1—C12—H12B	110.1
C4—C5—H5B	109.7	H12A—C12—H12B	108.4
O8—S1—O6—C12	−48.73 (13)	C6—O2—C4—C5	136.56 (13)
O7—S1—O6—C12	178.68 (11)	C6—O2—C4—C3	17.21 (15)
N1—S1—O6—C12	61.39 (12)	O3—C3—C4—O2	6.99 (15)
O8—S1—N1—N2	−173.57 (13)	C2—C3—C4—O2	124.63 (14)
O7—S1—N1—N2	−41.67 (15)	O3—C3—C4—C5	−110.49 (14)
O6—S1—N1—N2	71.03 (13)	C2—C3—C4—C5	7.15 (19)
C5—O1—C1—O5	−80.38 (16)	C1—O1—C5—C4	−70.46 (16)
C5—O1—C1—C12	159.94 (12)	O2—C4—C5—O1	−69.49 (16)
C5—O1—C1—C2	36.65 (18)	C3—C4—C5—O1	44.74 (17)
C9—O5—C1—O1	121.68 (13)	C4—O2—C6—O3	−35.14 (15)
C9—O5—C1—C12	−121.96 (14)	C4—O2—C6—C8	−150.14 (14)
C9—O5—C1—C2	−1.77 (16)	C4—O2—C6—C7	83.68 (15)
C9—O4—C2—C3	−154.60 (12)	C3—O3—C6—O2	39.69 (15)
C9—O4—C2—C1	−32.84 (15)	C3—O3—C6—C8	155.43 (13)
O1—C1—C2—O4	−99.81 (14)	C3—O3—C6—C7	−79.67 (16)
O5—C1—C2—O4	21.00 (15)	C2—O4—C9—O5	32.11 (15)
C12—C1—C2—O4	140.47 (13)	C2—O4—C9—C10	148.94 (13)
O1—C1—C2—C3	17.5 (2)	C2—O4—C9—C11	−86.21 (15)
O5—C1—C2—C3	138.33 (14)	C1—O5—C9—O4	−18.10 (16)
C12—C1—C2—C3	−102.20 (16)	C1—O5—C9—C10	−134.40 (14)
C6—O3—C3—C2	−150.97 (12)	C1—O5—C9—C11	100.94 (15)
C6—O3—C3—C4	−28.39 (15)	S1—O6—C12—C1	−133.17 (11)
O4—C2—C3—O3	−167.78 (12)	O1—C1—C12—O6	52.54 (16)
C1—C2—C3—O3	77.46 (16)	O5—C1—C12—O6	−67.06 (16)
O4—C2—C3—C4	76.26 (16)	C2—C1—C12—O6	177.58 (12)
C1—C2—C3—C4	−38.49 (19)		

Hydrogen-bond geometry (Å, °)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C7—H7A⋯O1[i]	0.98	2.65	3.473 (2)	141
C7—H7A⋯O5[i]	0.98	2.50	3.456 (2)	165
C5—H5B⋯O8[ii]	0.99	2.65	3.328 (2)	126
C12—H12A⋯O4[iii]	0.99	2.58	3.163 (2)	117

Symmetry codes: (i) $x-1, y, z$; (ii) $x, y-1, z$; (iii) $-x+1, y+1/2, -z+1/2$.

(I-298K)

Crystal data

$C_{12}H_{19}N_3O_8S$
$M_r = 365.36$
Orthorhombic, $P2_12_12_1$
$a = 8.0717$ (8) Å
$b = 9.1135$ (12) Å
$c = 22.506$ (3) Å
$V = 1655.6$ (3) Å^3
$Z = 4$
$F(000) = 768$

$D_x = 1.466$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9504 reflections
$\theta = 3.4–27.5°$
$\mu = 0.24$ mm^{-1}
$T = 298$ K
Cut block, colourless
$0.24 \times 0.22 \times 0.14$ mm

Data collection

Bruker D8 Venture dual source
   diffractometer
Radiation source: microsource
Detector resolution: 7.41 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan
   (*SADABS*; Krause *et al.*, 2015)
$T_{min} = 0.815$, $T_{max} = 0.959$

22913 measured reflections
3786 independent reflections
3523 reflections with $I > 2\sigma(I)$
$R_{int} = 0.067$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.4°$
$h = -10 \rightarrow 10$
$k = -11 \rightarrow 11$
$l = -29 \rightarrow 25$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.099$
$S = 1.04$
3786 reflections
221 parameters
0 restraints
Primary atom site location: structure-invariant
   direct methods
Secondary atom site location: difference Fourier
   map

Hydrogen site location: difference Fourier map
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0574P)^2 + 0.1732P]$
   where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}
Absolute structure: Flack x determined using
   1388 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: 0.07 (5)

Special details

Experimental. The crystal was mounted glued to the tip of a fine glass fibre, which was fastened in a copper mounting pin with electrical solder.
Data were collected at room temperature to investigate the possibility of the methyl group at C7 undergoing rapid spinning.

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

Refinement. Refinement progress was checked using *Platon* (Spek, 2020) and by an *R*-tensor (Parkin, 2000). The final model was further checked with the IUCr utility *checkCIF*.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.74935 (9)	0.79040 (6)	0.39549 (3)	0.05008 (19)
O1	0.68675 (18)	0.39774 (16)	0.38959 (6)	0.0334 (3)
O2	0.3760 (2)	0.3015 (2)	0.44310 (7)	0.0418 (4)
O3	0.29761 (19)	0.4019 (2)	0.35639 (7)	0.0444 (4)
O4	0.6115 (2)	0.22006 (19)	0.27062 (7)	0.0405 (4)
O5	0.81632 (19)	0.37532 (19)	0.29858 (8)	0.0406 (4)
O6	0.7900 (2)	0.65798 (18)	0.35461 (8)	0.0424 (4)
O7	0.9030 (3)	0.8324 (3)	0.42018 (11)	0.0739 (7)
O8	0.6469 (4)	0.8910 (2)	0.36562 (13)	0.0810 (7)
N1	0.6247 (3)	0.7208 (3)	0.44695 (12)	0.0586 (6)
N2	0.6998 (4)	0.6366 (3)	0.48301 (13)	0.0658 (7)
N3	0.7518 (6)	0.5639 (5)	0.51657 (17)	0.1031 (12)
C1	0.6730 (3)	0.4242 (2)	0.32874 (9)	0.0317 (4)
C2	0.5312 (3)	0.3413 (3)	0.29776 (9)	0.0345 (4)
H2	0.479985	0.403642	0.267422	0.041*
C3	0.3998 (2)	0.2823 (3)	0.33972 (9)	0.0356 (4)
H3	0.333367	0.207389	0.319499	0.043*
C4	0.4662 (3)	0.2211 (2)	0.39892 (10)	0.0349 (4)
H4	0.442711	0.115874	0.402132	0.042*
C5	0.6487 (3)	0.2499 (2)	0.40661 (10)	0.0354 (5)
H5A	0.679702	0.234428	0.447786	0.042*
H5B	0.711710	0.181983	0.382284	0.042*
C6	0.2387 (3)	0.3687 (3)	0.41482 (10)	0.0414 (5)
C7	0.0929 (3)	0.2650 (4)	0.41220 (13)	0.0581 (8)
H7A	0.005298	0.309049	0.389497	0.087*
H7B	0.054350	0.245317	0.451757	0.087*
H7C	0.126270	0.174873	0.393681	0.087*
C8	0.2014 (4)	0.5115 (4)	0.44567 (16)	0.0670 (9)
H8A	0.299085	0.571603	0.446096	0.101*
H8B	0.166869	0.492278	0.485738	0.101*
H8C	0.114454	0.561748	0.424853	0.101*
C9	0.7734 (3)	0.2684 (3)	0.25411 (10)	0.0420 (5)
C10	0.8905 (4)	0.1406 (3)	0.25816 (15)	0.0605 (7)
H10A	0.860101	0.067868	0.229299	0.091*
H10B	0.884762	0.098732	0.297250	0.091*
H10C	1.001458	0.173537	0.250531	0.091*
C11	0.7710 (4)	0.3396 (4)	0.19315 (12)	0.0612 (7)
H11A	0.742453	0.267603	0.163763	0.092*

H11B	0.878574	0.379166	0.184444	0.092*
H11C	0.690558	0.417191	0.192682	0.092*
C12	0.6536 (3)	0.5884 (2)	0.32299 (11)	0.0399 (5)
H12A	0.655642	0.616531	0.281430	0.048*
H12B	0.548641	0.619096	0.339866	0.048*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0602 (4)	0.0303 (3)	0.0598 (4)	−0.0049 (3)	0.0056 (3)	−0.0074 (2)
O1	0.0365 (7)	0.0311 (7)	0.0326 (7)	−0.0014 (6)	−0.0052 (6)	0.0011 (6)
O2	0.0355 (7)	0.0565 (10)	0.0333 (7)	0.0088 (8)	−0.0010 (6)	−0.0008 (7)
O3	0.0373 (8)	0.058 (1)	0.0377 (8)	0.0136 (7)	0.0045 (7)	0.0071 (7)
O4	0.0382 (7)	0.0418 (8)	0.0416 (8)	−0.0060 (7)	0.0071 (7)	−0.0148 (7)
O5	0.0312 (7)	0.0451 (9)	0.0455 (9)	−0.0025 (7)	0.0042 (6)	−0.0133 (7)
O6	0.0441 (9)	0.0327 (8)	0.0505 (9)	−0.0036 (6)	0.0048 (7)	−0.0046 (7)
O7	0.0710 (14)	0.0659 (14)	0.0848 (16)	−0.0266 (12)	0.0040 (12)	−0.0256 (12)
O8	0.110 (2)	0.0347 (10)	0.0983 (17)	0.0140 (12)	−0.0007 (16)	0.0037 (11)
N1	0.0585 (13)	0.0592 (14)	0.0583 (13)	0.0015 (12)	0.0130 (11)	−0.0079 (12)
N2	0.0774 (18)	0.0630 (16)	0.0570 (15)	−0.0066 (14)	0.0089 (14)	−0.0031 (13)
N3	0.127 (3)	0.105 (3)	0.077 (2)	0.003 (3)	0.000 (3)	0.026 (2)
C1	0.0312 (9)	0.0324 (10)	0.0315 (10)	0.0009 (8)	−0.0007 (8)	−0.0017 (8)
C2	0.0323 (10)	0.0420 (11)	0.0292 (10)	0.0006 (8)	−0.0005 (8)	−0.0045 (9)
C3	0.0276 (8)	0.0439 (11)	0.0353 (10)	−0.0019 (9)	−0.0002 (8)	−0.0058 (9)
C4	0.0332 (9)	0.0333 (10)	0.0382 (11)	0.0012 (8)	0.0028 (8)	0.0009 (9)
C5	0.0338 (10)	0.0305 (10)	0.0417 (11)	0.0041 (8)	−0.0017 (8)	0.0061 (8)
C6	0.0343 (10)	0.0544 (13)	0.0354 (11)	0.0097 (11)	0.0028 (9)	0.0025 (9)
C7	0.0346 (11)	0.091 (2)	0.0489 (14)	−0.0055 (13)	0.0014 (10)	0.0063 (15)
C8	0.075 (2)	0.0620 (18)	0.0640 (18)	0.0221 (16)	0.0206 (16)	−0.0047 (15)
C9	0.0373 (11)	0.0472 (12)	0.0416 (11)	−0.0022 (10)	0.0072 (9)	−0.013 (1)
C10	0.0526 (15)	0.0566 (15)	0.0722 (18)	0.0116 (14)	0.0114 (14)	−0.0184 (14)
C11	0.0600 (16)	0.0788 (19)	0.0449 (14)	−0.0095 (16)	0.0106 (13)	−0.0052 (13)
C12	0.0450 (12)	0.0331 (11)	0.0415 (12)	0.0021 (9)	−0.0049 (9)	0.0020 (9)

Geometric parameters (Å, º)

S1—O8	1.406 (3)	C4—C5	1.507 (3)
S1—O7	1.412 (2)	C4—H4	0.9800
S1—O6	1.5527 (17)	C5—H5A	0.9700
S1—N1	1.660 (3)	C5—H5B	0.9700
O1—C1	1.395 (3)	C6—C8	1.506 (4)
O1—C5	1.434 (3)	C6—C7	1.511 (4)
O2—C6	1.417 (3)	C7—H7A	0.9600
O2—C4	1.434 (3)	C7—H7B	0.9600
O3—C3	1.418 (3)	C7—H7C	0.9600
O3—C6	1.431 (3)	C8—H8A	0.9600
O4—C2	1.419 (3)	C8—H8B	0.9600
O4—C9	1.429 (3)	C8—H8C	0.9600

O5—C1	1.413 (3)	C9—C10	1.503 (4)
O5—C9	1.439 (3)	C9—C11	1.518 (4)
O6—C12	1.457 (3)	C10—H10A	0.9600
N1—N2	1.271 (4)	C10—H10B	0.9600
N2—N3	1.089 (5)	C10—H10C	0.9600
C1—C12	1.510 (3)	C11—H11A	0.9600
C1—C2	1.539 (3)	C11—H11B	0.9600
C2—C3	1.518 (3)	C11—H11C	0.9600
C2—H2	0.9800	C12—H12A	0.9700
C3—C4	1.540 (3)	C12—H12B	0.9700
C3—H3	0.9800		
O8—S1—O7	121.86 (16)	H5A—C5—H5B	108.2
O8—S1—O6	110.36 (14)	O2—C6—O3	104.16 (17)
O7—S1—O6	104.96 (12)	O2—C6—C8	108.8 (2)
O8—S1—N1	103.09 (17)	O3—C6—C8	107.9 (2)
O7—S1—N1	111.19 (15)	O2—C6—C7	110.9 (2)
O6—S1—N1	104.17 (11)	O3—C6—C7	110.8 (2)
C1—O1—C5	114.07 (16)	C8—C6—C7	113.7 (2)
C6—O2—C4	107.84 (16)	C6—C7—H7A	109.5
C3—O3—C6	105.87 (17)	C6—C7—H7B	109.5
C2—O4—C9	106.84 (16)	H7A—C7—H7B	109.5
C1—O5—C9	110.53 (16)	C6—C7—H7C	109.5
C12—O6—S1	117.92 (14)	H7A—C7—H7C	109.5
N2—N1—S1	112.8 (2)	H7B—C7—H7C	109.5
N3—N2—N1	173.9 (4)	C6—C8—H8A	109.5
O1—C1—O5	110.61 (17)	C6—C8—H8B	109.5
O1—C1—C12	105.30 (17)	H8A—C8—H8B	109.5
O5—C1—C12	110.87 (19)	C6—C8—H8C	109.5
O1—C1—C2	114.75 (18)	H8A—C8—H8C	109.5
O5—C1—C2	103.67 (16)	H8B—C8—H8C	109.5
C12—C1—C2	111.76 (18)	O4—C9—O5	104.36 (16)
O4—C2—C3	108.12 (18)	O4—C9—C10	108.7 (2)
O4—C2—C1	103.75 (16)	O5—C9—C10	109.3 (2)
C3—C2—C1	114.32 (17)	O4—C9—C11	110.8 (2)
O4—C2—H2	110.1	O5—C9—C11	110.0 (2)
C3—C2—H2	110.1	C10—C9—C11	113.2 (2)
C1—C2—H2	110.1	C9—C10—H10A	109.5
O3—C3—C2	107.36 (18)	C9—C10—H10B	109.5
O3—C3—C4	104.59 (17)	H10A—C10—H10B	109.5
C2—C3—C4	115.04 (17)	C9—C10—H10C	109.5
O3—C3—H3	109.9	H10A—C10—H10C	109.5
C2—C3—H3	109.9	H10B—C10—H10C	109.5
C4—C3—H3	109.9	C9—C11—H11A	109.5
O2—C4—C5	109.14 (18)	C9—C11—H11B	109.5
O2—C4—C3	103.79 (16)	H11A—C11—H11B	109.5
C5—C4—C3	112.11 (18)	C9—C11—H11C	109.5
O2—C4—H4	110.5	H11A—C11—H11C	109.5

C5—C4—H4	110.5	H11B—C11—H11C	109.5
C3—C4—H4	110.5	O6—C12—C1	108.13 (18)
O1—C5—C4	110.00 (17)	O6—C12—H12A	110.1
O1—C5—H5A	109.7	C1—C12—H12A	110.1
C4—C5—H5A	109.7	O6—C12—H12B	110.1
O1—C5—H5B	109.7	C1—C12—H12B	110.1
C4—C5—H5B	109.7	H12A—C12—H12B	108.4
O8—S1—O6—C12	−47.7 (2)	C6—O2—C4—C5	135.43 (19)
O7—S1—O6—C12	179.27 (18)	C6—O2—C4—C3	15.7 (2)
N1—S1—O6—C12	62.31 (19)	O3—C3—C4—O2	7.4 (2)
O8—S1—N1—N2	−173.4 (2)	C2—C3—C4—O2	124.95 (19)
O7—S1—N1—N2	−41.2 (3)	O3—C3—C4—C5	−110.21 (19)
O6—S1—N1—N2	71.3 (2)	C2—C3—C4—C5	7.3 (3)
C5—O1—C1—O5	−80.8 (2)	C1—O1—C5—C4	−69.5 (2)
C5—O1—C1—C12	159.36 (17)	O2—C4—C5—O1	−70.3 (2)
C5—O1—C1—C2	36.0 (2)	C3—C4—C5—O1	44.1 (2)
C9—O5—C1—O1	121.76 (19)	C4—O2—C6—O3	−33.2 (2)
C9—O5—C1—C12	−121.8 (2)	C4—O2—C6—C8	−148.0 (2)
C9—O5—C1—C2	−1.7 (2)	C4—O2—C6—C7	86.1 (2)
C9—O4—C2—C3	−154.34 (17)	C3—O3—C6—O2	38.0 (2)
C9—O4—C2—C1	−32.6 (2)	C3—O3—C6—C8	153.5 (2)
O1—C1—C2—O4	−99.94 (19)	C3—O3—C6—C7	−81.4 (2)
O5—C1—C2—O4	20.8 (2)	C2—O4—C9—O5	31.7 (2)
C12—C1—C2—O4	140.27 (19)	C2—O4—C9—C10	148.3 (2)
O1—C1—C2—C3	17.6 (3)	C2—O4—C9—C11	−86.6 (2)
O5—C1—C2—C3	138.32 (19)	C1—O5—C9—O4	−17.9 (2)
C12—C1—C2—C3	−102.2 (2)	C1—O5—C9—C10	−134.0 (2)
C6—O3—C3—C2	−150.25 (18)	C1—O5—C9—C11	101.1 (2)
C6—O3—C3—C4	−27.6 (2)	S1—O6—C12—C1	−135.17 (17)
O4—C2—C3—O3	−167.48 (17)	O1—C1—C12—O6	53.4 (2)
C1—C2—C3—O3	77.5 (2)	O5—C1—C12—O6	−66.3 (2)
O4—C2—C3—C4	76.6 (2)	C2—C1—C12—O6	178.62 (17)
C1—C2—C3—C4	−38.4 (3)		

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7A···O1ⁱ	0.96	2.70	3.531 (3)	146
C7—H7A···O5ⁱ	0.96	2.62	3.540 (3)	160
C5—H5B···O8ⁱⁱ	0.97	2.73	3.398 (3)	127
C12—H12A···O4ⁱⁱⁱ	0.97	2.63	3.234 (3)	121

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