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ISSN 2414-3146

(*R*)-3-(*tert*-Butoxycarbonyl)-5-methyl-1,2,3-oxathiazolidine 2,2-dioxide

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Received 10 June 2017
Accepted 11 June 2017

Edited by W. T. A. Harrison, University of Aberdeen, Scotland

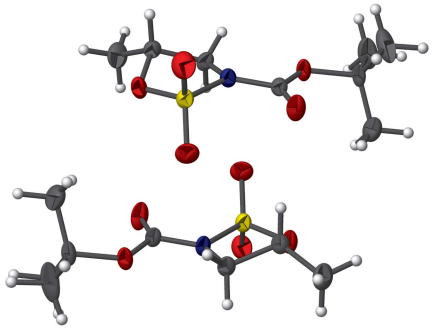
Keywords: crystal structure; C—H···O contacts; oxathiazolidine; sulfamidate.

CCDC reference: 1555404

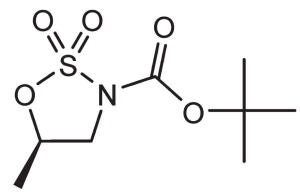
Structural data: full structural data are available from iucrdata.iucr.org

The chiral title compound, $C_8H_{15}NO_5S$, was obtained by cyclization of (*R*)-1-(*tert*-butoxycarbonylamino)-2-propanol with thionyl chloride and subsequent oxidation with sodium metaperiodate/ruthenium(IV) oxide. It crystallizes with two independent molecules in the asymmetric unit. In the crystal, C—H···O interactions link the molecules into a three-dimensional network.

3D view



Chemical scheme



Structure description

Cyclic sulfamidates are valuable reactive intermediates, because ring-opening reactions proceed with total inversion at the stereogenic centre (Meléndez & Lubell, 2003). The title compound represents such a building block derived from (*R*)-1-amino-2-propanol useful for the preparation of substituted β-phenylethylamines, an important class of pharmacologically active compounds (Hebeisen *et al.*, 2011). The configuration of the enantiomer has been assigned by reference to an unchanging chiral centre in the synthetic procedure and confirmed by anomalous-dispersion effects in diffraction measurements on the crystal: the Flack parameter was refined to 0.02 (2).

The title compound crystallizes with two independent molecules in the asymmetric unit. The five-membered rings adopt (O)C-envelope conformations, denoting the flap atoms, C1 and C6, are adjacent to the oxygen atoms. The methyl groups occupy equatorial positions (Fig. 1). The bonding geometries at the N atoms are close to planar, as the sums of the angles at N1 and N2 are 358.9 and 358.8°, respectively, and the N atoms lie only 0.092 and 0.094 Å out of the planes of the atoms to which they are bonded, as expected for an *N*-acyl fragment. In the crystal, C—H···O interactions (Table 1) are observed. The apolar *tert*-butyl groups and the polar sulfamidate rings are alternately arranged in layers parallel to the *bc*-plane (Fig. 2).

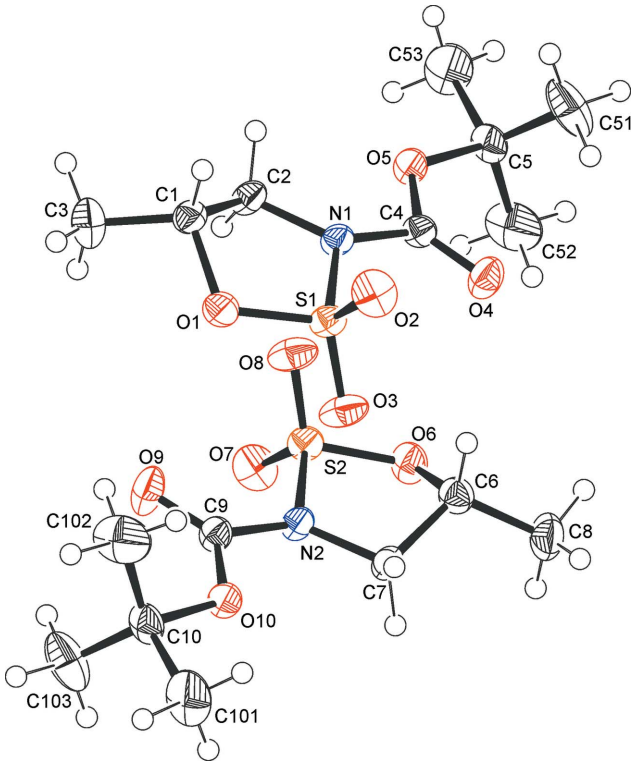


Figure 1
The molecular structure of the two independent molecules in the asymmetric unit of the title compound, showing the atom labels and 50% probability displacement ellipsoids for non-H atoms.

Related structures of N-substituted 1,2,3-oxathiazolidine 2,2-dioxides exhibit (O)C-envelope (Mata *et al.*, 2012; Jiménez-Osés *et al.*, 2009; Avenoza *et al.*, 2004; Nicolaou *et al.*, 2002), O-envelope (Son *et al.*, 2016; Gritsonie *et al.*, 1994), S-envelope (Achary *et al.*, 2016), and (N)C-envelope (Carreras *et al.*, 2007) conformations. These structures exhibited either close to planar (*N*-acyl-substituted, sum of angles >357°) or unequivocally pyramidal (*N*-alkyl-substituted, 335–345°) geometries at the N atoms.

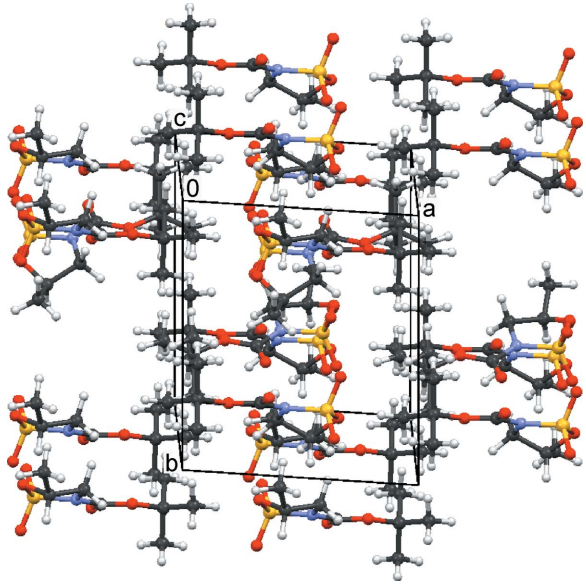


Figure 2
Alternating layers of apolar and polar moieties parallel to the *bc* plane.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7*A*···O4^{i}	0.99	2.40	3.360 (5)	164
C2—H2*B*···O9^{ii}	0.99	2.52	3.200 (4)	126
C2—H2*A*···O8	0.99	2.52	3.205 (4)	126
C1—H1···O9^{ii}	1.00	2.55	3.084 (4)	113
C8—H8*A*···O7^{iii}	0.98	2.57	3.547 (5)	174

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_8H_{15}NO_5S$
M_r	237.27
Crystal system, space group	Monoclinic, $P2_1$
Temperature (K)	193
a, *b*, *c* (Å)	9.4093 (3), 10.5822 (4), 12.2844 (5)
β (°)	107.640 (1)
V (Å^3)	1165.66 (7)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.28
Crystal size (mm)	0.06 × 0.05 × 0.02
Data collection	
Diffractometer	Bruker D8 QUEST PHOTON 100
Absorption correction	Multi-scan (*SADABS*; Bruker, 2012)
T_{min}, T_{max}	0.938, 0.971
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	33847, 4342, 4094
R_{int}	0.030
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.606
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.027, 0.073, 1.04
No. of reflections	4342
No. of parameters	272
No. of restraints	1
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.23, −0.28
Absolute structure	Flack *x* determined using 1855 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons et al., 2013)
Absolute structure parameter	0.02 (2)

Computer programs: *APEX2* and *SAINT* (Bruker, 2012), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *ORTEP-3 for Windows* (Farrugia, 2012) and *Mercury* (Macrae *et al.*, 2008).

Synthesis and crystallization

A solution of $SOCl_2$ (1.5 ml, 21 mmol) in CH_2Cl_2 (15 ml) was added to imidazole (4.7 g, 68 mmol) in CH_2Cl_2 (50 ml) at 0°C. After 90 min, (*R*)-1-(*tert*-butoxycarbonylamino)propan-2-ol (2.0 g, 11 mmol; Zhong *et al.*, 1998) in CH_2Cl_2 (25 ml) was added, and the mixture was stirred for 2 h. The suspension was mixed with H_2O (90 ml) for 15 min. The organic phase was washed with citric acid (5.7 g) in H_2O (50 ml), then with a mixture of saturated brine (30 ml) and H_2O (30 ml). A solution of $NaIO_4$ (6.3 g, 30 mmol) in H_2O (60 ml) was added, then

$RuO_2{\cdot}H_2O$ (100 mg), and the mixture was well stirred for 4 h at room temperature. The organic phase was washed with a solution of Na ascorbate (1.7 g) in H_2O (15 ml), dried over $MgSO_4$ and concentrated under reduced pressure to yield 2.20 g (81%) of colourless product. Suitable crystals were obtained by slow evaporation of a solution in CH_2Cl_2/heptane, m.p. 107–108°C. ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.51 (*s*, 9H), 1.55 (*d*, 3H), 3.62 (*t*, J = 9.8 Hz, 1H), 4.05 (*dd*, J = 5.6 and 9.9 Hz, 1H), 4.93 (*m*, 1H) p.p.m. ^{13}C NMR (75 MHz, $CDCl_3$): δ 18.2, 28.1 (3 C), 51.8, 76.3, 85.4, 148.8 p.p.m. IR (neat): ν 2983, 1716, 1365, 1337, 1258, 1199, 1144, 1089, 1025, 921, 852, 825, 764, 730, 685, 598, 545 cm^{-1}.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

References

Achary, R., Son, S.-M. A. J. I. & Lee, H. K. (2016). *CSD Commun.*: DAMLIP, DAMLOV.

Avenoza, A., Busto, J. H., Corzana, F., Jiménez-Osés, G. & Peregrina, J. M. (2004). *Chem. Commun.* pp. 980–981.

Bruker (2012). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Carreras, J., Avenoza, A., Busto, J. H. & Peregrina, J. M. (2007). *J. Org. Chem.* **72**, 3112–3115.

Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.

Gritsonie, P., Pilkington, M., Wallis, J. D. & Povey, D. C. (1994). *Acta Cryst.* C**50**, 763–765.

Hebeisen, P., Weiss, U., Alker, A. & Staempfli, A. (2011). *Tetrahedron Lett.* **52**, 5229–5233.

Jiménez-Osés, G., Avenoza, A., Busto, J. H., Rodríguez, F. & Peregrina, J. M. (2009). *Chem. Eur. J.* **15**, 9810–9823.

Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.

Mata, L., Avenoza, A., Busto, J. H., Corzana, F. & Peregrina, J. M. (2012). *Chem. Eur. J.* **18**, 15822–15830.

Meléndez, R. E. & Lubell, W. D. (2003). *Tetrahedron*, **59**, 2581–2616.

Nicolaou, K. C., Huang, X., Snyder, S. A., Rao, P. B., Bella, M. & Reddy, M. V. (2002). *Angew. Chem. Int. Ed.* **41**, 834–838.

Parsons, S., Flack, H. D. & Wagner, T. (2013). *Acta Cryst.* B**69**, 249–259.

Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.

Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.

Son, S.-M., Seo, Y. J. & Lee, H.-K. (2016). *Chem. Commun.* **52**, 4286–4289.

Zhong, B., Lu, X. & Silverman, R. B. (1998). *Bioorg. Med. Chem.* **6**, 2405–2419.

full crystallographic data

IUCrData (2017). **2**, x170869 [https://doi.org/10.1107/S2414314617008690]

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Crystal data

$C_8H_{15}NO_5S$
$M_r = 237.27$
Monoclinic, $P2_1$
$a = 9.4093$ (3) Å
$b = 10.5822$ (4) Å
$c = 12.2844$ (5) Å
$\beta = 107.640$ (1)°
$V = 1165.66$ (7) Å^3
$Z = 4$
$F(000) = 504$
$D_x = 1.352$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9948 reflections
$\theta = 2.5$–27.0°
$\mu = 0.28$ mm^{-1}
$T = 193$ K
Prism, colourless
0.06 × 0.05 × 0.02 mm

Data collection

Bruker D8 QUEST PHOTON 100 diffractometer
Radiation source: Incoatec Microfocus
Multi layered optics monochromator
Detector resolution: 10.4 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2012)
$T_{min} = 0.938$, $T_{max} = 0.971$
33847 measured reflections
4342 independent reflections
4094 reflections with $I > 2\sigma(I)$
$R_{int} = 0.030$
$\theta_{max} = 25.5°$, $\theta_{min} = 2.3°$
$h = -11 \rightarrow 11$
$k = -12 \rightarrow 12$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.027$
$wR(F^2) = 0.073$
$S = 1.04$
4342 reflections
272 parameters
1 restraint
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0377P)^2 + 0.3902P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}
Extinction correction: SHELXL2014 (Sheldrick, 2015b), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0255 (19)
Absolute structure: Flack *x* determined using 1855 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons et al., 2013)
Absolute structure parameter: 0.02 (2)

Special details

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.62330 (9)	0.67259 (7)	0.85287 (6)	0.0250 (2)
S2	0.37651 (9)	0.33171 (7)	0.65306 (6)	0.0261 (2)
O1	0.6732 (2)	0.5996 (3)	0.96929 (18)	0.0319 (6)
O3	0.6538 (3)	0.5935 (3)	0.7696 (2)	0.0386 (7)
O2	0.6802 (3)	0.7969 (3)	0.8659 (2)	0.0408 (7)
O4	0.3738 (3)	0.7477 (3)	0.66348 (19)	0.0358 (7)
O5	0.2039 (2)	0.6996 (2)	0.75543 (17)	0.0284 (6)
O6	0.3235 (2)	0.3893 (3)	0.53039 (18)	0.0329 (6)
O7	0.3186 (3)	0.2066 (3)	0.6462 (2)	0.0396 (7)
O8	0.3483 (3)	0.4151 (3)	0.7343 (2)	0.0393 (7)
O9	0.6277 (3)	0.2530 (3)	0.83941 (19)	0.0395 (8)
O10	0.7964 (2)	0.3050 (2)	0.74775 (18)	0.0292 (6)
N1	0.4436 (3)	0.6742 (3)	0.8454 (2)	0.0239 (6)
N2	0.5553 (3)	0.3329 (3)	0.6599 (2)	0.0244 (6)
C1	0.5647 (3)	0.6155 (3)	1.0342 (2)	0.0274 (6)
H1	0.5791	0.6992	1.0737	0.033*
C2	0.4151 (3)	0.6111 (4)	0.9426 (2)	0.0270 (8)
H2A	0.3822	0.5228	0.9235	0.032*
H2B	0.3382	0.6566	0.9672	0.032*
C3	0.5916 (5)	0.5106 (4)	1.1197 (3)	0.0455 (11)
H3A	0.5215	0.5181	1.1643	0.068*
H3B	0.5768	0.4292	1.0798	0.068*
H3C	0.6940	0.5158	1.1709	0.068*
C4	0.3387 (4)	0.7112 (3)	0.7447 (3)	0.0240 (8)
C5	0.0685 (4)	0.7318 (4)	0.6575 (3)	0.0294 (8)
C6	0.4405 (3)	0.4745 (3)	0.5123 (2)	0.0288 (6)
H6	0.4428	0.5559	0.5540	0.035*
C7	0.5840 (4)	0.4009 (4)	0.5643 (3)	0.0269 (8)
H7A	0.6031	0.3412	0.5082	0.032*
H7B	0.6703	0.4585	0.5917	0.032*
C8	0.4016 (4)	0.4982 (4)	0.3859 (3)	0.0416 (10)
H8A	0.4764	0.5541	0.3707	0.062*
H8B	0.3999	0.4177	0.3460	0.062*
H8C	0.3032	0.5381	0.3587	0.062*
C9	0.6606 (4)	0.2940 (4)	0.7593 (3)	0.0258 (8)
C10	0.9306 (4)	0.2727 (4)	0.8444 (3)	0.0326 (8)
C51	0.0728 (5)	0.8705 (5)	0.6290 (5)	0.0585 (14)
H51A	0.0771	0.9213	0.6966	0.088*
H51B	0.1612	0.8876	0.6050	0.088*

H51C	−0.0172	0.8925	0.5670	0.088*
C52	0.0619 (5)	0.6479 (5)	0.5580 (3)	0.0579 (14)
H52A	−0.0265	0.6690	0.4942	0.087*
H52B	0.1517	0.6602	0.5346	0.087*
H52C	0.0560	0.5594	0.5799	0.087*
C53	−0.0562 (4)	0.7038 (6)	0.7066 (4)	0.0559 (13)
H53A	−0.0487	0.7602	0.7714	0.084*
H53B	−0.1521	0.7173	0.6479	0.084*
H53C	−0.0492	0.6158	0.7325	0.084*
C101	1.0564 (4)	0.3039 (6)	0.7948 (4)	0.0598 (15)
H10A	1.1525	0.2862	0.8519	0.090*
H10B	1.0462	0.2522	0.7268	0.090*
H10C	1.0514	0.3936	0.7740	0.090*
C102	0.9364 (5)	0.3583 (5)	0.9441 (3)	0.0579 (14)
H10D	1.0246	0.3377	1.0082	0.087*
H10E	0.9421	0.4465	0.9215	0.087*
H10F	0.8464	0.3461	0.9671	0.087*
C103	0.9308 (5)	0.1343 (4)	0.8720 (4)	0.0543 (13)
H10G	1.0197	0.1143	0.9357	0.081*
H10H	0.8412	0.1139	0.8934	0.081*
H10I	0.9316	0.0845	0.8049	0.081*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0159 (4)	0.0338 (5)	0.0254 (4)	−0.0013 (4)	0.0062 (3)	−0.0021 (4)
S2	0.0167 (5)	0.0339 (5)	0.0265 (4)	−0.0012 (4)	0.0050 (3)	−0.0003 (4)
O1	0.0210 (12)	0.0445 (14)	0.0290 (11)	0.0092 (11)	0.0060 (9)	0.0047 (11)
O3	0.0284 (15)	0.0559 (17)	0.0358 (14)	0.0003 (13)	0.0162 (11)	−0.0112 (13)
O2	0.0295 (15)	0.0399 (16)	0.0505 (15)	−0.0098 (13)	0.0087 (12)	0.0007 (12)
O4	0.0256 (17)	0.055 (2)	0.0282 (13)	0.0015 (12)	0.0094 (11)	0.0107 (12)
O5	0.0155 (11)	0.0435 (16)	0.0247 (11)	0.0038 (11)	0.0040 (9)	0.0058 (10)
O6	0.0218 (12)	0.0430 (14)	0.0294 (12)	0.0014 (11)	0.0012 (9)	0.0050 (11)
O7	0.0268 (15)	0.0384 (16)	0.0514 (15)	−0.0076 (12)	0.0086 (11)	0.0013 (11)
O8	0.0280 (15)	0.0544 (17)	0.0402 (15)	−0.0016 (13)	0.0174 (12)	−0.0110 (13)
O9	0.0258 (17)	0.062 (2)	0.0289 (13)	−0.0045 (12)	0.0063 (12)	0.0175 (13)
O10	0.0172 (12)	0.0428 (17)	0.0272 (11)	0.0052 (11)	0.0063 (9)	0.0069 (11)
N1	0.0174 (14)	0.0322 (15)	0.0227 (13)	0.0013 (14)	0.0069 (10)	0.0015 (14)
N2	0.0155 (14)	0.0369 (15)	0.0204 (12)	0.0000 (14)	0.0045 (10)	0.0029 (14)
C1	0.0276 (14)	0.0324 (15)	0.0223 (13)	0.0031 (12)	0.0076 (11)	−0.0003 (11)
C2	0.0224 (18)	0.039 (2)	0.0200 (15)	−0.0015 (16)	0.0067 (13)	−0.0010 (14)
C3	0.050 (2)	0.045 (3)	0.037 (2)	0.006 (2)	0.0065 (19)	0.0146 (18)
C4	0.0183 (19)	0.0286 (19)	0.0241 (16)	0.0001 (14)	0.0049 (14)	−0.0009 (13)
C5	0.0138 (18)	0.039 (2)	0.0302 (17)	0.0030 (17)	−0.0018 (14)	0.0033 (17)
C6	0.0261 (14)	0.0303 (14)	0.0281 (14)	0.0016 (11)	0.0056 (11)	0.0009 (12)
C7	0.0259 (18)	0.035 (2)	0.0209 (15)	0.0020 (16)	0.0093 (14)	0.0039 (13)
C8	0.043 (2)	0.049 (3)	0.0293 (19)	0.005 (2)	0.0053 (17)	0.0119 (17)
C9	0.0211 (19)	0.0319 (19)	0.0227 (16)	0.0003 (15)	0.0044 (14)	0.0017 (14)

C10	0.0201 (19)	0.040 (2)	0.0328 (18)	0.0056 (18)	0.0009 (15)	0.0080 (17)
C51	0.034 (2)	0.045 (3)	0.083 (3)	0.010 (2)	−0.002 (2)	0.019 (2)
C52	0.038 (2)	0.072 (4)	0.047 (2)	0.010 (2)	−0.0111 (18)	−0.019 (2)
C53	0.023 (2)	0.086 (4)	0.058 (2)	0.006 (2)	0.0109 (19)	0.016 (3)
C101	0.016 (2)	0.091 (4)	0.071 (3)	0.011 (2)	0.0110 (19)	0.034 (3)
C102	0.035 (2)	0.079 (4)	0.046 (2)	0.009 (3)	−0.0090 (18)	−0.019 (2)
C103	0.035 (2)	0.043 (3)	0.071 (3)	0.003 (2)	−0.006 (2)	0.015 (2)

Geometric parameters (Å, º)

S1—O2	1.410 (3)	C5—C51	1.512 (6)
S1—O3	1.417 (3)	C6—C8	1.504 (4)
S1—O1	1.567 (2)	C6—C7	1.522 (4)
S1—N1	1.666 (3)	C6—H6	1.0000
S2—O8	1.416 (3)	C7—H7A	0.9900
S2—O7	1.425 (3)	C7—H7B	0.9900
S2—O6	1.561 (2)	C8—H8A	0.9800
S2—N2	1.659 (3)	C8—H8B	0.9800
O1—C1	1.483 (3)	C8—H8C	0.9800
O4—C4	1.206 (4)	C10—C103	1.504 (6)
O5—C4	1.320 (4)	C10—C102	1.510 (6)
O5—C5	1.502 (4)	C10—C101	1.522 (5)
O6—C6	1.491 (4)	C51—H51A	0.9800
O9—C9	1.199 (4)	C51—H51B	0.9800
O10—C9	1.333 (4)	C51—H51C	0.9800
O10—C10	1.487 (4)	C52—H52A	0.9800
N1—C4	1.385 (4)	C52—H52B	0.9800
N1—C2	1.461 (4)	C52—H52C	0.9800
N2—C9	1.382 (4)	C53—H53A	0.9800
N2—C7	1.470 (4)	C53—H53B	0.9800
C1—C3	1.496 (5)	C53—H53C	0.9800
C1—C2	1.513 (4)	C101—H10A	0.9800
C1—H1	1.0000	C101—H10B	0.9800
C2—H2A	0.9900	C101—H10C	0.9800
C2—H2B	0.9900	C102—H10D	0.9800
C3—H3A	0.9800	C102—H10E	0.9800
C3—H3B	0.9800	C102—H10F	0.9800
C3—H3C	0.9800	C103—H10G	0.9800
C5—C52	1.497 (5)	C103—H10H	0.9800
C5—C53	1.502 (5)	C103—H10I	0.9800
O2—S1—O3	118.69 (18)	N2—C7—H7A	111.1
O2—S1—O1	110.94 (16)	C6—C7—H7A	111.1
O3—S1—O1	107.29 (17)	N2—C7—H7B	111.1
O2—S1—N1	109.95 (16)	C6—C7—H7B	111.1
O3—S1—N1	112.98 (15)	H7A—C7—H7B	109.0
O1—S1—N1	94.22 (13)	C6—C8—H8A	109.5
O8—S2—O7	118.11 (18)	C6—C8—H8B	109.5

O8—S2—O6	111.31 (18)	H8A—C8—H8B	109.5
O7—S2—O6	107.53 (16)	C6—C8—H8C	109.5
O8—S2—N2	111.30 (16)	H8A—C8—H8C	109.5
O7—S2—N2	111.98 (16)	H8B—C8—H8C	109.5
O6—S2—N2	93.91 (13)	O9—C9—O10	127.8 (3)
C1—O1—S1	111.72 (18)	O9—C9—N2	122.6 (3)
C4—O5—C5	120.4 (2)	O10—C9—N2	109.5 (3)
C6—O6—S2	110.25 (17)	O10—C10—C103	110.3 (3)
C9—O10—C10	120.3 (3)	O10—C10—C102	108.8 (3)
C4—N1—C2	127.0 (3)	C103—C10—C102	113.8 (4)
C4—N1—S1	119.3 (2)	O10—C10—C101	101.9 (3)
C2—N1—S1	112.6 (2)	C103—C10—C101	110.7 (4)
C9—N2—C7	126.6 (3)	C102—C10—C101	110.6 (4)
C9—N2—S2	119.1 (2)	C5—C51—H51A	109.5
C7—N2—S2	113.1 (2)	C5—C51—H51B	109.5
O1—C1—C3	107.2 (3)	H51A—C51—H51B	109.5
O1—C1—C2	103.5 (2)	C5—C51—H51C	109.5
C3—C1—C2	114.9 (3)	H51A—C51—H51C	109.5
O1—C1—H1	110.3	H51B—C51—H51C	109.5
C3—C1—H1	110.3	C5—C52—H52A	109.5
C2—C1—H1	110.3	C5—C52—H52B	109.5
N1—C2—C1	103.6 (2)	H52A—C52—H52B	109.5
N1—C2—H2A	111.0	C5—C52—H52C	109.5
C1—C2—H2A	111.0	H52A—C52—H52C	109.5
N1—C2—H2B	111.0	H52B—C52—H52C	109.5
C1—C2—H2B	111.0	C5—C53—H53A	109.5
H2A—C2—H2B	109.0	C5—C53—H53B	109.5
C1—C3—H3A	109.5	H53A—C53—H53B	109.5
C1—C3—H3B	109.5	C5—C53—H53C	109.5
H3A—C3—H3B	109.5	H53A—C53—H53C	109.5
C1—C3—H3C	109.5	H53B—C53—H53C	109.5
H3A—C3—H3C	109.5	C10—C101—H10A	109.5
H3B—C3—H3C	109.5	C10—C101—H10B	109.5
O4—C4—O5	128.7 (3)	H10A—C101—H10B	109.5
O4—C4—N1	122.0 (3)	C10—C101—H10C	109.5
O5—C4—N1	109.3 (3)	H10A—C101—H10C	109.5
C52—C5—O5	109.6 (3)	H10B—C101—H10C	109.5
C52—C5—C53	111.5 (4)	C10—C102—H10D	109.5
O5—C5—C53	102.1 (3)	C10—C102—H10E	109.5
C52—C5—C51	112.6 (4)	H10D—C102—H10E	109.5
O5—C5—C51	109.3 (3)	C10—C102—H10F	109.5
C53—C5—C51	111.2 (4)	H10D—C102—H10F	109.5
O6—C6—C8	107.2 (2)	H10E—C102—H10F	109.5
O6—C6—C7	103.2 (3)	C10—C103—H10G	109.5
C8—C6—C7	115.2 (3)	C10—C103—H10H	109.5
O6—C6—H6	110.3	H10G—C103—H10H	109.5
C8—C6—H6	110.3	C10—C103—H10I	109.5
C7—C6—H6	110.3	H10G—C103—H10I	109.5

N2—C7—C6	103.4 (2)	H10H—C103—H10I	109.5
O2—S1—O1—C1	90.0 (2)	C5—O5—C4—O4	1.2 (6)
O3—S1—O1—C1	−138.9 (2)	C5—O5—C4—N1	−179.1 (3)
N1—S1—O1—C1	−23.2 (2)	C2—N1—C4—O4	−170.2 (4)
O8—S2—O6—C6	83.9 (2)	S1—N1—C4—O4	−3.3 (5)
O7—S2—O6—C6	−145.3 (2)	C2—N1—C4—O5	10.1 (5)
N2—S2—O6—C6	−30.8 (2)	S1—N1—C4—O5	176.9 (2)
O2—S1—N1—C4	76.9 (3)	C4—O5—C5—C52	61.1 (4)
O3—S1—N1—C4	−58.2 (3)	C4—O5—C5—C53	179.5 (3)
O1—S1—N1—C4	−169.0 (3)	C4—O5—C5—C51	−62.7 (4)
O2—S1—N1—C2	−114.4 (3)	S2—O6—C6—C8	165.0 (2)
O3—S1—N1—C2	110.5 (3)	S2—O6—C6—C7	43.0 (3)
O1—S1—N1—C2	−0.3 (3)	C9—N2—C7—C6	−152.5 (3)
O8—S2—N2—C9	62.5 (3)	S2—N2—C7—C6	14.8 (3)
O7—S2—N2—C9	−72.2 (3)	O6—C6—C7—N2	−33.4 (3)
O6—S2—N2—C9	177.2 (3)	C8—C6—C7—N2	−149.9 (3)
O8—S2—N2—C7	−105.9 (3)	C10—O10—C9—O9	−4.5 (6)
O7—S2—N2—C7	119.5 (3)	C10—O10—C9—N2	178.1 (3)
O6—S2—N2—C7	8.8 (3)	C7—N2—C9—O9	170.5 (4)
S1—O1—C1—C3	160.6 (2)	S2—N2—C9—O9	3.9 (5)
S1—O1—C1—C2	38.8 (3)	C7—N2—C9—O10	−11.9 (5)
C4—N1—C2—C1	−170.2 (3)	S2—N2—C9—O10	−178.6 (2)
S1—N1—C2—C1	22.2 (3)	C9—O10—C10—C103	64.5 (5)
O1—C1—C2—N1	−35.6 (3)	C9—O10—C10—C102	−61.0 (4)
C3—C1—C2—N1	−152.2 (3)	C9—O10—C10—C101	−177.9 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7*A*···O4^{i}	0.99	2.40	3.360 (5)	164
C2—H2*B*···O9^{ii}	0.99	2.52	3.200 (4)	126
C2—H2*A*···O8	0.99	2.52	3.205 (4)	126
C1—H1···O9^{ii}	1.00	2.55	3.084 (4)	113
C8—H8*A*···O7^{iii}	0.98	2.57	3.547 (5)	174

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