# Comparison of the C—H···O bonding in two crystalline phases of 1,4-dithiane 1,1,4,4-tetraoxide

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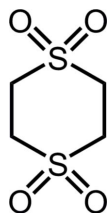
The crystal structures of two crystalline phases of 1,4-dithiane 1,1,4,4-tetraoxide, $C_4H_8O_4S_2$, have been determined in order to examine the nature of possible intermolecular hydrogen bonds. Phase 1 is monoclinic, space group $C2/m$, with unit-cell dimensions of $a = 9.073\,(8)$, $b = 7.077\,(6)$, $c = 5.597\,(5)$ Å and $\beta = 105.89\,(1)°$. The molecule adopts $2/m$ symmetry and all of the molecules are related by translation and thus have the same orientation. Phase 2 is also monoclinic but in space group $P2_1/n$ with unit-cell dimensions of $a = 7.1305\,(5)$, $b = 5.7245\,(4)$, $c = 8.3760\,(6)$ Å and $\beta = 91.138\,(2)°$. In this phase, the molecule sits on an inversion center and the molecules within the unit cell adopt quite different orientations. In both phases, examination of the potential C—H···O hydrogen bonds around each of the independent oxygen atoms (one axial and the other equatorial) shows the general O···H patterns to be quite similar with each oxygen atom in contact with four neighboring H atoms, and each H atom contacting two neighboring O atoms. While none of the H···O contacts is particularly short (all are greater than 2.5 Å), each molecule has 32 such contacts that form an extensive intermolecular network. A ^{1}H NMR spectrum of the compound dissolved in DMSO shows a singlet of 8H at δ 3.677 which indicates that the C—H bonds are only moderately polarized by the single adjacent –SO_2– moiety: strongly polarized C—H bonds have δ values in the 5–6 range [Li & Sammes (1983). *J. Chem. Soc. Perkin Trans. 1*, pp. 1303–1309]. The phase 1 crystal studied was non-merohedrally twinned.

1. Chemical context

Some years ago, multiple studies of C—H···X (X = N, O) *intra*molecular hydrogen bonds were carried out on a series of 1,3-dithiane 1,1,3,3-tetraoxides which had various substituents at the 2 position located between the two SO_2 groups. The remaining C—H bond in the 2 position is strongly polarized given the electron-withdrawing properties of the two adjacent sulfone groups. The substituents bonded at the 2 position contained nitrogen or oxygen electron-pair donors which, with proper chain lengths, were able to form an intramolecular hydrogen bond to the polar hydrogen atom.
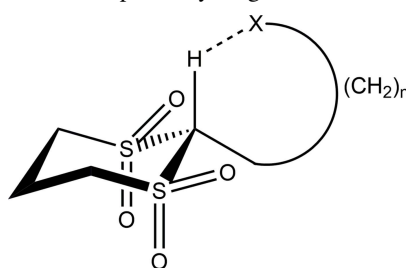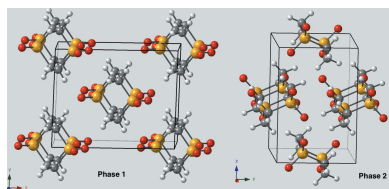
The chemistry and NMR/IR spectroscopic information of a wide variety of compounds were reported in a number of papers (see Li & Sammes, 1983, and references therein). Focusing on those compounds with significant shifts of the polar methine hydrogen in the ^{1}H-NMR spectra, their crystal structure determinations clearly demonstrated the formation of *intra*molecular hydrogen bonds (Harlow *et al.*, 1984). Never explored, however, was the nature of the C—H···O interactions likely to be found in the unsubstituted compound itself. As a matter of curiosity, we consequently decided to undertake the crystal structure determinations of the two possible (1,3- and 1,4-) dithiane tetraoxides and the unique 1,3,5-trithiane hexaoxide. All three of the compounds have unusually high melting/decomposition temperatures and we wanted to explore and compare the nature of the *inter*molecular C—H···O interactions in this group of uncomplicated compounds. As a start on this project, we report herein the completion of the structures of two crystalline phases of 1,4-dithiane 1,1,4,4-tetraoxide, a compound which has no dipole moment, has the same 1:2 O:H ratio as water, and decomposes above 627 K.

2. Structural commentary

1,4-Dithiane 1,1,4,4-tetraoxide contains two crystalline phases as determined from an X-ray diffraction pattern of the as-synthesized powder. When sublimed, crystals of both phases were also produced and it was only by chance that the two laboratories involved picked different phases. Fig. 1 compares the molecular *ORTEP* drawings of the molecules in the two phases. The molecule in phase 1 adopts 2/*m* symmetry while in phase 2 the molecule sits on a center of symmetry. The intramolecular bond distances and angles for the two phases are comparable.
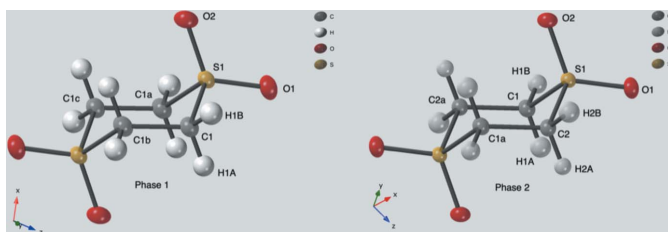

Figure 1
ORTEP drawings (50% probability) of the 1,4-dithiane 1,1,4,4-tetraoxide molecule in crystalline phases 1 and 2. The molecule in phase 1 has 2/*m* symmetry; in phase 2, it has a center of inversion. All of the unique atoms are labeled as are the symmetry-related carbon atoms to emphasize the different symmetries. Symmetry codes for phase 1: (a) *x*, 1 − *y*, *z*; (b) 1 − *x*, *y*, −*z*; (c) 1 − *x*, 1 − *y*, −*z*. Symmetry code for phase 2: (a) −*x*, −*y*, 1 − *z*.
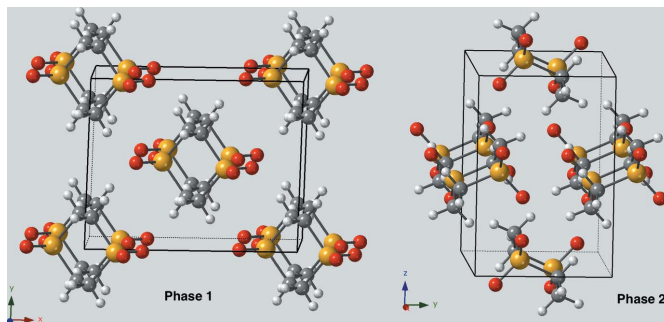

Figure 2
Packing diagrams for crystalline phase 1 as viewed nearly along the *c* axis and phase 2 as viewed nearly along the *a*-axis. In phase 1, all the molecules are related by translation and thus have the same orientation. In phase 2, the molecules have two different orientations.

3. Supramolecular features

Packing diagrams (Fig. 2) reveal that the packing for the two forms is quite different. In phase 1, all of the molecules are related by simple translational symmetries and thus all the molecules have the same orientation. In phase 2, the molecules have two different orientations in a somewhat herringbone fashion. Thus, one might expect any C—H···O contacts to be quite different for the two phases but, in fact, they are very similar. Figs. 3 and 4 compare the environments of O1 (equatorial oxygen atom) and O2 (axial oxygen atom).
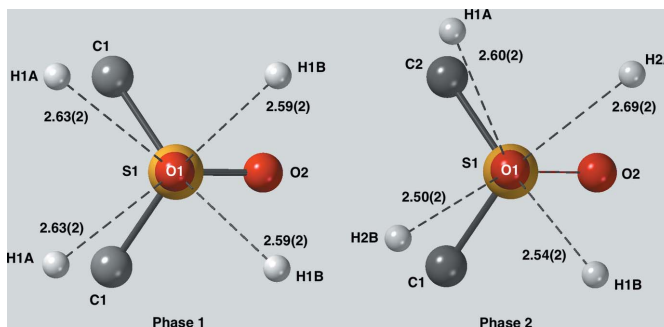

Figure 3
Environment of the equatorial oxygen atom, O1, in phases 1 and 2. Although the packing of the molecules is quite different, the arrangement of the C—H···O contacts in both phases is seen to be very similar.
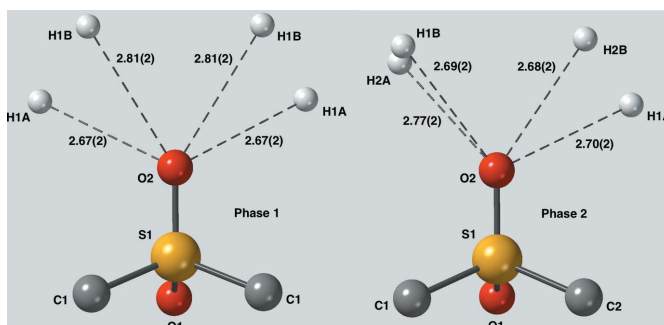

Figure 4
Environment of the axial oxygen atom, O2, in phases 1 and 2. In this case, the environments are still similar, but less so than for equatorial O1.

In all cases, each oxygen atom is in contact with four hydrogen atoms arranged in a distorted square. Probably for steric reasons, the distortion is less for the equatorial oxygen atom than for the axial oxygen atom.

Each oxygen atom in both phases 'sees' four hydrogen atoms while each hydrogen atom 'sees' two oxygen atoms. This bifurcation of the hydrogen contacts means that none of the H⋯O distances is particularly short. It should also be pointed out that each methylene group has only one neighboring sulfone group, which would limit the polarization of the C—H bonds compared to our previous studies where the C—H bond of interest sat between two sulfone groups. Thus, very short C—H⋯O bonds were not expected. The exact details of the C—H⋯O contacts are given in Table 1. Thus, while there are no really short C—H⋯O contacts (none less than 2.50 Å), every donor and every acceptor plays a role in forming a extensive network of contacts in which each molecule has a total of 32 interactions with its neighbors.

The shortest C—H⋯O contacts tend to be between the equatorial oxygen atoms, O1, and the equatorial hydrogen atoms labeled with the suffix B. These also come with C—H⋯O angles that are closest to being linear, 148 to 160°. Presumably the difference between axial and equatorial H⋯O contacts is mostly due to steric effects, the equatorial atoms being more accessible. The shorter contacts can undoubtedly be classified as true C—H⋯O hydrogen bonds using, as a guide, the seminal study of weak hydrogen bonds by Desiraju & Steiner (1999). The remaining bonds are probably better described as mostly electrostatic in nature. However, as Desiraju & Steiner point out, there are no hard limits for determining what may, and may not, be a true hydrogen bond.

4. Database survey

A Cambridge Crystallographic Database survey of 1,4-dithiane reveals over 200 structures with that base motif (CSD v. 5.40 + 1 update; Groom *et al.*, 2016). A more modest survey, with one oxygen bonded to each sulfur yields 33 results, of which 1,4-dithiane 1,4-dioxide has two polymorphs [DTHDOX and DTHDOX01 (Shearer, 1959; Takemura *et al.*, 2014) and DTHDSX (Montgomery, 1960)]. There is only one reported structure that incorporates 1,4-dithiane 1,1,4,4-tetraoxide into its structure, *viz.* 5,6,7-triphenyl-2,3-dihydro-6*H*-phospholo[3,4-*b*][1,4]dithiine 1,1,4,4,6-pentaoxide (GACCUK; Fadhel *et al.*, 2010). One of the five oxygen atoms is located on the phosphorus, while the remaining four are on the sulfur atoms of the sulfone moiety.

5. Synthesis and crystallization

Following literature procedures (Schultz *et al.*, 1963), a 100 mL round-bottom flask was charged with 1,4-thiane (Sigma–Aldrich; 1.005 g, 8.4 mmol) in 25 mL glacial acetic acid. To this were added 10 mL 30% hydrogen peroxide solution (excess) in 25 mL of glacial acetic acid. The solution was heated to 323 K for 12 h under stirring over an oil bath. The white solid that formed was filtered and washed with water (3 × 25 mL)

Table 1
Intermolecular contacts (Å, °) as potential C⋯H⋯O hydrogen bonds for phases 1 and 2.

Atoms	bond H⋯O	angle C—H⋯O	angle S—O⋯H
Phase 1			
C1—H1A⋯O1[i]	2.63 (2)	148 (2)	123 (2)
C1—H1B⋯O1[ii]	2.59 (2)	157 (2)	127 (2)
C1—H1A⋯O2[iii]	2.67 (2)	112 (2)	114 (2)
C1—H1B⋯O2[iv]	2.81 (2)	122 (2)	145 (2)
Phase 2			
C1—H1A⋯O1[i]	2.60 (2)	151 (1)	123 (1)
C1—H1B⋯O1[ii]	2.54 (2)	160 (1)	127 (1)
C2—H2A⋯O1[i]	2.69 (2)	149 (1)	122 (1)
C2—H2B⋯O1[iii]	2.50 (2)	155 (1)	129 (1)
C1—H1A⋯O2[iv]	2.70 (2)	110 (1)	111 (1)
C1—H1B⋯O2[v]	2.69 (2)	122 (1)	140 (1)
C2—H2A⋯O2[vi]	2.77 (2)	100 (1)	127 (1)
C2—H2B⋯O2[vii]	2.68 (2)	125 (1)	144 (1)

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

and diethyl ether (3 × 25 mL) (yield: 1.325 g, 86%). Crystals suitable for structural analysis were grown by sublimation of the solid. NMR data were recorded on a Bruker Avance 400 MHz with d_6-DMSO as solvent, referenced to residue proteo-DMSO. TGA/DSC data showed decomposition occurring from 627 to 739 K.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The refinement of phase 1 used two sets of reflections as the crystal was non-merohedrally twinned. Non-hydrogen atoms were refined with anisotropic atomic displacement parameters. All hydrogen atoms were refined freely.

Table 2
Experimental details.

	Phase 1	Phase 2
Crystal data		
Chemical formula	$C_4H_8O_4S_2$	$C_4H_8O_4S_2$
M_r	184.22	184.22
Crystal system, space group	Monoclinic, $C2/m$	Monoclinic, $P2_1/n$
Temperature (K)	233	150
a, b, c (Å)	9.073 (8), 7.077 (6), 5.597 (5)	7.1308 (5), 5.7245 (4), 8.3760 (6)
β (°)	105.894 (10)	91.138 (2)
V (Å^3)	345.6 (5)	341.84 (4)
Z	2	2
Radiation type	Mo $K\alpha$	Synchrotron, $\lambda = 0.7288$ Å
μ (mm^{-1})	0.72	0.78
Crystal size (mm)	$0.43 \times 0.35 \times 0.35$	$0.04 \times 0.03 \times 0.02$
Data collection		
Diffractometer	APEXII CCD	Bruker D8 Photon-2
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.747, 0.787	0.811, 0.862
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	433, 433, 428	14904, 1041, 957
R_{int}	–	0.036
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.652	0.714
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.023, 0.067, 1.19	0.028, 0.065, 1.11
No. of reflections	433	1041
No. of parameters	37	62
H-atom treatment	All H-atom parameters refined	All H-atom parameters refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.32, −0.32	0.39, −0.44

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *SHELXL* (Sheldrick, 2015), *OLEX2* (Dolomanov *et al.*, 2009), *CrystalMaker* (Palmer, 2014) and *publCIF* (Westrip, 2010).

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Comparison of the C—H⋯O bonding in two crystalline phases of 1,4-dithiane 1,1,4,4-tetraoxide

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Computing details

For both structures, data collection: *APEX3* (Bruker, 2016); cell refinement: *SAINT* (Bruker, 2016); data reduction: *SAINT* (Bruker, 2016); program(s) used to solve structure: *OLEX2* (Dolomanov *et al.*, 2009); program(s) used to refine structure: *SHELXL* (Sheldrick, 2015); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009), *CrystalMaker* (Palmer, 2014). Software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009) and *publCIF* (Westrip, 2010) for 14-disulphone-phase1; *OLEX2* (Dolomanov *et al.*, 2009) for 14-disulphone-phase2.

1,4-Dithiane 1,1,4,4-tetraoxide (14-disulphone-phase1)

Crystal data

$C_4H_8O_4S_2$	$F(000) = 192$
$M_r = 184.22$	$D_x = 1.770$ Mg m^{-3}
Monoclinic, $C2/m$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 9.073$ (8) Å	Cell parameters from 999 reflections
$b = 7.077$ (6) Å	$\theta = 3.7–27.7°$
$c = 5.597$ (5) Å	$\mu = 0.72$ mm^{-1}
$\beta = 105.894$ (10)°	$T = 233$ K
$V = 345.6$ (5) Å^3	Irregular block, colorless
$Z = 2$	$0.43 \times 0.35 \times 0.35$ mm

Data collection

APEXII CCD diffractometer	433 independent reflections
$\theta/2\theta$ scans	428 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)	$\theta_{max} = 27.6°$, $\theta_{min} = 3.7°$
	$h = -11 \rightarrow 11$
$T_{min} = 0.747$, $T_{max} = 0.787$	$k = 0 \rightarrow 9$
433 measured reflections	$l = 0 \rightarrow 7$

Refinement

Refinement on F^2	0 restraints
Least-squares matrix: full	Primary atom site location: dual
$R[F^2 > 2\sigma(F^2)] = 0.023$	Secondary atom site location: difference Fourier map
$wR(F^2) = 0.067$	
$S = 1.19$	Hydrogen site location: difference Fourier map
433 reflections	All H-atom parameters refined
37 parameters	

$w = 1/[\sigma^2(F_o^2) + (0.0297P)^2 + 0.2882P]$
  where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.32$ e Å^{-3}
$\Delta\rho_{min} = -0.32$ e Å^{-3}

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.

Refinement. Refined as a 2-component twin.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.65320 (6)	0.500000	0.24835 (9)	0.01721 (19)
O1	0.6955 (2)	0.500000	0.5165 (3)	0.0273 (4)
O2	0.77466 (18)	0.500000	0.1266 (3)	0.0258 (4)
C1	0.53198 (17)	0.3028 (2)	0.1408 (3)	0.0198 (3)
H1A	0.454 (2)	0.305 (3)	0.225 (4)	0.024 (5)*
H1B	0.590 (2)	0.200 (3)	0.192 (4)	0.028 (5)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0144 (3)	0.0179 (3)	0.0173 (3)	0.000	0.0010 (2)	0.000
O1	0.0270 (8)	0.0319 (10)	0.0183 (8)	0.000	−0.0017 (7)	0.000
O2	0.0166 (8)	0.0289 (9)	0.0326 (9)	0.000	0.0080 (7)	0.000
C1	0.0193 (7)	0.0158 (7)	0.0217 (8)	−0.0011 (5)	0.0015 (6)	0.0026 (6)

Geometric parameters (Å, º)

S1—O1	1.444 (2)	C1—C1^{ii}	1.523 (3)
S1—O2	1.4456 (19)	C1—H1A	0.949 (19)
S1—C1	1.7768 (19)	C1—H1B	0.90 (2)
S1—C1^i	1.7768 (19)		
O1—S1—O2	118.03 (11)	S1—C1—H1A	106.9 (12)
O1—S1—C1^i	108.42 (7)	S1—C1—H1B	105.9 (13)
O1—S1—C1	108.42 (7)	C1^{ii}—C1—S1	112.07 (9)
O2—S1—C1^i	108.74 (8)	C1^{ii}—C1—H1A	113.1 (11)
O2—S1—C1	108.74 (8)	C1^{ii}—C1—H1B	110.6 (13)
C1—S1—C1^i	103.52 (12)	H1A—C1—H1B	108.0 (17)
O1—S1—C1—C1^{ii}	174.03 (12)	C1^i—S1—C1—C1^{ii}	59.03 (17)
O2—S1—C1—C1^{ii}	−56.47 (15)		

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

1,4-Dithiane 1,1,4,4-tetraoxide (14-disulphone-phase2)

Crystal data

$C_4H_8O_4S_2$	$F(000) = 192$
$M_r = 184.22$	$D_x = 1.790$ Mg m^{-3}
Monoclinic, $P2_1/n$	Synchrotron radiation, $\lambda = 0.7288$ Å
$a = 7.1308$ (5) Å	Cell parameters from 4318 reflections
$b = 5.7245$ (4) Å	$\theta = 29.1$–3.8°
$c = 8.3760$ (6) Å	$\mu = 0.78$ mm^{-1}
$\beta = 91.138$ (2)°	$T = 150$ K
$V = 341.84$ (4) Å^3	Tablet, colorless
$Z = 2$	$0.04 \times 0.03 \times 0.02$ mm

Data collection

Bruker D8 Photon-2 diffractometer	14904 measured reflections
Radiation source: synchrotron	1041 independent reflections
Detector resolution: 10.34 pixels mm^{-1}	957 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.036$
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)	$\theta_{max} = 31.4°$, $\theta_{min} = 3.8°$
	$h = -10\rightarrow10$
	$k = -8\rightarrow8$
$T_{min} = 0.811$, $T_{max} = 0.862$	$l = -11\rightarrow11$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.028$	All H-atom parameters refined
$wR(F^2) = 0.065$	$w = 1/[\sigma^2(F_o^2) + (0.0197P)^2 + 0.3362P]$
$S = 1.11$	where $P = (F_o^2 + 2F_c^2)/3$
1041 reflections	$(\Delta/\sigma)_{max} = 0.001$
62 parameters	$\Delta\rho_{max} = 0.39$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.44$ e Å^{-3}
Primary atom site location: dual	

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 (Å^2)

	x	y	z	$U_{iso}*/U_{eq}$
S1	0.19891 (5)	0.14315 (6)	0.54564 (4)	0.01294 (10)
O1	0.35411 (15)	0.14742 (19)	0.65934 (13)	0.0194 (2)
O2	0.19584 (15)	0.31562 (19)	0.41982 (12)	0.0186 (2)
C1	0.1857 (2)	−0.1390 (2)	0.45787 (17)	0.0149 (3)
C2	−0.0137 (2)	0.1639 (3)	0.65211 (17)	0.0147 (3)
H1A	0.186 (3)	−0.247 (4)	0.541 (2)	0.024 (5)*
H1B	0.299 (3)	−0.156 (3)	0.401 (2)	0.020 (5)*
H2A	−0.006 (3)	0.049 (3)	0.734 (2)	0.016 (4)*
H2B	−0.008 (3)	0.314 (4)	0.696 (2)	0.024 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.01212 (16)	0.01260 (16)	0.01412 (17)	−0.00078 (11)	0.00029 (11)	−0.00062 (12)
O1	0.0153 (5)	0.0218 (5)	0.0208 (5)	−0.0010 (4)	−0.0042 (4)	−0.0031 (4)
O2	0.0209 (5)	0.0156 (5)	0.0194 (5)	−0.0018 (4)	0.0024 (4)	0.0038 (4)
C1	0.0135 (6)	0.0134 (6)	0.0177 (6)	0.0010 (5)	0.0011 (5)	−0.0018 (5)
C2	0.0146 (6)	0.0157 (6)	0.0137 (6)	−0.0002 (5)	0.0013 (5)	−0.0022 (5)

Geometric parameters (Å, º)

S1—O1	1.4459 (11)	C1—H1A	0.93 (2)
S1—O2	1.4439 (11)	C1—H1B	0.95 (2)
S1—C1	1.7762 (14)	C2—H2A	0.952 (19)
S1—C2	1.7778 (14)	C2—H2B	0.94 (2)
C1—C2^i	1.5265 (19)		
O1—S1—C1	108.75 (7)	C2^i—C1—H1A	112.1 (12)
O1—S1—C2	108.51 (7)	C2^i—C1—H1B	111.5 (11)
O2—S1—O1	118.00 (7)	H1A—C1—H1B	108.6 (16)
O2—S1—C1	108.65 (7)	S1—C2—H2A	106.2 (11)
O2—S1—C2	108.65 (7)	S1—C2—H2B	103.3 (12)
C1—S1—C2	103.29 (7)	C1^i—C2—S1	111.98 (10)
S1—C1—H1A	107.1 (13)	C1^i—C2—H2A	113.8 (11)
S1—C1—H1B	105.4 (12)	C1^i—C2—H2B	110.6 (12)
C2^i—C1—S1	111.72 (10)	H2A—C2—H2B	110.4 (16)

Symmetry code: (i) −*x*, −*y*, −*z*+1.