

2*H*,10*H*-1,4-Dioxepino[5',6':4,5]thieno[3,2-*e*][1,4]dioxepine-5,7(3*H*,9*H*)-dione

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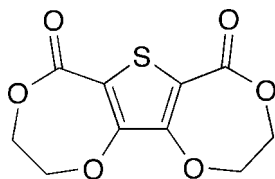
Received 29 October 2007; accepted 29 October 2007

Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; disorder in main residue; R factor = 0.066; wR factor = 0.151; data-to-parameter ratio = 10.8.

In the title compound, $\text{C}_{10}\text{H}_8\text{O}_6\text{S}$, which was synthesized by the intramolecular cyclization of diethyl 3,4-bis(2-hydroxyethoxy)thiophene-2,5-dicarboxylate, the thiophene portion lies on a mirror plane. The crystal structure is stabilized by intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds.

Related literature

For the synthesis of the starting reagent, see: Halfpenny *et al.* (2000). For the antibacterial activity of 1,4-dioxepin-5-one compounds, see: Ito *et al.* (1997); Rao (1996). For their polymerization abilities, see: Mathisen *et al.* (1989). For related literature on molecular structures including a 1,4-dioxepin-5-one ring, see: Blaser & Stoeckli-Evans (1991); Brassy *et al.* (1977); Connolly *et al.* (1984); Lamothe & Fuchs (1993); Kawahara *et al.* (1988); Mulzer *et al.* (1996); Xu *et al.* (2000). For literature on $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds, see: Batchelor *et al.* (2000); Biradha *et al.* (1993); Taylor & Kennard (1982).



Experimental

Crystal data

$\text{C}_{10}\text{H}_8\text{O}_6\text{S}$
 $M_r = 256.22$
 Orthorhombic, $Pnma$
 $a = 20.301(2)$ Å
 $b = 6.9037(8)$ Å
 $c = 7.3463(8)$ Å

$V = 1029.6(2)$ Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.33$ mm⁻¹
 $T = 296(2)$ K
 $0.25 \times 0.25 \times 0.10$ mm

Data collection

Rigaku/MSC Mercury CCD diffractometer
 Absorption correction: none
 7769 measured reflections

1246 independent reflections
 1222 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.038$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.066$
 $wR(F^2) = 0.151$
 $S = 1.12$
 1246 reflections
 115 parameters

39 restraints
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.24$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.28$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C6}-\text{H6A}\cdots\text{O4}^i$	0.97	2.59	3.506 (7)	158
$\text{C10}-\text{H10A}\cdots\text{O4}^{ii}$	0.97	2.47	3.403 (6)	160
$\text{C6}-\text{H6B}\cdots\text{O5}^{iii}$	0.97	2.53	3.262 (7)	132
$\text{C6}-\text{H6B}\cdots\text{O1}^{iv}$	0.97	2.61	3.422 (7)	141

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

Data collection: *CrystalClear* (Molecular Structure Corporation & Rigaku, 2001); cell refinement: *CrystalClear*; data reduction: *TEXSAN* (Molecular Structure Corporation & Rigaku, 2000); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

This work was supported by a Grant-in-Aid (Nos. 17750037 and 19550034) from the Ministry of Education, Culture, Sports, Science and Technology, Japan. We thank the Instrument Center of the Institute for Molecular Science for the X-ray structure analysis.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2356).

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Acta Cryst. (2007). E63, o4568-o4569 [doi:10.1107/S1600536807054001]

2*H*,10*H*-1,4-Dioxepino[5',6':4,5]thieno[3,2-*e*][1,4]dioxepine-5,7(3*H*,9*H*)-dione

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Comment

Heterocyclic compounds containing a 1,4-dioxepin-5-one ring (7-membered lactone) are expected to exhibit antibacterial activity (Ito *et al.*, 1997; Rao, 1996). In addition, the compounds are of interest in terms of their polymerization abilities (Mathisen *et al.*, 1989). The title compound, (I), is the first example of a thiophene derivative with two fused 1,4-dioxepin-5-one rings and its molecular and crystal structures are described here.

The compound (I) crystallizes in the *Pnma* space group with one molecule in the asymmetric unit (Fig. 1). The bond lengths and angles are within the normal ranges (Table 1). The molecule lies on the mirror plane except the O2, C6, C9 and C10 atoms and the H atoms bonded to the C atoms, which are crystallographically disordered about the plane over each site with 0.5 of occupancy.

In the crystal structure, the molecules stack along the *b* axis, where no molecular overlap was observed (Fig. 2). The distance between the molecular planes is 3.45 Å. As shown in Table 2 and Fig. 2, the stacks are linked by intermolecular C—H...O hydrogen bonds (Taylor & Kennard, 1982; Biradha *et al.*, 1993; Batchelor *et al.*, 2000).

Experimental

The title compound (I) was synthesized by the intramolecular cyclization of diethyl 3,4-bis(2-hydroxyethoxy)-2,5-thiophenedicarboxylate (II). Compound (II) was prepared as follows: A mixture of diethyl 3,4-dihydroxythiophene-2,5-dicarboxylate (Halfpenny *et al.*, 2000) (6.55 g, 25.2 mmol) and caesium fluoride (11.5 g, 75.7 mmol) in dry acetonitrile (150 ml) was stirred for 1 h under nitrogen. A solution of ethylene glycol monotosylate (12.0 g, 55.5 mmol) in acetonitrile (50 ml) was added dropwise and the mixture was refluxed for 62 h. After cooling, the reaction mixture was filtered and the filtrate was concentrated. The residue was dissolved in dichloromethane and the solution was washed with water. The organic solution was dried over Na₂SO₄ and concentrated. The resulting solid was chromatographed on alumina gel (from AcOEt to AcOEt/EtOH = 1:1) and silica gel (CH₂Cl₂/AcOEt = from 7:3 to 1:1) to give compound (II) (3.96 g, 45%) as colorless needles. Physical data for (II): m.p. 356–357 K; IR (KBr, cm⁻¹): 3306, 1715, 1493, 1370, 1296, 1256, 1076, 1047; ¹H NMR (CDCl₃, δ p.p.m.): 1.38 (t, *J* = 7.1 Hz, 6H), 3.83 (br s, 6H), 4.33–4.41 (m, 8H); ¹³C NMR (CDCl₃, δ p.p.m.): 14.2, 61.0, 61.9, 76.4, 120.2, 153.1, 160.9; MS (EI): *m/z* 348 (*M*⁺), 302, 286, 256, 212, 168. Anal. Calcd. For C₁₄H₂₀O₈S: C, 48.27; H, 5.79. Found: C, 48.28; H, 5.68.

Compound (I) was prepared as follows: A mixture of compound (II) (367 mg, 1.05 mmol) and *p*-toluenesulfonic acid (20 mg) in toluene was refluxed with a Dean-Stark apparatus for 20 h. The white precipitate was filtered to give compound (I) (245 mg, 91%). Physical data for (I): m.p. >573 K; IR (KBr, cm⁻¹): 1692, 1522, 1462, 1406, 1373, 1325, 1161, 1100, 1044, 1015, 968, 752; ¹H NMR (DMSO-*d*₆, δ p.p.m.): 4.64–4.69 (m, 8H); ¹³C NMR (DMSO-*d*₆, δ p.p.m.): 162.8, 145.6,

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113.1, 71.4, 67.4; MS (EI): m/z 256 (M^+), 212, 185. Anal. Calcd. For $C_{10}H_8O_6S$: C, 46.87; H, 3.15. Found: C, 46.90; H, 3.10. Colorless crystals of (I) suitable for X-ray analysis were grown from an acetone solution.

Refinement

All H atoms were positioned geometrically refined using a riding model with C—H = 0.97 Å and with $U_{iso}(H) = 1.2$ times $U_{eq}(C)$.

Figures

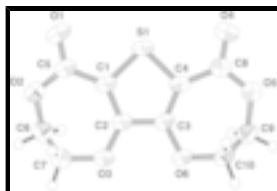


Fig. 1. The molecular structure of (I), with atom labels and 50% probability displacement ellipsoids for non-H atoms and H atoms are shown as small spheres of arbitrary radii. The crystallographically disordered atoms for O2, C6, C9 and C10 are omitted for clarity.

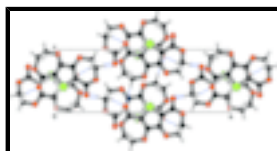


Fig. 2. The packing diagram of (I), viewed along the b axis. Dashed lines indicate intermolecular C—H...O hydrogen bonds.

2*H*,10*H*-1,4-Dioxepino[5',6':4,5]thieno[3,2-*e*][1,4]dioxepine-5,7(3*H*,9*H*)-dione

Crystal data

$C_{10}H_8O_6S$

$M_r = 256.22$

Orthorhombic, $Pnma$

Hall symbol: -P 2ac 2n

$a = 20.301$ (2) Å

$b = 6.9037$ (8) Å

$c = 7.3463$ (8) Å

$V = 1029.6$ (2) Å³

$Z = 4$

$F_{000} = 528$

$D_x = 1.653$ Mg m⁻³

Melting point: >573 K

Mo $K\alpha$ radiation

$\lambda = 0.71070$ Å

Cell parameters from 2531 reflections

$\theta = 3.0$ – 27.5°

$\mu = 0.33$ mm⁻¹

$T = 296$ (2) K

Prism, colorless

$0.25 \times 0.25 \times 0.10$ mm

Data collection

Rigaku/MSM Mercury CCD diffractometer

Radiation source: Rotating Anode

Monochromator: Graphite Monochromator

Detector resolution: 14.6199 pixels mm⁻¹

$T = 296$ (2) K

φ & ω scans

1246 independent reflections

1222 reflections with $I > 2\sigma(I)$

$R_{int} = 0.038$

$\theta_{max} = 27.5^\circ$

$\theta_{min} = 3.4^\circ$

$h = -26 \rightarrow 23$

Absorption correction: none
7769 measured reflections

$k = -8 \rightarrow 8$
 $l = -6 \rightarrow 9$

Refinement

Refinement on F^2

Secondary atom site location: difference Fourier map

Least-squares matrix: full

Hydrogen site location: inferred from neighbouring sites

$R[F^2 > 2\sigma(F^2)] = 0.066$

H-atom parameters constrained

$wR(F^2) = 0.151$

$$w = 1/[\sigma^2(F_o^2) + (0.1532P)^2 + 1.2875P]$$

where $P = (F_o^2 + 2F_c^2)/3$

$S = 1.12$

$(\Delta/\sigma)_{\max} = 0.003$

1246 reflections

$\Delta\rho_{\max} = 0.24 \text{ e } \text{\AA}^{-3}$

115 parameters

$\Delta\rho_{\min} = -0.28 \text{ e } \text{\AA}^{-3}$

39 restraints

Extinction correction: none

Primary atom site location: structure-invariant direct methods

Special details

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

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
S1	0.03443 (5)	0.2500	0.59051 (10)	0.0415 (3)	
O1	0.16771 (17)	0.2500	0.7229 (4)	0.0830 (12)	
O2	0.22262 (18)	0.1835 (6)	0.4789 (5)	0.0626 (14)	0.50
O3	0.12917 (14)	0.2500	0.1296 (3)	0.0647 (9)	
O4	-0.10643 (16)	0.2500	0.5961 (4)	0.0637 (8)	
O5	-0.13039 (15)	0.2500	0.3096 (4)	0.0825 (12)	
O6	0.00129 (15)	0.2500	0.0697 (3)	0.0829 (13)	
C1	0.10475 (18)	0.2500	0.4587 (4)	0.0393 (7)	
C2	0.08960 (18)	0.2500	0.2764 (4)	0.0436 (8)	
C3	0.02063 (18)	0.2500	0.2444 (4)	0.0446 (8)	
C4	-0.01558 (18)	0.2500	0.4004 (4)	0.0382 (7)	
C5	0.1665 (2)	0.2500	0.5615 (5)	0.0551 (10)	
C6	0.2204 (3)	0.1130 (11)	0.2935 (8)	0.0624 (15)	0.50
H6A	0.1911	0.0020	0.2890	0.075*	0.50
H6B	0.2641	0.0684	0.2601	0.075*	0.50

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C7	0.1991 (2)	0.2500	0.1616 (6)	0.0687 (13)	
H7A	0.2215	0.2234	0.0476	0.082*	0.50
H7B	0.2123	0.3784	0.2011	0.082*	0.50
C8	-0.08665 (19)	0.2500	0.4417 (5)	0.0452 (8)	
C9	-0.1102 (3)	0.1692 (10)	0.1254 (7)	0.0619 (15)	0.50
H9A	-0.0861	0.0488	0.1399	0.074*	0.50
H9B	-0.1486	0.1453	0.0502	0.074*	0.50
C10	-0.0673 (3)	0.3208 (10)	0.0416 (7)	0.0599 (18)	0.50
H10A	-0.0769	0.3347	-0.0871	0.072*	0.50
H10B	-0.0738	0.4449	0.1008	0.072*	0.50

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0580 (6)	0.0389 (5)	0.0277 (4)	0.000	0.0007 (3)	0.000
O1	0.077 (2)	0.135 (3)	0.0370 (14)	0.000	-0.0137 (14)	0.000
O2	0.0498 (19)	0.092 (4)	0.0462 (18)	-0.0018 (18)	-0.0067 (15)	0.0049 (18)
O3	0.0526 (16)	0.111 (3)	0.0304 (12)	0.000	0.0031 (11)	0.000
O4	0.0643 (18)	0.082 (2)	0.0452 (15)	0.000	0.0140 (13)	0.000
O5	0.0471 (17)	0.154 (4)	0.0467 (16)	0.000	0.0001 (13)	0.000
O6	0.0514 (17)	0.168 (4)	0.0289 (13)	0.000	-0.0037 (11)	0.000
C1	0.0520 (19)	0.0342 (17)	0.0317 (14)	0.000	-0.0017 (13)	0.000
C2	0.054 (2)	0.047 (2)	0.0293 (15)	0.000	0.0005 (13)	0.000
C3	0.050 (2)	0.054 (2)	0.0292 (15)	0.000	-0.0043 (13)	0.000
C4	0.0517 (18)	0.0304 (15)	0.0326 (15)	0.000	-0.0003 (13)	0.000
C5	0.057 (2)	0.070 (3)	0.0378 (18)	0.000	-0.0049 (16)	0.000
C6	0.046 (3)	0.084 (4)	0.057 (3)	0.004 (3)	0.004 (2)	-0.008 (3)
C7	0.051 (2)	0.107 (4)	0.048 (2)	0.000	0.0029 (18)	0.000
C8	0.052 (2)	0.0413 (19)	0.0425 (18)	0.000	0.0015 (15)	0.000
C9	0.055 (3)	0.080 (4)	0.051 (3)	0.003 (3)	-0.012 (2)	-0.011 (3)
C10	0.056 (3)	0.090 (5)	0.034 (2)	0.010 (3)	-0.011 (2)	0.004 (2)

Geometric parameters (\AA , $^\circ$)

S1—C1	1.725 (4)	C2—C3	1.420 (5)
S1—C4	1.727 (3)	C3—C4	1.361 (5)
O1—C5	1.186 (4)	C4—C8	1.474 (5)
O2—C6	1.447 (6)	C5—O2 ⁱ	1.370 (5)
O2—C5	1.370 (5)	C6—C7	1.421 (7)
O3—C2	1.345 (4)	C6—H6A	0.9700
O3—C7	1.439 (5)	C6—H6B	0.9700
O4—C8	1.204 (4)	C7—C6 ⁱ	1.421 (7)
O5—C8	1.315 (5)	C7—H7A	0.9700
O5—C9 ⁱ	1.520 (6)	C7—H7B	0.9700
O5—C9	1.520 (6)	C9—C10	1.494 (8)
O6—C3	1.342 (4)	C9—H9A	0.9700
O6—C10	1.491 (6)	C9—H9B	0.9700
O6—C10 ⁱ	1.491 (6)	C10—H10A	0.9700

C1—C2	1.375 (4)	C10—H10B	0.9700
C1—C5	1.463 (5)		
C1—S1—C4	91.88 (16)	O2—C6—H6B	108.4
C6—O2—C5	120.2 (4)	C7—C6—H6B	108.4
C2—O3—C7	117.3 (3)	H6A—C6—H6B	107.5
C8—O5—C9 ⁱ	118.3 (3)	C6 ⁱ —C7—C6	83.4 (6)
C8—O5—C9	118.3 (3)	C6 ⁱ —C7—O3	114.3 (3)
C3—O6—C10	113.9 (3)	C6—C7—O3	114.3 (3)
C3—O6—C10 ⁱ	113.9 (3)	C6 ⁱ —C7—H7A	124.9
C2—C1—C5	134.0 (3)	C6—C7—H7A	108.7
C2—C1—S1	111.2 (3)	O3—C7—H7A	108.7
C5—C1—S1	114.8 (2)	C6—C7—H7B	108.7
O3—C2—C1	130.4 (3)	O3—C7—H7B	108.7
O3—C2—C3	117.2 (3)	H7A—C7—H7B	107.6
C1—C2—C3	112.4 (3)	O4—C8—O5	118.0 (4)
O6—C3—C4	130.3 (3)	O4—C8—C4	121.4 (3)
O6—C3—C2	116.5 (3)	O5—C8—C4	120.6 (3)
C4—C3—C2	113.2 (3)	C10—C9—O5	105.5 (4)
C3—C4—C8	134.6 (3)	C10—C9—H9A	110.6
C3—C4—S1	111.3 (3)	O5—C9—H9A	110.6
C8—C4—S1	114.1 (2)	C10—C9—H9B	110.6
O1—C5—C1	122.2 (4)	O5—C9—H9B	110.6
O1—C5—O2 ⁱ	115.2 (4)	H9A—C9—H9B	108.8
C1—C5—O2 ⁱ	119.0 (3)	O6—C10—C9	104.9 (4)
O1—C5—O2	115.2 (4)	O6—C10—H10A	110.8
C1—C5—O2	119.0 (3)	C9—C10—H10A	110.8
O2—C6—C7	115.3 (5)	O6—C10—H10B	110.8
O2—C6—H6A	108.4	C9—C10—H10B	110.8
C7—C6—H6A	108.4	H10A—C10—H10B	108.8
C4—S1—C1—C2	0.0	S1—C1—C5—O2 ⁱ	-157.5 (2)
C4—S1—C1—C5	180.0	C2—C1—C5—O2	-22.5 (2)
C7—O3—C2—C1	0.0	S1—C1—C5—O2	157.5 (2)
C7—O3—C2—C3	180.0	C6—O2—C5—O1	158.5 (4)
C5—C1—C2—O3	0.0	C6—O2—C5—C1	-0.5 (5)
S1—C1—C2—O3	180.0	C6—O2—C5—O2 ⁱ	-101.9 (4)
C5—C1—C2—C3	180.0	C5—O2—C6—C7	60.1 (6)
S1—C1—C2—C3	0.0	O2—C6—C7—C6 ⁱ	23.1 (6)
C10—O6—C3—C4	21.0 (3)	O2—C6—C7—O3	-90.6 (5)
C10 ⁱ —O6—C3—C4	-21.0 (3)	C2—O3—C7—C6 ⁱ	-46.9 (4)
C10—O6—C3—C2	-159.0 (3)	C2—O3—C7—C6	46.9 (4)
C10 ⁱ —O6—C3—C2	159.0 (3)	C9 ⁱ —O5—C8—O4	155.4 (3)
O3—C2—C3—O6	0.0	C9—O5—C8—O4	-155.4 (3)
C1—C2—C3—O6	180.0	C9 ⁱ —O5—C8—C4	-24.6 (3)
O3—C2—C3—C4	180.0	C9—O5—C8—C4	24.6 (3)
C1—C2—C3—C4	0.0	C3—C4—C8—O4	180.0
O6—C3—C4—C8	0.0	S1—C4—C8—O4	0.0

supplementary materials

C2—C3—C4—C8	180.0	C3—C4—C8—O5	0.0
O6—C3—C4—S1	180.0	S1—C4—C8—O5	180.0
C2—C3—C4—S1	0.0	C8—O5—C9—C10	-75.3 (4)
C1—S1—C4—C3	0.0	C9 ⁱ —O5—C9—C10	27.0 (4)
C1—S1—C4—C8	180.0	C3—O6—C10—C9	-68.2 (4)
C2—C1—C5—O1	180.0	C10 ⁱ —O6—C10—C9	30.7 (4)
S1—C1—C5—O1	0.0	O5—C9—C10—O6	101.2 (4)
C2—C1—C5—O2 ⁱ	22.5 (2)		

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

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C6—H6A \cdots O4 ⁱⁱ	0.97	2.59	3.506 (7)	158
C10—H10A \cdots O4 ⁱⁱⁱ	0.97	2.47	3.403 (6)	160
C6—H6B \cdots O5 ^{iv}	0.97	2.53	3.262 (7)	132
C6—H6B \cdots O1 ^v	0.97	2.61	3.422 (7)	141

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

Fig. 1

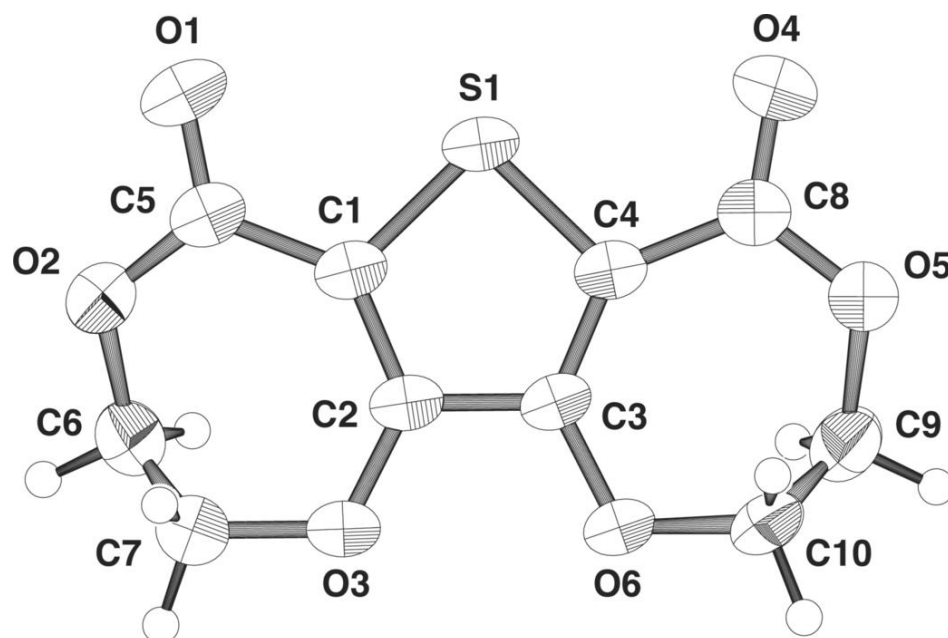


Fig. 2

