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Structure of 2-Selenoxoperhydro-2*H*-thieno[3,4-*d*][1,3]dithiole 5,5-Dioxide

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Abstract. $C_5H_6O_2S_3Se$, $M_r = 273$, monoclinic, $P2_1/a$, $a = 10.599$ (2), $b = 6.861$ (1), $c = 11.889$ (4) Å, $\beta = 100.6$ (2)°, $V = 849.8$ (7) Å^3, $Z = 4$, $D_x = 2.14$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 50.0$ cm^{-1}, $F(000) = 536$, $T = 294$ K, final $R = 0.048$ for 1176 observed reflections. The C=Se bond length is 1.795 (9) Å. The molecule adopts a butterfly-like shape where the five-membered rings, which form the wings, have envelope conformations. The dihedral angle between the mean planes of the five-membered rings is 73.0 (3)°.

Experimental. A crystal of dimensions 0.35 × 0.35 × 0.18 mm was used for X-ray crystal structure determination. Cell parameters were obtained by least-squares refinement of 25 reflections ($8 \leq 2\theta \leq 16°$) on an Enraf–Nonius CAD-4 diffractometer. Mo $K\alpha$ radiation, monochromatized by reflection from a graphite crystal, was used for data collection. Using the ω–2θ scan technique, 1715 reflections were collected ($2\theta < 50°$), of which 1493 were unique ($R_{\rm int} = 0.021$) and 1176 were considered observed by the criterion $I > 2.5\sigma(I)$. The *hkl* range was $-12 \leq h \leq 12$, $0 \leq k \leq 8$, $0 \leq l \leq 14$. The standard reflection decay was 1.04%. Lorentz and polarization corrections were applied but not absorption, decay or secondary extinction.

The structure was solved by direct methods with the *MULTAN*11/84 program (Main, Germain & Woolfson, 1984). Subsequent weighted Fourier syntheses gave the remaining non-H atoms. Full-matrix refinement on *F* was carried out with the *SHELX*76 least-squares program (Sheldrick, 1976) using anisotropic thermal parameters for non-H atoms. H atoms were refined riding on their C atoms with a global isotropic temperature factor. The refinement process converged at $R = 0.048$, $wR = 0.050$ with $w = 1/[\sigma^2(F) + 0.00015F^2]$, $S = 1.32$ using 101 variable parameters. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV) except for those of H atoms (Stewart, Davidson & Simpson, 1965). The largest shift/e.s.d. value in the final cycle was 0.005. Maximum and minimum final difference Fourier map peaks were 0.9 and -0.7 e Å^{-3}, respectively. Final atomic parameters are listed in Table 1,† while bond lengths and bond angles are shown in Table 2. Fig. 1 shows a perspective view of the asymmetric unit with the atom-numbering scheme which was depicted using *PLUTO* (Motherwell & Clegg, 1978).

Table 1. *Fractional atomic coordinates* ($\times 10^4$) *with their e.s.d.'s in parentheses and their equivalent isotropic temperature factors* (Å^2)

$$B_{\rm eq} = (8\pi^2/3)\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$
Se	5391 (1)	2346 (1)	951 (1)	3.74
S1	7691 (2)	−332 (4)	851 (2)	3.28
S3	7008 (2)	402 (4)	3083 (2)	3.41
S6	8211 (2)	−4307 (3)	3614 (2)	2.97
O1	9056 (7)	−5936 (9)	3916 (6)	4.33
O2	6961 (7)	−4376 (11)	3917 (6)	4.32
C2	6694 (8)	827 (13)	1621 (9)	3.16
C4	8617 (8)	−626 (13)	3131 (8)	2.93
C5	8988 (8)	−2068 (12)	4110 (8)	2.99
C7	8021 (9)	−3791 (13)	2094 (8)	3.20
C8	8592 (7)	−1756 (13)	2037 (7)	2.75
H4	9296 (8)	563 (13)	3244 (8)	4.35
H8	9563 (7)	−1977 (13)	1905 (7)	4.35
H51	10016 (8)	−2261 (12)	4301 (8)	4.35
H52	8657 (8)	−1563 (12)	4866 (8)	4.35
H71	7019 (9)	−3804 (13)	1696 (8)	4.35
H72	8537 (9)	−4845 (13)	1677 (8)	4.35

Related literature. The carbon—selenium bond length is 1.795 (9) Å which is in the range of the C(sp^2)=Se bond lengths [1.71–1.99 Å: Guziec (1987); 1.71–1.95 Å, mean value 1.87 (5) Å: Cambridge Structural Database, version 1/93 (Allen, Kennard & Taylor, 1983)]. The remaining bond distances have expected values. The molecule adopts a butterfly-like shape where the five-membered rings have envelope conformations, C4 being the atom that departs most from the mean plane of each ring. The dihedral angle between the mean planes of the five-membered rings

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† Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55917 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: NA1018]

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Table 2. *Bond distances* (Å), *angles* (°) *and hydrogen-bond geometry* (Å, °) *with e.s.d.'s in parentheses*

C2—Se	1.795 (9)	O2—S6	1.437 (7)
C4—S3	1.836 (9)	C5—S6	1.790 (9)
C2—S3	1.733 (10)	C7—S6	1.816 (9)
C2—S1	1.716 (10)	C4—C5	1.523 (13)
C8—S1	1.833 (8)	C8—C4	1.511 (12)
O1—S6	1.436 (7)	C7—C8	1.528 (12)

C2—S3—C4	95.8 (4)	C8—C4—S3	107.6 (6)
C8—S1—C2	97.0 (4)	C8—C4—C5	106.6 (7)
O2—S6—O1	118.2 (4)	S3—C2—Se	122.2 (6)
C5—S6—O1	111.0 (4)	S1—C2—Se	122.1 (6)
C5—S6—O2	109.6 (4)	S1—C2—S3	115.7 (5)
C7—S6—O1	110.5 (4)	C4—C8—S1	108.2 (6)
C7—S6—O2	108.5 (5)	C7—C8—S1	111.5 (6)
C7—S6—C5	96.9 (4)	C7—C8—C4	112.0 (7)
C4—C5—S6	105.5 (6)	C8—C7—S6	104.5 (6)
C5—C4—S3	112.4 (6)		

D—H···*X*	*D*···*X*	H···*X*	*D*—H···*X*
C5—H51···O1^{i}	3.14 (1)	2.49 (1)	118 (1)
C5—H51···O2^{ii}	3.35 (1)	2.46 (1)	139 (1)
C5—H52···O2^{iii}	3.28 (1)	2.26 (1)	157 (1)
C4—H4···O1^{iv}	3.36 (1)	2.56 (1)	130 (1)
C7—H72···Se^{ii}	3.92 (1)	2.86 (1)	167 (1)

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

is 73.0 (3)°. The shortest intermolecular distances are summarized in Table 2.

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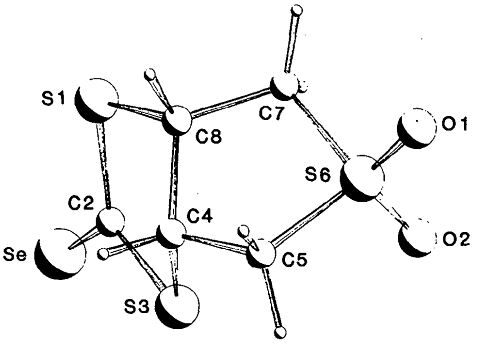

Fig. 1. Perspective view of the molecule with the atom-numbering scheme.

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