

thermal parameters; H atoms of CH and CH₃ groups riding on bonded C with a fixed isotropic temperature factor $U = 0.06 \text{ \AA}^2$, $\sum w(\Delta F)^2$ minimized, $w = [\sigma^2(F_o) + 0.00224(F_o)^2]^{-1}$, where σ is the standard deviation of observed amplitudes based on counting statistics. In the last cycle $(\Delta/\sigma)_{\text{max}} = 0.102$; $\Delta\rho$ from -0.26 to 0.24 e \AA^{-3} ; final $R = 0.059$, $wR = 0.076$ and $S = 1.16$. Scattering factors from *International Tables for X-ray Crystallography* (1974). All computations performed on a Nova 4S computer and plots drawn on a Tektronix plotter with the *SHELXTL* system of programs.

Atomic coordinates are given in Table 1.* A perspective view of the molecule and the atom labelling are shown in Fig. 1. Bond distances, angles and intermolecular approaches are listed in Table 2. The arrangement of the molecules in the unit cell is shown in Fig. 2.

* Lists of structure amplitudes, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51627 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structures of Sulfur Analogues of Precocenes. I. 2,2,6-Trimethyl-2H-1-benzothiopyran 1,1-Dioxide

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Abstract. C₁₂H₁₄O₂S, $M_r = 222.30$, orthorhombic, $P2_12_12_1$, $a = 7.357$ (2), $b = 10.186$ (3), $c = 15.253$ (2) Å, $V = 1143.0$ (8) Å³, $Z = 4$, $D_x = 1.292 \text{ g cm}^{-3}$, $\lambda(\text{Mo K}\alpha) = 0.71073$ Å, $\mu = 2.48 \text{ cm}^{-1}$, $F(000) = 472$, $T = 298 \text{ K}$, $R = 0.050$ for 663 observed reflections. The S atom is tetrahedral with a dihedral angle between planes C–S–C and O–S–O of 89.4 (4)°. The S–C(*sp*²) and S–C(*sp*³) bond distances are 1.751 (8) and 1.799 (9) Å respectively.

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Related literature. Recinine was first isolated from the seeds of *Recinus communis* by Tuson (1864) and was chemically characterized by Späth & Koller (1923, 1925). It was of interest to determine the crystal structure of (1) to ascertain its conformation and molecular geometry.

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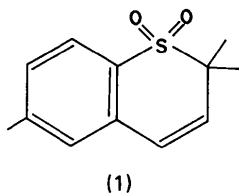
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Table 1. *Crystallographic summary* (1)

(a) Data collection ^{i,ii}	
Mode	$\omega-2\theta$
Scan rate (° min ⁻¹)	1.9–6.7
θ range (°)	0–22
Range of hkl	$-7 < h < 7$ $k < 10$ $l < 16$
Total reflections measured	1001
Unique reflections	948
R_{int}	0.01
Crystal dimensions (mm)	0.17 × 0.23 × 0.37
(b) Structure refinement ⁱⁱⁱ	
Reflections used [$I > 3\sigma(I)$]	663
No. of variables	137
R, wR	0.050, 0.054
Max. shift/e.s.d.	0.002
Max., min. density in difference map (e Å ⁻³)	0.30, -0.26
S	19.08

Notes: (i) Unit-cell parameters by least-squares refinement of the setting angles of 25 reflections with $9.5 < \theta < 16.8^\circ$. (ii) Enraf-Nonius CAD-4 diffractometer with a graphite monochromator was used. One standard reflection (600) measured every hour showed no significant variation. No correction for absorption. (iii) Function minimized was $\sum w(|F_o| - |F_c|)^2$, where $w^{-1} = [\sigma^2(F_o) + 0.002F_o^2]$.

(1965); programs used: *SHELX76* (Sheldrick, 1976), *ORTEP* (Johnson, 1965). Most of the calculations were performed on a VAX 11/780 computer of the Instituto de Física e Química de São Carlos.



Atomic coordinates are listed in Table 2,* bond lengths and angles in Table 3. Shortest intermolecular distances: O(1)···C(5)(0.5-x, 1-y, z-0.5) = 3.313 (8), O(2)···C(8)(1-x, y-0.5, 1.5-z) = 3.48 (1) Å. Fig. 1 is a perspective drawing of the molecule illustrating atom labeling.

Related literature. Structural data of several cyclic sulfones have been published by Yasuoka, Kasai, Tanaka, Nagai & Tokura (1972), Yasuoka, Kai & Kasai (1975), Towns & Simonsen (1975), Ealick, van der Helm, Ramalingam, Thyvelikakath & Berlin (1977), Ealick, van der Helm & Baker (1979) and Desiraju & Kamala (1983).

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Table 2. *Fractional atomic coordinates and isotropic temperature factors* (Å²)

	x	y	z	B _{iso} *
S	0.3433 (3)	0.3887 (2)	0.8164 (1)	3.73 (8)
O(1)	0.3135 (8)	0.4159 (5)	0.7256 (3)	4.7 (2)
O(2)	0.5162 (7)	0.3343 (6)	0.8419 (3)	5.1 (3)
C(2)	0.167 (1)	0.2822 (8)	0.8567 (5)	3.7 (3)
C(3)	0.166 (1)	0.2900 (8)	0.9556 (6)	4.2 (3)
C(4)	0.207 (1)	0.392 (1)	1.0024 (4)	4.3 (4)
C(5)	0.252 (1)	0.6378 (8)	1.0137 (4)	3.3 (3)
C(6)	0.287 (1)	0.760 (1)	0.9765 (6)	3.9 (4)
C(7)	0.328 (1)	0.7657 (7)	0.8879 (5)	3.9 (3)
C(8)	0.343 (1)	0.6533 (9)	0.8392 (4)	3.4 (3)
C(9)	0.306 (1)	0.5330 (8)	0.8761 (5)	2.9 (3)
C(10)	0.258 (1)	0.5225 (7)	0.9667 (5)	3.0 (3)
C(11)	0.2813 (9)	0.8796 (7)	1.0339 (5)	5.9 (4)
C(12)	-0.016 (1)	0.3276 (7)	0.8208 (4)	5.6 (4)
C(13)	0.2118 (9)	0.1425 (8)	0.8244 (4)	6.6 (4)

$$* B_{iso} = \frac{4}{3} \sum_i \sum_j B_{ij}(\mathbf{a}_i, \mathbf{a}_j).$$

Table 3. *Interatomic bond distances* (Å) and angles (°)

S-O(1)	1.429 (5)	C(4)-C(10)	1.48 (1)
S-O(2)	1.441 (6)	C(5)-C(6)	1.39 (1)
S-C(2)	1.799 (9)	C(5)-C(10)	1.38 (1)
S-C(9)	1.751 (8)	C(6)-C(7)	1.39 (1)
C(2)-C(3)	1.51 (1)	C(6)-C(11)	1.50 (1)
C(2)-C(12)	1.53 (1)	C(7)-C(8)	1.37 (1)
C(2)-C(13)	1.54 (1)	C(8)-C(9)	1.38 (1)
C(3)-C(4)	1.30 (1)	C(9)-C(10)	1.43 (1)
O(1)-S-O(2)	118.1 (3)	C(3)-C(4)-C(10)	125.1 (8)
O(1)-S-C(2)	109.7 (4)	C(6)-C(5)-C(10)	123.0 (7)
O(1)-S-C(9)	108.5 (4)	C(5)-C(6)-C(7)	118.4 (8)
O(2)-S-C(2)	108.2 (4)	C(5)-C(6)-C(11)	118.9 (7)
O(2)-S-C(9)	108.7 (4)	C(7)-C(6)-C(11)	122.7 (8)
C(2)-S-C(9)	102.4 (4)	C(6)-C(7)-C(8)	120.8 (8)
S-C(2)-C(3)	108.2 (6)	C(7)-C(8)-C(9)	120.5 (7)
S-C(2)-C(12)	109.3 (6)	S-C(9)-C(8)	120.3 (6)
S-C(2)-C(13)	107.1 (6)	S-C(9)-C(10)	118.6 (6)
C(3)-C(2)-C(12)	109.8 (7)	C(8)-C(9)-C(10)	120.7 (7)
C(3)-C(2)-C(13)	111.6 (7)	C(4)-C(10)-C(5)	124.4 (7)
C(12)-C(2)-C(13)	110.7 (7)	C(4)-C(10)-C(9)	118.9 (7)
C(2)-C(3)-C(4)	126.2 (8)	C(5)-C(10)-C(9)	116.6 (7)

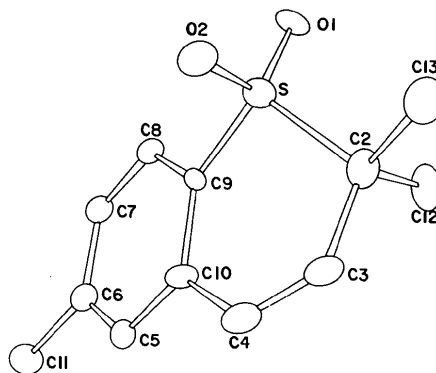


Fig. 1. Perspective view of the molecule showing atom labeling.

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Structures of Sulfur Analogues of Precocenes. II. 6,7-Dimethoxy-2,2-dimethyl-3,4-epoxy-3,4-dihydro-2H-1-benzothiopyran 1,1-Dioxide

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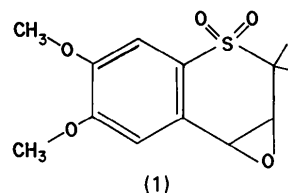
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Abstract. C₁₃H₁₆O₅S, $M_r = 284.33$, monoclinic, $P2_1/n$, $a = 12.180$ (5), $b = 6.535$ (2), $c = 16.893$ (7) Å, $\beta = 91.27$ (4)°, $V = 1344.3$ (9) Å³, $Z = 4$, $D_x = 1.405$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 2.42$ cm⁻¹, $F(000) = 600$, $T = 298$ K, $R = 0.053$ for 1242 observed reflections. The S atom is tetrahedral with a dihedral angle between planes C–S–C and O–S–O of 90.3 (2)°. The S–C(*sp*²) and S–C(*sp*³) bond distances are 1.756 (4) and 1.827 (5) Å respectively.

Experimental. The synthesis of compound (1) has been reported previously (Ferreira & Catani, 1987). The data-collection and refinement parameters are summarized in Table 1. The structure was solved using standard direct methods and difference Fourier techniques. In final cycles of full-matrix least-squares refinement all non-H atoms anisotropic. H atoms included, as fixed contributors, at positions found in difference synthesis, all with a common isotropic temperature factor that refined to $U = 0.083$ Å². Scattering factors for non-H atoms from Cromer & Mann (1968) with corrections for anomalous dispersion from Cromer & Liberman (1970), for H from Stewart, Davidson & Simpson (1965); programs used: *SHELX76* (Sheldrick, 1976), *ORTEP* (Johnson, 1965). Most of the calculations were performed on a VAX 11/

780 computer of the Instituto de Física e Química de São Carlos.



Atomic coordinates are listed in Table 2,* bond lengths and angles in Table 3. Shortest intermolecular distances: O(1)···C(3)($x, y-1, z$) = 3.315 (6), and O(2)···C(13)($1-x, -y, -z$) = 3.294 (6) Å. Fig. 1 is a perspective drawing of the molecule illustrating atom labeling.

Related literature. Structural data of several cyclic sulfones have been published by Yasuoka, Kasai,

* Lists of H-atom positions, anisotropic thermal parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51656 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.