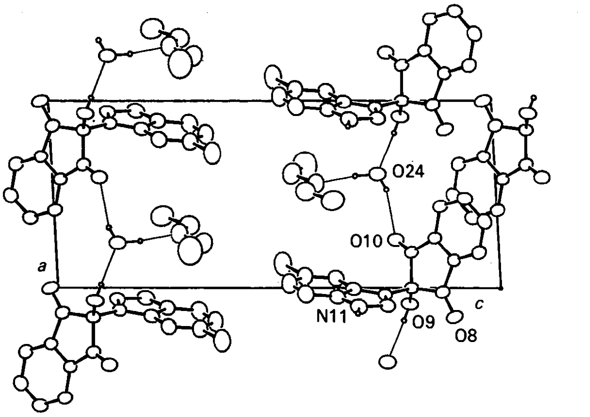

Fig. 2. Dessin de la structure vue selon [010]. Seuls sont représentés les atoms d'hydrogène qui participent à des liaisons hydrogène. La liaison N(11)—H(11)···O(9^{ii}) n'apparaît pas sur la figure.

indique les numéros attribués aux atomes de l'unité asymétrique et les lettres utilisées pour désigner les cycles. La Fig. 2 représente la structure vue selon [010].

Littérature associée. Structure de l'acide (indolyl-3)-acétique (Chandrasekhar & Raghunathan, 1982). Structure cristalline et moléculaire de l'(hydroxy-4 benzyl) pindolol (Weber & Petcher, 1977). Ester méthylique de l'acide α-(indolyl-3)-3 nitro-2 acrylique (Andrianov, Struchkov & Babievsky, 1982). Ethyl-2 phényl-2 indanedione-1,3 (Bravic, Gaultier & Hauw, 1974). Indanedione-1,3 (Bravic, Bechtel, Gaultier & Hauw, 1976). Etude par diffraction neutronique du nitro-2 indanedione-1,3 dihydrate (Selenius & Lundgren, 1980). Structure de deux acyl-2 indanediones-1,3 (Korp, Bernal & Lemke, 1980).

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Structures of Sulfur Analogues of Precocenes. III. 6-Methoxy-2,2-dimethyl-2*H*-1-benzothiopyran 1,1-Dioxide

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Abstract. $C_{12}H_{14}O_3S$, $M_r = 238{\cdot}31$, monoclinic, $P2_1/n$, $a = 6{\cdot}629\,(2)$, $b = 17{\cdot}233\,(3)$, $c = 10{\cdot}494\,(3)$ Å, $\beta = 92{\cdot}45\,(3)°$, $V = 1197{\cdot}7\,(9)$ Å^3, $Z = 4$, $D_x = 1{\cdot}321$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0{\cdot}21$ mm^{-1}, $F(000) = 504$, $T = 298$ K, $R = 0{\cdot}043$ for 1595 observed reflections. The S atom is tetrahedral with a dihedral angle between planes C—S—C and O—S—O of 89·1 (1)°. The S—C(sp^2) and S—C(sp^3)

Table 1. *Crystallographic summary for* (1)

(*a*) Data collection[i,ii]	
Mode	ω–2θ
Scan rate (° min^{-1})	2·6–6·7
θ range (°)	0–25
Range of *hkl*	$-7 < h < 7$, $0 < k < 20$, $0 < l < 12$
Total reflections measured	2292
Unique reflections	2102
R_{int}	0·011
Crystal dimensions (mm)	~0·28 × 0·50 × 0·50
(*b*) Structure refinement[iii]	
Reflections used $[I > 3\sigma(I)]$[iv]	1595
No. of variables	146
R, *wR*	0·043, 0·050
Max. shift/e.s.d.	0·005
Max., min. density in final difference map (e Å^{-3})	0·22, −0·28
S	2·02

Notes: (i) Unit-cell parameters by least-squares refinement of the setting angles of 25 reflections with $13 < \theta < 25°$. (ii) Enraf–Nonius CAD-4 diffractometer with a graphite monochromator. Two standard reflections ($2,\overline{10},0$ and 462), measured every hour showed no significant variation. No correction for absorption. (iii) Function minimized was $\sum w(|F_o| - |F_c|)^2$, where $w^{-1} = [\sigma^2(F_o) + 0{\cdot}001F_o^2]$. (iv) Reflections 263 and 032 were also excluded during refinement.

Table 2. *Fractional atomic coordinates and equivalent isotropic temperature factors* (Å^2)

	x	*y*	*z*	B_{iso}*
S	0·2747 (1)	0·1501 (0)	−0·2166 (1)	3·75 (3)
O(1)	0·3251 (3)	0·2297 (1)	−0·2363 (2)	5·55 (8)
O(2)	0·3933 (3)	0·0931 (1)	−0·2784 (2)	5·69 (8)
O(3)	0·3276 (3)	0·1156 (1)	0·3380 (2)	5·19 (7)
C(2)	0·0109 (4)	0·1375 (1)	−0·2591 (3)	3·99 (9)
C(3)	−0·1072 (4)	0·1818 (2)	−0·1635 (3)	4·4 (1)
C(4)	−0·0569 (4)	0·1892 (2)	−0·0409 (3)	4·3 (1)
C(5)	0·1488 (4)	0·1490 (1)	0·1517 (3)	3·94 (9)
C(6)	0·3224 (4)	0·1183 (2)	0·2083 (2)	3·85 (9)
C(7)	0·4788 (4)	0·0929 (2)	0·1354 (2)	4·02 (9)
C(8)	0·4571 (4)	0·0996 (2)	0·0037 (2)	3·77 (9)
C(9)	0·2864 (3)	0·1312 (1)	−0·0524 (2)	3·11 (8)
C(10)	0·1264 (4)	0·1568 (1)	0·0204 (2)	3·35 (8)
C(11)	−0·0410 (5)	0·0513 (2)	−0·2551 (3)	5·9 (1)
C(12)	−0·0222 (5)	0·1705 (2)	−0·3942 (3)	5·8 (1)
C(13)	0·5149 (5)	0·0964 (2)	0·4016 (3)	6·1 (1)

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

bond distances are 1·752 (2) and 1·799 (3) 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 $U = 0{\cdot}092$ Å^2. 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.

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

S—O(1)	1·429 (2)	C(3)—C(4)	1·321 (4)
S—O(2)	1·431 (2)	C(4)—C(10)	1·461 (4)
S—C(2)	1·799 (3)	C(5)—C(6)	1·378 (4)
S—C(9)	1·752 (2)	C(5)—C(10)	1·386 (4)
O(3)—C(6)	1·361 (3)	C(6)—C(7)	1·385 (4)
O(3)—C(13)	1·423 (4)	C(7)—C(8)	1·388 (3)
C(2)—C(3)	1·506 (4)	C(8)—C(9)	1·366 (3)
C(2)—C(11)	1·526 (4)	C(9)—C(10)	1·404 (3)
C(2)—C(12)	1·535 (4)		
O(1)—S—O(2)	117·2 (1)	C(3)—C(4)—C(10)	124·2 (2)
O(1)—S—C(2)	108·1 (1)	C(6)—C(5)—C(10)	121·2 (2)
O(1)—S—C(9)	108·7 (1)	O(3)—C(6)—C(5)	115·4 (2)
O(2)—S—C(2)	110·6 (1)	O(3)—C(6)—C(7)	123·6 (2)
O(2)—S—C(9)	108·4 (1)	C(5)—C(6)—C(7)	120·9 (2)
C(2)—S—C(9)	102·9 (1)	C(6)—C(7)—C(8)	118·4 (2)
C(6)—O(3)—C(13)	117·5 (2)	C(7)—C(8)—C(9)	120·7 (2)
S—C(2)—C(3)	107·5 (2)	S—C(9)—C(8)	119·9 (2)
S—C(2)—C(11)	109·2 (2)	S—C(9)—C(10)	118·3 (2)
S—C(2)—C(12)	106·5 (2)	C(8)—C(9)—C(10)	121·4 (2)
C(3)—C(2)—C(11)	110·5 (2)	C(4)—C(10)—C(5)	121·8 (2)
C(3)—C(2)—C(12)	111·7 (2)	C(4)—C(10)—C(9)	120·9 (2)
C(11)—C(2)—C(12)	111·3 (2)	C(5)—C(10)—C(9)	117·3 (2)
C(2)—C(3)—C(4)	125·5 (2)		

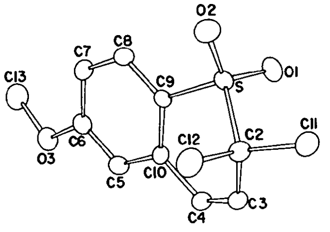
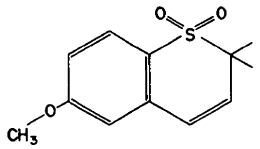


Fig. 1. Perspective view of the molecule showing the atom labeling.



(1)

Atomic coordinates are listed in Table 2,* bond lengths and bond angles in Table 3. Shortest intermolecular distances: O(1)···C(5)($0{\cdot}5 + x$, $0{\cdot}5 - y$, $z - 0{\cdot}5$) = 3·251 (3) and O(2)···C(13)($1 - x$, $-y$, $-z$) = 3·574 (4) Å. Fig. 1 is a perspective drawing of the molecule illustrating the atom labeling.

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* 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 51869 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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, Ramalingan, Thyvelikakath & Berlin (1977), Ealick, van der Helm & Baker (1979), Desiraju & Kamala (1983) and Castellano, De Simone, Zukerman-Schpector, Ferreira & Catani (1989).

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Acta Cryst. (1989). **C45**, 1448–1450

Structures of Sulfur Analogues of Precocenes. IV. 7-Ethoxy-6-methoxy-2,2-dimethyl-2*H*-1-benzothiopyran 1,1-Dioxide

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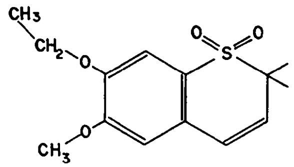
(*Received* 23 *February* 1989; *accepted* 31 *March* 1989)

Abstract. $C_{14}H_{18}O_4S$, $M_r = 282{\cdot}36$, monoclinic, $P2_1/c$, $a = 11{\cdot}372\,(2)$, $b = 10{\cdot}544\,(3)$, $c = 12{\cdot}122\,(3)$ Å, $\beta = 106{\cdot}23\,(2)°$, $V = 1396\,(1)$ Å^3, $Z = 4$, $D_x = 1{\cdot}344$ Mg m^{-3}, $\lambda(\mathrm{Mo}\ K\alpha) = 0{\cdot}71073$ Å, $\mu = 0{\cdot}19$ mm^{-1}, $F(000) = 600$, $T = 298$ K, $R = 0{\cdot}042$ for 1265 observed reflections. The S atom is tetrahedral with a dihedral angle between planes C—S—C and O—S—O of 90·0 (1)°. The S—C(sp^2) and S—C(sp^3) bond distances are 1·758 (4) and 1·800 (3) 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 $U = 0{\cdot}077$ Å^2. 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.



(1)