

The interatomic distances and angles in the hexachloronorborene moiety for the four related compounds are similar except that the bonds from C(3) and C(6) to C(11) are somewhat shorter, the angle C(3)–C(11)–C(6) is greater and the C(11)–Cl(1) bond is significantly longer in isodrin (1.86 Å compared with the mean of 1.77 Å). Long C–Cl bonds are, however, not uncommon among the members of the cyclodiene series, but only where Cl is substituted at C(7) or C(10), for example isobenzan (1.84 Å), heptachlor (1.85 Å) and heptachlor epoxide (1.817 Å) (Hovmöller, Smith & Kennard, 1978). In the non-chlorinated residue, the angle C(7)–C(12)–C(10) [101.1 (2)°] is significantly larger than that found in aldrin [93.7 (5)°]. This is probably due to the shortness of C(7)–C(12) (1.44 compared with 1.54 Å) as a result of the interaction between the two sets of double bonds within the molecule. The mid-point double bond to double bond distances for isodrin and aldrin are 2.86 and 4.50 Å respectively.

The overall shape of isodrin is more equidimensional than that of aldrin, with the mid-point of C(8)=C(9) to Cl(1) and Cl(2) being 5.23 and 4.93 Å respectively. The longest distances across the approximate mirror plane [Cl(3)–Cl(6)] are similar (5.49, 5.53 Å), but this is expected because of the relatively constant size and shape found for all members of the cyclodiene series. There were no close intermolecular constants; consequently, only van der Waals forces hold the solid together (Fig. 2).

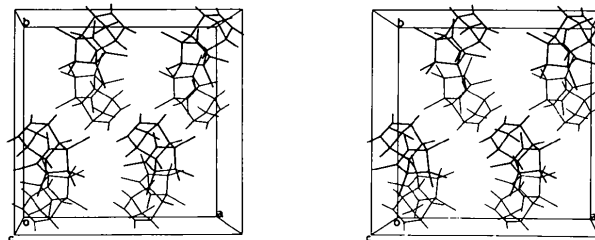


Fig. 2. Stereoview of the packing of isodrin viewed down the *c* axis.

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## Thiochroman-4-one 1,1-Dioxide

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**Abstract.** C<sub>9</sub>H<sub>8</sub>O<sub>3</sub>S, *M<sub>r</sub>* = 196.21, orthorhombic, *Pca*2<sub>1</sub>, *Z* = 4. At *T* = 298 K: *a* = 14.901 (1), *b* = 7.7875 (3), *c* = 7.4373 (3) Å, *V* = 863.0 Å<sup>3</sup>, *D<sub>c</sub>* = 1.510, *D<sub>m</sub>* = 1.508 Mg m<sup>-3</sup>. At *T* = 113 K: *a* = 15.014 (9), *b* = 7.678 (5), *c* = 7.281 (5) Å, *V* = 839.3 Å<sup>3</sup>. Final *R* value for all 935 intensity data is 0.026. The structure solution shows a small difference between the S–O distances: 1.440 (2) and 1.450 (2) Å while the

two S–C distances are the same: 1.763 (2) and 1.766 (2) Å.

**Introduction.** Thiochroman-4-one 1,1-dioxide is a keto sulfone which serves as precursor for a number of polycyclic pyrazoles, isoxazoles and imines which show a variety of bactericidal and drug potentiation effects. Thiochroman-4-one 1,1-dioxide itself shows 100%

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Table 1. Atomic coordinates ( $x \times 10^5$ ,  $y$ ,  $z \times 10^4$ ) for S, O and C atoms

Standard deviations for the least significant digit are given in parentheses.

	$x$	$y$	$z$		$x$	$y$	$z$
S(1)	4222 (3)	2128.0 (6)	-1000*	C(8)	13724 (17)	4876 (3)	-2168 (4)
C(2)	7502 (15)	-76 (3)	-1037 (4)	C(9)	14376 (15)	3211 (3)	-1443 (3)
C(3)	15374 (17)	-348 (3)	260 (4)	C(10)	22674 (14)	2454 (3)	-1057 (4)
C(4)	23631 (16)	655 (3)	-252 (3)	O(1A)	-1743 (14)	2469 (3)	-2506 (3)
C(5)	30337 (15)	3414 (3)	-1429 (4)	O(1B)	1322 (14)	2571 (3)	841 (3)
C(6)	29781 (17)	5076 (3)	-2140 (4)	O(4)	30993 (13)	61 (2)	46 (3)
C(7)	21458 (18)	5823 (3)	-2515 (4)				

\*  $z$  coordinate of S atom fixed.Table 2. Atomic coordinates ( $\times 10^3$ ) and thermal parameters for H atoms

The H atoms are given the number of the atoms to which they are bonded.

	$x$	$y$	$z$	$B$ ( $\text{\AA}^2$ )		$x$	$y$	$z$	$B$ ( $\text{\AA}^2$ )
H(2A)	90 (2)	-30 (4)	-231 (5)	1.3 (6)	H(4)	358 (2)	285 (4)	-112 (6)	1.8 (7)
H(2B)	28 (3)	-82 (6)	-84 (8)	3.7 (9)	H(5)	349 (3)	591 (5)	-217 (7)	3.2 (8)
H(3A)	168 (2)	-153 (4)	32 (5)	0.9 (5)	H(6)	205 (2)	688 (4)	-317 (6)	1.7 (6)
H(3B)	138 (2)	9 (5)	149 (6)	2.6 (8)	H(7)	81 (3)	507 (4)	-251 (6)	2.8 (8)

inhibition of *Bacillus subtilis* at a concentration less than  $91 \mu\text{g ml}^{-1}$  and 17% inhibition of KB cell growth at  $50 \mu\text{g ml}^{-1}$ . The title compound was prepared at room temperature by addition of 30% hydrogen peroxide to thiochroman-4-one in glacial acetic acid (Ramalingam *et al.*, 1977).

All data were taken on a Nonius CAD-4 automatic diffractometer,  $\lambda(\text{Cu } K\alpha) = 1.54178 \text{ \AA}$ , for intensity data and  $\lambda(\text{Cu } K\alpha_1) = 1.54051 \text{ \AA}$  for least-squares cell dimensions. The crystal system was shown to be orthorhombic with systematic extinctions:  $0kl$  ( $l = 2n + 1$ ) and  $h0l$  ( $h = 2n + 1$ ), indicating the space group to be either  $Pca2_1$  or  $Pcam$ . The refinement of the structure showed the space group to be  $Pca2_1$ . Intensity data were measured at 113 K. A total of 935 intensities [931 with  $I > 2\sigma(I)$ ] comprising all unique data with  $2\theta < 150^\circ$  were measured twice each using  $\theta$ - $2\theta$  scan techniques as two separate data sets. The two data sets were averaged prior to the absorption correction ( $\mu = 3.091 \text{ mm}^{-1}$ ). The data were corrected for Lorentz and polarization effects and individual structure factor amplitudes and their relative weights were derived (van der Helm & Poling, 1976).

The positions of the S, C and O atoms were obtained from the interpretation of a sharpened Patterson map. The H atoms were located from a difference Fourier map based on the partially refined positions of the S, C and O atoms. All nonhydrogen atoms were included in the full-matrix least-squares refinement with anisotropic thermal parameters while the H atoms were included with isotropic thermal parameters. In the last stages of the refinement, a correction was applied for the anomalous scattering of the S atom. The correct space group polarity was determined by refining the two sets of atomic parameters. The final  $R = (\sum ||F_o| -$

$|F_c|)| / \sum |F_o|$  was 0.026 for all data. The magnitude of  $[\sum w(|F_o| - |F_c|)^2 / (m - n)]^{1/2}$ , where  $m$  is the number of reflections and  $n$  is the number of parameters refined, was 0.810. The quantity  $\sum w(|F_o| - |F_c|)^2$  was minimized using the *ORFLS* program (Busing, Martin & Levy, 1962). The atomic scattering factors used for S, C and O atoms, and the real and imaginary part of the anomalous scattering for the S atoms were from *International Tables for X-ray Crystallography* (1962, 1974). For the H atoms the values given by Stewart, Davidson & Simpson (1965) were used. The final atomic parameters are given in Tables 1 and 2.\*

**Discussion.** The six-membered heterocycle was found to exist in an envelope conformation with C(2) farthest from the plane of the ring. This description can be seen

\* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34015 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

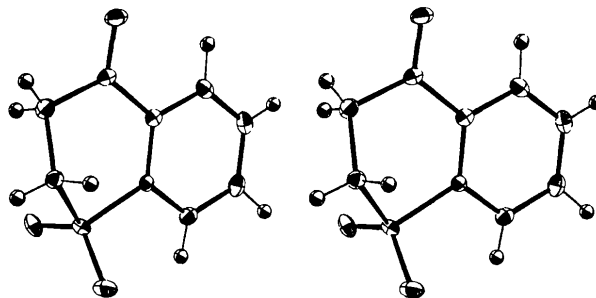


Fig. 1. Stereoscopic drawing of thiochroman-4-one 1,1-dioxide (Johnson, 1965).

from the internal torsion angles S(1)—C(9)  $-24.2$ , C(9)—C(10)  $0.6$ , C(10)—C(4)  $-3.4$ , C(4)—C(3)  $36.3$ , C(3)—C(2)  $-62.4$  and C(2)—S(1)  $52.5^\circ$ , and from the stereoscopic view of the molecule shown in Fig. 1. The molecule is relatively planar with the S—O(1A) bond approximately in the plane and the S—O(1B) bond approximately perpendicular to the plane.

The bond distances and angles are shown in Figs. 2 and 3. The two S—O bond distances showed a small difference of  $0.01 \text{ \AA}$  ( $5\sigma$ ). Such differences have been observed in other sulfones in six-membered rings (Ealick, van der Helm, Ramalingan, Thyvelikakath & Berlin, 1977; Towns & Simonsen, 1975; Andreetti, Bocelli, Coghi & Sgarabotto, 1976). The bond distances of the S atom are similar to those observed in 1,9-dihydro[1]benzothiopyrano[4,3-*c*]pyrazole 5,5-dioxide (Ealick *et al.*, 1977); the S—O distances in the present compound are about  $0.006 \text{ \AA}$  longer and the S—C distances  $0.006 \text{ \AA}$  shorter, while in both structures the S—O bond which is approximately perpendicular to the molecular plane is the longer of the two S—O distances. Also similar is the unexpected observation that the two S—C distances are not significantly

different in length, although one involves an  $sp^2$ -hybridized atom, C(9), while the other involves an  $sp^3$ -hybridized atom, C(2). The  $R$  value for the incorrect polarity was  $0.032$  and significantly higher than  $0.026$  calculated for the parameters given in Tables 1 and 2. It is interesting to note that the change in polarity seriously affects the S—O distances which were calculated to be S—O(1A)  $1.468 \text{ \AA}$  and S—O(1B)  $1.416 \text{ \AA}$  compared to  $1.440$  and  $1.450 \text{ \AA}$  in the correct polarity.

The smallest interatomic distances are observed for the three O atoms which are involved in contacts with H atoms. The contact distances are smaller than the sum of the van der Waals radii: O(4)···H(2A)( $\frac{1}{2} - x, y, \frac{1}{2} + z$ ):  $2.46 \text{ \AA}$ , O(1A)···H(6)( $-\frac{1}{2} + x, 1 - y, z$ ):  $2.37 \text{ \AA}$  and O(1B)···H(8)( $-x, 1 - y, \frac{1}{2} + z$ ):  $2.59 \text{ \AA}$ . In all three cases, the angle C—H···O is larger than  $150^\circ$ .

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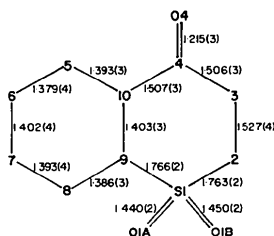


Fig. 2. Bond distances ( $\text{\AA}$ ) and numbering scheme.

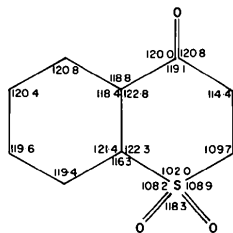


Fig. 3. Bond angles ( $^\circ$ ). The standard deviations are between  $0.1$  and  $0.2^\circ$ . Additional bond angles are O(1A)—S(1)—C(2):  $109.7^\circ$  and O(1B)—S(1)—C(9):  $108.5^\circ$ .