

constrained to threefold symmetry about the N(2)—C bonds and C—H was constrained to 1.03 Å. Only the angles of rotation of the methyl groups about the N(2)—C bonds, the N—C—H angle and the overall isotropic temperature factor for the methyl H atoms were allowed to vary. The resultant angle N—C—H = 103 (3)°; the resultant angles of rotations of methyl groups brought H(73) and H(83) into the plane defined by the benzene ring (see Fig. 1). The position of H(72) from this refinement is (0.66, 0.51, 0.10) which is in a good agreement with its position from the final Fourier map. With the methyl groups thus constrained, the agreement factors ($R = 0.165$ and $R_w = 0.035$ for all atoms freely refined) rose to $R = 0.261$ and $R_w = 0.085$. Fractional coordinates of methyl H atoms differ by an average 1.6σ [max. 4.4σ for H(72)] from their unconstrained counterparts. Max. shift = 0.62 Å [for H(72)], mean [without H(72)] = 0.27 Å. Computer programs: *XRAY* (Stewart, Kruger, Ammon, Dickinson & Hall, 1972), *ORFLS* (Busing, Martin & Levy, 1962), *ORFFE* (Busing, Martin & Levy, 1964), *ORTEP* (Johnson, 1971).

Discussion. Fractional coordinates for both X-ray and neutron measurements are listed in Table 1;* the bond distances and angles are plotted in Fig. 1, which also shows the molecular conformation and the numbering of the atoms. The molecular packing is shown in Fig. 2.

The hydrogen bridge $I \cdots H(1) - N(1)$ is linear because all three atoms are in special positions (0, y , 0.25). Although the anisotropy of the thermal ellipsoid of H(1) is very high and its largest amplitude is in the direction

* Lists of anisotropic thermal parameters, structure factors and a more detailed table of interatomic distances and angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39259 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

of the $I \cdots H(1) - N(1)$ bond, attempts to introduce statistically disordered H(1) positions failed on a very high correlation and the divergence of the refinement.

Comparison of three available sets of atomic coordinates (the X-ray study of TO, and the present X-ray and neutron measurements) shows that for non-hydrogen atoms the average estimated standard deviations are in the ratio 12.7:1:2.4 respectively. Atomic positions of I, N, C atoms resulting from these three refinements agree on average within 0.75σ (σ taken for neutron data) and differ by 2.1σ at the most.

The lower accuracy of the neutron measurement was imposed by the allocated time, the small size of the sample and a very high background caused by the incoherent scattering of H atoms in the sample.

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Structure of 6-Chloro-4-phenyl-1,2,3-benzoxathiazine 2,2-Dioxide, $C_{13}H_8ClNO_3S$

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Abstract. $M_r = 293.7$, orthorhombic, *Pbca*, $a = 8.418$ (1), $b = 14.708$ (6), $c = 20.564$ (3) Å, $V = 2546.1$ Å³, $Z = 8$, $D_m = 1.50$, $D_x = 1.53$ Mg m⁻³, Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å, $\mu = 0.407$ mm⁻¹, $F(000) = 1200.0$, room temperature, $R = 0.060$ for 1272 observed reflections. Bond lengths and angles are

in agreement with expected values. The oxathiazine ring exhibits a distorted half-chair conformation; the chlorophenyl and benzo groups are each planar, with a dihedral angle between these planes of -150.3 (2)°. There are no intermolecular contacts less than van der Waals distances.

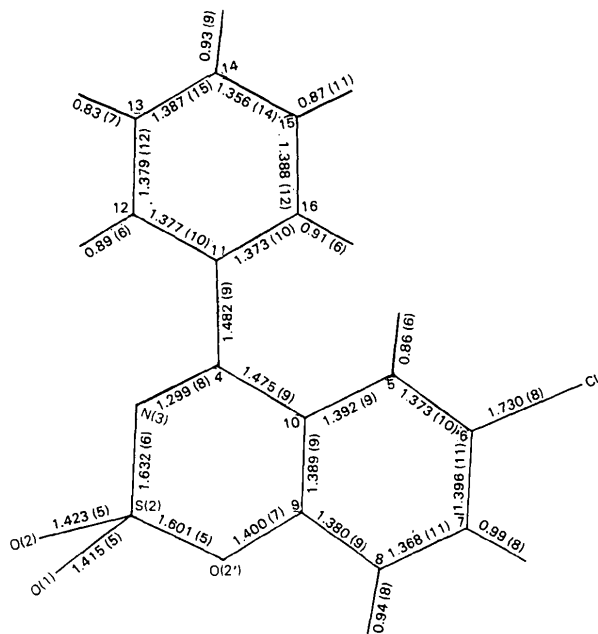


Fig. 1. Atomic numbering and the bond distances (Å) in the molecule with e.s.d.'s in parentheses.

Table 1. Positional parameters ($\times 10^4$; $\times 10^3$ for H) and isotropic thermal parameters ($\text{Å}^2 \times 10^3$)

$$U_{eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33}).$$

	x	y	z	U_{eq}/U_{iso}
O(1)	7619 (6)	4112 (3)	2218 (2)	48 (3)
S(2)	8343 (2)	3145 (1)	2019 (1)	41 (1)
O(2')	7205 (6)	2760 (4)	1594 (2)	57 (3)
O(2)	9924 (6)	3280 (4)	1793 (2)	43 (3)
N(3)	8360 (7)	2557 (4)	2692 (3)	41 (3)
C(4)	8573 (8)	2973 (4)	3242 (3)	34 (4)
C(5)	9604 (9)	4384 (5)	3791 (4)	43 (4)
C(5)	9878 (9)	5304 (5)	3782 (4)	47 (5)
C(6)	10979 (3)	5813 (2)	4392 (1)	85 (2)
C(7)	9321 (10)	5840 (6)	3269 (5)	55 (6)
C(8)	8544 (10)	5442 (5)	2759 (4)	52 (5)
C(9)	8351 (4)	4511 (5)	2758 (3)	38 (4)
C(10)	8828 (8)	3965 (4)	3274 (3)	33 (4)
C(11)	8558 (8)	2403 (5)	3837 (3)	37 (4)
C(12)	7747 (10)	2694 (6)	4380 (4)	54 (5)
C(13)	7658 (13)	2155 (8)	4928 (4)	80 (7)
C(14)	8399 (13)	1313 (8)	4930 (5)	73 (7)
C(15)	9210 (12)	1024 (7)	4399 (6)	63 (7)
C(16)	9268 (11)	1563 (5)	3845 (5)	66 (6)
H(5)	1002 (8)	409 (4)	412 (3)	18 (2)*
H(7)	954 (9)	650 (5)	333 (4)	66 (3)*
H(8)	808 (10)	576 (5)	241 (4)	75 (3)*
H(12)	724 (7)	322 (4)	439 (3)	19 (2)*
H(13)	714 (9)	236 (5)	524 (4)	41 (3)*
H(14)	841 (11)	104 (6)	534 (4)	92 (3)*
H(15)	964 (10)	493 (7)	435 (6)	130 (5)*
H(16)	964 (9)	135 (5)	346 (3)	31 (3)*

* U_{iso} .

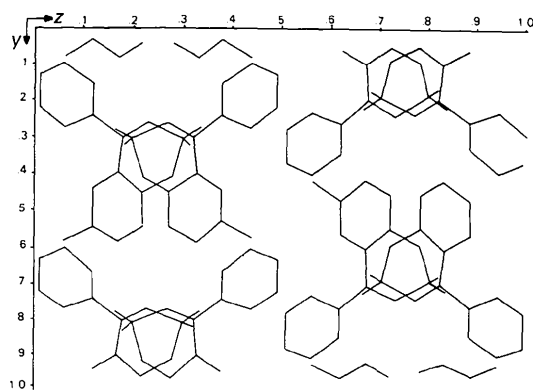


Fig. 2. Packing of the molecules viewed along [100].

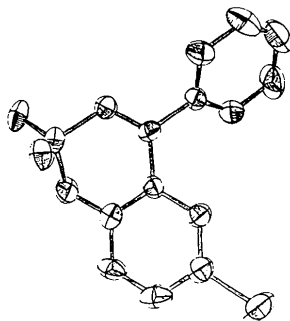


Fig. 3. ORTEP drawing of the molecule.

Table 2. Bond angles ($^\circ$) with e.s.d.'s in parentheses

C(1)-C(6)-C(5)	120.4 (6)	C(4)-C(11)-C(12)	119.9 (6)
C(1)-C(6)-C(7)	118.9 (6)	C(4)-C(11)-C(16)	121.1 (7)
C(5)-C(6)-C(7)	120.6 (8)	C(4)-N(3)-S(2)	119.3 (5)
C(6)-C(5)-C(10)	120.4 (7)	C(12)-C(11)-C(16)	119.0 (7)
C(6)-C(7)-C(8)	119.9 (7)	C(11)-C(12)-C(13)	120.9 (9)
C(5)-C(10)-C(9)	117.5 (6)	C(11)-C(16)-C(15)	120.6 (9)
C(5)-C(10)-C(4)	122.6 (6)	C(12)-C(13)-C(14)	119.4 (10)
C(9)-C(10)-C(4)	119.7 (6)	C(13)-C(14)-C(15)	120.3 (1)
C(10)-C(9)-C(8)	122.7 (7)	C(14)-C(15)-C(16)	120.0 (10)
C(10)-C(9)-O(1)	119.4 (6)	N(3)-S(2)-O(1)	105.0 (3)
C(10)-C(4)-C(11)	121.6 (6)	N(3)-S(2)-O(2')	108.5 (3)
C(10)-C(4)-N(3)	121.6 (6)	N(3)-S(2)-O(2)	110.0 (3)
C(8)-C(9)-O(1)	117.9 (7)	O(1)-S(2)-O(2')	104.8 (3)
C(9)-C(8)-C(7)	118.8 (8)	O(2)-S(2)-O(1)	108.4 (3)
C(9)-O(1)-S(2)	114.0 (4)	O(2')-S(2)-O(2)	119.2 (3)
C(11)-C(4)-N(3)	116.8 (6)		

Introduction. The title compound was supplied by Kamal & Sattur (1981) and an X-ray investigation was undertaken in order to ascertain the geometry of the molecule.

Experimental. Crystal $0.5 \times 0.3 \times 0.3$ mm, preliminary examination by photographic methods, density by flotation in zinc bromide solution, cell dimensions from 25 high-angle reflections, Enraf-Nonius CAD-4 automatic diffractometer, 3806 reflections collected, ω - 2θ scans, 2891 unique, $R_{int} = 0.045$, 2718 reflections considered observed with $I \geq 2\sigma(I)$; intensity variation of standards negligible; Lorentz and polarization corrections but not absorption; structure determined by direct methods using *MULTAN78* (Main,

Hull, Lessinger, Germain, Declercq & Woolfson, 1978), non-H atoms refined on F using full-matrix least squares with *SHELX* (Sheldrick, 1976), H atoms found in difference Fourier map, refined with isotropic temperature factors equal to those of their bonded C atoms; refinement of overall scale factor, positional and thermal parameters (anisotropic for non-hydrogen atoms) converged to $R = 0.060$ for 1272 reflections (index range h 0 to 9, k 0 to 16, l 0 to 22; $\sin\theta \leq 0.39$); unit weights used throughout refinement; max. Δ in final LS cycle = 1.1σ , $\Delta\rho = \pm 0.4 \text{ e } \text{\AA}^{-3}$; scattering factors from *SHELX*.

Discussion. Atomic coordinates are listed in Table 1* and bond angles in Table 2. The labelling of the atoms in the molecule and the bond lengths are shown in Fig. 1. The packing of the molecules viewed along a is illustrated in Fig. 2. Fig. 3 is an *ORTEP* (Johnson, 1965) drawing of the molecule; the conformation is defined by the torsion angles.* The phenyl ring is planar, the algebraic sum of the ring torsion angles being 0.08° . The chlorophenyl ring is also nearly planar. The average deviation of the atoms from the least-squares plane is 0.032 \AA with the chlorine included, and 0.027 \AA without it. The dihedral angle between the

planes of the two phenyl rings is $-150.3(2)^\circ$ and that between the chlorophenyl ring and the heterocyclic ring is $53.3(4)^\circ$. S(2) and the exocyclic atoms bonded to it show a significant departure from the plane. The oxathiazine ring, containing two double bonds, exhibits a distorted half-chair conformation. There are no intermolecular contacts shorter than the sums of the van der Waals radii. The crystal structure therefore contains discrete molecules.

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* Lists of structure factors, anisotropic thermal parameters, bond angles involving H atoms and torsion angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39242 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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9,10-Dihydro-9,10-epidioxyanthracene, $\text{C}_{14}\text{H}_{10}\text{O}_2$

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Abstract. $M_r = 210.23$, monoclinic, $P2_1/a$, $a = 15.944(10)$, $b = 5.863(10)$, $c = 11.432(10) \text{ \AA}$, $\beta = 108.2(2)^\circ$, $V = 1015.20 \text{ \AA}^3$, $Z = 4$, $D_m = 1.356(4)$, $D_x = 1.375 \text{ Mg m}^{-3}$, $\text{Cu K}\alpha$, $\lambda = 1.5418 \text{ \AA}$, $\mu = 0.75 \text{ mm}^{-1}$, $F(000) = 440$, $T = 293 \text{ K}$, $R = 0.062$ for 798 observed reflexions [$I > 3\sigma(I)$]. Molecules in the crystal are discrete and held together by van der Waals forces. The intramolecular dimensions are normal for anthraquinone-type compounds. The two benzo rings make a dihedral angle of $52.66(5)^\circ$.

Introduction. A dilute solution of anthracene in carbon disulphide forms a peroxide when irradiated with ultra-violet light; crystals of the title compound are deposited on evaporation of the solvent (Dufraisse & Gérard, 1935). These crystals are stable over long periods in a corked phial in a refrigerator or in a cool room, but decompose in a few hours at elevated temperature or in an X-ray beam forming a mixture of anthrone and anthraquinone (Lonsdale, Nave & Stephens, 1966). The decomposition is explosive at the