

Fig. 1. Perspective view of the molecule.

Calculations were performed on a PC/AT computer. The structure is defined in Table 1 and Fig. 1.\*

\* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54261 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

**Related literature.** Structure of leucosine hydrobromide and isoboldine hydrobromide: Brown & Hall (1977); structure of bulbocapnine methiodide: Wei, Basu, Einstein & Hingerty (1984); structure of bulbocapnine: Ribár, Mészáros, Gašić, Kanyó & Engel (1991).

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## Structure of 1H-3,4-Dihydro-2,3-benzothiazine 2,2-Dioxide

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**Abstract.**  $C_8H_9NO_2S$ ,  $M_r = 183.23$ , monoclinic,  $P2_1/c$ ,  $a = 9.492$  (3),  $b = 10.443$  (2),  $c = 9.348$  (3) Å,  $\beta = 119.04$  (5)°,  $V = 810$  (1) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.502$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71073$  Å,  $\mu =$

$0.337$  mm<sup>-1</sup>,  $F(000) = 384$ ,  $T = 293$  K,  $R = 0.036$  for 894 observed reflections. A distorted tetrahedral geometry is observed about sulfur, with an O—S—O angle of 118.9 (1)° and a dihedral angle between planes N—S—C and O—S—O of 90.4 (1)°. The S—N distances [1.603 (4) Å] are shorter than those previously found in related compounds. The N atom is in a distorted pyramidal conformation, with the H atom located almost perpendicular to the heterocyclic ring. Molecules related by  $\bar{x} + 1$ ,  $\bar{y} + 1$ ,  $\bar{z}$  form hydrogen-bonded dimers through a pair of N—H⋯O type hydrogen bonds [N⋯O 3.033 (5), H⋯O

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2.20 (4) Å and N—H...O angle of 178. (3)°]. No structures have previously been reported for the 2,3-benzothiazine system.

**Experimental.** The synthesis of the title compound has been reported previously (Orazi, Corral & Bravo, 1986). The crystal obtained by slow cooling (330–300 K) in ethanol and used for data collection was 0.1 × 0.3 × 0.4 mm. Intensities measured with Nonius CAD-4 diffractometer,  $\omega$ -2 $\theta$  scans with scan width (0.80 + 0.35 tan  $\theta$ )° and scan speed of 4.00–6.67° min<sup>-1</sup>; graphite-monochromated Mo  $K\alpha$  radiation. Unit-cell parameters determined from least squares on setting angles of 24 reflections with 8.6 < 2 $\theta$  < 20.2°;  $hkl$  range: -1 <  $h$  < 9, 0 <  $k$  < 15, -12 <  $l$  < 12,  $\theta_{\max}$  = 23°. One standard reflection (140) varied  $\pm 2.2\%$  of mean intensity over data collection; 1344 reflections measured, 1057 unique,  $R_{\text{int}}$  = 0.0225, 894 observed on the criterion  $I > 2\sigma(I)$ ; Lorentz-polarization and geometrical absorption corrections were applied (average transmission factors 0.95, with max. 0.97 and min. 0.90 for  $\bar{1}2\bar{1}$  and 622 reflections respectively). Structure solved by (centrosymmetric) direct methods and refined by least squares. After a full-matrix least-squares refinement (on  $F$ ), with all non-H atoms anisotropic, the H atoms were included at positions based on those found in difference synthesis and refined with three common isotropic thermal parameters for CH<sub>2</sub>, fused benzene and the nitrogen H atoms respectively. Function minimized  $\sum[w(|F_o| - |F_c|)^2]$  with  $w = [\sigma^2(F_o) + 0.001F_o^2]^{-1}$ , for the 140 parameters refined. Refinement converged to a final  $R = 0.036$  and  $wR = 0.04$ ; inspection of  $F_c$  and  $F_o$  values indicated a correction for secondary extinction was required:  $F_{c,\text{corr}} = F_c / |1.0 - 10^{-4}\chi F_c^2 / \sin(\theta)|$ ,  $\chi = 0.0258$  in the final run. Final  $(\Delta/\sigma)_{\max} = 0.002$ ,  $\Delta\rho_{\min} = -0.25$  and  $\Delta\rho_{\max} = 0.20$  e Å<sup>-3</sup> on final difference map. Atomic 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). Most of the calculations were performed on a MicroVAX II computer at the Departamento de Física de la Universidad Nacional de La Plata. Programs used: *SHELX76* (Sheldrick, 1976), *SHELXS86* (Sheldrick, 1985) and *ORTEP* (Johnson, 1965). Fractional atomic parameters with equivalent isotropic thermal parameters for the non-H atoms are given in Table 1.\* The numbering scheme is shown in Fig. 1. Fig. 2

\* Lists of structure factors, anisotropic thermal parameters, bond lengths and angles involving H atoms, torsion angles, least-squares planes, and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54362 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional positional parameters for non-H atoms and isotropic thermal parameters with e.s.d.'s in parentheses

	x	y	z	$B_{\text{eq}}$ * (Å <sup>2</sup> )
C(1)	0.3777 (4)	0.4721 (3)	0.2598 (4)	3.1 (1)
S(2)	0.5072 (1)	0.5807 (1)	0.2350 (1)	3.25 (3)
O(1)	0.6264 (3)	0.5092 (2)	0.2151 (3)	4.74 (8)
O(2)	0.5598 (3)	0.6740 (2)	0.3625 (2)	4.21 (7)
N(3)	0.3911 (3)	0.6498 (3)	0.0646 (4)	3.68 (9)
C(4)	0.2482 (4)	0.7128 (4)	0.0500 (4)	3.4 (1)
C(5)	0.0087 (4)	0.6885 (3)	0.0907 (4)	3.2 (1)
C(6)	-0.0773 (4)	0.6349 (3)	0.1587 (4)	3.4 (1)
C(7)	-0.0127 (4)	0.5328 (3)	0.2641 (4)	3.5 (1)
C(8)	0.1355 (4)	0.4855 (3)	0.2976 (4)	3.1 (1)
C(9)	0.2215 (3)	0.5369 (3)	0.2261 (3)	2.47 (8)
C(10)	0.1586 (3)	0.6418 (3)	0.1235 (3)	2.59 (8)

\* Defined according to Hamilton (1959).

Table 2. Bond distances (Å) and bond angles (°) with e.s.d.'s in parentheses

C(1)—S(2)	1.768 (5)	C(1)—C(9)	1.517 (5)
S(2)—O(1)	1.442 (3)	S(2)—O(2)	1.429 (2)
S(2)—N(3)	1.603 (4)	N(3)—C(4)	1.454 (5)
C(4)—C(10)	1.521 (6)	C(5)—C(6)	1.374 (6)
C(5)—C(10)	1.390 (5)	C(6)—C(7)	1.377 (4)
C(7)—C(8)	1.376 (5)	C(8)—C(9)	1.390 (6)
C(9)—C(10)	1.386 (5)		
S(2)—C(1)—C(9)	111.0 (2)	S(2)—N(3)—C(4)	117.2 (4)
C(1)—C(9)—C(10)	123.1 (3)	C(1)—S(2)—O(1)	108.9 (2)
O(1)—S(2)—O(2)	118.9 (1)	N(3)—C(4)—C(10)	116.0 (3)
C(8)—C(9)—C(10)	118.8 (3)	C(1)—S(2)—O(2)	108.2 (2)
C(6)—C(5)—C(10)	122.0 (3)	C(4)—C(10)—C(5)	117.8 (3)
C(1)—S(2)—N(3)	103.5 (2)	C(5)—C(6)—C(7)	119.2 (3)
C(4)—C(10)—C(9)	123.5 (3)	C(6)—C(7)—C(8)	119.4 (4)
C(5)—C(10)—C(9)	118.7 (3)	O(1)—S(2)—N(3)	108.1 (2)
C(7)—C(8)—C(9)	121.8 (3)	O(2)—S(2)—N(3)	108.4 (2)
C(1)—C(9)—C(8)	118.1 (3)		

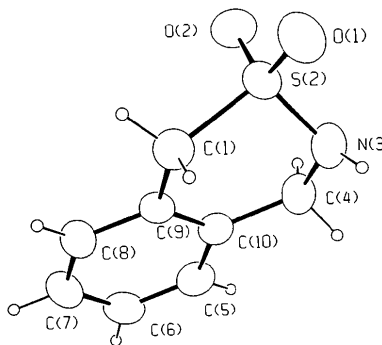


Fig. 1. Perspective drawing of title compound indicating atom labeling. Thermal ellipsoids are drawn at the 50% probability level.

shows a stereoscopic view of the unit cell. Table 2 shows bond lengths and angles within the molecule. The S—N distance is shorter than that previously found in other related compounds (mean value 1.625 Å):  $\beta$ -sulfanilamide (O'Connell & Maslen, 1967), thiazine ring without fused benzene as sulthiame (Aupers, Carlisle & Lindley, 1974; Camerman & Camerman, 1975) and the 1,2-benzothiazines (Kojić-Prodić & Ružić-Toroš, 1982; Norris, Berke,

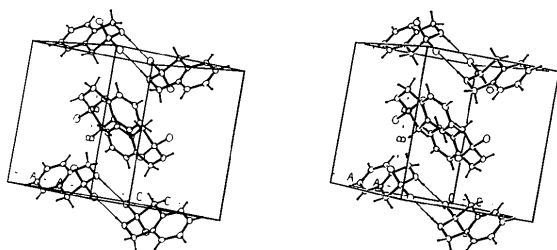


Fig. 2. A stereoscopic view of the unit cell. Hydrogen bonding in the dimers is shown by thin lines.

Bordner & Lombardino, 1985; Golic & Leban, 1987). The pyramidal coordination around the N is distorted due to intermolecular contact, with angles C—N—H 120 (3), S—N—C 117.2 (4) and S—N—H 110 (2)°. The H atom belonging to the NH group is located almost perpendicular to the heterocyclic ring; the C(1)—S(2)—N(3)—H(31) and C(10)—C(4)—N(3)—H(31) torsion angles are  $-85$  (3) and  $97$  (3)°, respectively. The heterocyclic thiazine ring adopts a half-chair conformation.

**Related literature.** A search in the June 1990 edition of the Cambridge Structural Database (Allen, Kennard & Taylor, 1983) showed that no other 2,3-benzothiazines have been structurally characterized until now. However, 1,2-benzothiazines are relatively common.

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## 4-Oxo-5-phenyl-2,3-dihydro-3a-azaphenalen-6-yl Benzenesulfonate

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**Abstract.**  $C_{24}H_{19}NO_4S$ ,  $M_r = 417.5$ , monoclinic,  $P2_1/n$ ,  $a = 10.5374$  (4),  $b = 12.9206$  (6),  $c = 14.6462$  (6) Å,  $\beta = 98.863$  (5)°,  $V = 1970.3$  Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.407$  g cm<sup>-3</sup>,  $\lambda(Mo K\alpha) = 0.71073$  Å,  $\mu = 1.9$  cm<sup>-1</sup>,  $F(000) = 872$ ,  $T = 295$  K, final  $R = 0.0515$  for 2794 observed reflections with  $F > 4\sigma_c(F)$  ( $\sigma_c$  from counting statistics only). The ring system is puckered, owing to the presence of the methylene-C

atoms, and has C—C distances ranging from 1.380 (4) to 1.518 (3) Å. The trigonal N atom forms two essentially equivalent C—N distances [mean 1.391 (3) Å], with a third to a neighbouring methylene-C atom at 1.488 (3) Å. The exocyclic ketonic carbonyl distance is 1.225 (2) Å and the sulfonate-S atom forms two essentially equivalent terminal S—O distances [mean 1.428 (2) Å], while