Mn—O bond length in the present structure is 1.610 (6) Å which is closer to the reported values for the potassium (Palenik, 1967) and caesium (Prout & Nassimbeni, 1966) salts [1.607 (5) (without libration correction) and 1.63 Å, respectively].

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Structure of 3-Methyl-3,4-dihydro-1H-2,3-benzothiazine 2,2-Dioxide

By Blas E. Rivero* and Mario A. Bianchet†

Departamento de Física, Facultad de Ciencias Exactas, Universidad Nacional de La Plata, 47 y 115, CC 67 (1900) La Plata, Argentina

and Rodolfo D. Bravo‡

Departamento de Ciencias Básicas, Universidad Nacional de Lujan, rutas 5 y 7, (6700) Lujan, Argentina

Abstract. $C_9H_{11}NO_2S$, $M_r = 197.26$, triclinic, $P\bar{1}$, $a = 9.355$ (4), $b = 10.001$ (3), $c = 10.787$ (5) Å, $\alpha = 86.93$ (3), $\beta = 88.88$ (3), $\gamma = 69.56$ (3)°, $V = 944$ (1) Å^3, $Z = 4$, $D_x = 1.387$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 0.294$ mm^{-1}, $F(000) = 416$, $T = 293$ K, $R = 0.045$ for 2057 observed reflections. There are two crystallographically independent molecules per asymmetric unit and the heterocyclic ring in both takes a twisted half-chair conformation. The S atoms are tetrahedrally coordinated with a dihedral angle between the C—S—N and O—S—O planes of 89.51 (14)°. The methyl groups are almost axial with torsional angles C(1A,B)—S(2A,B)—N(3A,B)—C(11A,B) of 70.73 (31) and 74.52 (35)°, respectively.

Experimental. The synthesis of the title compound has been reported previously (Orazi, Corral & Bravo, 1986). Crystals obtained by slow cooling (330–300 K) of a solution in ethanol. The crystal used for

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* Member of Carrera del Investigador Científico, CICPBA, Argentina.

† Present address: Department of Biophysics and Biophysical Chemistry, The Johns Hopkins University, School of Medicine, 725 N. Wolfe St, Baltimore, MD 21205, USA.

‡ Present address: Departamento de Química, Facultad de Ciencias Exactas, Universidad Nacional de La Plata, 47 y 115, (1900) La Plata, Argentina.

data collection had dimensions 0.1 × 0.3 × 0.5 mm. Intensities measured with a Nonius CAD-4 diffractometer; ω–2θ scanning mode, with scan width $(0.80 + 0.35\tan\theta)°$ and scan speed of 2.50–6.67° min^{-1}; graphite-monochromated Mo $K\alpha$ radiation. Unit-cell parameters determined from least squares on setting angles of 24 reflections with $8.5 < 2\theta < 19°$; range of hkl: $-1 < h < 11$, $-10 < k < 11$, $-12 < l < 12$, $\theta_{max} = 25°$. One standard reflection ($\bar{1}07$) varied $\pm 2.1\%$ of mean intensity over data collection; 3965 reflections measured, 2945 unique, $R_{int} = 0.018$, 2057 considered observed on the criterion $I > 3\sigma(I)$; Lorentz–polarization and geometrical absorption corrections were applied (average transmission factor 0.95, with maximum 0.97 and minimum 0.92 for 120 and $\bar{1},0,12$ reflections, respectively). Structure solved by centrosymmetric direct methods and refined by least squares. After a full-matrix least-squares refinement, with all non-H atoms anisotropic, the H atoms in the heterocyclic ring and the benzene ring were included at positions based on those found in a difference synthesis; refined in the final run with two common isotropic thermal parameters for CH_2 and fused-benzene H atoms. The methyl-group H atoms in both molecules were geometrically placed (C—H 1.08 Å); the methyl group was treated as a rigid group with a common isotropic factor for its H atoms. Function minimized $\sum[w(|F_o| - |F_c|)^2]$ with

Table 1. *Fractional positional parameters and equivalent isotropic thermal parameters* (Å^2) *for* non-H *atoms with e.s.d.'s in parentheses*

$$B_{eq} = (4/3)\Sigma_i\Sigma_j B_{ij} \mathbf{a}_i.\mathbf{a}_j \text{ (Hamilton, 1959)}.$$

	x	y	z	B_{eq}
C(1A)	0·1863 (4)	0·1926 (4)	0·2458 (3)	3·8 (2)
S(2A)	0·05912 (10)	0·2164 (1)	0·1216 (1)	3·79 (4)
O(1A)	−0·0180 (3)	0·1160 (3)	0·1350 (3)	5·8 (2)
O(2A)	−0·0290 (3)	0·3654 (3)	0·1066 (2)	5·0 (1)
N(3A)	0·1723 (3)	0·1747 (3)	0·0015 (3)	3·8 (1)
C(4A)	0·2596 (5)	0·2721 (4)	−0·0169 (4)	4·3 (2)
C(5A)	0·4780 (5)	0·3221 (4)	0·0756 (5)	5·2 (2)
C(6A)	0·5624 (6)	0·3375 (5)	0·1732 (6)	6·5 (3)
C(7A)	0·5207 (6)	0·3117 (5)	0·2931 (6)	6·2 (3)
C(8A)	0·3973 (5)	0·2689 (4)	0·3147 (4)	4·9 (2)
C(9A)	0·3131 (4)	0·2499 (3)	0·2156 (3)	3·4 (1)
C(10A)	0·3526 (4)	0·2792 (3)	0·0934 (3)	3·6 (2)
C(11A)	0·2651 (5)	0·0224 (4)	−0·0064 (4)	4·9 (2)
C(1B)	−0·0867 (4)	0·9028 (4)	0·3586 (4)	4·1 (2)
S(2B)	0·0952 (1)	0·79309 (9)	0·41000 (9)	3·86 (4)
O(1B)	0·2000 (3)	0·8674 (3)	0·3884 (3)	5·8 (2)
O(2B)	0·0819 (3)	0·7381 (3)	0·5326 (2)	5·2 (1)
N(3B)	0·1375 (3)	0·6607 (3)	0·3190 (3)	3·8 (1)
C(4B)	0·0305 (4)	0·5820 (4)	0·3335 (4)	4·4 (2)
C(5B)	−0·2425 (5)	0·6016 (4)	0·3338 (4)	4·4 (2)
C(6B)	−0·3972 (5)	0·6755 (5)	0·3401 (4)	5·0 (2)
C(7B)	−0·4491 (5)	0·8200 (5)	0·3497 (4)	5·2 (2)
C(8B)	−0·3469 (4)	0·8904 (4)	0·3564 (4)	4·3 (2)
C(9B)	−0·1909 (4)	0·8184 (3)	0·3504 (3)	3·1 (1)
C(10B)	−0·1376 (4)	0·6709 (3)	0·3378 (3)	3·2 (1)
C(11B)	0·1794 (6)	0·6875 (5)	0·1909 (4)	5·9 (2)

$w = [\sigma^2(F_o) + 0.002F_o{}^2]^{-1}$, for a total of 293 parameters refined. Refinement converged to a final $R = 0.045$ and $wR = 0.048$; inspection of F_c and F_o values indicated a correction for secondary extinction was necessary, $F_{corr} = F_c/|1.0 - 10^{-4}\chi F_c{}^2/\sin\theta|$, $\chi = 0.0199$ in the final run. Final $\Delta/\sigma = 0.03$, $\Delta\rho_{min} = -0.28$ and $\Delta\rho_{max} = 0.22$ e Å^{-3} 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 (Departamento de Física de la Universidad Nacional de La Plata). Programs used: *SHELX*76 (Sheldrick, 1976), *SHELXS*86 (Sheldrick, 1985) and *ORTEP* (Johnson, 1965). Fractional atomic parameters and equivalent isotropic thermal parameters for the non-H atoms are given in Table 1.* The atom-numbering scheme is shown in Fig. 1. Table 2 gives bond lengths and angles within the molecule.

Related literature. The average S—C(sp^3) and S—N(sp^2) bond distances are 1·757 (7) and 1·634 (6) Å respectively. The S—N and S—C distances and geometry are in reasonable agreement with those

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, bond distances and angles involving H atoms, least-squares-planes data, and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54383 (24 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

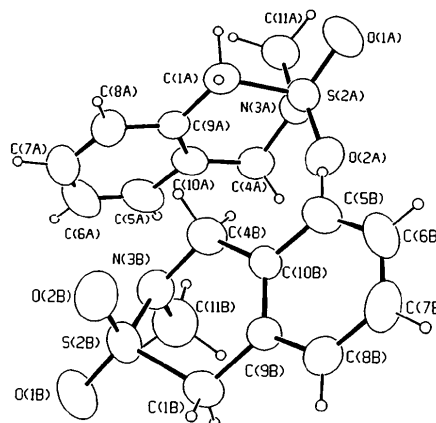|				
C(1A)—S(2A)	1·761 (4)	C(1B)—S(2B)	1·753 (3)
C(1A)—C(9A)	1·510 (6)	C(1B)—C(9B)	1·502 (7)
S(2A)—O(1A)	1·427 (4)	S(2B)—O(1B)	1·431 (4)
S(2A)—O(2A)	1·431 (3)	S(2B)—O(2B)	1·425 (3)
S(2A)—N(3A)	1·634 (3)	S(2B)—N(3B)	1·622 (3)
N(3A)—C(4A)	1·478 (7)	N(3B)—C(4B)	1·475 (6)
N(3A)—C(11A)	1·473 (4)	N(3B)—C(11B)	1·465 (6)
C(4A)—C(10A)	1·507 (6)	C(4B)—C(10B)	1·514 (5)
C(10A)—C(5A)	1·390 (6)	C(10B)—C(5B)	1·388 (6)
C(5A)—C(6A)	1·375 (8)	C(5B)—C(6B)	1·378 (6)
C(6A)—C(7A)	1·381 (9)	C(6B)—C(7B)	1·363 (7)
C(7A)—C(8A)	1·376 (8)	C(7B)—C(8B)	1·376 (6)
C(8A)—C(9A)	1·399 (6)	C(8B)—C(9B)	1·386 (5)
C(9A)—C(10A)	1·405 (5)	C(9B)—C(10B)	1·396 (5)
S(2A)—C(1A)—C(9A)	113·2 (2)	S(2B)—C(1B)—C(9B)	111·0 (2)
C(1A)—S(2A)—C(10A)	122·9 (3)	C(1B)—C(9B)—C(10B)	123·0 (4)
O(1A)—S(2A)—O(2A)	119·0 (2)	O(1B)—S(2B)—O(2B)	118·5 (2)
O(1A)—S(2A)—C(1A)	110·3 (2)	O(1B)—S(2B)—C(1B)	109·3 (2)
O(1A)—S(2A)—N(3A)	107·8 (2)	O(1B)—S(2B)—N(3B)	109·1 (2)
O(2A)—S(2A)—C(1A)	108·1 (2)	O(2B)—S(2B)—C(1B)	108·5 (2)
O(2A)—S(2A)—N(3A)	107·3 (2)	O(2B)—S(2B)—N(3B)	107·4 (2)
S(2A)—N(3A)—C(4A)	111·2 (3)	S(2B)—N(3B)—C(4B)	112·0 (2)
S(2A)—N(3A)—C(11A)	116·1 (3)	S(2B)—N(3B)—C(11B)	117·0 (3)
C(8A)—C(9A)—C(1A)	117·6 (3)	C(8B)—C(9B)—C(1B)	118·4 (3)
C(10A)—C(9A)—C(8A)	119·4 (4)	C(10B)—C(9B)—C(8B)	118·7 (4)
C(10A)—C(5A)—C(6A)	122·2 (5)	C(10B)—C(5B)—C(6B)	121·5 (4)
N(3A)—S(2A)—C(1A)	103·1 (2)	N(3B)—S(2B)—C(1B)	102·9 (2)
N(3A)—C(4A)—C(10A)	114·9 (3)	N(3B)—C(4B)—C(10B)	116·7 (3)
C(4A)—N(3A)—C(11A)	114·1 (3)	C(4B)—N(3B)—C(11B)	115·5 (4)
C(4A)—C(10A)—C(9A)	122·2 (4)	C(4B)—C(10B)—C(9B)	122·7 (4)
C(4A)—C(10A)—C(5A)	119·6 (4)	C(4B)—C(10B)—C(5B)	118·4 (3)
C(5A)—C(6A)—C(7A)	119·3 (6)	C(7B)—C(6B)—C(5B)	119·6 (5)
C(5A)—C(10A)—C(9A)	118·2 (4)	C(9B)—C(10B)—C(5B)	118·8 (3)
C(6A)—C(7A)—C(8A)	120·4 (6)	C(8B)—C(7B)—C(7B)	119·9 (4)
C(7A)—C(8A)—C(9A)	120·5 (4)	C(9B)—C(8B)—C(7B)	121·6 (4)

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

reported for related compounds: β-sulfanilamide (O'Connell & Maslen, 1967); thiazine ring without fused benzene, sulthiame (Aupers, Carlisle & Lindley, 1974; Camerman & Camerman, 1975); and 1,2-benzothiazines (Kojić-Prodić & Ružić-Toroš, 1982; Golič & Leban, 1987). The bond lengths and angles of the title compound are in good agreement with those of other 2,3-benzothiazines; the S—N bond distance is slightly larger (~1·5%) than the S—N distance [1·603 (4) Å] reported for 3,4-dihydro-1H-2,3-benzothiazine 2,2-dioxide (Rivero, Bianchet & Bravo, 1991).

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Structure of 6,13-Bis(butylthio)-5,12-dioxa-6,13-dithioxo-6a,7,13a,14-tetraaza-6,13-diphosphadibenz[a,h]anthracene

BY JIN-LING WANG,* MING SUN AND FANG MING MIAO

Department of Chemistry, Tianjin Normal University, Tianjin, People's Republic of China

AND QI-JIE CHEN AND SHU-JUAN JIN

Institute of Elemento-Organic Chemistry, Nankai University, Tianjin, People's Republic of China

Abstract. $C_{22}H_{26}N_4O_2P_2S_4$, $M_r = 568.66$, monoclinic, $A2/a$ (non-standard setting), $a = 14.941$ (5), $b = 7.050$ (1), $c = 24.832$ (4) Å, $\beta = 90.48$ (2)°, $V = 2616$ (2) Å^3, $Z = 4$, $D_x = 1.444$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 4.963$ cm^{-1}, $F(000) = 1184$, room temperature, $R = 0.039$, $wR = 0.044$ for 1017 observed reflections with $I > 3\sigma(I)$. The molecule is centrosymmetric. The six-membered oxazaphosphorinan ring has a chair conformation, the P=S double bond is equatorial and the P—S single bond is axial. The dihedral angle between the oxazaphosphorinan ring and the phenyl ring is 15.0°.

Experimental. The title compound was synthesized as indicated in *Related literature*. Crystals were obtained by slow evaporation from a trichloromethane solution. A crystal with dimensions 0.2 × 0.2 × 0.3 mm was mounted on a glass fibre. Accurate cell parameters were obtained from centred setting angles of 25 reflections in the range 10 < θ < 19°. An

Enraf–Nonius CAD-4 diffractometer with graphite-monochromated Mo $K\alpha$ radiation ($\lambda = 0.71073$ Å) was used. Space group $A2/a$ with the b axis unique is a non-standard setting with equivalent positions $[(0, 0, 0; 0, \frac{1}{2}, \frac{1}{2}) + (x, y, z; -x, -y, -z; \frac{1}{2} - x, y, -z; \frac{1}{2} + x, y, -z)]$. Diffraction intensities in the range 2 < θ < 25° were measured using the ω–2θ scan mode, index range h: −16→16, k: 0→8, l: 0→26. Deviations of three standard reflections, measured after each group of 200 reflections, was less than 3.1%. 2304 unique reflections were collected, of which 1017 were considered observed with $I > 3\sigma(I)$. The intensities were corrected for Lorentz, polarization and absorption effects (with transmission coefficients in the range 0.82–0.97). The structure was solved by direct methods (*MULTAN*78; Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) and the positions of all the H atoms were found in a $\Delta\rho$ map at $R = 0.064$. The structure was refined by full-matrix least-squares calculations on F, using unit weights, anisotropic temperature factors for non-H atoms and isotropic temperature factors

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