

Intermolecular van der Waals interactions may prove to be important in the binding of an antagonist to the PAF-membrane receptor site. Additional investigations are now in progress in our laboratory to determine the utility of this X-ray crystallographic approach to the design of potent and specific antagonists and a drug-receptor site binding model.

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## Structure of ( $\pm$ )-3-[1-Hydroxy-1-(4-methylphenyl)ethyl]-6-phenyl-1,2,4,5-tetrazine

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**Abstract.**  $C_{17}H_{16}N_4O$ ,  $M_r = 292.34$ , monoclinic,  $C2/c$ ,  $a = 22.827$  (7),  $b = 6.004$  (4),  $c = 23.276$  (5) Å,  $\beta = 108.16$  (3)°,  $V = 3031.3$  Å<sup>3</sup>,  $Z = 8$ ,  $D_x = 1.28$  g cm<sup>-3</sup>,

$\lambda(\text{Mo } K\alpha) = 0.71073$  Å,  $\mu = 0.8$  cm<sup>-1</sup>,  $F(000) = 1232$ ,  $T = 294$  K,  $R = 0.063$  for 879 unique observed reflections. Bond lengths and angles lie within expected

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Table 1. Coordinates ( $\times 10^4$ ) and equivalent isotropic temperature factors ( $\text{\AA}^2 \times 10^3$ ) for non-H atoms with e.s.d.'s in parentheses

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	$U_{eq}$
C3	9398 (3)	64 (10)	5798 (2)	42 (2)
N2	8841 (2)	-508 (10)	5805 (2)	68 (2)
N1	8556 (2)	-2157 (10)	5437 (3)	70 (2)
C6	8833 (3)	-3066 (11)	5069 (2)	47 (2)
N5	9398 (2)	-2404 (8)	5050 (2)	45 (1)
N4	9679 (2)	-842 (8)	5423 (2)	48 (1)
C8A	9752 (3)	1879 (10)	6203 (2)	46 (2)
C9A	9358 (3)	2991 (10)	6544 (3)	59 (2)
O1A	9898 (2)	3529 (6)	5834 (2)	55 (1)
C1A	10334 (3)	918 (11)	6653 (2)	45 (2)
C2A	10340 (3)	-1175 (12)	6910 (3)	59 (2)
C3A	10859 (4)	-1967 (14)	7323 (3)	75 (3)
C4A	11407 (3)	-767 (17)	7504 (3)	74 (3)
C7A	11967 (4)	-1773 (17)	7964 (4)	118 (4)
C5A	11405 (3)	1333 (15)	7257 (3)	71 (3)
C6A	10870 (3)	2132 (12)	6829 (3)	58 (2)
C1B	8532 (2)	-4946 (11)	4692 (2)	44 (2)
C2B	7904 (3)	-5307 (13)	4582 (3)	73 (3)
C3B	7624 (3)	-7103 (15)	4239 (3)	89 (3)
C4B	7953 (4)	-8468 (14)	3989 (3)	81 (3)
C5B	8582 (3)	-8116 (12)	4091 (3)	67 (2)
C6B	8853 (3)	-6326 (11)	4432 (3)	55 (2)

Table 2. Interatomic distances ( $\text{\AA}$ ) and angles ( $^\circ$ )

N2-C3	1.322 (7)	N4-C3	1.349 (6)
C8A-C3	1.502 (8)	N1-N2	1.338 (7)
C6-N1	1.329 (7)	N5-C6	1.363 (6)
C1B-C6	1.464 (8)	N4-N5	1.303 (6)
C9A-C8A	1.525 (8)	O1A-C8A	1.417 (6)
C1A-C8A	1.527 (8)	C2A-C1A	1.389 (8)
C6A-C1A	1.372 (8)	C3A-C2A	1.358 (9)
C4A-C3A	1.391 (10)	C7A-C4A	1.514 (10)
C5A-C4A	1.385 (9)	C6A-C5A	1.397 (9)
C2B-C1B	1.393 (8)	C6B-C1B	1.366 (7)
C3B-C2B	1.375 (9)	C4B-C3B	1.360 (9)
C5B-C4B	1.396 (9)	C6B-C5B	1.364 (8)
N4-C3-N2	123.8 (6)	C8A-C3-N2	120.8 (5)
C8A-C3-N4	115.3 (5)	N1-N2-C3	117.5 (5)
C6-N1-N2	118.8 (5)	N5-C6-N1	123.1 (6)
C1B-C6-N1	118.3 (6)	C1B-C6-N5	118.6 (5)
N4-N5-C6	117.6 (4)	N5-N4-C3	119.1 (5)
C9A-C8A-C3	110.8 (5)	O1A-C8A-C3	108.0 (4)
O1A-C8A-C9A	107.3 (5)	C1A-C8A-C3	109.8 (5)
C1A-C8A-C9A	109.7 (5)	C1A-C8A-O1A	111.1 (5)
C2A-C1A-C8A	121.8 (6)	C6A-C1A-C8A	120.6 (6)
C6A-C1A-C2A	117.6 (6)	C3A-C2A-C1A	120.7 (6)
C4A-C3A-C2A	122.4 (7)	C7A-C4A-C3A	119.3 (9)
C5A-C4A-C3A	117.3 (7)	C5A-C4A-C7A	123.4 (9)
C6A-C5A-C4A	119.9 (7)	C5A-C6A-C1A	121.9 (7)
C2B-C1B-C6	119.6 (6)	C6B-C1B-C6	121.1 (5)
C6B-C1B-C2B	119.2 (6)	C3B-C2B-C1B	119.6 (6)
C4B-C3B-C2B	120.1 (7)	C5B-C4B-C3B	120.8 (7)
C6B-C5B-C4B	118.3 (7)	C5B-C6B-C1B	121.8 (6)

ranges. The tetrazine and phenyl rings are planar. The structure contains one intermolecular hydrogen bond, O1A...N5 ( $x, 1+y, z$ ) 3.06 (1)  $\text{\AA}$ .

**Introduction.** 3,6-Disubstituted-1,2,4,5-tetrazines are commonly symmetrically substituted because it is difficult to prepare species with different substituents in the 3- and 6-positions. The title compound  $\{(\pm)\text{-}3\text{-}[1\text{-hydroxy-1-(4-methylphenyl)ethyl}]\text{-6-phenyl-1,2,4,5-tetrazine}\}$  (Bowie, Gardner, Neilson, Watson, Mahmood & Ridd, 1972), in addition to being unsymmetrically substituted, has an asymmetric centre on the 3-substituent and was prepared as part of our studies on 1,2,4,5-tetrazines and their rearrangements (Mahmood, 1973).

**Experimental.** The compound was synthesized by the action of hydrazine hydrate on a mixture of benzamidine and  $(\pm)\text{-}2\text{-hydroxy-2-}p\text{-tolylpropionamidine hydrochloride}$ . It crystallized as intensely red plates from methanol, m.p. 395–397 K. Cell constants and an orientation matrix for data collection determined on a CAD-4 diffractometer by a least-squares treatment of the setting angles of 25 reflections in the range  $4 < \theta < 19^\circ$  (Frenz, 1978). Crystal dimensions 0.12  $\times$  0.20  $\times$  0.33 mm; intensities of reflections with indices  $h$  0 to 21,  $k$  -7 to 0,  $l$  -22 to 21 with  $2 < 2\theta < 46^\circ$  measured;  $\omega$ - $2\theta$  scans; graphite-monochromatized Mo  $K\alpha$  radiation; intensities of three reflections chosen as standards monitored every 2 h and showed no evidence of crystal decay; 1399 reflections measured, 1203 unique; 879 had  $F > 6\sigma(F)$  and used in the structure solution and refinement. Data corrected for

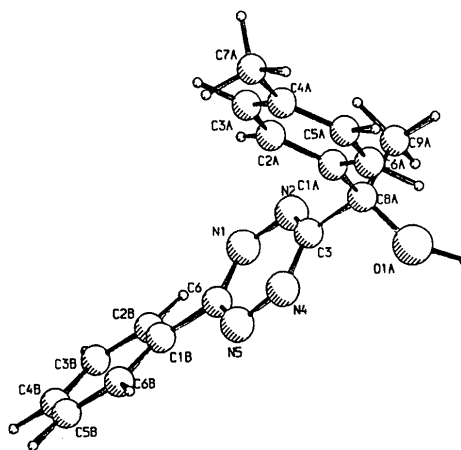


Fig. 1. View of the molecule showing atomic numbering.

Lorentz and polarization effects but not for absorption. Phase problem solved using *SHELXS86* (Sheldrick, 1986). An *E* map revealed all non-H atoms. Refinement (on *F*) by blocked-matrix least squares with *SHELX76* (Sheldrick, 1976). Non-H atoms refined anisotropically; H atoms attached to C atoms included at calculated positions (C-H 1.08  $\text{\AA}$ ); the hydroxyl H atom was located from a difference synthesis; H atoms were given isotropic temperature factors 1.5 times those of their parent atoms. 199 parameters refined,  $R = 0.063$ ,  $wR = 0.082$ ,  $w = 1.00/[\sigma(F)^2 + 0.004192F^2]$ .  $\Delta/\sigma < 0.003$ ; max. difference synthesis

peak 0.23, min.  $-0.26 \text{ e } \text{\AA}^{-3}$ . Other programs used: *XANADU* (Roberts & Sheldrick, 1975) and *PLUTO* (Motherwell & Clegg, 1978). Scattering factors from *International Tables for X-ray Crystallography* (1974). No correction for secondary extinction.

**Discussion.** Atomic coordinates are given in Table 1,\* with bond lengths and angles in Table 2. The atom numbering is shown in Fig. 1. The bond lengths and angles show no unusual features. The dimensions of the 3-[1-hydroxy-1-(4-methylphenyl)ethyl]-1,2,4,5-tetrazine moiety in the molecule agree well with those reported for (*R,R*)-(+)-3,6-bis[1-hydroxy-1-(4-methylphenyl)ethyl]-1,2,4,5-tetrazine (Low, Neilson & Scrimgeour, 1986). The phenyl and tetrazine rings are planar within experimental error [max. deviations: phenyl *A* 0.01 (2), phenyl *B* 0.01 (2), tetrazine 0.02 (2) Å]. The angles between the normals to the planes of these rings are 83 (1) and 17 (1)° for tetrazine/phenyl *A* and tetrazine/phenyl *B* respectively. The phenyl *A*/phenyl *B* angle is 67 (1)°. There is one hydrogen bond in the structure,  $\text{O1A} \cdots \text{N5}(x, 1+y, z)$ ,

\* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51794 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

3.06 (1) Å, with O1A—HO1 1.11, HO1...N5 1.99 Å, angle at H = 158°.

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## Structure of 1,3-Dimethylimidazolium-2-dithiocarboxylate

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**Abstract.**  $\text{C}_6\text{H}_8\text{N}_2\text{S}_2$ ,  $M_r = 172.24$ , monoclinic,  $P2_1/n$ ,  $a = 7.117$  (1),  $b = 10.572$  (2),  $c = 10.739$  (3) Å,  $\beta = 99.66$  (1)°,  $Z = 4$ ,  $V = 796.5$  (2) Å<sup>3</sup>,  $D_x(130 \text{ K}) = 1.44 \text{ g cm}^{-3}$ ,  $\text{Mo } K\alpha$ ,  $\lambda = 0.71069$  Å,  $\mu = 5.7 \text{ cm}^{-1}$ ,  $F(000) = 360$ ,  $T = 130 \text{ K}$ ,  $R = 0.029$ , 1201 unique reflections with  $I > 3\sigma(I)$ . The molecule consists of a planar imidazolium ring with the C atoms of the two methyl groups and the dithiocarboxylate group lying within the plane of the ring. The  $\text{CS}_2$  group is nearly perpendicular to the ring, with a torsion angle of 71.7°.

**Introduction.** We report the structure of a heterocyclic dithiocarboxylate zwitterion. Metal complexes of 1,1-

dithiolate ligands have been of interest for a long time. We have recently reported complexes of this zwitterionic dithiocarboxylate ligand with nickel triad metals (Borer, Kong & Sinn, 1986) and other soft acid metals (Borer, Kong, Keihl & Forkey, 1987).

**Experimental.** 1,3-Dimethylimidazolium-2-dithiocarboxylate obtained as prisms from water according to the procedures of Keihl & Borer (1984), crystal dimensions 0.18 × 0.30 × 0.40 mm, Syntex  $P2_1$  diffractometer (UC, Davis), locally modified LT-1 apparatus,  $T = 130 \text{ K}$ ,  $\text{Mo } K\alpha$  radiation, graphite monochromator, cell dimensions from least-squares fit