

Fig. 3. Stereoscopic view of a unit cell, *a* axis projection, with *c* axis vertical and *b* axis horizontal.

hybridization to  $sp^2$ . The angle between the thiazole ring and the adjacent aromatic ring is  $7.2^\circ$ . Also the S–N (1.692 Å) and C–C (1.480 Å) bonds show more single-bond character than in the other two structures. The N–S–N bond angle has a value of  $97.2^\circ$  which is close to those of compounds (i) and (ii) ( $98.6^\circ$  and  $99.4^\circ$ ). The bond lengths and angles in the aromatic ring [C(2)–C(3)–C(4)–C(5)–C(6)–C(7)] are normal. There are four water molecules in the cell and each is triply disordered.

The S → O bond length of  $1.478 \pm 5$  Å is normal for a sulfoxide. There is only one short intermolecular

distance of significant value: N(*x*, *y*, *z*)–O(1) (1 + *x*, *y*, *z*), 2.65 Å.

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### 4-[2-(Methylthio)dibenzo[*b*, *f*]thiepin-11-yl]-1-piperazinylopropanol Hemihydrate (Oxyprothepine)

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**Abstract.** Monoclinic,  $P2_1/c$ ,  $a = 20.648$  (5),  $b = 5.924$  (2),  $c = 17.627$  (5) Å,  $\beta = 93.67$  (1) $^\circ$ , 25 $^\circ$ C,  $C_{22}H_{28}N_2OS_2 \cdot \frac{1}{2}H_2O$ , F.W. = 409.3,  $Z = 4$ .

**Introduction.** This compound is a potent neuroleptic usually known as oxyprothepine. Slow evaporation of a solution in ethanol yielded transparent crystals. Preliminary Weissenberg photographs showed systematic absences ( $h0l$ :  $l = 2n + 1$ ) corresponding to space group  $P2_1/c$  or  $Pc$ . The first was chosen on the basis of the intensity distribution.

The lattice parameters were obtained by the measurement of the Bragg angle of ten reflexions on a Nonius CAD-4 computer-controlled diffractometer. The ex-

perimental conditions used during the measurement of the intensities are given in Table 1.

Table 1. *Experimental conditions*

Source Cu  $K\alpha$ ;  $\lambda = 1.5418$  Å;  $\omega$ - $2\theta$  scan; graphite monochromator;  $\Delta 2\theta = 0.7 + 0.3 \text{ tg } \theta$ ;  $\theta_{\min} = 2^\circ$ ;  $\theta_{\max} = 70^\circ$ ; aperture =  $3.0 + 0.5 \text{ tg } \theta$   
 Confidence level: 2.5  
 Total number of independent reflexions: 3585  
 Total observed: 2400

The structure was solved by direct methods with a modified version of *MULTAN* (Germain, Main & Woolfson, 1971) and refined by block-diagonal least-

Table 2. *Final positional and thermal parameters* ( $\times 10^4$ ) (with standard deviations in parentheses)
$$B = \exp [-(B_{11}h^2 + B_{22}k^2 + B_{33}l^2 + B_{12}hk + B_{13}hl + B_{23}kl)] .$$

	<i>x</i>	<i>y</i>	<i>z</i>	$B_{11}$	$B_{22}$	$B_{33}$	$B_{23}$	$B_{13}$	$B_{12}$
C(1)	1447 (2)	8152 (10)	3228 (3)	14	308	21	-7	-3	23
C(2)	844 (2)	7400 (10)	3447 (3)	16	332	23	-20	-2	0
C(3)	613 (3)	5314 (11)	3200 (3)	18	322	28	7	-1	-2
C(4)	959 (3)	4084 (10)	2695 (3)	22	267	25	13	-2	3
C(5)	1551 (2)	4847 (9)	2458 (3)	20	237	21	16	-1	29
S(6)	1897 (1)	3134 (3)	1755 (1)	37	215	30	14	17	55
C(7)	2009 (3)	5221 (10)	1056 (3)	24	276	21	-3	9	49
C(8)	1652 (3)	5126 (11)	357 (3)	23	353	26	-21	3	47
C(9)	1721 (3)	6851 (12)	-168 (3)	23	387	23	5	2	58
C(10)	2132 (3)	8640 (11)	2 (3)	28	356	23	7	2	54
C(11)	2508 (3)	8701 (11)	694 (3)	25	366	17	20	10	40
C(12)	2450 (2)	6979 (11)	1225 (3)	18	313	20	9	6	26
C(13)	2865 (3)	6951 (12)	1965 (3)	18	443	19	10	4	34
C(14)	2499 (2)	7847 (10)	2646 (3)	14	292	20	25	2	15
C(15)	1821 (2)	6867 (9)	2753 (3)	15	262	18	23	-3	18
N(16)	2878 (2)	7628 (7)	3384 (2)	15	199	20	28	-1	6
C(17)	3499 (2)	8845 (10)	3401 (3)	18	251	27	32	-5	-31
C(18)	3786 (3)	8912 (10)	4227 (3)	21	230	31	5	-7	-14
N(19)	3883 (2)	6611 (8)	4529 (2)	16	259	22	8	-4	2
C(20)	3270 (2)	5344 (10)	4462 (3)	17	287	23	28	-6	-16
C(21)	2996 (3)	5296 (9)	3636 (3)	22	195	25	40	-8	-7
C(22)	4131 (3)	6771 (12)	5334 (3)	22	384	22	-17	-9	22
C(23)	4315 (3)	4447 (12)	5674 (3)	22	417	28	66	-6	5
C(24)	4679 (3)	4678 (17)	6457 (4)	20	781	26	44	-9	-24
O(25)	5275 (2)	5930 (10)	6398 (2)	23	563	33	-52	-9	25
S(26)	420 (1)	9204 (4)	4039 (1)	20	484	47	-140	23	-38
C(27)	-401 (4)	8234 (21)	3930 (7)	22	900	101	-295	41	-20
O(28)	5000 (0)	9377 (30)	7500 (0)						

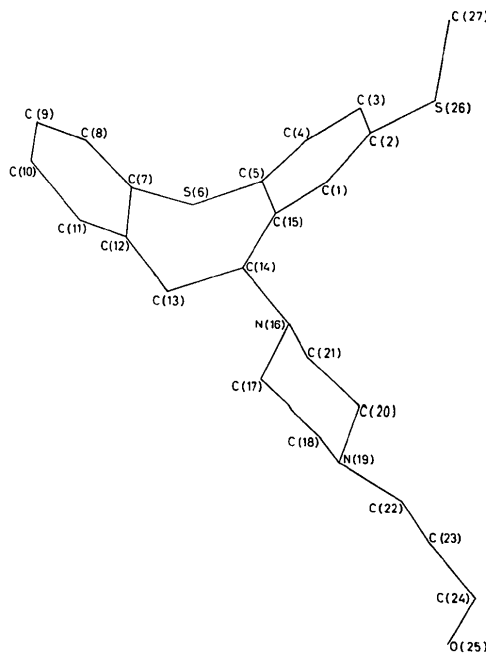
$B = 16.50 \text{ \AA}^2$

Table 2 (cont.) *Coordinates of the hydrogen atoms* ( $\times 10^4$ ) $B = 4.5 \text{ \AA}^2$  for all atoms.

	<i>x</i>	<i>y</i>	<i>z</i>
H(1)	1637	9736	3388
H(3)	188	4697	3330
H(4)	776	2631	2470
H(8)	1348	3859	299
H(9)	1456	6760	-699
H(10)	2169	10053	-373
H(11)	2870	10098	770
H(131)	3281	7941	1912
H(132)	3019	5205	2068
H(14)	2426	9553	2560
H(171)	3382	10499	3208
H(172)	3828	8191	3079
H(181)	4210	9764	4266
H(182)	3489	9999	4531
H(201)	3383	3837	4660
H(202)	2955	6195	4773
H(211)	2590	4521	3636
H(212)	3308	4499	3330
H(221)	3803	7619	5680
H(222)	4549	7804	5317
H(231)	4615	3538	5323
H(232)	3898	3387	5720
H(241)	4834	2980	6672
H(242)	4376	5782	6803
H(25)	5607	5219	6091
H(271)	-440	6568	4199
H(272)	-681	9424	4214
H(273)	-561	8183	3323

squares calculations with the program written by Ahmed, Hall, Pippy & Huber (1966). The positions of the hydrogen atoms were obtained from a difference map, which also contained a strong peak [O(28)] about

five times larger than the best defined hydrogen atom. From its position and distances to the hydroxyl groups of the neighbouring molecules it was assumed to be a water molecule. The positions and temperature factors of the hydrogen atoms were not refined.

Fig. 1. Conformation and atom numbering scheme of  $C_{22}H_{28}N_2OS_2$ .

A structure-factor calculation including all atoms resulted in  $R = \sum |F_o| - |F_c| / \sum |F_o| = 0.05$  for all observed reflexions. Scattering factors were taken from *International Tables for X-ray Crystallography* (1962). The final coordinates and standard deviations are given in Table 2.\*

**Discussion.** The conformation of the molecule and the atom numbering are shown in Fig. 1, the bond distances and angles in Table 3.

\* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30628 (21 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1 NZ, England.

Table 3. *Intramolecular bond distances (Å) and angles (°) (with standard deviations in parentheses)*

C(1)—C(2)	1.401 (7)	C(1)—H(1)	1.05
C(1)—C(15)	1.400 (7)	C(3)—H(3)	0.99
C(2)—C(3)	1.384 (9)	C(4)—H(4)	1.01
C(2)—S(26)	1.764 (6)	C(8)—H(8)	0.98
C(3)—C(4)	1.385 (8)	C(9)—H(9)	1.05
C(4)—C(5)	1.391 (8)	C(10)—H(10)	1.07
C(5)—S(6)	1.785 (5)	C(11)—H(11)	1.12
C(5)—C(15)	1.406 (8)	C(13)—H(131)	1.05
S(6)—C(7)	1.772 (6)	C(13)—H(132)	1.09
C(7)—C(8)	1.395 (8)	C(14)—H(14)	1.03
C(7)—C(12)	1.404 (8)	C(17)—H(171)	1.06
C(8)—C(9)	1.393 (9)	C(17)—H(172)	0.99
C(9)—C(10)	1.379 (9)	C(18)—H(181)	1.01
C(10)—C(11)	1.404 (8)	C(18)—H(182)	1.06
C(11)—C(12)	1.395 (8)	C(20)—H(201)	0.98
C(12)—C(13)	1.512 (7)	C(20)—H(202)	1.01
C(13)—C(14)	1.554 (7)	C(21)—H(211)	0.96
C(14)—C(15)	1.537 (7)	C(21)—H(212)	0.99
C(14)—N(16)	1.480 (6)	C(22)—H(221)	1.06
N(16)—C(17)	1.468 (7)	C(22)—H(222)	1.06
N(16)—C(21)	1.467 (7)	C(23)—H(231)	1.05
C(17)—C(18)	1.537 (8)	C(23)—H(232)	1.07
C(18)—N(19)	1.472 (7)	C(24)—H(241)	1.11
N(19)—C(20)	1.470 (7)	C(24)—H(242)	1.11
N(19)—C(22)	1.480 (7)	O(25)—H(25)	0.99
C(20)—C(21)	1.528 (7)	C(27)—H(271)	1.10
C(22)—C(23)	1.539 (10)	C(27)—H(272)	1.06
C(23)—C(24)	1.535 (9)	C(27)—H(273)	1.10
C(24)—O(25)	1.447 (9)		
S(26)—C(27)	1.788 (9)		
C(2)—C(1)—C(15)	121.8 (5)	C(11)—C(10)—H(10)	117
C(1)—C(2)—C(3)	119.4 (5)	C(10)—C(11)—H(11)	117
C(1)—C(2)—S(26)	117.0 (4)	C(12)—C(11)—H(11)	123
C(3)—C(2)—S(26)	123.6 (4)	C(12)—C(13)—H(131)	110
C(2)—C(3)—C(4)	119.4 (5)	C(12)—C(13)—H(132)	107
C(3)—C(4)—C(5)	121.4 (5)	C(14)—C(13)—H(131)	109
C(4)—C(5)—S(6)	114.9 (4)	C(14)—C(13)—H(132)	110
C(4)—C(5)—C(15)	120.1 (5)	H(131)—C(13)—H(132)	108
S(6)—C(5)—C(15)	125.0 (4)	C(13)—C(14)—H(14)	107
C(5)—S(6)—C(7)	99.3 (3)	C(15)—C(14)—H(14)	105
S(6)—C(7)—C(8)	120.1 (4)	N(16)—C(14)—H(14)	106
S(6)—C(7)—C(12)	118.8 (4)	N(16)—C(17)—H(171)	105
C(8)—C(7)—C(12)	121.2 (5)	N(16)—C(17)—H(172)	115
C(7)—C(8)—C(9)	119.1 (5)	C(18)—C(17)—H(171)	110
C(8)—C(9)—C(10)	120.5 (6)	C(18)—C(17)—H(172)	108
C(9)—C(10)—C(11)	120.5 (6)	H(171)—C(17)—H(172)	109
C(10)—C(11)—C(12)	119.9 (5)	C(17)—C(18)—H(181)	111
C(7)—C(12)—C(11)	118.8 (5)	C(17)—C(18)—H(182)	107
C(7)—C(12)—C(13)	120.0 (5)	N(19)—C(18)—H(181)	110

Table 3 (cont.)

C(11)—C(12)—C(13)	121.2 (5)	N(19)—C(18)—H(182)	117
C(12)—C(13)—C(14)	112.7 (5)	H(181)—C(18)—H(182)	101
C(13)—C(14)—C(15)	117.2 (4)	N(19)—C(20)—H(201)	105
C(13)—C(14)—N(16)	113.1 (4)	N(19)—C(20)—H(202)	106
C(15)—C(14)—N(16)	107.1 (4)	C(21)—C(20)—H(201)	113
C(1)—C(15)—C(5)	117.5 (5)	C(21)—C(20)—H(202)	108
C(1)—C(15)—C(14)	114.1 (4)	H(201)—C(20)—H(202)	114
C(5)—C(15)—C(14)	128.3 (5)	N(16)—C(21)—H(211)	109
C(14)—N(16)—C(17)	112.8 (4)	N(16)—C(21)—H(212)	113
C(14)—N(16)—C(21)	114.7 (4)	C(20)—C(21)—H(211)	106
C(17)—N(16)—C(21)	109.2 (4)	C(20)—C(21)—H(212)	108
N(16)—C(17)—C(18)	108.4 (4)	H(211)—C(21)—H(212)	112
C(17)—C(18)—N(19)	110.7 (4)	N(19)—C(22)—H(221)	113
C(18)—N(19)—C(20)	110.3 (4)	N(19)—C(22)—H(222)	104
C(18)—N(19)—C(22)	108.6 (4)	C(23)—C(22)—H(221)	110
C(20)—N(19)—C(22)	110.6 (4)	C(23)—C(22)—H(222)	110
N(19)—C(20)—C(21)	110.5 (4)	H(221)—C(22)—H(222)	107
N(16)—C(21)—C(20)	108.4 (4)	C(22)—C(23)—H(231)	112
N(19)—C(22)—C(23)	112.2 (5)	C(22)—C(23)—H(232)	112
C(22)—C(23)—C(24)	111.4 (5)	C(24)—C(23)—H(231)	108
C(23)—C(24)—O(25)	110.5 (6)	C(24)—C(23)—H(232)	109
C(2)—S(26)—C(27)	104.1 (4)	H(231)—C(23)—H(232)	104
C(2)—C(1)—H(1)	122	C(23)—C(24)—H(241)	110
C(15)—C(1)—H(1)	116	C(23)—C(24)—H(242)	107
C(2)—C(3)—H(3)	123	O(25)—C(24)—H(241)	105
C(4)—C(3)—H(3)	117	O(25)—C(24)—H(242)	104
C(3)—C(4)—H(4)	120	H(241)—C(24)—H(242)	120
C(5)—C(4)—H(4)	118	C(24)—O(25)—H(25)	116
C(7)—C(8)—H(8)	115	S(26)—C(27)—H(271)	110
C(9)—C(8)—H(8)	126	S(26)—C(27)—H(272)	106
C(8)—C(9)—H(9)	119	S(26)—C(27)—H(273)	110
C(10)—C(9)—H(9)	121	H(271)—C(27)—H(272)	110
C(9)—C(10)—H(10)	122	H(271)—C(27)—H(273)	112
		H(272)—C(27)—H(273)	110

The molecule has a folded conformation similar to that of phenothiazine derivatives, *e.g.* chlorpromazine (McDowell, 1969) and thiethylperazine (McDowell, 1970). However, the folding is much more pronounced in the present compound. The angle between the planes of the benzene rings is  $104^\circ$  as opposed to  $140^\circ$  in the phenothiazine derivatives. There is some analogy between the conformation of the side chain of thiethylperazine, spiperone (Koch, 1973) and spirilene (Koch & Evrard, 1973) and one side of the piperidine ring in oxyprothepine as shown by the comparison of the relevant torsion angles in Table 4.

Table 4. *Torsion angles (°) in corresponding fragments of oxyprothepine (A), spiperone (B), spirilene (C) and thiethylperazine (D)*

	A	B	C	D
C(22)—N(19)—C(20)—C(21)	177	168	173	164
N(19)—C(20)—C(21)—N(16)	-60	-64	-70	-54
C(20)—C(21)—N(16)—C(14)	-169	170	-176	175
C(21)—N(16)—C(14)—C(15)	64	175	176	66
N(16)—C(14)—C(15)—C(1)	69	-13	-28	20

The molecules are held together by hydrogen bonds and form centrosymmetric dimers: N(19)—O(25) [O(25) at  $1-x, 1-y, 1-z$ ],  $2.885 \text{ \AA}$ , and O(25)—N(19) [N(19) at  $1-x, 1-y, 1-z$ ]. Moreover the hydroxyl groups of neighbouring dimers are connected through the water molecule: O(28)—O(25) [O(25) at  $x, y, z$ ],  $2.899 \text{ \AA}$ , and O(28)—O(25) [O(25) at  $1-x, y, \frac{3}{2}-z$ ].

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## Copper(II) Bis-(*N,N*-dimethyldithiocarbamate)

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**Abstract.**  $[(\text{CH}_3)_2\text{NCS}_2]_2\text{Cu}$ , monoclinic,  $C2/c$ ,  $a = 15.243$  (3),  $b = 9.538$  (2),  $c = 8.086$  (2) Å,  $\beta = 101.31$  (1)°,  $Z = 4$ ,  $D_c = 1.75$ ,  $D_m = 1.76$  g cm<sup>-3</sup>, final  $R = 0.060$ . Magnetic gram susceptibility  $\chi = 2.66 \times 10^{-6}$  cgsu at 300 K. Crystals tend to grow as intimate twins, one individual of the twin being related to the other by reflexion across the  $bc$  plane; the  $b$  and  $c$  axes are common. The copper atom lies on a centre of symmetry and is octahedrally coordinated to six sulphur atoms [Cu–S(1) = 2.302 (2), Cu–S(2) = 2.319 (2), Cu–S(1') = 3.159 (2) Å]; the two longer bonds are to sulphur atoms in adjacent symmetry-related molecules.

**Introduction.** Black crystals were grown from a 1:1 methanol–ethanol mixed solvent system. Intensities and cell dimensions were measured on a computer-controlled Picker four-circle diffractometer with a crystal of dimensions 0.18 × 0.15 × 0.11 mm. Systematic absences are  $hkl$ ,  $h+k \neq 2n$  and  $h0l$ ,  $l \neq 2n$  confirming space group  $Cc$  or  $C2/c$ . Precession photographs of the  $h0l$  and  $h1l$  layers indicated that the crystal grew as a twin. One individual of the twin is the mirror image of the other; the  $b$  and  $c$  axes, which are common, define the mirror plane. Reflexions from the major twin (83% based on  $F^2$ ) are related to those from the minor twin by reflexion across the  $a^*b^*$  plane. Thus the  $hk0$  reflexions from each twin overlap in the  $a^*b^*$  plane while reflexions in the remainder of reciprocal space are in general well separated ( $\beta > 90^\circ$ ). Explicit calculation showed that for the  $hkl$ ,  $l \neq 0$  reflexions, partial overlap should only occur in the  $hk8$  layer. Data were collected by the  $\theta$ – $2\theta$  scan technique to a maximum  $2\theta$  value of 55° with Nb-filtered Mo  $K\alpha$  radiation ( $\lambda = 0.7107$  Å, scan rate = 2.0° min<sup>-1</sup>, total scan width = 0.6° extended for spectral dispersion). 1333 reflexions were measured of which 733 ( $> 2.3\sigma$ ) were classed

as observed. The values for  $I$  and  $\sigma(I)$  were corrected for Lorentz and polarization effects. No absorption corrections were made; the maximum error that this would introduce in  $F$  is  $\pm 2\%$  ( $\mu = 26.1$  cm<sup>-1</sup>).

All non-hydrogen atom positions were located by routine Patterson and Fourier syntheses assuming space group  $C2/c$ . Refinement of the structure was carried out by full-matrix least-squares techniques; the function minimized was  $\sum w(|F_o| - |F_c|)^2$  and each reflexion was given weight  $[= 1/\sigma^2(F)]$  in terms of  $\sigma(F) = \sigma(I)/(Lp)2F_o$ . Separate scale factors for the  $hk0$  and  $hkl$  ( $l \neq 0$ ) reflexions were used; in this way contributions of the minor twin to the  $hk0$  reflexions were largely accounted for. Refinement of the non-hydrogen atom positions with anisotropic temperature factors for all atoms except the methyl carbon atoms converged at  $R = 0.076$ . A difference synthesis gave the positions of all the hydrogen atoms; during further refinement the hydrogen atom positions were allowed to vary but their isotropic temperature factors were fixed at a constant value of  $B = 5.00$  Å<sup>2</sup>.

Table 1. Atomic parameters

(a) Fractional coordinates with e.s.d.'s

	$x/a$	$y/b$	$z/c$
Cu	0.0	0.0	0.5
S(1)	0.1184 (1)	–0.0311 (2)	0.3654 (3)
S(2)	0.0256 (1)	–0.2395 (2)	0.5227 (3)
N	0.1497 (5)	–0.3048 (7)	0.3415 (9)
C(1)	0.1041 (5)	–0.2063 (9)	0.4003 (10)
C(2)	0.2122 (7)	–0.2718 (13)	0.2338 (16)
C(3)	0.1351 (8)	–0.4531 (13)	0.3650 (19)
H(21)	0.251 (6)	–0.359 (8)	0.231 (10)
H(22)	0.193 (7)	–0.224 (10)	0.159 (12)
H(23)	0.252 (6)	–0.209 (10)	0.280 (12)
H(31)	0.097 (7)	–0.479 (10)	0.294 (12)
H(32)	0.195 (6)	–0.500 (10)	0.381 (11)
H(33)	0.092 (6)	–0.455 (10)	0.434 (12)