

as a monodentate oxygen donor (Blake, Gould & Winpenny, 1991).

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## Structure of *trans*-Perhydro-4,1-benzoxazepin-2-one

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**Abstract.**  $C_9H_{15}NO_2$ ,  $M_r = 169.225$ , monoclinic,  $P2_1/n$ ,  $a = 12.148$  (5),  $b = 5.2735$  (9),  $c = 14.559$  (4) Å,  $\beta = 106.48$  (3)°,  $V = 894.4$  (5) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.256$  Mg m<sup>-3</sup>, m.p. 430–431 K,  $\lambda(\text{Mo } K\alpha) = 0.7107$  Å,  $\mu = 0.52$  cm<sup>-1</sup>,  $F(000) = 368$ ,  $T = 293$  K, final  $R = 0.082$  for 842 reflections with  $F > 2\sigma(F)$ . The bond lengths and angles are normal. There is an intermolecular hydrogen bond N(1)—H $\cdots$ O(10): N(1) $\cdots$ O(10) = 2.943 (6), H $\cdots$ O(10) = 1.999 Å, N(1)—H $\cdots$ O(10) = 144.4 (3)°. Conformational analysis of the cyclohexane ring indicates a nearly ideal chair conformation <sup>1</sup>C<sub>4</sub>. The 4,1-benzoxazepine hetero-ring also adopts a chair conformation.

**Experimental.** Diffraction measurements were carried out using a Nicolet *R3m/E* automatic diffractometer, with graphite-monochromated Mo  $K\alpha$  radiation. A crystal of dimensions 0.12 × 0.17 × 0.44 mm was used for the X-ray data collection at 293 K. The lattice parameters and orientation matrix were obtained from 25 reflections in the range  $2 < 2\theta < 32^\circ$ . Intensity data were measured by the  $\omega$ - $2\theta$  scan technique, scan width 2°, scan speed to 29.3° min<sup>-1</sup>(max.). Check reflections 404 and 101 were monitored every 100 reflections. 1538 unique reflections were measured [842 with  $F > 2\sigma(F)$ ].  $h, k, l$

range: 0 to 13, 0 to 16 and -16 to 16,  $\theta_{\max} = 25^\circ$ . Neither absorption nor extinction corrections were applied. Structures were solved by using *SHELXTL* (Sheldrick, 1984) and refined by *SHELX76* (Sheldrick, 1976). Full-matrix refinement,  $\sum w(\Delta F)^2$  minimized, anisotropic non-H atoms. Positions of H atoms were generated from the assumed geometries, checked in Fourier maps and refined in the riding mode with an overall isotropic temperature factor [final value 0.079 (4) Å<sup>2</sup>] (110 parameters). Final  $R = 0.082$ ,  $wR = 0.069$ ,  $w = [\sigma^2(F) + 9 \times 10^{-4}(F)^2]^{-1}$ . The value of the  $R$  factor reflects the poor quality of the crystal sample. Difference Fourier maps showed no significant peaks (maximum 0.34, minimum -0.28 e Å<sup>-3</sup>). Maximum  $\Delta/\sigma$  is 0.189. Scattering factors were taken as in *SHELX76*. Calculations were performed on the Nicolet *R3m/E* diffractometer system with structure solution package Nicolet *SHELXTL* and on an IBM 43/41 computer.

The final atomic coordinates for non-H atoms are listed in Table 1.\* Fig. 1 shows a perspective view of

\* 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 54249 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional atomic coordinates ( $\times 10^4$ ) and equivalent isotropic temperature factors ( $\times 10^3$ ) with e.s.d.'s in parentheses

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

	x	y	z	$U_{eq}$ ( $\text{\AA}^2$ )
N(1)	959 (3)	7596 (7)	9637 (3)	41 (1)
C(2)	148 (4)	7073 (10)	8819 (3)	41 (1)
C(3)	72 (4)	8728 (11)	7957 (3)	56 (2)
O(4)	1103 (3)	8872 (7)	7699 (2)	62 (1)
C(5)	1849 (5)	10832 (10)	8175 (3)	56 (2)
C(5A)	2499 (4)	10262 (10)	9221 (3)	48 (2)
C(6)	3319 (5)	12463 (11)	9594 (4)	60 (2)
C(7)	4003 (5)	12164 (11)	10632 (4)	74 (2)
C(8)	3223 (5)	11760 (10)	11277 (4)	60 (2)
C(9)	2417 (4)	9545 (10)	10912 (3)	51 (2)
C(9A)	1706 (4)	9862 (9)	9865 (3)	40 (1)
O(10)	-528 (3)	5292 (7)	8770 (2)	54 (1)

Table 2. Bond lengths ( $\text{\AA}$ ) and angles ( $^\circ$ ) with e.s.d.'s in parentheses

N(1)—C(2)	1.341 (5)	C(5A)—C(6)	1.527 (7)
N(1)—C(9A)	1.480 (5)	C(5A)—C(9A)	1.538 (6)
C(2)—C(3)	1.509 (6)	C(6)—C(7)	1.511 (7)
C(2)—O(10)	1.237 (5)	C(7)—C(8)	1.526 (7)
C(3)—O(4)	1.408 (6)	C(8)—C(9)	1.520 (7)
O(4)—C(5)	1.419 (5)	C(9)—C(9A)	1.533 (6)
C(5)—C(5A)	1.532 (6)		
C(2)—N(1)—C(9A)	127.9 (4)	C(6)—C(5A)—C(9A)	110.2 (4)
N(1)—C(2)—C(3)	118.4 (5)	C(5A)—C(6)—C(7)	113.2 (5)
N(1)—C(2)—O(10)	121.1 (4)	C(6)—C(7)—C(8)	111.7 (5)
O(10)—C(2)—C(3)	120.5 (5)	C(7)—C(8)—C(9)	110.1 (4)
C(2)—C(3)—O(4)	113.8 (4)	C(8)—C(9)—C(9A)	112.9 (4)
C(3)—O(4)—C(5)	113.7 (4)	C(9)—C(9A)—C(5A)	110.4 (4)
O(4)—C(5)—C(5A)	114.8 (4)	C(9)—C(9A)—N(1)	105.8 (4)
C(5)—C(5A)—C(6)	107.7 (4)	C(5A)—C(9A)—N(1)	115.1 (4)
C(5)—C(5A)—C(9A)	113.5 (4)		

the molecule. Bond lengths and angles are given in Table 2. Puckering parameters (Cremer & Pople, 1975) and asymmetry factors (Kálmán, Czugler & Simon, 1982) for benzoxazepine [ $q_1 = 0.328$  (6),  $q_2 = 0.632$  (6)  $\text{\AA}$ ,  $\varphi_2 = 146.8$  (5),  $\varphi_3 = 102.4$  (5) $^\circ$ ,  $fC_s = 0.084$  (12),  $fC_2 = 0.100$  (14)  $\text{\AA}$ ] and the cyclohexane ring [ $Q = 0.569$  (6)  $\text{\AA}$ ,  $\varphi = 84.7$  (8) $^\circ$ ,  $\theta = 178.7$  (6) $^\circ$ ] indicated that both rings have a nearly ideal chair conformation: seven-membered-ring chair conformation (Boessenkool & Boeyens, 1980) and  ${}^1C_4$  (Boeyens, 1978) respectively. H atoms at C(5A) and C(9A) are in *trans* positions, as predicted by NMR (Bernáth, Fülöp & Sohár, 1987). The equatorial position of the NH group and the C(5)-methylene group with respect to the cyclohexane ring are also confirmed. The crystal packing is illustrated in Fig. 2. The molecules are connected through intermolecular hydrogen bonds of the N—H $\cdots$ O type to form dimers. The corresponding bond lengths and angles are H $\cdots$ O(10) = 1.999, N(1) $\cdots$ O(10) = 2.943 (6)  $\text{\AA}$ , N(1)—H $\cdots$ O(10) = 144.4 (3) $^\circ$ .

**Related literature.** Pharmacological studies of the saturated heterocycle analogues of benzodiazepine-like structures (Sternbach, 1979) indicated their

favourable biological activity (Bernáth, Fülöp, Ecsery, Blazsó & Minker, 1983), so the systematic synthesis of this group of compounds was initiated. *cis*- and *trans*-2-hydroxymethyl-1-cyclohexylamine were cyclized with ethyl chloroacetate in the presence of sodium hydroxide in order to synthesize stereoisomeric perhydro-4,1-benzoxazepin-2-ones (Bernáth, Fülöp & Sohár, 1987).

The title compound was obtained as the single product in the reaction with the *trans*-aminoalcohol, while reaction of the *cis*-aminoalcohol resulted in a mixture of a seven-membered hetero-ring and a dimeric compound containing a 14-membered hetero-ring (Kalmán, Argay, Fülöp & Bernáth, to be published). The difference in reaction course depending on the *cis* or *trans* character of the reactants prompted a study of the crystal and molecular structures of the reaction products.

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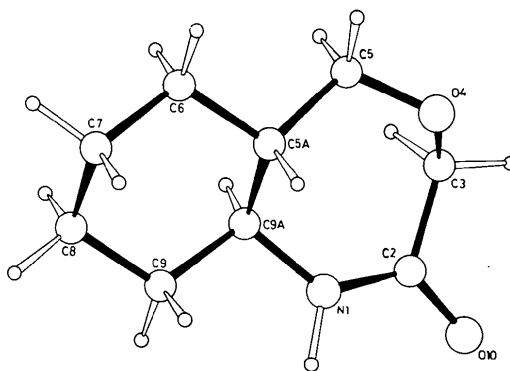


Fig. 1. A perspective view of the molecule, showing the atomic numbering. The H atoms are shown, but not labelled.

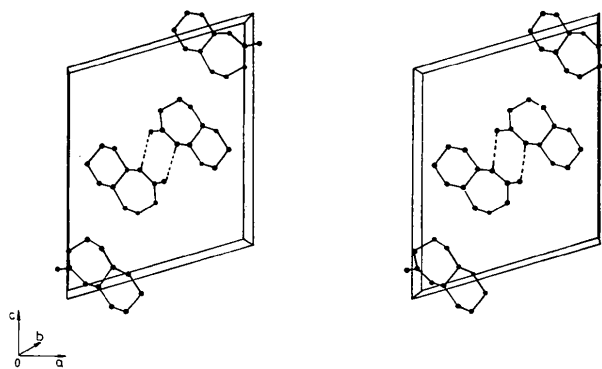


Fig. 2. Stereoscopic drawing of the molecular packing in the unit cell. Hydrogen bonds are indicated by dashed lines.

on Small Molecules of the International Union of Crystallography. The X-ray diffraction facility was established through funds provided by the National Science Foundation and by the Boeing Company.

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## Structure of 10-Benzyl-2-[(3,4-dimethoxyphenyl)methyl]phenothiazine-1-carbonitrile

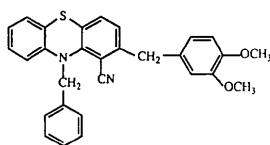
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**Abstract.**  $C_{29}H_{24}N_2O_2S$ ,  $M_r = 464.58$ , triclinic,  $P\bar{1}$ ,  $a = 9.967$  (3),  $b = 10.912$  (3),  $c = 12.258$  (3) Å,  $\alpha = 71.07$  (2),  $\beta = 67.26$  (2),  $\gamma = 75.61$  (2)°,  $V = 1151.4$  (8) Å<sup>3</sup>,  $Z = 2$ ,  $D_x = 1.34$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71073$  Å,  $\mu = 1.62$  cm<sup>-1</sup>,  $F(000) = 488$ ,  $T = 230$  K,  $R = 0.0375$  for 3734 observed reflections. The molecule adopts the *synclinal* conformation in which the 10-benzyl group occupies the pseudo axial position; the dihedral angle between the two benzo rings is 142.7 (1)°.

**Experimental.** The title compound (1) was prepared by the reaction of lithio-3,4-(dimethoxyphenyl)-acetonitrile and 10-benzyl-2-chlorophenothiazine with diisopropylamide in tetrahydrofuran. Colorless prismatic crystals were recrystallized from ethyl acetate. Unit-cell parameters by least-squares fit of



(1)

24 reflections in the range  $21 < 2\theta < 30^\circ$ , crystal dimensions  $0.35 \times 0.25 \times 0.20$  mm, space group  $P\bar{1}$ ; Siemens *R3m/V* diffractometer was used for data collection, graphite-monochromated Mo  $K\alpha$  radiation,  $\theta/2\theta$  scan mode, scan rate  $3.0\text{--}15.0^\circ \text{ min}^{-1}$  depending upon the intensity, 5889 reflections measured in the range  $3.0 \leq 2\theta \leq 56.0^\circ$ ,  $0 \leq h \leq 14$ ,  $-15$

$\leq k \leq 15$ ,  $-17 \leq l \leq 17$ , 5579 independent,  $R_{\text{int}} = 0.014$ , 3734 observed reflections with  $I > 3\sigma(I)$ ,  $\sigma(I)$  from counting statistics. Three standard reflections remeasured after every 150 reflections showed a maximum fluctuation in intensity of  $\pm 0.86\%$  during the data collection. Lorentz and polarization effects corrected, but no extinction or absorption correction. Structure was solved by direct methods with *SHELXTL-Plus* (Sheldrick, 1990), refined by full-matrix least squares, anisotropically for all non-H atoms. All H atoms were located in difference Fourier maps and refined isotropically. The function minimized was  $\sum w(|F_o| - F_c)^2$  with  $w = 1/[\sigma^2(F_o) + 0.00025(F_o)^2]$ . Final  $R = 0.0375$ ,  $wR = 0.0436$ ,  $S = 1.62$  for 3734 reflections with 403 parameters refined. In the last cycle,  $(\Delta/\sigma)_{\text{max}} = 0.002$ , the maximum and minimum difference Fourier residuals are 0.19 and  $-0.22 \text{ e } \text{Å}^{-3}$ , respectively. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV).

Positional and thermal parameters for non-H atoms are given in Table 1,\* while the bond lengths and bond angles are given in Table 2. Fig. 1 is an *ORTEP* (Johnson, 1965) drawing of the title compound and Fig. 2 shows the packing in the unit cell.

\* Lists of structure factors, anisotropic thermal parameters, torsion angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54224 (25 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.