KARLE, I. L. & BROCKWAY, L. O. (1944). *J. Am. Chem. Soc.* **66**, 1974.
LEHMANN, M. S. & LARSEN, F. K. (1974). *Acta Cryst.* A**30**, 580–584.
MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN*80. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.
RAMACHANDRAN, G. N. & SASISEKHARAN, V. (1968). *Adv. Protein Chem.* **23**, 283.
SCHMID, E. D. & BROSA, B. (1972). *J. Chem. Phys.* **56**, 6267–6268.
SHELDRICK, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.
TROTTER, J. (1961). *Acta Cryst.* **14**, 1135–1140.

Acta Cryst. (1987). C**43**, 2391–2393

Structure of 1,2-Dipiperidinoethane Mono-*N*-oxide Trihydrate

BY MARIUSZ JASKÓLSKI

Department of Crystallography, Faculty of Chemistry, A. Mickiewicz University, ul. Grunwaldzka 6, 60–780 *Poznań, Poland*

(*Received* 4 *March* 1987; *accepted* 16 *July* 1987)

Abstract. *N*-(2-Piperidinoethyl)piperidine *N*-oxide trihydrate, $C_{12}H_{24}N_2O.3H_2O$, $M_r = 267{\cdot}1$, monoclinic, $P2_1/n$, $a = 13{\cdot}696\ (2)$, $b = 7{\cdot}5819\ (5)$, $c = 14{\cdot}721\ (2)$ Å, $\beta = 99{\cdot}10\ (1)°$, $V = 1509{\cdot}5\ (3)$ Å^3, $Z = 4$, $D_x = 1{\cdot}17$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 0{\cdot}71$ mm^{-1}, $F(000) = 592$, $T = 292$ K, $R = 0{\cdot}044$ for 1570 observed reflexions. The overall conformation of the molecule is similar to that of the *transoidal* form characteristic of the bis-*N*-oxide derivative: the piperidine rings are in a chair form, the $-CH_2-$ substituents at the N atoms are equatorial, the C—N—C—C torsion angles around the exocyclic N—C bonds are characteristic of *gauche* and *trans* conformations at both N atoms, the *N* substituents at the C—C bridge are *trans*. The water molecules play distinctly different roles in the structure. One forms only two H bonds (as a donor) with water molecules, one participates in two O(*W*)—H···O(*W*) interactions and in a relatively weak O(*W*)—H···N(2) hydrogen bond and the third one accepts two H bonds from water molecules and forms two relatively strong H bonds with *N*-oxide O atoms. Being involved in two H bonds, the *N*-oxide O atom is a much more active H-bond acceptor than the amino N atom which accepts only one, relatively weak, H bond.

Introduction. This paper continues the studies on the derivatives of 1,1′-ethylenebispiperidine (PEtP) in the aspects of their conformation and H bonding in the crystalline state. The $PEtP(NO)_2$ molecule [1,1′-ethylenebis(piperidine 1-oxide)] and its diprotonated cation [in $PEtP(NO)_2.2HClO_4$] were found to be crystallographically centrosymmetric in their crystal structures (Jaskólski, Kosturkiewicz, Mickiewicz-Wichłacz & Wiewiórowski, 1979; Jaskólski, 1982). It was concluded from those structures that in the $PEtP(NO)_2$ skeleton the piperidine rings maintain their chair conformations, the O atoms are axial and the diaminoethylene bridge has a *gauche*$^+$-*trans*-*gauche*$^-$ conformation (O—N—C—C—N—O). This conformation was termed *'transoidal'* as it has the *N*-oxide O atoms on the ends of the N—C—C—N bridge *trans* oriented. However, the monoprotonated $PEtP(NO)_2H^+$ cation [in $PEtP(NO)_2.HClO_4$; Jaskólski, Gdaniec & Kosturkiewicz, 1982; Jaskólski, Olovsson, Tellgren & Mickiewicz-Wichłacz, 1982] turned out to be *cisoidal* with the two N—O groups bridged by a very short intramolecular O···H···O hydrogen bond [2·426 (3) Å] and with appreciable deformation in the N—C—C—N bridge owing to internal strain. A similar situation was found in the monoprotonated mono-*N*-oxide analog [$PEtP(NO).HClO_4.H_2O$; Jaskólski & Kosturkiewicz, 1984] where the $PEtP(NO)H^+$ cation is *cisoidal* with a very short intramolecular O···H···N hydrogen bond [2·551 (4) Å]. It was, therefore, of interest to investigate if the mono-*N*-oxide free-base PEtP(NO) molecule will also adopt a conformation similar to that of $PEtP(NO)_2$. This paper reports the X-ray crystal structure of $PEtP(NO).3H_2O$.

Experimental. Hygroscopic crystals (elongated plates) from water. Crystal 0·25 × 0·3 × 0·4 mm covered with thin film of vaseline and mounted on a Syntex $P2_1$ diffractometer, Cu $K\alpha$ radiation. Cell parameters from least-squares treatment of setting angles of 15 reflexions ($8{\cdot}2 \le 2\theta \le 31{\cdot}3°$). θ:2θ profiles measured for 2214 unique $+h+k\pm l$ reflexions with $2\theta \le 115°$ [$\sin\theta/\lambda$ (max.) = 0·547 Å^{-1}; $0 \le h \le 14$, $0 \le k \le 8$, $-16 \le l \le 15$], profile analysis according to Lehmann & Larsen (1974). No significant intensity variation (< 2·7%) for two standard reflexions measured every 1·5 h. No

0108-2701/87/122391-03$01.50

Table 1. *Final fractional coordinates and equivalent isotropic thermal parameters* (Å^2)

$B_{eq} = \frac{1}{3}\sum_i\sum_j B_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	*y*	*z*	B_{eq}
C(22)	0·3796 (2)	0·5677 (4)	0·2391 (2)	3·83 (7)
C(23)	0·3073 (2)	0·4634 (4)	0·2860 (2)	4·79 (8)
C(24)	0·3511 (2)	0·4215 (4)	0·3844 (2)	4·99 (9)
C(25)	0·4508 (2)	0·3316 (4)	0·3882 (2)	4·72 (8)
C(26)	0·5179 (2)	0·4393 (4)	0·3363 (2)	3·95 (7)
N(2)	0·4726 (1)	0·4685 (3)	0·2404 (1)	2·92 (5)
C(2)	0·5392 (2)	0·5680 (3)	0·1903 (2)	3·19 (7)
C(1)	0·6164 (2)	0·4451 (3)	0·1624 (2)	2·93 (6)
N(1)	0·6970 (1)	0·5384 (2)	0·1224 (1)	2·58 (5)
C(12)	0·6566 (2)	0·6215 (3)	0·0310 (2)	3·16 (6)
C(13)	0·7385 (2)	0·7062 (4)	−0·0119 (2)	3·79 (7)
C(14)	0·8192 (2)	0·5751 (4)	−0·0238 (2)	4·08 (7)
C(15)	0·8576 (2)	0·4856 (3)	0·0674 (2)	3·65 (7)
C(16)	0·7738 (2)	0·4025 (3)	0·1079 (2)	3·04 (6)
O(1)	0·7402 (1)	0·6678 (2)	0·1840 (1)	2·86 (4)
O(*W*1)	0·8382 (1)	0·5025 (2)	0·3391 (1)	4·18 (5)
O(*W*2)	0·1024 (2)	0·3532 (3)	0·4755 (1)	5·72 (6)
O(*W*3)	1·0384 (1)	0·5805 (3)	0·3221 (2)	5·93 (6)

absorption or extinction correction. 1577 observed reflexions with $I \geq 1{\cdot}96\sigma(I)$. Structure solved with *MULTAN*. Full-matrix least-squares refinement on F; weighting scheme: $w = (F_o/F_l)^2$ for $|F_o| < F_l$, $w = 1$ for $F_l \leq |F_o| \leq F_H$, $w = (F_H/F_o)^2$ for $|F_o| > F_H$ with $F_l = 4{\cdot}35$ and $F_H = 17{\cdot}5$. Positions of H atoms refined using difference-Fourier maps calculated between refinements of anisotropic non-H atoms; fixed isotropic temperature factors for H atoms (one unit greater than B_{iso} of their carriers). Seven strong reflexions judged to suffer seriously from extinction (020, 10$\bar{1}$, 105, 110, 122, 21$\bar{1}$, 40$\bar{2}$) excluded from final refinement. Final convergence: $R = 0{\cdot}044$, $wR = 0{\cdot}051$, $S = 4{\cdot}36$, $(\Delta/\sigma)_{max} = 0{\cdot}03$, largest absolute value of electron density in final ΔF map $= 0{\cdot}15$ e Å^{-3}. Computer programs: Syntex *XTL* system (Syntex, 1973) and local programs (Jaskólski, 1979); thermal-ellipsoid molecular illustration drawn using *ORTEP* (Johnson, 1976). Atomic scattering factors as included in the *XTL* system.

Discussion. Atomic coordinates are listed in Table 1.* Non-H bond lengths and angles are given in Fig. 1. They agree very well with the standard values. The C—H bond lengths range from 0·92 to 1·12 Å with a mean of 1·02 Å. A stereoscopic thermal-ellipsoid representation of the PEtP(NO) molecule is given in Fig. 2. The molecular conformation can be characterized as follows: (i) both piperidine rings are in a chair conformation; (ii) the exocyclic $-CH_2-$ substituents at the piperidyl N atoms are equatorial; (iii) the C—C—N—C torsion angles around the N(1)—C(1) bond are *gauche* [C(2)—C(1)—N(1)—C(12)] and *trans* [C(2)—C(1)—N(1)—C(16)] and the same situation is observed for N(2)—C(2) [in other words, the N(1)—O(1) bond and the lone electron pair at N(2) are *gauche* with respect to the C(1)—C(2) bridge]; (iv) the N(1)—C(1)—C(2)—N(2) torsion angle is *trans*. The PEtP(NO) molecule has a pseudo-centrosymmetric form similar to the perfectly centrosymmetric (*transoidal*) conformation observed for the $PEtP(NO)_2$ molecule [in $PEtP(NO)_2.4H_2O$] and the $PEtP(NO)_2 2H^+$ dication [in $PEtP(NO)_2.2HClO_4$]. Most of the departures from perfect symmetry (measured by torsion angles) are less

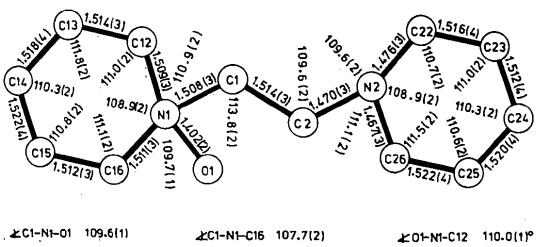

Fig. 1. Bond distances (Å) and angles (°) in the PEtP(NO) molecule (H atoms are not shown).

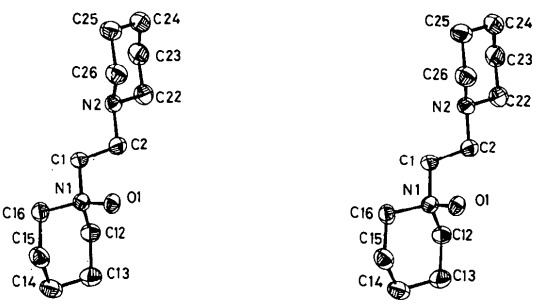

Fig. 2. Stereoscopic thermal-ellipsoid view of the molecule (H atoms have been omitted). The thermal ellipsoids are drawn at the 50% probability level.

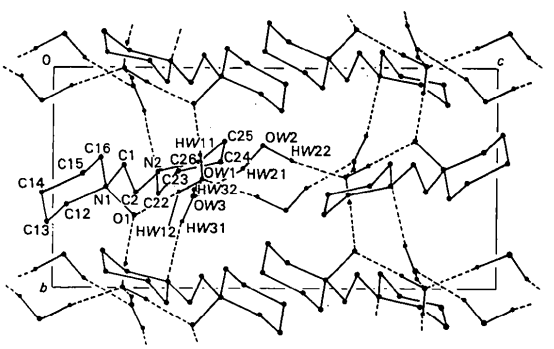

Fig. 3. Projection of the structure down **a**. H bonds are shown as broken lines. H atoms attached to C have been omitted.

*Lists of structure factors, anisotropic thermal parameters, H-atom parameters and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44257 (25 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

than 3°. The main deviations are limited to rotations around the N–C–C–N bridge [171·8 (3) instead of 180°] and around the N(2)–C(2) bond. The conformation around this bond [C(26)–N(2)–C(2)–C(1) 79·5 (2), C(22)–N(2)–C(2)–C(1) −160·3 (2)°] differs by *ca* 13° from that at the N(1)–C(1) bond, this flexibility evidently being due to the lack of an O substituent at the N(2) atom. Comparison of corresponding torsion angles indicates that the independent 'halves' of $PEtP(NO)_2$ and $PEtP(NO)_2H^+$ and the 'N(1) half' of PEtP(NO) have nearly the same conformation. In conclusion, one can say that the PEtP(NO) molecule adopted a *transoidal* form similar to the *transoidal* conformation characteristic of the $PEtP(NO)_2$ skeleton.

In contrast to $PEtP(NO)_2.4H_2O$, PEtP(NO) crystallizes as the trihydrate. All water molecules are involved in H bonding as double donors (Fig. 3). Water 1 is a donor in H bonds with two *N*-oxide acceptors. Water 3 uses one of its H atoms in an H bond with water 1 and the other one is donated to N(2). Water 2 is a donor in two H bonds with the other two H_2O molecules. The three water molecules show quite different acceptor properties. Water 1 accepts two H bonds, water 3 one H bond and water 2 does not show any acceptor activity. The remaining acceptor centers are located on the PEtP(NO) molecule: N(2) is a single acceptor and O(1) accepts two H bonds. It is interesting to note that the *N*-oxide O atom is much more active as an acceptor (it accepts two relatively strong H bonds) than the N(2) amino atom (which accepts only one, relatively weak, H bond). Geometrical parameters for the H bonds are reported in Table 2.

Table 2. *Geometry of the* H *bonds*

	D–H (Å)	H···*A* (Å)	*D*···*A* (Å)	∠*D*–H···*A* (°)
O(*W*1)–H(*W*11)···O(1^{i})	0·92	1·84	2·755 (2)	176
O(*W*1)–H(*W*12)···O(1)	0·96	1·81	2·760 (2)	171
O(*W*3)–H(*W*31)···N(2^{ii})	0·98	2·11	3·080 (3)	172
O(*W*3)–H(*W*32)···O(*W*1)	1·12	1·78	2·854 (3)	160
O(*W*2)–H(*W*21)···O(*W*3^{iii})	1·05	1·87	2·866 (3)	159
O(*W*2)–H(*W*22)···O(*W*1^{iv})	1·09	1·85	2·933 (2)	173

Symmetry code: (i) 1·5−*x*, *y*−0·5, 0·5−*z*; (ii) 1·5−*x*, 0·5+*y*, 0·5−*z*; (iii) *x*−1, *y*, *z*; (iv) 1−*x*, 1−*y*, 1−*z*.

I thank Professors Z. Kosturkiewicz and M. Wiewiórowski for their interest in this work and Mgr D. Mickiewicz-Wichłacz for growing the crystals. This work was partly supported by project CPBP 01.12.10.4.

References

JASKÓLSKI, M. (1979). Thesis, A. Mickiewicz Univ., Poznań, Poland.

JASKÓLSKI, M. (1982). *Pol. J. Chem.* **56**, 187–193.

JASKÓLSKI, M., GDANIEC, M. & KOSTURKIEWICZ, Z. (1982). *Pol. J. Chem.* **56**, 297–305.

JASKÓLSKI, M. & KOSTURKIEWICZ, Z. (1984). *Acta Cryst.* C**40**, 869–871.

JASKÓLSKI, M., KOSTURKIEWICZ, Z., MICKIEWICZ-WICHŁACZ, D. & WIEWIÓROWSKI, M. (1979). *J. Mol. Struct.* **52**, 77–94.

JASKÓLSKI, M., OLOVSSON, I., TELLGREN, R. & MICKIEWICZ-WICHŁACZ, D. (1982). *Acta Cryst.* B**38**, 291–294.

JOHNSON, C. K. (1976). *ORTEP*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

LEHMANN, M. S. & LARSEN, F. K. (1974). *Acta Cryst.* A**30**, 580–584.

Syntex (1973). *XTL Structure Determination System*. Syntex Analytical Instruments, Cupertino, California, USA.

Acta Cryst. (1987). C**43**, 2393–2398

Structure of Remoxipride, a New Antipsychotic Agent. Comparison of Base and Hydrochloride Forms

BY BIRGITTA STENSLAND

Department of Structural Chemistry, Arrhenius Laboratory, University of Stockholm, S-106 91 Stockholm, Sweden

AND THOMAS HÖGBERG AND STEN RÄMSBY

Research and Development Laboratories, Astra Alab AB, S-151 85 Södertälje, Sweden

(*Received* 9 *February* 1987; *accepted* 24 *July* 1987)

Abstract. (−)-(*S*)-3-Bromo-*N*-[(1-ethyl-2-pyrrolidinyl)methyl]-2,6-dimethoxybenzamide, remoxipride base form, $C_{16}H_{23}BrN_2O_3$, $M_r = 371{\cdot}27$, monoclinic, $P2_1$, $a = 13{\cdot}605$ (1), $b = 14{\cdot}302$ (1), $c = 9{\cdot}794$ (2) Å, $\beta = 103{\cdot}67$ (1)°, $V = 1851{\cdot}7$ (4) Å^3, $Z = 4$, $D_x = 1{\cdot}332$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 31{\cdot}44$ cm^{-1}, $F(000) = 768$, room temperature, $R = 0{\cdot}066$ for 2499 reflections. The absolute-configuration analysis, from

0108-2701/87/122393-06$01.50