

Fig. 1. Thermal ellipsoid plot of 1,9-diacetyl-3,5,7-trinitro-1,3,5,7,9-pentaazanonane with ellipsoids drawn at the 20% probability level. Only one of the two similar molecules in the asymmetric unit is shown. The open bonds indicate hydrogen bonding to H(1'), H(9'), O(23'), and O(11') of molecule 2 or its symmetry equivalents.

C atoms applied to bonded hydrogens, C—H = 0.96 Å. $U(H) = 1.2 U_{eq}(C)$, amine hydrogens refined isotropically. $(\Delta/\sigma)_{max} = 0.40$, $R = 0.038$, $wR = 0.045$, $S = 1.737$. Final difference Fourier excursions 0.14 and $-0.31 e \text{ \AA}^{-3}$. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).^{*} Atom numbering for Tables 1 and 2, which

^{*} Lists of structure factors, anisotropic thermal parameters and hydrogen coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51747 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Crystal Studies of Heterocyclic Compounds Containing One Oxygen and Two Nitrogen Atoms. XIV. 4,5,6,7,8,9-Hexahydro-1-oxa-5,8-diazadibenzo[*b*,*j*]cycloundecene

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Abstract. C₁₆H₁₈N₂O, $M_r = 254.3$, monoclinic, $P2_1/n$, $a = 5.035(2)$, $b = 9.928(3)$, $c = 26.831(9)$ Å, $\beta =$

$92.10(3)^\circ$, $V = 1340.2(8)$ Å³, $Z = 4$, $D_x = 1.260(8)$ g cm⁻³, $\lambda(\text{Cu K}\alpha) = 1.54178$ Å, $\mu = 5.48$ cm⁻¹, $F(000) = 544$. Diffractometer data at room temperature, $R = 0.033$ for 1711 reflections with $I > 2.5\sigma(I)$. Neglecting the oxygen and benzene C

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give atom coordinates, and bond distances, bond angles and selected torsion angles, respectively, follows that shown in Fig. 1; molecule 2 is numbered similarly and differentiated by the inclusion of an apostrophe.

Related literature. For the structure of a similar compound, 1,7-diacetyl-2,4,6-trinitro-2,4,6-triazahexane, see Cobbleddick & Small (1973*a–c*). For a similarly substituted tetraazaazanonane, see Gilardi & George (1989).

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atoms, the heterocyclic ring is in a twist-like conformation. The benzene rings fused to the heterocycle ring form a dihedral angle of $95.4(1)^\circ$. The neighbouring molecules are linked together by an intermolecular hydrogen bond of type $N-H\cdots N$, with distances $N\cdots N$ $3.171(4)$ and $N\cdots H$ $3.32(2)$ Å and angle $N-H\cdots N$ $171(1)^\circ$. The molecules linked by hydrogen bonds are arranged parallel to the twofold axes.

Experimental. This paper is a continuation of the study of the structure-biological activity relationship of dibenzoxazacycloalkenes. The title compound was obtained by hydrogenolysis of 6,9-ditosyldibenzo- $[b,j]$ perhydro-1-oxa-5,8-diazacycloundecane with metallic sodium in boiling *n*-pentanol. The formula was confirmed by MS, IR and NMR spectra. The compound shows neuroleptic activity (Glinka, 1986).

Colourless, thin tabular crystals from ethanol at room temperature; crystal size $0.1 \times 0.2 \times 0.4$ mm, Stoe diffractometer using $\theta-2\theta$ scan technique; unit-cell parameters from 38 reflections, $\theta_{\max} = 22.3^\circ$, Cu $K\alpha$ radiation, range of h , k and l $0 \leftrightarrow 5$, $0 \leftrightarrow 11$, $-30 \leftrightarrow 30$, respectively. Total of 1998 unique reflections measured to $\sin\theta/\lambda = 0.56 \text{ \AA}^{-1}$, data not corrected for absorption. Standard reflections $0,0,12$ and 042 , maximum

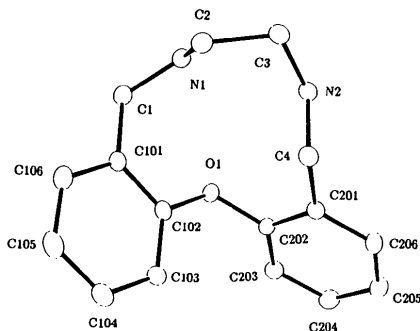


Fig. 1. The structure of the molecule with the atom-numbering scheme.

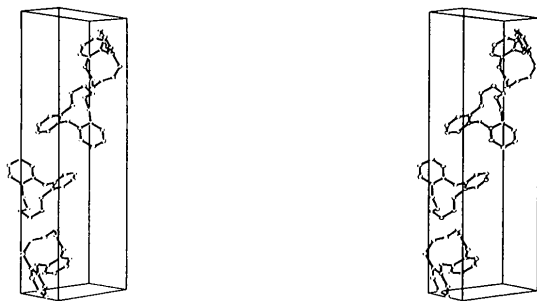


Fig. 2. A stereoview of the molecular packing, viewed down the a axis.

Table 1. Final positional and thermal parameters ($\times 10^4$) with e.s.d.'s in parentheses

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

	x	y	z	$U_{eq} (\text{\AA}^2)$
O1	6269 (2)	-994 (1)	1191 (0)	402 (6)
C1	7535 (4)	-3498 (2)	1640 (1)	472 (11)
N1	7857 (3)	-2628 (2)	2077 (1)	439 (9)
C2	10602 (4)	-2262 (2)	2205 (1)	486 (11)
C3	10687 (4)	-911 (2)	2475 (1)	490 (11)
N2	9061 (3)	76 (2)	2198 (1)	421 (8)
C4	10247 (4)	545 (2)	1742 (1)	405 (10)
C101	8720 (3)	-3025 (2)	1160 (1)	399 (9)
C102	8063 (3)	-1785 (2)	942 (1)	364 (9)
C103	9084 (4)	-1397 (2)	492 (1)	440 (10)
C104	10833 (4)	-2224 (2)	256 (1)	554 (12)
C105	11552 (4)	-3444 (2)	465 (1)	599 (13)
C106	10505 (4)	-3827 (2)	910 (1)	511 (12)
C201	8206 (3)	1173 (2)	1392 (1)	364 (9)
C202	6351 (3)	405 (2)	1119 (1)	358 (8)
C203	4466 (4)	986 (2)	801 (1)	441 (10)
C204	4366 (4)	2375 (2)	752 (1)	489 (11)
C205	6160 (4)	3159 (2)	1017 (1)	504 (11)
C206	8060 (4)	2563 (2)	1332 (1)	462 (11)

Table 2. Interatomic distances (Å), bond angles ($^\circ$) and selected torsion angles ($^\circ$) with e.s.d.'s in parentheses

O1-C102	1.387 (2)	C102-C103	1.383 (2)
O1-C202	1.403 (2)	C103-C104	1.375 (3)
C1-C101	1.514 (2)	C105-C106	1.377 (3)
C1-N1	1.461 (2)	C106-C101	1.390 (2)
N1-C2	1.461 (2)	C201-C202	1.392 (2)
C2-C3	1.524 (3)	C202-C203	1.379 (2)
C3-N2	1.463 (2)	C203-C204	1.387 (3)
N2-C4	1.458 (2)	C204-C205	1.370 (3)
C4-C201	1.502 (2)	C205-C206	1.387 (3)
C101-C102	1.397 (2)		
C102-O1-C202	118.1 (1)	C103-C104-C105	120.1 (2)
C101-C1-N1	117.6 (1)	C104-C105-C106	119.5 (2)
C1-N1-C2	114.4 (2)	C105-C106-C101	122.3 (2)
N1-C2-C3	109.9 (2)	C4-C201-C202	122.1 (1)
C2-C3-N2	110.0 (1)	C4-C201-C206	121.1 (1)
C3-N2-C4	113.7 (1)	C202-C201-C206	116.8 (2)
N2-C4-C201	111.4 (1)	O1-C202-C201	119.4 (1)
C1-C101-C102	122.3 (2)	O1-C202-C203	118.4 (2)
C1-C101-C106	120.9 (2)	C201-C202-C203	122.0 (2)
C106-C101-C102	116.8 (2)	C202-C203-C204	119.7 (2)
O1-C102-C101	116.4 (1)	C203-C204-C205	119.7 (2)
O1-C102-C103	122.2 (1)	C204-C205-C206	120.0 (2)
C101-C102-C103	121.4 (2)	C205-C206-C201	121.8 (2)
C102-C103-C104	120.0 (2)		
O1-C102-C101-C1	0.5 (3)	C3-N2-C4-C201	161.1 (2)
C102-C101-C1-N1	-56.3 (3)	N2-C4-C201-C202	-74.2 (3)
C101-C1-N1-C2	-54.5 (3)	C4-C201-C202-O1	3.7 (3)
C1-N1-C2-C3	152.8 (2)	C201-C202-O1-C102	-82.2 (2)
N1-C2-C3-N2	-49.1 (3)	C202-O1-C102-C101	154.4 (2)
C2-C3-N2-C4	-73.1 (2)		

change of 4.0% and 1.5% respectively, 1711 reflections with $I > 2.5\sigma(I)$ used in calculations; solution by direct methods using *SHELX86* (Sheldrick, 1986), refinement using *SHELX76* (Sheldrick, 1976), H atoms located from difference Fourier map, refinement by full-matrix least-squares procedure on F magnitudes, 244 parameters. Refinement to final $R = 0.033$, $S = 0.4643$, unit weights. Largest peaks on a final

difference Fourier map 0.11 and $-0.16 \text{ e } \text{Å}^{-3}$, ratio of $(\Delta/\sigma)_{\text{max}} = 0.002$. Scattering factors from *SHELX76*. The molecule and the numbering scheme are shown in Fig. 1, molecular packing in Fig. 2, positional parameters and equivalent values of the anisotropic temperature factors for the non-H atoms are given in Table 1,* interatomic distances, angles and torsion angles in Table 2.

*Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond distances and angles involving H atoms, have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51775 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Related literature. Stępień, Wajsman, Grabowski, Glinka & Perrin (1987); Olszak, Stępień, Wajsman, Grabowski, Glinka & Lecocq (1987).

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Structure of the Antimalarial Halofantrine Hydrochloride

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Abstract. 1,3-Dichloro- α -[2-(dibutylamino)ethyl]-6-(trifluoromethyl)-9-phenanthrenemethanol hydrochloride, C₂₆H₃₁Cl₂F₃NO⁺.Cl⁻, $M_r = 536.9$, monoclinic, $P2_1/n$, $a = 8.169$ (3), $b = 32.924$ (13), $c = 22.775$ (6) Å, $\beta = 98.99$ (3)°, $V = 6050.2$ Å³, $Z = 8$, $D_x = 1.18 \text{ g cm}^{-3}$, $\text{Cu K}\alpha$, $\lambda = 1.54178$ Å, $\mu = 15.51 \text{ cm}^{-1}$, $F(000) = 2240$, room temperature, final $R = 18.3\%$ for 2899 reflections with $|F_o| > 3\sigma$. The crystal structure of halofantrine hydrochloride was determined to 1.0 to 1.1 Å resolution. The high R factor is due to poor crystal quality. In order to have a crystal with sufficient thickness for data collection, it was necessary to use a crystal that had grown in layers. The high R factor is also due to a disordered CF₃ group, a disordered solvent channel, and high thermal factors on the long hydrocarbon chains. The two halofantrine conformers stack such that the phenanthrene rings are nearly on top of each other with the chlorine and CF₃ groups on opposite sides and with the hydrocarbon side chains projected away from each other, but on the same side of the phenanthrene rings. Atoms in the phenanthrene rings of the two stacked conformers are separated by

3.4 to 3.7 Å. On each of the halofantrine conformers, one of the n -butyl groups extends in a linear fashion whereas the other n -butyl group is bent back towards the phenanthrene ring. The crystal structure contains a pseudo twofold axis, parallel to the b axis, which intersects Cl(3) and passes through the center ring of the phenanthrene ring system of both conformers. Cl(3) is hydrogen bonded with O(1) (3.14 Å) and O(51) (3.09 Å) and perhaps makes a hydrogen-bond distance of 3.16 Å with a peak of electron density not found at full weight (symbolized Os, part of disordered solvent). Six disordered solvent peaks lie in a channel parallel to

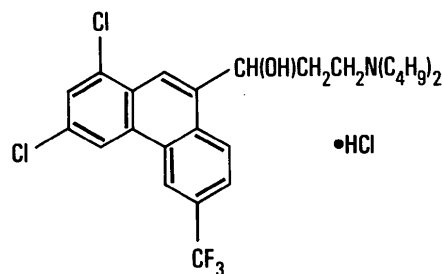


Fig. 1. Chemical structure of halofantrine hydrochloride.

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