

Fig. 2. Packing diagram viewed down **b**.

distances and angles which are within normal ranges, except the angle O—C(2)—C(1). In all molecules this angle [mean value 105.8 (1)°] deviates from the regular tetrahedral value. The only difference between the three molecules is the orientation of the phenyl ring relative to the C—O axis.

The molecular packing is illustrated in Fig. 2. This shows that the O atoms of the three molecules in the asymmetric unit are linked by hydrogen bonds producing spirals parallel to the **b** direction; molecule (II) is linked with a molecule (III) from a unit cell translated over **b**. The distances between O atoms involved in a hydrogen bond are similar with a mean value of

2.767 (2) Å; the angles are also within normal ranges. There are no intermolecular C—C distances less than 3.6 Å and the shortest H—H contact is 2.21 (4) Å. In each molecule the hydroxyl-H atom is positioned between C(3) and C(11), which results in a mean distance H(OH)—C(3) of 2.64 (4) Å. The position of the hydroxyl-H atoms is the same as that found in IR studies (Visser & Van der Maas, 1983).

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Structure of a Chloral–Benzamidoxime Adduct

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Abstract. *O*-(2,2,2-Trichloro-1-hydroxyethyl)-benzamidoxime, C₉H₉Cl₃N₂O₂, *M_r* = 283.5, monoclinic, *P*2₁/*a*, *a* = 11.881 (7), *b* = 9.338 (4), *c* = 12.098 (5) Å, β = 118.01 (4)°, *V* = 1185 Å³, *Z* = 4, *D_x* = 1.59 g cm⁻³, λ(Mo *K*α) = 0.71069 Å, μ = 7.6 cm⁻¹, *F*(000) = 576, *T* ~ 173 K, *R* = 0.028 for 1537 unique observed reflections with *I* > σ(*I*). The molecule has the expected hemiacetal structure. It is nearly planar and the bond lengths and angles are normal. The trichloromethyl group is nearly tetra-

hedral. The molecules form dimers through O—H...N hydrogen bonds.

Introduction. A variety of evidence (Srivastava, Freire, Chaves, Beltrão & Carpenter, 1986, references therein) suggests that the reaction of an arylamidoxime with an aldehyde proceeds by attack of the acetaldehyde at the O atom to form an O-hemiacetal intermediate, rather than by attack at the amide N atom to form an N-hemiacetal. The postulated hemiacetal intermediates

are mostly too unstable to isolate. However, the aldehyde chloral (2,2,2-trichloroethanal) had been reported (Falck, 1886) to form a stable adduct with benzamidoxime. The structure of this adduct was determined to see if it is, in fact, the expected O-hemiacetal.

Experimental. Synthesized by Srivastava *et al.* (1986) from benzamidoxime and chloral according to Falck (1886), and recrystallized from benzene. Colorless single crystal, 0.24 × 0.56 × 0.66 mm, mounted on glass fiber with quick-setting epoxy cement. Nicolet R3m/E diffractometer system with low-temperature device (about 170 K), graphite monochromator; unit-cell parameters by least-squares refinement of 25 reflections ($20 \leq 2\theta \leq 30^\circ$), θ - 2θ scans at variable rates; $2\theta_{\max} = 45^\circ$ for the range $0 \leq h \leq 12$, $0 \leq k \leq 10$, $-13 \leq l \leq 11$; three reflections monitored every 100 reflections with 2% decrease in intensity over the course of data collection; 1561 measured unique intensities (not including space-group absences), 1537 unique data with $I > \sigma(I)$ used for refinement; numerical absorption correction based on indexed and measured faces (maximum and minimum transmission factors 0.840, 0.682). Structure by direct methods, all H atoms found on difference map; for refinement, all C—H and N—H bond lengths fixed at 0.96 Å and refined as rigid groups with ideal geometry; anisotropic thermal parameters for all non-H atoms and fixed isotropic parameters for H atoms (20% greater than that of carrying atom). Refined by cascade block-diagonal least squares on F with maximum $(\sin\theta)/\lambda = 0.54 \text{ \AA}^{-1}$; refinement of 148 parameters converged to $R = 0.028$, $wR = 0.044$; $w = 1/[\sigma^2(F) + 0.00020F^2]$ where $\sigma^2(F)$ is from counting statistics; goodness of fit = 2.45 (divided by slope of normal probability plot, 1.31); $(\Delta/\sigma)_{\max} = 0.13$ in final cycle; highest peak in final difference map 0.20 e \AA^{-3} ; deepest hole -0.25 e \AA^{-3} ; atomic scattering factors from *International Tables for X-ray Crystallography* (1974); all calculations were performed on a Data General Eclipse S140 computer using the *SHELXTL* 4.1 (Sheldrick, 1984) program package.

Discussion. Atomic coordinates and equivalent isotropic thermal parameters are listed in Table 1.* Fig. 1 is a thermal-ellipsoid plot of the molecule showing the atom-numbering scheme. The molecule has the expected hemiacetal structure. The trichloromethyl group is nearly tetrahedral with mean C—Cl bond length 1.771 Å

* Tables of bond lengths and bond angles for non-H atoms, anisotropic thermal parameters, H-atom parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43602 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

and r.m.s. deviation 0.006 Å. The benzene ring is nearly regular with mean C—C bond length 1.385 Å and r.m.s. deviation 0.006 Å. Bond lengths and angles are all within normal ranges. Fig. 2 shows a stereoview of the contents of one unit cell. The molecules are linked into dimers across centers of inversion by pairs of O—H...N hydrogen bonds of length 2.775 Å; otherwise no intermolecular contacts are exceptional.

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Table 1. Atom coordinates ($\times 10^4$) and equivalent isotropic temperature factors ($\text{\AA}^2 \times 10^3$)

	x	y	z	U_{eq}^*
Cl(1)	2732 (1)	7922 (1)	1769 (1)	29 (1)
Cl(2)	1655 (1)	5105 (1)	1552 (1)	36 (1)
Cl(3)	1144 (1)	7326 (1)	2923 (1)	30 (1)
C(1)	2304 (2)	6593 (2)	2551 (2)	22 (1)
C(2)	3494 (2)	6118 (2)	3751 (2)	19 (1)
C(3)	5512 (2)	8291 (2)	6178 (2)	18 (1)
C(4)	6868 (2)	8412 (2)	7144 (2)	19 (1)
C(5)	7807 (2)	8515 (2)	6781 (2)	23 (1)
C(6)	9076 (2)	8623 (2)	7683 (2)	28 (1)
C(7)	9397 (2)	8598 (2)	8934 (2)	32 (1)
C(8)	8461 (2)	8504 (2)	9300 (2)	31 (1)
C(9)	7186 (2)	8425 (2)	8412 (2)	24 (1)
N(1)	5229 (1)	7253 (2)	5380 (2)	19 (1)
N(2)	4675 (2)	9247 (2)	6195 (2)	26 (1)
O(1)	3194 (1)	5055 (1)	4359 (1)	23 (1)
O(2)	3898 (1)	7375 (1)	4480 (1)	23 (1)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

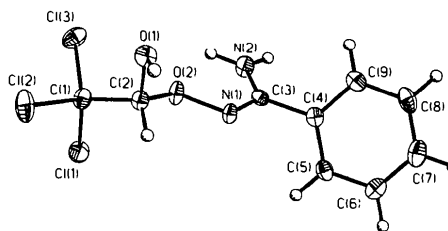


Fig. 1. Thermal ellipsoid plot of *O*-(2,2,2-trichloro-1-hydroxyethyl)benzamidoxime.

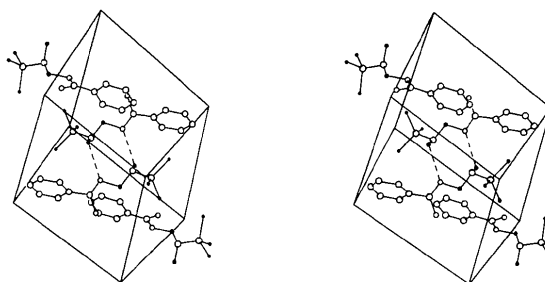


Fig. 2. Stereoview of the unit-cell contents. The dashed lines indicate the O—H...N hydrogen bonds that link the molecules into dimers.

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Molecular Structure Analysis of Benzamide Neuroleptics and Analogs. X. *exo*-2,3-Dimethoxy-*N*-(9-phenylmethyl-9-azabicyclo[3.3.1]non-3-yl)benzamide Hydrochloride

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Abstract. $C_{24}H_{31}N_2O_3^+Cl^-$, $M_r = 430.9$, monoclinic, $P2_1/c$, $a = 10.518$ (1), $b = 23.624$ (3), $c = 9.268$ (1) Å, $\beta = 93.03$ (2)°, $V = 2299.7$ Å³, $Z = 4$, $D_x = 1.24$ g cm⁻³, $Mo K\alpha$, $\lambda = 0.71069$ Å, $\mu = 1.53$ cm⁻¹, $F(000) = 920$, $T = 293$ K, $R = 0.03$ for 1400 observed reflections. The dimethoxyphenyl and amide groups are coplanar and held together by means of an intramolecular H bond as usually observed in the active antidopaminergic benzamides. Unexpectedly, the *N*-benzyl side chain is in an axial position with respect to the piperidinyl ring which holds the benzamide group. This piperidinyl moiety adopts a more planar geometry compared with the corresponding part in the tropapride, the well known nortropane benzamide analog.

Introduction. This X-ray structure determination is part of a general study on conformational properties of dopamine antagonists which bind to a subpopulation of DA receptors, the so-called D_2 receptors, in a sodium-dependent manner. Compared with tropapride (Durant, De Beys, Collin & Evrard, 1986), a very potent benzamide analog, the title compound has one more carbon in the piperidine bridge. This results in a slight decrease of activity. Therefore, it seemed to be of interest to analyze the conformation of this analog and in particular to clarify the orientation of the benzyl group at the ternary endocyclic N atom.

Experimental. This compound crystallized from a methanol–ethyl acetate mixture at room temperature. Colourless prismatic crystal $0.40 \times 0.10 \times 0.10$ mm for all X-ray measurements. Enraf–Nonius CAD-4 diffractometer. Lattice parameters from least-squares

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refinement of 25 medium-angle reflections. No absorption or extinction correction. No intensity variation of standard reflection. $4 \leq 2\theta \leq 46^\circ$. 3180 independent reflections measured ($-11 \leq h \leq 11$, $0 \leq k \leq 26$, $0 \leq l \leq 10$), 1400 observed [$I \geq 2.5\sigma(I)$]. Structure solved by direct methods (*SHELX76*, Sheldrick, 1976). All non-H atoms found in the best FOM *E* map. Full-matrix least-squares refinement on *F* using *SHELX76*. All H atoms located on difference Fourier map and not refined. Anisotropic temperature factors (U_{ij}) for all non-H atoms and isotropic ones for H atoms (corresponding to the isotropic temperature factors of the carrier atoms incremented by 0.02). $R = 0.03$, $wR = 0.03$, $w = 1.0/[\sigma^2(F) + 0.005F^2]$, $(\Delta/\sigma)_{\max} = -0.053$ [U_{33} of C(27)], $S = 0.74$; max. and min. heights in final difference Fourier synthesis 0.18 and -0.14 e Å⁻³. Scattering factors from *SHELX76*. Structural analysis by *XRAY76* (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976).

Discussion. Atomic parameters are given in Table 1.* Fig. 1 shows the atom numbering and bond lengths and angles. Fig. 2 is a stereoview of the unit cell.

The existence of an intramolecular hydrogen bond between N(17) and O(26) [O(26)⋯N(17) 2.698 (4), H(17)⋯O(26) 1.882 (2) Å, N(17)–H(17)⋯O(26) 124.8 (2)°] leads to the formation of a virtual six-membered ring like those occurring in the active

* 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 43533 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.