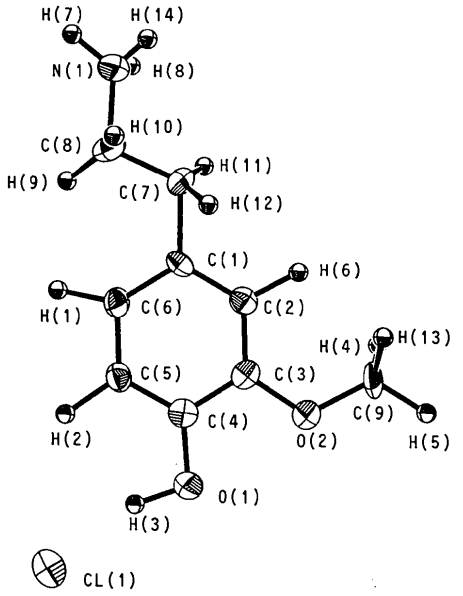

Fig. 1. Perspective view of 3-methoxytyramine hydrochloride, along with the atomic numbering used.

Fig. 2 shows a stereoscopic drawing of the crystal packing. The hydrogen bonds are listed in Table 2. The Cl^- ion is hydrogen bonded to the O(1) atom of the phenyl ring and N(1) atom of the side chain. Each 3-methoxytyramine molecule is joined to neighbouring molecules by a hydrogen-bond network involving the hydroxyl group, the charged amino group and the Cl^- ion.

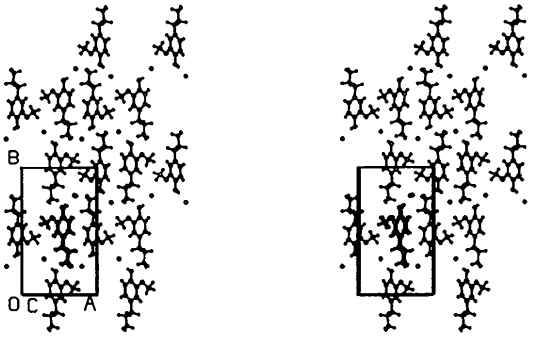

Fig. 2. A stereoscopic view of the structure, viewed along the *c* axis.

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Structure of Two Forms of Hordenine

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Abstract. 4-[2-(Dimethylamino)ethyl]phenol, $C_{10}H_{15}NO$, $M_r = 165{\cdot}24$, orthorhombic form: $Pna2_1$, $a = 9{\cdot}113\ (6)$, $b = 17{\cdot}356\ (4)$, $c = 6{\cdot}155\ (1)$ Å, $V = 973{\cdot}5$ Å^3, $Z = 4$, $D_x = 1{\cdot}127$ Mg m^{-3}, Mo $K\alpha$ radiation, $\lambda = 0{\cdot}71073$ Å, $\mu = 0{\cdot}068$ mm^{-1}, $F(000) = 360$, $T = 293\ (1)$ K, $R = 0{\cdot}041$ for 882 observed reflections with $I > 3\sigma(I)$; monoclinic form: $P2_1/n$, $a = 13{\cdot}80\ (2)$, $b = 12{\cdot}37\ (2)$, $c = 5{\cdot}95\ (1)$ Å, $\beta = 99{\cdot}7\ (1)°$, $V = 1001$ Å^3, $Z = 4$, $D_m = 1{\cdot}12$ (by flotation in CCl_4/*n*-hexane), $D_x = 1{\cdot}10$ Mg m^{-3}, Cu $K\alpha$ radiation, $\lambda = 1{\cdot}5418$ Å, $\mu = 0{\cdot}524$ mm^{-1}, $F(000) = 360$, $T = 293\ (1)$ K, $R = 0{\cdot}056$ for 1026 observed reflections. In

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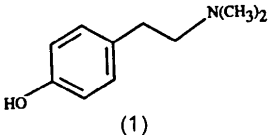
both structures, the dimethylaminoethyl side chain is fully extended (*anti* conformation). However, the hydroxyl H atom is *cis* to C(9) in the orthorhombic form and *trans* to C(9) in the monoclinic form.

Introduction. Hordenine, (1), has been found in a number of cacti along with other hallucinogenic alkaloids (Nean, Sato & Howald, 1972) and in many species of plants (Smith, 1977).



(1)

The 3,4-dihydroxy derivatives of phenylethylamines, catecholamines, are known to occur as zwitterions in the crystalline state (Andersen, 1975). A similar zwitterionic state had been observed in phenylephrine, the 3-hydroxy derivative of phenylethylamine (Andersen, 1976). Because of the inductive effect of the side chain, the molecular structure of 4-hydroxy derivatives of phenylethylamine might differ from their 3-hydroxy analogues, as is evident from their different pK values (Antikainen & Witikainen, 1973). Therefore, the crystal structure of hordenine, a 4-hydroxyphenylethylamine derivative, was determined to establish the nature of this compound in the crystalline state. A monoclinic structure was initially determined from photographic data (Parvez, 1977). In this paper we report this structure as well as the structure of an orthorhombic form.

Experimental. Orthorhombic: Colorless crystals of (1) were obtained from its sulfate (Sigma Inc.) by treatment with an aqueous solution of sodium carbonate and recrystallized from methanol by slow evaporation at room temperature. A crystal with approximate dimensions 0·42 × 0·27 × 0·18 mm was used for data collection on an Enraf–Nonius CAD-4 diffractometer equipped with graphite-monochromatized Mo $K\alpha$ radiation. Accurate cell constants and a crystal orientation matrix were determined by a least-squares fit of the setting angles of 25 reflections with $10 < \theta < 15°$. Intensity data were collected by the $\omega/2\theta$ scan method using variable scan speed (0·91–3·3° min^{-1}) in the range $2 < \theta < 30°$. The intensities of three reflections, chosen as standards, were monitored at 2 h intervals and did not decrease significantly. Intensities of 1679 independent reflections (h 0–12, k 0–23, l 0–8) were measured of which 882 had $I > 3\sigma(I)$ and were used in the structure solution and refinement. Data were corrected for Lorentz and polarization effects but not for absorption.

Monoclinic: Colorless crystals of (1) were obtained from its sulfate (ICN Pharmaceutical Inc.) by treatment with an aqueous solution of sodium carbonate and recrystallized from diethyl ether. Three-dimensional data were recorded on multiple-film equi-inclination Weissenberg photographs of layers $hk0$ to $hk5$ using Ni-filtered Cu $K\alpha$ radiation. The intensity data were measured by the SRC Microdensitometer Service, Rutherford Laboratory, England, and corrected for Lorentz and polarization effects; absorption correction was deemed unnecessary. 1026 independent non-zero structure amplitudes requiring six layer scale factors were employed in the structure solution and refinement.

The structures were solved using direct phasing procedures in *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and refined by full-matrix least-squares calculations on F's. H atoms were located from difference Fourier syntheses and refined with isotropic temperature factors, all other atoms were refined with anisotropic thermal parameters. For the orthorhombic crystal, the refinement converged with R and wR 0·041 and 0·042, respectively, where $w = [\sigma^2(F_o) + (0{\cdot}020F_o)^2]^{-1}$, $(\Delta/\sigma)_{max} < 0{\cdot}01$, $\Delta\rho$ in final ΔF map $-0{\cdot}33$ to 0·26 e Å^{-3} and $S = 2{\cdot}014$. For the monoclinic crystal, convergence was achieved with $R = wR = 0{\cdot}065$, using the weighting scheme $w = (10 + |F_o| + 0{\cdot}001|F_o|^3)^{-1}$, $(\Delta/\sigma)_{max} < 0{\cdot}1$, $\Delta\rho$ in final ΔF map $\pm 0{\cdot}30$ e Å^{-3} and $S = 1{\cdot}50$. Scattering factors used in the calculations were taken from Cromer & Mann (1968) and Stewart, Davidson & Simpson (1965). Computer programs used in this study were from the Enraf–Nonius *SDP Structure Determination Package* (B. A. Frenz & Associates Inc., 1985) and *ORTEP*II (Johnson, 1976).

Discussion. Final fractional coordinates and mean isotropic temperature factors with e.s.d.'s are given in Table 1,* and molecular dimensions are in Table 2. Fig. 1 shows *ORTEP* drawings of both forms of hodenine while the crystal structures indicating H bonds are represented in Fig. 2.

The molecular dimensions in the crystal structures of hordenine are normal in both forms. The fully extended side chain comprising C(1), C(7), C(8), N is planar in both structures. The two planes [C(1)–C(6) and C(1), C(7), C(8), N] are at right angles to each other; the dihedral angle between the two planes in the orthorhombic and monoclinic forms being

* Lists of structure factors, anisotropic temperature factors, positional parameters of H atoms and molecular dimensions involving H atoms for both structures have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53721 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional coordinates and mean isotropic temperature factors* (Å^2) *with e.s.d.'s in parentheses*

	x	y	z	U_{eq}
Orthorhombic				
O	1·3741 (2)	0·3665 (1)	1·3234*	0·076 (1)†
N	0·5521 (2)	0·3638 (1)	0·9636 (3)	0·053 (1)†
C(1)	0·9284 (3)	0·4026 (1)	1·2143 (4)	0·054 (1)†
C(2)	0·9803 (3)	0·3682 (2)	1·4007 (5)	0·072 (1)†
C(3)	1·1283 (3)	0·3569 (2)	1·4355 (5)	0·073 (2)†
C(4)	1·2295 (3)	0·3785 (1)	1·2807 (4)	0·055 (1)†
C(5)	1·1795 (3)	0·4133 (1)	1·0954 (4)	0·060 (1)†
C(6)	1·0313 (3)	0·4251 (1)	1·0640 (5)	0·058 (1)†
C(7)	0·7672 (3)	0·4150 (1)	1·1717 (5)	0·065 (1)†
C(8)	0·7046 (2)	0·3508 (1)	1·0350 (5)	0·056 (1)†
C(9)	0·5429 (3)	0·4278 (2)	0·8110 (5)	0·069 (1)†
C(10)	0·4970 (3)	0·2939 (2)	0·8607 (7)	0·093 (2)†
Monoclinic				
O	0·0868 (2)	−0·1648 (2)	0·3346 (5)	0·059 (2)‡
N	0·5116 (2)	0·2207 (2)	0·8886 (5)	0·039 (2)‡
C(1)	0·2800 (3)	0·0850 (3)	0·5689 (6)	0·044 (2)‡
C(2)	0·2107 (3)	0·0507 (3)	0·6981 (6)	0·045 (2)‡
C(3)	0·1450 (3)	−0·0322 (3)	0·6243 (6)	0·046 (2)‡
C(4)	0·1488 (3)	−0·0833 (3)	0·4182 (6)	0·043 (2)‡
C(5)	0·2174 (3)	−0·0497 (4)	0·2888 (7)	0·054 (3)‡
C(6)	0·2818 (3)	0·0338 (4)	0·3640 (7)	0·054 (3)‡
C(7)	0·3508 (3)	0·1758 (3)	0·6481 (8)	0·052 (2)‡
C(8)	0·4447 (3)	0·1339 (3)	0·7932 (7)	0·043 (2)‡
C(9)	0·5578 (3)	0·2744 (4)	0·7172 (7)	0·057 (3)‡
C(10)	0·5868 (3)	0·1756 (3)	1·0704 (7)	0·051 (2)‡

* Fixed z coordinate to define the origin.
† $U_{eq} = (U_{11} + U_{22} + U_{33})/3$.
‡ $U_{eq} = (U_{11} + U_{22} + U_{33} + 2U_{13}\cos\beta)/3$.

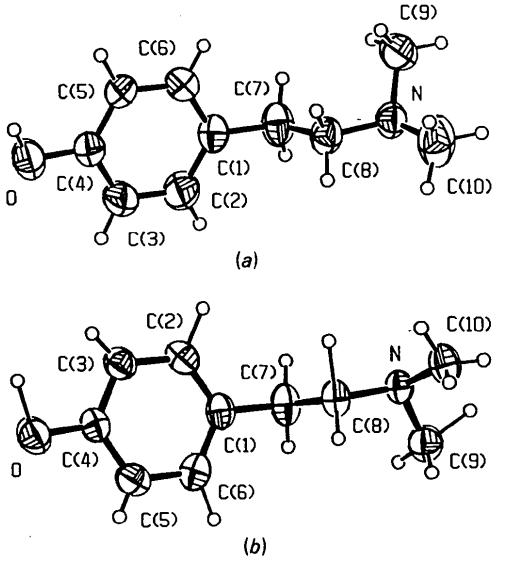


Fig. 1. *ORTEP* drawings of hordenine: (*a*) orthorhombic form; (*b*) monoclinic form.

95·5 (2) and 90·1 (3)°, respectively. In each structure one methyl carbon atom, C(10), is in the plane of the side chain, *i.e.* the side chain is fully extended as far as the C(10) *N*-methyl group, the other methyl carbon atom, C(9), is *cis* oriented with respect to the O—H bond on the ring in the orthorhombic form and *trans* oriented in the monoclinic system.

Table 2. *Molecular bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

	Ortho-rhombic	Monoclinic		Ortho-rhombic	Monoclinic
O—C(4)	1·360 (3)	1·359 (4)	C(2)—C(3)	1·380 (4)	1·388 (5)
N—C(8)	1·474 (3)	1·464 (5)	C(3)—C(4)	1·379 (4)	1·373 (5)
N—C(9)	1·457 (3)	1·441 (5)	C(4)—C(5)	1·368 (3)	1·374 (5)
N—C(10)	1·457 (3)	1·467 (5)	C(5)—C(6)	1·381 (3)	1·385 (6)
C(1)—C(2)	1·378 (4)	1·383 (6)	C(7)—C(8)	1·509 (3)	1·517 (5)
C(1)—C(6)	1·373 (3)	1·363 (6)	O'···N	2·75 (1[i])	2·69 (1[ii])
C(1)—C(7)	1·508 (3)	1·509 (5)			

	Ortho-rhombic	Monoclinic		Ortho-rhombic	Monoclinic
C(8)—N—C(9)	111·3 (2)	113·3 (3)	O—C(4)—C(3)	118·3 (3)	123·0 (3)
C(8)—N—C(10)	109·1 (2)	108·8 (3)	O—C(4)—C(5)	123·5 (2)	118·4 (3)
C(9)—N—C(10)	109·5 (2)	109·9 (3)	C(3)—C(4)—C(5)	118·3 (2)	118·6 (3)
C(2)—C(1)—C(6)	116·7 (2)	117·2 (3)	C(4)—C(5)—C(6)	120·5 (2)	121·0 (3)
C(2)—C(1)—C(7)	122·8 (2)	122·1 (3)	C(1)—C(6)—C(5)	122·1 (2)	121·5 (3)
C(6)—C(1)—C(7)	120·5 (2)	120·7 (3)	C(1)—C(7)—C(8)	111·1 (2)	111·5 (3)
C(1)—C(2)—C(3)	121·8 (3)	122·1 (3)	N—C(8)—C(7)	114·2 (2)	112·8 (3)
C(2)—C(3)—C(4)	120·5 (3)	119·6 (3)	O'—H(O')···N	175·4[i]	150[ii]

Primed atoms are related to the unprimed atoms by the symmetry transformations: (i) $1 + x, y, z$; (ii) $\frac{1}{2} - x, \frac{1}{2} + y, 1\frac{1}{2} - z$.

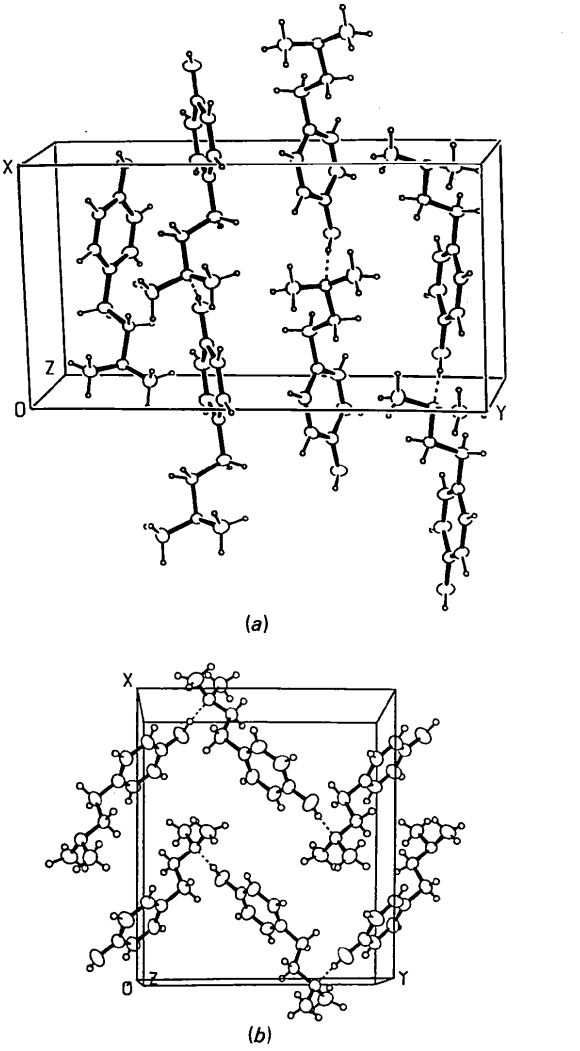


Fig. 2. Crystal structure of hodenine showing H bonds: (*a*) orthorhombic form; (*b*) monoclinic form.

In both structures, the hordenine molecules are hydrogen bonded in a head-to-tail fashion (Fig. 2), with N···O 2·75 (1) Å in the orthorhombic form and 2·69 (1) Å in the monoclinic form, giving rise to four or two chains of molecules per unit cell, running parallel to the *a* and *b* axes, respectively.

The position of H(O) is critical as its attachment determines whether the molecule is zwitterionic or neutral. As the atom was located and successfully refined in both structures, the molecule is shown to be non-ionic. This is in agreement with acid ionization constants in aqueous solution which show the *p*-OH to be less acidic than a protonated amino group.

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Platelet Activating Factor Antagonists. Structure of *N,N'*-Bis(3,4,5-trimethoxybenzoyl)-2-piperazinylmethyl 2,2-Dimethylpropanoate

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Abstract. Racemic title compound, $C_{30}H_{40}N_2O_{10}$, M_r = 588·65, triclinic, $P\bar{1}$, a = 10·154 (7), b = 11·820 (9), c = 15·038 (8) Å, α = 96·02 (5), β = 107·54 (4), γ = 110·34 (5)°, V = 1569 (2) Å^3, Z = 2, D_x = 1·25 g cm^{-3}, Cu $K\alpha$, λ = 1·5418 Å, μ = 7·4 cm^{-1}, $F(000)$ = 628, T = 293 K, R = 0·0470, wR = 0·0481 for 1961 unique observed reflections. The piperazine ring adopts a chair conformation and the molecule shows limited flexibility of the pseudo-twofold-related trimethoxybenzoyl moieties. The planes through the piperazine and the two trimethoxyphenyl rings are oriented almost perpendicular to each other. Apart from a few possible weak hydrogen bonds, the molecules are held together by weak π overlap and van der Waals forces.

Introduction. Platelet activating factor (PAF, PAF-acether) is an autacoid mediator implicated in a diverse range of pathological conditions including inflammation, various types of vascular disorders and shock (Godfroid & Braquet, 1986; Braquet & Godfroid, 1987; Braquet, Touqui, Shen & Vargaftig, 1987). Since the first development of PAF antagonists in 1983 a broad variety of naturally and synthetically derived compounds have been increasingly studied for their remarkable and different biological PAF activities. The characterization of the high-affinity PAF receptor site and molecular design of PAF antagonists are of prime interest for therapeutical purposes in the development of new and efficacious pharmaceutical agents. Recently (Dive, Godfroid, Lamotte-Brasseur, Batt, Heymans, Dupont & Braquet, 1989; Godfroid, Dive, Lamotte-Brasseur & Heymans, 1990), the conformational and electronic properties of some heterogeneous but potent antagonists have been studied in order to find the major and common features that may be of relevance for the control of biological activity. These

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