Table 3. *Comparison between unmethylated and methylated cytosine structures*

Compound	Whether protonated	Whether stacking by ring–ring overlap present	Reference
Unmethylated compounds			
Cytosine	No	Yes	(*a*)
Cytosine.H_2O	No	No	(*b*)
Cytosine.HCl	Yes	Yes, charge on N(3) delocalized by a strong N(3)—H···Cl hydrogen bond	(*c*)
Cytidine	No	No, O(4′) stacks on base ring	(*d*)
2′-Deoxycytidine.HCl	Yes	Yes, very little overlap exists	(*e*)
5′-dCMP*.H_2O	Yes	No	(*f*)
5′-dCMP* Na_2.7H_2O	No	Yes	(*g*)
5′-dCMP* Na_2.11H_2O	No	Yes	(*h*)
Methylated compounds			
5-Methylcytosine.0·5H_2O	No	No	(*i*)
5-Methylcytosine.HCl	Yes	No	(*j*)
5-Methyl-2-thiocytosine.0·5H_2O	No	No	(*k*)
5-Methylcytidine	No	No	Present work
5-Methyl-2′-dCMP.2H_2O	Yes	No	(*l*)

References: (*a*) Barker & Marsh (1964); (*b*) Jeffrey & Kinoshita (1963); (*c*) Mandel (1977); (*d*) Furberg, Peterson & Rømming (1965); (*e*) Subramanian & Hunt (1970); (*f*) Viswamitra, Swaminatha Reddy, Lin & Sundaralingam (1971); (*g*) Pandit, Seshadri & Viswamitra (1983); (*h*) Viswamitra & Pandit (1983); (*i*) Grainger & Bailey (1981); (*j*) Padmaja, Ramakumar & Viswamitra (1987); (*k*) Ravichandran, Chacko, Ponnuswamy & Trotter (1985); (*l*) Lalitha, Ramakumar & Viswamitra (1989).

* dCMP = deoxycytidine monophosphate.

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Structure of 3-Methoxytyramine Hydrochloride

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Abstract. 2-(4-Hydroxy-3-methoxyphenyl)ethylammonium chloride, $C_9H_{14}NO_2^+.Cl^-$, $M_r = 203{\cdot}67$, orthorhombic, $P2_12_12_1$, $a = 10{\cdot}586\ (2)$, $b = 17{\cdot}885\ (2)$, $c = 5{\cdot}190\ (2)$ Å, $V = 982{\cdot}6\ (4)$ Å^3, $Z = 4$, $D_x = 1{\cdot}377$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}354$ mm^{-1}, $F(000) = 432$, $T = 296$ K. The final values for R and wR were 0·037 and 0·039, respectively, for 1053 observed reflections. The amino side chain is in the fully extended *trans* conformation and lies in the same plane as the phenyl ring. There is a hydrogen-bonding network involving the *p*-hydroxyl group, the protonated amino group and the Cl^- ions.

Introduction. The crystal structure of catecholamines, such as dopamine (Bergin & Carlström, 1968; Giesecke, 1980), adrenaline (Andersen, 1975*a*),

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noradrenaline (Andersen, 1975*b*; Carlström & Bergin, 1967) and other related compounds (Barlow, Johnson, Howard, Walton & Koellner, 1989; Seiler, Markstein, Walkinshaw & Boelsterli, 1989) have been determined. It is important to study the detailed structure of catecholamine metabolites to obtain a more complete structural understanding of the catecholamine action as well as the mechanism of its metabolism. We report here the crystal structure of the first metabolic product of dopamine, 3-methoxytyramine, which is produced by methylation of dopamine.

Experimental. The title compound was crystallized from 50% methanol solution (0·2 mol dm^{-3}) as platelets, 0·4 × 0·3 × 0·2 mm; Rigaku AFC5R automated four-circle diffractometer with graphite-monochromated Mo $K\alpha$ radiation. Lattice parameters determined by least-squares fit of 2θ values of 18 reflections ($20{\cdot}75 < 2\theta < 25{\cdot}49°$); intensity data up to $2\theta = 50{\cdot}0°$ collected, ω–2θ scan, scan speed 16·0° (ω) min^{-1}, scan width $(1{\cdot}26 + 0{\cdot}30\tan\theta)°$, the ratio of peak counting time to background counting time was 2:1 at 50 kV and 180 mA; h 0–12, k 0–21, l 0–6. 1053 [705 with $I > 3\sigma(I)$] independent reflections measured; three reference reflections monitored every 100 reflections showed no crystal deterioration; Lorentz, polarization and absorption corrections (max. and min. transmission factors 1·11, 0·85) applied. Structure solved by direct methods with *MITHRIL* (Gilmore, 1984) and *DIRDIF* (Beurskens, 1984), refined by least squares with anisotropic thermal parameters for all non-H atoms; H atoms, located from difference Fourier map, included in refinement with isotropic thermal parameters. Final $R = 0{\cdot}037$, $wR = 0{\cdot}039$, $S = 1{\cdot}41$; $\sum w(|F_o| - |F_c|)^2$ minimized; $w = 4F_o^2/\sigma^2(F_o^2)$, $(\Delta/\sigma)_{max} = 0{\cdot}18$, the maximum and minimum peaks on the final difference Fourier map corresponded to 0·17 and $-0{\cdot}22$ e Å^{-3}. Atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974, Vol. IV); all numerical calculations performed using the *TEXSAN* crystallographic software package (Molecular Structure Corporation, 1985).

Discussion. Final atomic parameters of non-H atoms are listed in Table 1.* The bond lengths and angles are in Table 2. A perspective view of the molecule with the atomic numbering scheme is shown in Fig. 1. The overall conformation of the molecule corresponds to the β-rotameric conformation of dopamine which is necessary for high dopamine activity (Seiler, Markstein, Walkinshaw & Boelsterli, 1989; Cannon, 1975). The molecule has a charged amino group which also appears to be important for catechol amine activity (Barlow & Hamilton, 1962). The amino side chain is extended fully in the *trans* conformation and oriented in the same plane as the phenyl ring {torsion angles: τ_1 [N(1)—C(8)—C(7)—C(1)] 178·8 (6), τ_2 [C(2)—C(1)—C(7)—C(8)] −175·8 (6)°}. The planarity of the molecule contrasts remarkably with the structure of catecholamines, all of which have the side chains oriented more perpendicular to the phenyl ring plane [dopamine hydrochloride, τ_1 173·2, τ_2 77·6° (Giesecke, 1980); adrenaline, τ_1 171·6, τ_2 −96·4° (Andersen, 1975*a*); noradrenaline hydrochloride, τ_1 176·1, τ_2 81·5° (Carlström & Bergin, 1967)]. The 3-methoxy group is also oriented in the phenyl ring plane [torsion angle C(4)—C(3)—O(2)—C(9) −173·1 (5)°].

Table 1. *Atomic coordinates for non-*H *atoms* (× 10^4) *with e.s.d.'s in parentheses*

	x	y	z	B_{eq}*(Å^2)
Cl(1)	2065 (1)	−2780 (8)	8709 (3)	3·44 (6)
O(1)	530 (4)	−1821 (2)	12399 (9)	3·0 (2)
O(2)	−805 (4)	−1002 (2)	15488 (7)	2·8 (2)
N(1)	1015 (6)	2460 (3)	856 (1)	3·6 (3)
C(1)	551 (5)	491 (3)	1139 (1)	2·1 (2)
C(2)	−141 (5)	148 (3)	1333 (1)	2·1 (2)
C(3)	−139 (4)	−617 (3)	1363 (1)	2·0 (2)
C(4)	575 (5)	−1068 (3)	1197 (1)	2·2 (2)
C(5)	1280 (5)	−733 (3)	1010 (1)	2·3 (2)
C(6)	1261 (5)	43 (3)	976 (1)	2·3 (2)
C(7)	456 (7)	1332 (3)	1107 (1)	2·9 (3)
C(8)	1154 (7)	1641 (3)	884 (2)	3·1 (3)
C(9)	−1667 (6)	−593 (4)	1707 (1)	3·3 (3)

* The B values are the equivalent isotropic temperature factors calculated from $B_{eq} = (4/3)(B_{11}a^2 + B_{22}b^2 + B_{33}c^2 + ac\cos\beta)$.

Table 2. *Bond lengths* (Å) *and angles* (°) *between non-*H *atoms, and hydrogen-bond lengths* (Å)

O(1)—C(4)	1·365 (6)	C(1)—C(7)	1·518 (7)
O(2)—C(3)	1·381 (6)	C(2)—C(3)	1·376 (6)
O(2)—C(9)	1·428 (7)	C(3)—C(4)	1·400 (7)
N(1)—C(8)	1·479 (8)	C(4)—C(5)	1·364 (7)
C(1)—C(2)	1·387 (7)	C(5)—C(6)	1·399 (8)
C(1)—C(6)	1·386 (7)	C(7)—C(8)	1·478 (9)

C(3)—O(2)—C(9)	118·2 (4)	O(1)—C(4)—C(3)	116·8 (5)
C(2)—C(1)—C(6)	118·2 (4)	O(1)—C(4)—C(5)	124·6 (5)
C(2)—C(1)—C(7)	119·0 (5)	C(3)—C(4)—C(5)	118·6 (5)
C(6)—C(1)—C(7)	122·8 (5)	C(4)—C(5)—C(6)	121·1 (5)
C(1)—C(2)—C(3)	121·4 (5)	C(1)—C(6)—C(5)	120·4 (5)
O(2)—C(3)—C(2)	125·0 (5)	C(1)—C(7)—C(8)	115·1 (5)
O(2)—C(3)—C(4)	114·6 (4)	N(1)—C(8)—C(7)	113·3 (6)
C(2)—C(3)—C(4)	120·3 (5)		

A (at x, y, z)	D	Symmetry	$A\cdots D$
Cl(1)	O(1)	x, y, z	3·042 (4)
Cl(1)	N(1)	$\frac{1}{2}-x, -y, \frac{1}{2}+z$	3·286 (7)
Cl(1)	N(1)	$\frac{1}{2}-x, -y, -\frac{1}{2}+z$	3·406 (8)
Cl(1)	N(1)	$-x, -\frac{1}{2}+y, \frac{3}{2}-z$	3·493 (7)

* Lists of structure factors, anisotropic thermal parameters for non-H atoms, and coordinates and isotropic thermal parameters for H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53717 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

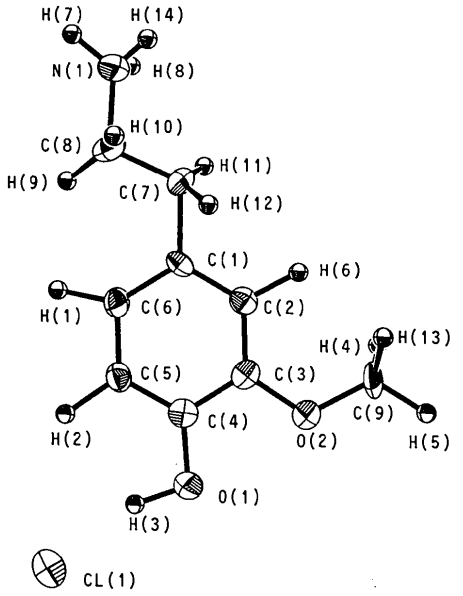

Fig. 1. Perspective view of 3-methoxytyramine hydrochloride, along with the atomic numbering used.

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.

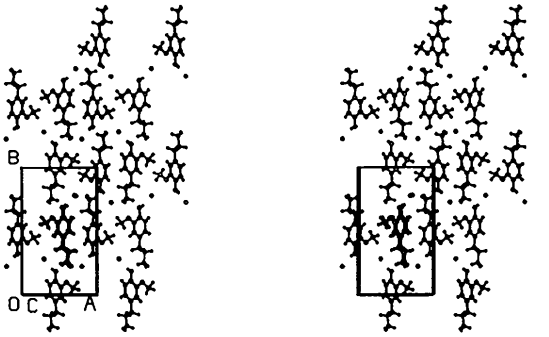

Fig. 2. A stereoscopic view of the structure, viewed along the *c* axis.

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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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