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Structure of 3-Methoxytyramine Perchlorate

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Abstract. 2-(4-Hydroxy-3-methoxyphenyl)ethylammonium perchlorate, $C_9H_{14}NO_2^+.ClO_4^-$, M_r = 267.67, triclinic, $P\bar{1}$, a = 8.075 (2), b = 10.086 (3), c = 7.465 (2) Å, α = 92.17 (3), β = 93.91 (3), γ = 96.58 (2)°, V = 601.9 (3) Å^3, Z = 2, D_m = 1.483 (1), D_x = 1.477 g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, μ = 3.29 cm^{-1}, $F(000)$ = 278, T = 296 K, final R = 0.068 for 891 reflections [$I > 3\sigma(I)$]. The molecule has a fully extended amino side chain of a *trans* configuration, and the plane of the side chain is oriented nearly perpendicular to the phenyl ring plane. There is a hydrogen-bonding network involving the 3-methoxy group, the 4-hydroxy group, the protonated amino group and O atoms of the perchlorate ions.

Experimental. Platelets of the title compound were crystallized from 50% methanol solution. A crystal 0.2 × 0.05 × 0.5 mm was used for data collection on a Rigaku AFC-5*R* automated four-circle diffractometer with graphite-monochromated Mo $K\alpha$ radiation. Lattice parameters were determined from 2θ values of 24 reflections ($20.4 < 2\theta < 34.4°$). Intensity data were collected to 2θ = 50.0°, using ω–2θ scan, scan speed 32.0°(ω) min^{-1}, scan width (1.05 + 0.30tanθ)°, the ratio of peak counting time to background counting time was 2:1; h 0–8, k −11–11, l −8–8. 1900 reflections were measured of which 1844 were unique and 891 with $I > 3\sigma(I)$ were used for the analysis. Three reference reflections monitored every 100 reflections showed no crystal deterioration. Lorentz, polarization and absorption (maximum and minimum transmission factors 0.87, 1.11) corrections were applied. The structure was solved by direct methods with *MITHRIL* (Gilmore, 1984) and *DIRDIF* (Beurskens, 1984), and refined by least squares with anisotropic thermal parameters for all non-H atoms; H atoms were located from a

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

$B_{eq} = (B_{11}a^2 + B_{22}b^2 + B_{33}c^2 + B_{13}ac\cos\beta)$.

	x	y	z	B_{eq}
Cl(1)	0.7885 (5)	0.0854 (3)	0.2555 (4)	4.2 (1)
O(1)	0.447 (1)	0.7971 (6)	0.274 (1)	4.8 (4)
O(2)	0.685 (1)	0.6431 (6)	0.3247 (8)	4.1 (4)
O(3)	0.900 (1)	0.055 (1)	0.140 (1)	14 (1)
O(4)	0.751 (2)	−0.007 (1)	0.362 (2)	23 (1)
O(5)	0.836 (2)	0.197 (1)	0.353 (2)	16 (1)
O(6)	0.637 (2)	0.080 (1)	0.142 (2)	12 (1)
N(1)	0.255 (2)	0.0181 (9)	0.241 (2)	4.4 (6)
C(1)	0.337 (1)	0.3897 (8)	0.164 (1)	3.1 (5)
C(2)	0.493 (2)	0.4423 (9)	0.228 (1)	3.0 (5)
C(3)	0.531 (1)	0.5775 (8)	0.264 (1)	2.8 (5)
C(4)	0.411 (2)	0.6617 (9)	0.241 (1)	3.1 (5)
C(5)	0.254 (2)	0.607 (1)	0.180 (2)	4.7 (7)
C(6)	0.211 (2)	0.474 (1)	0.141 (1)	3.5 (5)
C(7)	0.300 (2)	0.241 (1)	0.123 (1)	4.0 (6)
C(8)	0.291 (2)	0.1639 (9)	0.288 (1)	3.5 (5)
C(9)	0.815 (2)	0.565 (2)	0.368 (2)	5.0 (7)

Table 2. *Bond lengths* (Å), *bond angles* (°) *and hydrogen-bond geometry* (Å)

Cl(1)—O(3)	1.339 (9)	C(1)—C(2)	1.36 (1)
Cl(1)—O(4)	1.27 (1)	C(1)—C(6)	1.40 (1)
Cl(1)—O(5)	1.326 (9)	C(1)—C(7)	1.51 (1)
Cl(1)—O(6)	1.43 (1)	C(2)—C(3)	1.37 (1)
O(1)—C(4)	1.37 (1)	C(3)—C(4)	1.37 (1)
O(2)—C(3)	1.38 (1)	C(4)—C(5)	1.36 (2)
O(2)—C(9)	1.41 (1)	C(5)—C(6)	1.37 (2)
N(1)—C(8)	1.49 (1)	C(7)—C(8)	1.49 (1)

O(3)—Cl(1)—O(4)	113 (1)	O(2)—C(3)—C(2)	126.5 (9)
O(3)—Cl(1)—O(5)	113.8 (7)	O(2)—C(3)—C(4)	112.9 (9)
O(3)—Cl(1)—O(6)	102.7 (6)	C(2)—C(3)—C(4)	121 (1)
O(4)—Cl(1)—O(5)	108.1 (9)	O(1)—C(4)—C(3)	122 (1)
O(4)—Cl(1)—O(6)	102 (1)	O(1)—C(4)—C(5)	121 (1)
O(5)—Cl(1)—O(6)	117.0 (8)	C(3)—C(4)—C(5)	118 (1)
C(3)—O(2)—C(9)	118.0 (9)	C(4)—C(5)—C(6)	124 (1)
C(2)—C(1)—C(6)	119.5 (9)	C(1)—C(6)—C(5)	117 (1)
C(2)—C(1)—C(7)	120 (1)	C(1)—C(7)—C(8)	112.7 (8)
C(6)—C(1)—C(7)	121 (1)	N(1)—C(8)—C(7)	110.7 (9)
C(1)—C(2)—C(3)	121.0 (9)		

D	*A*	Symmetry	*D*···*A*
O(1)	O(2)	(i)	2.62 (1)
N(1)	O(1)	(ii)	2.87 (1)
N(1)	O(3)	(iii)	2.98 (2)
N(1)	O(4)	(iv)	2.98 (2)

Symmetry code: (i) x, y, z; (ii) $x, -1+y, z$; (iii) $-1+x, y, z$; (iv) $-1-x, -y, -1-z$.

0108-2701/92/091698-02$06.00

difference Fourier map and included in the refinement with isotropic thermal parameters. $\sum w(|F_o| - |F_c|)^2$ was minimized, $w = 4F_o^2/\sigma^2(F_o^2)$. Number of parameters refined = 214; final $R = 0.068$, $wR = 0.084$; $(\Delta/\sigma)_{max} = 0.05$, $S = 2.60$. The maximum and minimum peaks in the final difference Fourier map were 0.41 and -0.31 e Å^{-3}. Atomic scattering factors and anomalous-dispersion corrections were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). All numerical calculations were performed using the *TEXSAN* crystallographic software package of the Molecular Structure Corporation (1985). Final atomic parameters of non-H atoms are listed in Table 1; selected bond lengths, angles and hydrogen bonds are listed in Table 2.* A perspective view of 3-methoxytyramine perchlorate is shown in Fig. 1 with the atomic numbering scheme.

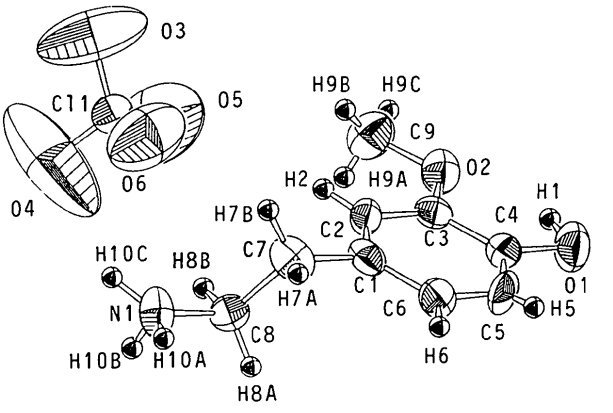

Fig. 1. Perspective view of 3-methoxytyramine perchlorate with the atomic numbering used.

Related literature. 3-Methoxytyramine is the first metabolic product of dopamine; the crystal structure of the hydrochloride has been reported (Okabe, Mori & Sasaki, 1991). The amino side-chain orientation resembles those of catecholamines, all of which have the side chains oriented perpendicular to the phenyl ring plane [dopamine hydrochloride (Giesecke, 1980); adrenaline (Andersen, 1975); noradrenaline hydrochloride (Carlström & Bergin, 1967)], but differs from that of 3-methoxytyramine hydrochloride in which the side chain lies in the same plane as the phenyl ring.

* 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 55106 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS0581]

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Structure of an Acetone Solvate of 7,9-Diacetyl-2,5-dinitro-2,5,7,9-tetraazabicyclo[4.3.0]nonane

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Abstract. $C_9H_{14}N_6O_6.\frac{1}{2}C_3H_6O$, $M_r = 331.29$, monoclinic, $C2/c$, $a = 29.512$ (5), $b = 9.142$ (1), $c = 10.872$ (2) Å, $\beta = 92.46$ (1)°, $V = 2930.5$ (8) Å^3, $Z = 8$, $D_x = 1.502$ Mg m^{-3}, λ(Cu $K\alpha$) = 1.54184 Å, $\mu = 1.04$ mm^{-1}, $F(000) = 1392$, $T = 295$ K, final $R = 0.043$, $wR = 0.047$ for 1708 independent observed reflections. Torsions and bends have destroyed the symmetry of this symmetrically substituted heterocycle. The torsion angles of the six-membered ring indicate a very distorted boat conformation, with the two N atoms at the bow and stern positions of the boat. The nitro-substituted N atoms are substantially pyramidalized, with bend angles between the N—N bonds and the adjacent CNC planes of 17.3 and 38.5°. The atoms of the five-membered ring fit a flattened envelope conformation, with an N atom

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