C10N	0.3204 (3)	−0.3140 (3)	1.332 (1)	0.049 (2)
C11	0.4966 (3)	0.1870 (3)	1.0184 (9)	0.045 (2)
C12	0.4813 (3)	0.2607 (3)	1.0988 (10)	0.050 (2)
C13	0.4620 (3)	0.3081 (3)	0.9151 (9)	0.040 (2)
C14	0.3994 (2)	0.2767 (3)	0.7914 (9)	0.034 (2)
C15	0.3741 (3)	0.3365 (3)	0.6497 (9)	0.047 (2)
C16	0.3811 (3)	0.4012 (3)	0.795 (1)	0.058 (2)
C17	0.4332 (4)	0.3786 (3)	0.973 (1)	0.054 (2)
C18	0.5166 (3)	0.0767 (3)	0.660 (1)	0.055 (2)
C19	0.5275 (3)	0.3232 (3)	0.776 (1)	0.060 (2)

Table 4. *Selected geometric parameters* (Å) *for* BENA

O1—C3	1.234 (7)	C2—C3	1.459 (9)
O2—C17	1.201 (8)	C3—C4	1.464 (9)
O1N—C2N	1.333 (8)	C4—C5	1.328 (8)
C1—C2	1.324 (8)	C5—C6	1.501 (8)
C1—C10	1.511 (8)		

For both compounds, data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1992). Program(s) used to solve structures: *SHELXS*86 (Sheldrick, 1990) for ALNA; *SIR*88 (Burla *et al.*, 1989) for BENA. For both compounds, program(s) used to refine structures: *TEXSAN LS*; software used to prepare material for publication: *TEXSAN FINISH*.

Thanks are due to Chinoin Pharmaceuticals for supporting the project with manpower and instrumentation. The authors are grateful to Professor A. Kálmán for helpful discussions on problems of isostructurality.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, bond distances and angles involving non-H atoms for ALNA and complete geometry for BENA have been deposited with the IUCr (Reference: KA1098). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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DL-4-Hydroxy-3-methoxymandelic Acid

NOBUO OKABE, TAMAMI SUGA AND YOSHIKO KOHYAMA

Faculty of Pharmaceutical Sciences, Kinki University, Kowakae 3-4-1, Higashi Osaka, Osaka 577, Japan

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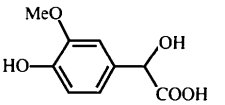
Abstract

In the title compound, DL-2-(4-hydroxy-3-methoxyphenyl)-2-hydroxyacetic acid, $C_9H_{10}O_5$, the acetic acid side chain adopts a roughly perpendicular orientation with respect to the phenyl ring. The molecules are linked together through hydrogen bonds of the type O—H···O.

Comment

The crystal structures of the catecholamines dopamine (Bergin & Carlström, 1968; Giesecke, 1980), adrenaline (Andersen, 1975*a*), noradrenaline (Andersen, 1975*b*; Carlström & Bergin, 1967) and their analogues (Barlow, Johnson, Howard, Walton & Koellner, 1989; Seiler, Markstein, Walkinshaw & Boelsterli, 1989) have been determined. It is also important to clarify the detailed structure of catecholamine metabolites in order to study catecholamine action as well as metabolism. In this respect the structures of the dopamine metabolites 3-methoxytyramine (Okabe, Mori & Sasaki, 1991; Okabe & Mori, 1992) and homovanillic acid (Okabe, Hatanaka & Sasaki, 1991), and the noradrenaline metabolite normetanephrine (Pattanayek, Dattagupta, Bhattacharyya & Saha, 1984) have been reported. We report here the crystal structure of the title compound, (I), which is the principal metabolite of adrenaline and noradrenaline (Grodsky, 1983).



(I)

The acetic acid side chain is oriented roughly perpendicularly to the phenyl ring [torsion angle C(2)—C(1)—C(7)—C(8) −63.8 (2)°]. This conformational feature of the molecule resembles that observed for catecholamines and the corresponding amines (Barlow, Johnson, Howard, Walton & Koellner, 1989) as well as the catecholamine metabolites normetanephrine (Pattanayek, Dattagupta, Bhattacharyya & Saha, 1984), homovanillic acid (Okabe, Hatanaka & Sasaki, 1991) and 3-methoxytyramine (Okabe & Mori, 1992). Two hydroxyl groups and the carboxyl group participate

in hydrogen bonding: O(7)···O(4)($\frac{1}{2}-x$, $\frac{1}{2}+y$, $\frac{3}{2}-z$) 2.761 (2), O(4)···O(82)($1-x$, $-y$, $2-z$) 2.931 (2) and O(81)···O(7)($\frac{1}{2}-x$, $-\frac{1}{2}+y$, $\frac{5}{2}-z$) 2.718 (2) Å.

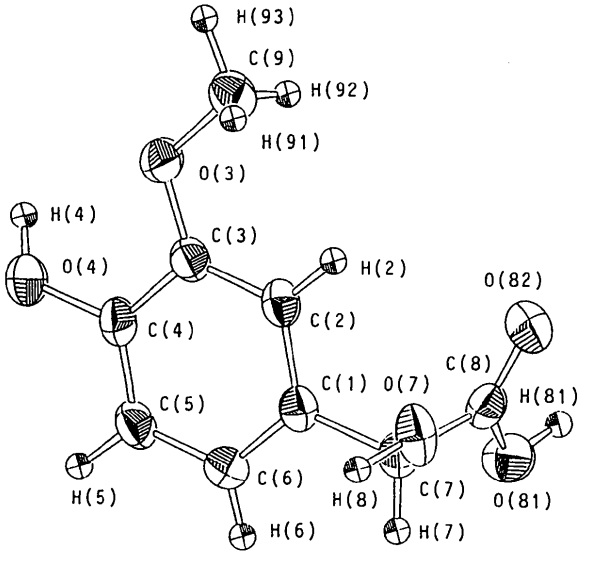


Fig. 1. Perspective view of the title compound with the atomic numbering scheme. Displacement ellipsoids are shown at the 50% probability level and H atoms are drawn as spheres of arbitrary size. The molecule is viewed down the *b* axis.

Experimental

Crystals were obtained by evaporation from 70% ethanol. The density D_m was measured by flotation in CCl_4/C_6H_6.

Crystal data

$C_9H_{10}O_5$
$M_r = 198.18$
Monoclinic
$P2_1/n$
$a = 10.086\ (3)$ Å
$b = 8.829\ (2)$ Å
$c = 10.503\ (2)$ Å
$\beta = 103.43\ (2)°$
$V = 909.6\ (4)$ Å^3
$Z = 4$
$D_x = 1.447$ Mg m^{-3}
$D_m = 1.439\ (3)$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 15.25$–$19.30°$
$\mu = 0.112$ mm^{-1}
$T = 296$ K
Prism
0.30 × 0.30 × 0.20 mm
Colorless

Data collection

Rigaku AFC-5*R* diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2349 measured reflections
2229 independent reflections
1553 observed reflections [$I > 1.5\sigma(I)$]
$R_{int} = 0.021$
$\theta_{max} = 27.5°$
$h = 0 \rightarrow 13$
$k = 0 \rightarrow 11$
$l = -13 \rightarrow 13$
3 standard reflections monitored every 150 reflections
frequency: 60 min
intensity decay: 0.40%

Refinement

Refinement on F
$R = 0.047$
$wR = 0.054$
$S = 1.71$
1553 reflections
127 parameters
H-atom parameters not refined
$w = 4F_o^2/\sigma^2(F_o^2)$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.22$ e Å^{-3}
$\Delta\rho_{min} = -0.29$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	B_{eq}
O(3)	0.5745 (1)	0.1564 (2)	0.8562 (1)	3.48 (6)
O(4)	0.4195 (2)	0.0196 (2)	0.6483 (1)	3.98 (7)
O(7)	0.2028 (2)	0.3758 (2)	1.0824 (1)	3.62 (6)
O(81)	0.1685 (1)	0.0097 (2)	1.1893 (1)	3.62 (7)
O(82)	0.3408 (2)	0.1719 (2)	1.2553 (1)	3.45 (6)
C(1)	0.2410 (2)	0.1644 (2)	0.9448 (2)	2.55 (7)
C(2)	0.3802 (2)	0.1871 (2)	0.9560 (2)	2.61 (7)
C(3)	0.4401 (2)	0.1391 (2)	0.8576 (2)	2.61 (7)
C(4)	0.3617 (2)	0.0654 (2)	0.7485 (2)	2.81 (8)
C(5)	0.2251 (2)	0.0407 (3)	0.7385 (2)	3.11 (8)
C(6)	0.1646 (2)	0.0920 (3)	0.8366 (2)	3.07 (8)
C(7)	0.1760 (2)	0.2198 (2)	1.0537 (2)	2.75 (8)
C(8)	0.2383 (2)	0.1336 (2)	1.1786 (2)	2.67 (8)
C(9)	0.6599 (2)	0.2301 (3)	0.9668 (2)	3.7 (1)

Table 2. *Selected geometric parameters* (Å, °)

O(3)—C(3)	1.368 (2)	C(1)—C(2)	1.397 (3)
O(3)—C(9)	1.432 (2)	C(1)—C(6)	1.374 (3)
O(4)—C(4)	1.377 (2)	C(1)—C(7)	1.524 (3)
O(7)—C(7)	1.422 (3)	C(2)—C(3)	1.379 (3)
O(81)—C(8)	1.320 (2)	C(3)—C(4)	1.393 (3)
O(82)—C(8)	1.203 (2)	C(4)—C(5)	1.375 (3)
C(7)—C(8)	1.521 (3)	C(5)—C(6)	1.389 (3)
C(3)—O(3)—C(9)	116.9 (2)	C(3)—C(4)—C(5)	120.3 (2)
C(2)—C(1)—C(6)	119.7 (2)	C(4)—C(5)—C(6)	119.8 (2)
C(2)—C(1)—C(7)	119.3 (2)	C(1)—C(6)—C(5)	120.5 (2)
C(6)—C(1)—C(7)	121.0 (2)	O(7)—C(7)—C(1)	112.1 (2)
C(1)—C(2)—C(3)	120.0 (2)	O(7)—C(7)—C(8)	106.4 (2)
O(3)—C(3)—C(2)	125.5 (2)	C(1)—C(7)—C(8)	108.8 (2)
O(3)—C(3)—C(4)	114.7 (2)	O(81)—C(8)—O(82)	124.7 (2)
C(2)—C(3)—C(4)	119.8 (2)	O(81)—C(8)—C(7)	111.5 (2)
O(4)—C(4)—C(3)	120.4 (2)	O(82)—C(8)—C(7)	123.8 (2)
O(4)—C(4)—C(5)	119.3 (2)		

Data collection and cell refinement: *Rigaku MSC/AFC Data Collection and Refinement Software* (Rigaku Corporation, 1988). Data reduction: *TEXSAN* (Molecular Structure Corporation, 1985). Structure solution: *SHELXS86* (Sheldrick, 1985) and *DIRDIF* (Beurskens, 1984). Structure refinement and molecular graphics: *TEXSAN*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, including torsion angles, have been deposited with the IUCr (Reference: AS1143). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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3-(*p*-Hydroxyphenyl)propionic Acid

NOBUO OKABE AND TAMAMI SUGA

Faculty of Pharmaceutical Sciences, Kinki University, Kowakae 3-4-1, Higashiosaka, Osaka 577, Japan

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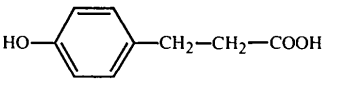
Abstract

The title compound, $C_9H_{10}O_3$, has a fully extended propionic side chain in a *trans* configuration; the plane of the side chain is almost perpendicular to the phenyl-ring plane. The molecules are held together by two kinds of hydrogen bonds between hydroxyl groups and between carboxyl groups.

Comment

p-Hydroxyphenylpropionic acid, (I), is well known as one of the intermediates of tyrosine metabolites such as *p*-hydroxyphenyl pyruvic acid, *p*-hydroxyphenyllactic acid, *p*-hydroxylphenylacrylic acid or *p*-hydroxyphenylacetic acid. The excretion of *p*-hydroxyphenylpropionic acid increases in patients with gastrointestinal diseases such as cystic fibrosis, coeliac disease or intestinal resection (van der Heiden, Wauters, Ketting, Duran & Wadman, 1971). On the other hand, *p*-hydroxyphenylpropionic acid inhibits peptic hydrolysis by pepsin (Schlamowitz, Shaw & Jackson, 1968). It also binds strongly to peroxidases which catalyse the oxidation of a large number of organic substances (Casella *et al.*, 1991). The present study was performed to find basic conformational features of the title compound for further investigation of its physiological function.



(I)

The molecule has a fully extended propionic side chain in a *trans* configuration [C(1)—C(7)—C(8)—C(9) = 177.8 (2)°]. The plane of the side chain is almost perpendicular to the phenyl plane [C(6)—C(1)—C(7)—C(8) = 112.6 (2)°]. Molecules are held together by two kinds of O—H···O intermolecular hydrogen bonds between two hydroxyl groups and between two carboxyl groups: O(4)—H(4)···O(4)(1 − *x*, 1 − *y*, 2 − *z*) 2.927 (3); O(91)—H(91)···O(92)(2 − *x*, 1 − *y*, 1 − *z*) 2.662 (2) Å.

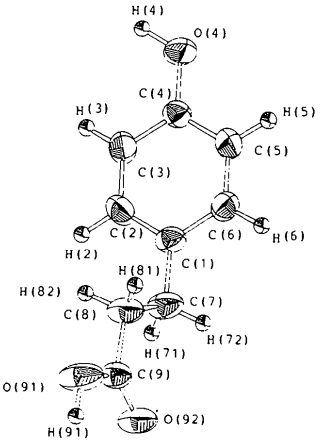


Fig. 1. Perspective view of the title compound with the atomic numbering. Ellipsoids for non-H atoms correspond to 50% probability.

Experimental

Crystal data

$C_9H_{10}O_3$
M_r = 166.18
Monoclinic
$P2_1/c$
a = 11.356 (2) Å
b = 5.358 (1) Å
c = 14.122 (2) Å
β = 105.94°
V = 826.3 (3) Å^3
Z = 4
D_x = 1.336 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 25 reflections
θ = 22.6–24.75°
μ = 0.094 mm^{-1}
T = 296 K
Needle
0.40 × 0.20 × 0.10 mm
Colourless
Crystal source: evaporation from water