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3-(*p*-Hydroxyphenyl)propionic Acid

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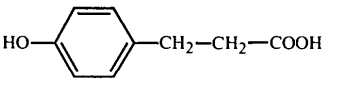
Abstract

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.

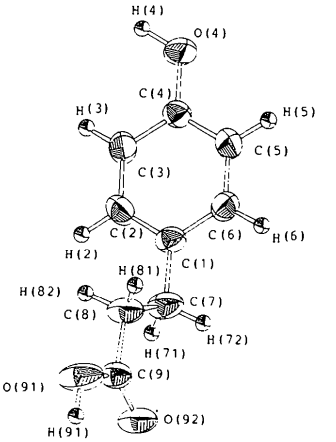


Fig. 1. Perspective view of the title compound with the atomic numbering. Ellipsoids for non-H atoms correspond to 50% probability.

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

Data collection

Rigaku AFC-5*R* diffractometer	$\theta_{max} = 27.5°$
$\omega/2\theta$ scans	$h = 0 \rightarrow 14$
Absorption correction: none	$k = 0 \rightarrow 6$
2197 measured reflections	$l = -18 \rightarrow 16$
2099 independent reflections	3 standard reflections monitored every 150 reflections
1436 observed reflections $[I > 2\sigma(I)]$	frequency: 100 min
$R_{int} = 0.011$	intensity decay: 0.2%

Refinement

Refinement on F	$(\Delta/\sigma)_{max} = 0.007$
$R = 0.050$	$\Delta\rho_{max} = 0.25$ e Å^{-3}
$wR = 0.063$	$\Delta\rho_{min} = -0.54$ e Å^{-3}
$S = 2.44$	Extinction correction: none
1436 reflections	Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)
110 parameters	
H-atom parameters not refined	
$w = 4F_o^2/\sigma^2(F_o^2)$	

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(4)	0.5150 (1)	0.2463 (3)	0.9667 (1)	4.41 (6)
O(91)	0.8791 (1)	0.6570 (3)	0.5324 (1)	5.56 (8)
O(92)	0.9793 (1)	0.3118 (3)	0.5893 (1)	4.45 (6)
C(1)	0.7515 (2)	0.2227 (4)	0.7873 (1)	3.27 (7)
C(2)	0.7621 (2)	0.4047 (4)	0.8581 (1)	3.93 (8)
C(3)	0.6842 (2)	0.4147 (4)	0.9180 (1)	3.76 (8)
C(4)	0.5936 (2)	0.2381 (3)	0.9068 (1)	3.04 (7)
C(5)	0.5801 (2)	0.0538 (4)	0.8370 (1)	3.51 (7)
C(6)	0.6593 (2)	0.0477 (3)	0.7777 (1)	3.55 (7)
C(7)	0.8353 (2)	0.2222 (4)	0.7207 (2)	4.37 (9)
C(8)	0.8163 (2)	0.4444 (4)	0.6544 (1)	3.91 (8)
C(9)	0.8995 (2)	0.4622 (4)	0.5886 (1)	3.27 (7)

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

O(4)—C(4)	1.389 (2)	C(2)—C(3)	1.383 (3)
O(91)—C(9)	1.294 (2)	C(3)—C(4)	1.375 (3)
O(92)—C(9)	1.211 (2)	C(4)—C(5)	1.374 (3)
C(1)—C(2)	1.378 (3)	C(5)—C(6)	1.388 (3)
C(1)—C(6)	1.384 (3)	C(7)—C(8)	1.494 (3)
C(1)—C(7)	1.511 (3)	C(8)—C(9)	1.499 (2)
C(2)—C(1)—C(6)	117.7 (2)	C(4)—C(5)—C(6)	119.2 (2)
C(2)—C(1)—C(7)	120.5 (2)	C(1)—C(6)—C(5)	121.5 (2)
C(6)—C(1)—C(7)	121.8 (2)	C(1)—C(7)—C(8)	112.4 (2)
C(1)—C(2)—C(3)	121.9 (2)	C(7)—C(8)—C(9)	115.6 (2)
C(2)—C(3)—C(4)	119.2 (2)	O(91)—C(9)—O(92)	123.4 (2)
O(4)—C(4)—C(3)	119.6 (2)	O(91)—C(9)—C(8)	113.2 (2)
O(4)—C(4)—C(5)	119.7 (2)	O(92)—C(9)—C(8)	123.4 (2)
C(3)—C(4)—C(5)	120.7 (2)		

Data collection and cell refinement: *MSC/AFC Data Collection and Refinement Software* (Rigaku Corporation, 1988). Data reduction: *TEXSAN* (Molecular Structure Corporation, 1985). Programs used to solve structure: *MITHRIL* (Gilmore, 1984) and *DIRDIF* (Beurskens, 1984). Program used to refine structure: *TEXSAN*. Molecular graphics: *ORTEP*II (Johnson, 1976).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, torsion angles and bond distances and angles involving H atoms have been deposited with the IUCr (Reference: AS1155). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Ethyl Pentaspiro[2.0.2.0.0.2.0.2.0.1]tetradeca-14-ylideneacetate, $C_{18}H_{22}O_2$

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Abstract

The title molecule has a *Z* configuration at the exocyclic C=C double bond. The four-membered ring and the COOEt group are almost coplanar. The unusual distribution of bond lengths in the polycyclic system is a result of the electron-withdrawing effect of the COOEt group.

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