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2-Hydroxy-3-(1*H*-indol-3-yl)propenoic Acid

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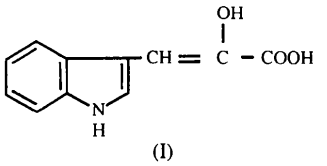
Abstract

The title compound, $C_{11}H_9NO_3$, has a fully extended propenoic side chain. The molecules are held together by two types of intermolecular hydrogen bonds between the carboxy groups and between the enol and carboxy groups. The O···O distances of the former and the latter hydrogen bonds are 2.760 (3) and 2.727 (3) Å, respectively.

Comment

3-(Indol-3-yl)-2-oxopropanoic acid (indolepyruvic acid) is a well known endogenous metabolite of tryptophan, deaminated by tryptophan aminotransferase. It has anxiolytic activity in mice similar to that of diazepam (Lapin & Politi, 1993) and is known to regulate the modulation of brain kynurenic acid content (Moroni *et al.*, 1991). Patients with phenylketonuria excrete increased quantities of indole acetate and indole lactate, formed by reduction of indolepyruvic acid (Martin *et al.*, 1983). Therefore, it is important to elucidate its accurate structure and conformation for physicochemical understanding of its metabolic pathway. Accordingly, we aimed to determine the crystal structure of indolepyruvic acid and revealed it as its enol tautomer, (I).



(I)

The molecular structure of (I) is shown in Fig. 1, with the unit-cell packing is shown in Fig. 2. The structural formula of indolepyruvic acid is usually given in its ketonic form. In contrast, the crystal structure determination in this study shows (I) to be the enol tautomer. As a result of tautomerism, the π electron in the double bond of the keto group is transferred to C10=C11 [1.325 (4) Å]. The indole ring and the carboxy group are *trans* to each other about the C10=C11 double bond. The three torsion angles O1—C11—C10—C3 −1.1 (4), C3—C10—C11—C12 179.4 (3) and C10—C11—C12—O3 −11.1 (4)° indicate a nearly planar and fully extended conformation.

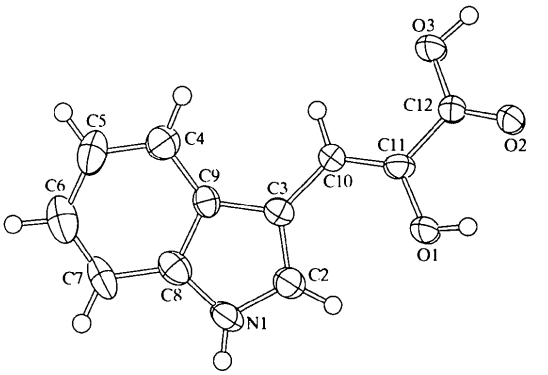


Fig. 1. *ORTEP*II (Johnson, 1976) drawing of the title compound with the atomic numbering scheme. Ellipsoids for non-H atoms correspond to 50% probability.

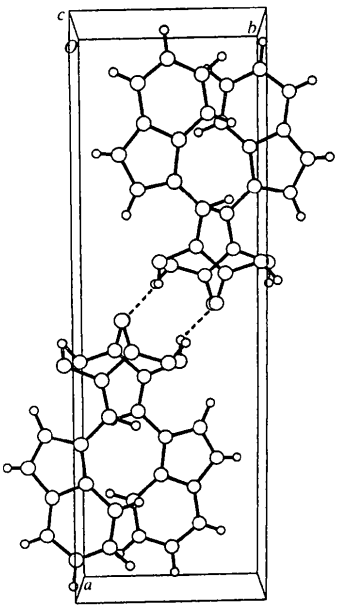


Fig. 2. Packing diagram of the title compound viewed approximately down the *a* axis.

At the present time, no structural information is available about the enzymatic recognition scheme of indolepyruvic acid by lactate dehydrogenase (1.1.1.27) for producing the further metabolite, indolelactic acid. The planar conformation of the enolic form of indolepyruvic acid determined in this study may be considered as an enzymatically recognizable form.

No stacking interaction between indole rings is observed. The crystal structure is stabilized by intermolecular hydrogen bonds between carboxy groups and between the hydroxy and carboxy groups. The N—H group in the indole ring does not participate in hydrogen bonding.

Experimental

The very thin brown plate crystal used for analysis was obtained by the slow evaporation of an ethanol solution of the title compound at room temperature.

Crystal data

$C_{11}H_9NO_3$
M_r = 203.20
Monoclinic
$P2_1/c$
a = 20.074 (2) Å
b = 6.488 (3) Å
c = 7.180 (4) Å
β = 96.68 (2)°
V = 928.8 (9) Å^3
Z = 4
D_x = 1.453 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 25 reflections
θ = 15.8–19.5°
μ = 0.100 mm^{-1}
T = 296 K
Plate
0.5 × 0.5 × 0.1 mm
Brown

Data collection

Rigaku AFC-5R diffractometer
ω–2θ scans
Absorption correction: none
2513 measured reflections
2334 independent reflections
1376 reflections with $I > \sigma(I)$

R_{int} = 0.059
θ_{max} = 27.5°
$h = -25 \rightarrow 25$
$k = -8 \rightarrow 0$
$l = 0 \rightarrow 9$
3 standard reflections
every 150 reflections
intensity decay: none

Refinement

Refinement on F^2
$R(F)$ = 0.059
$wR(F^2)$ = 0.120
S = 1.66
1376 reflections
148 parameters
H atoms: see below
$w = 4F_o^2/\sigma^2(F_o^2)$

$(\Delta/\sigma)_{max}$ = 0.013
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. *Selected geometric parameters (Å, °)*

O1—C11	1.368 (3)	C3—C10	1.453 (4)
O2—C12	1.213 (3)	C10—C11	1.325 (4)
O3—C12	1.317 (3)	C11—C12	1.477 (4)
C2—C3—C10	126.5 (3)	O1—C11—C10	121.5 (3)
C9—C3—C10	126.6 (3)	C10—C11—C12	125.3 (3)
C3—C10—C11	126.3 (3)	O2—C12—O3	123.6 (3)

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O2^{i}	0.96 (4)	1.80 (4)	2.760 (3)	175 (3)
O1—H11···O2^{ii}	0.88 (3)	1.97 (4)	2.727 (3)	143 (3)

Symmetry codes: (i) $1-x, -1-y, -z$; (ii) $1-x, -y, -z$.

All H atoms were located from difference Fourier maps. Only the H atoms of the O—H and N—H groups were refined isotropically because of the low data/parameter ratio; others were fixed at calculated positions.

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

Supplementary data for this paper are available from the IUCr electronic archives (Reference: JZ1267). Services for accessing these data are described at the back of the journal.

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N—H···N Hydrogen Bonding in the Four Independent Molecules of (2*S*,4*S*,5*R*)-(−)-2-(1*H*-Imidazol-2-yl)-3,4-dimethyl-5-phenyl-1,3-oxazolidine, with C—H···π_{arene}, C—H···O and C—H···$\pi_{C=C}$ Interactions

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Abstract

The title compound, $C_{14}H_{17}N_3O$, prepared from (1*R*,2*S*)-(−)-ephedrine, crystallizes in space group $P2_1$ with four molecules in the asymmetric unit. The molecules, in pairs, take part in intermolecular N—H···N hydrogen