Supplementary data for this paper are available from the IUCr electronic archives (Reference: AB1514). Services for accessing these data are described at the back of the journal.

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1*H*-Indole-3-propionic Acid

NOBUO OKABE AND YUKARI ADACHI

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

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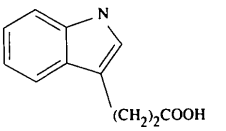
Abstract

The title compound, $C_{11}H_{11}NO_2$, has a fully extended propionic acid side chain. The molecules are held together as dimers by centrosymmetric pairs of intermolecular hydrogen bonds between the carboxyl groups. The O···O distance is 2.649 (2) Å.

Comment

The title compound, (I), has antibacterial activity against *Pseudomonas solanacearum* which causes a serious disease of many crop plants belonging to the family Solanaceae (Toyoda *et al.*, 1991; Matsuda *et al.*, 1993). It is also known as a hormone-type plant growth regulator like indole-3-acetic acid (Fargasova, 1994). Until now, its functional mechanism was unclear. Therefore, it is important to clarify its structure and conformation in order to investigate its function. Accordingly, the precise crystal structure of the title compound has been redetermined; its original structure determination was reported from film data, with an *R* value of 0.16 (Lahiri *et al.*, 1978). The non-standard space group $P2_1/a$ was chosen to agree with the original determination, which had unit-cell dimensions a = 12.38, b = 5.25, c = 14.39 Å and β = 95°.



(I)

The molecular structure of the title compound with the atomic labeling is shown in Fig. 1. The indole ring and the carboxyl group are in an *anti* conformation. The torsion angles, O(1)—C(12)—C(11)—C(10) −179.1 (1) and C(9)—C(3)—C(10)—C(11) −178.1 (2)°, indicate that the molecule has a nearly planar fully extended conformation. It may be important to take the planar conformation of indole-3-propionic acid into consideration for investigating the mechanism of the antibacterial or plant growth regulatory action. No stacking interactions between indole rings are observed. The crystal structure is stabilized by van der Waals interactions and hydrogen bonds between carboxyl groups, O(1)—H(3)···O(2)(1 − x, −y, −z) 2.649 (2) Å.

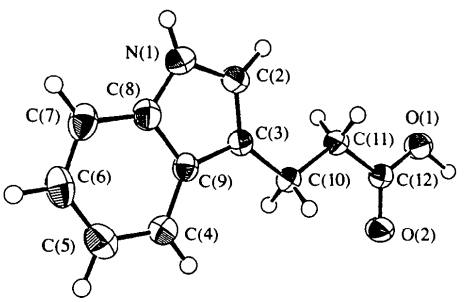


Fig. 1. *ORTEP*II (Johnson, 1976) drawing of the title compound with the atomic numbering scheme. Ellipsoids for non-H atoms correspond to 50% probability.

Experimental

The colorless pillar-shaped crystal used for analysis was obtained by the slow evaporation of a 90% ethanol solution of the title compound at room temperature.

Crystal data

$C_{11}H_{11}NO_2$
M_r = 189.21
Monoclinic
$P2_1/a$
a = 12.346 (3) Å
b = 5.244 (3) Å
c = 14.362 (2) Å
β = 94.77 (2)°
V = 926.6 (5) Å^3
Z = 4
D_x = 1.356 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 25 reflections
θ = 20.89–24.11°
μ = 0.088 mm^{-1}
T = 296 K
Pillar
0.4 × 0.3 × 0.2 mm
Colorless

Data collection

Rigaku AFC-5*R* diffractometer	$R_{int} = 0.009$
ω–2θ scans	$\theta_{max} = 27.45°$
Absorption correction: none	$h = 0 \rightarrow 16$
2456 measured reflections	$k = 0 \rightarrow 6$
2353 independent reflections	$l = -18 \rightarrow 18$
1561 reflections with $I > 1.5\sigma(I)$	3 standard reflections every 150 reflections intensity decay: none

Refinement

Refinement on F	$(\Delta/\sigma)_{max} = 0.001$
$R = 0.042$	$\Delta\rho_{max} = 0.15$ e Å^{-3}
$wR = 0.045$	$\Delta\rho_{min} = -0.27$ e Å^{-3}
$S = 1.63$	Extinction correction: none
1561 reflections	Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)
171 parameters	
H atoms refined isotropically	
$w = 4F_o^2/\sigma^2(F_o^2)$	

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

O(1)—C(12)	1.291 (2)	C(3)—C(9)	1.438 (2)
O(2)—C(12)	1.243 (2)	C(3)—C(10)	1.494 (2)
N(1)—C(2)	1.380 (2)	C(10)—C(11)	1.517 (2)
N(1)—C(8)	1.375 (2)	C(11)—C(12)	1.493 (2)
C(2)—C(3)	1.358 (2)		
C(2)—N(1)—C(8)	108.9 (1)	C(3)—C(9)—C(4)	133.1 (2)
N(1)—C(2)—C(3)	110.2 (2)	C(3)—C(9)—C(8)	107.7 (1)
C(2)—C(3)—C(9)	106.1 (1)	C(3)—C(10)—C(11)	113.3 (1)
C(2)—C(3)—C(10)	129.1 (1)	C(10)—C(11)—C(12)	115.1 (1)
C(9)—C(3)—C(10)	124.8 (1)	O(1)—C(12)—O(2)	122.5 (1)
N(1)—C(8)—C(7)	130.7 (2)	O(1)—C(12)—C(11)	113.8 (1)
N(1)—C(8)—C(9)	107.0 (1)	O(2)—C(12)—C(11)	122.3 (1)

All H atoms were located from difference Fourier maps and included in the refinement calculations isotropically. The B_{eq} values of H atoms ranged from 3.4 (4) Å^2 for H(11*B*) to 9.0 (7) Å^2 for H(3), and the C—H bond distances from 0.88 (2) Å for N(1)—H(1) to 1.02 (2) Å for C(10)—H(10*A*).

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: FR1089). Services for accessing these data are described at the back of the journal. A packing diagram has also been deposited.

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The Absolute Configuration of *cis*-(α*S*,1*S*,2*R*)-2-Methyl-1-(α-methylbenzylamino)cyclohexanecarboxamide

FRANZ-JOSEF VOLK,[a] GÜNTER MATTERN,[b] EDGAR WECKERT[b] AND AUGUST W. FRAHM[a]

[a]*Lehrstuhl für Pharmazeutische Chemie, Universität Freiburg, Hermann Herder Straße 9, D-79104 Freiburg, Germany, and* [b]*Institut für Kristallographie, Universität Karlsruhe (TH), Kaiserstraße 12, D-76128 Karlsruhe, Germany. E-mail: edgar.weckert@physik.uni-karlsruhe.de*

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Abstract

The title compound, $C_{16}H_{24}N_2O$, was obtained during our investigations on the asymmetric Strecker synthesis of non-proteinogenous α-quarternary α-amino acids with vicinal chiral centres. The X-ray structure determination was carried out in order to verify the absolute configuration, which was determined *via* experimentally measured triplet phases.

Comment

During our systematic investigations on an asymmetric Strecker synthesis starting from (*RS*)-2-methylcyclohexanone, *S*-(−)-α-methylbenzylamine and trimethylsilyl cyanide, we obtained either kinetically or thermodynamically controlled diastereomeric mixtures of the four feasible 2-methyl-1-(α-methylbenzylamino)cyclohexanecarbonitriles as crucial intermediates (Volk & Frahm, 1996). These were hydrolysed to the corresponding α-amino amide mixtures containing 11–15% of the title compound (I), (Fig. 1), which was isolated by means of column chromatography, followed by Lobar chromatography and preparative HPLC. The complete NMR data set of (I) has been published elsewhere (Volk & Frahm, 1996). Its relative stereochemistry was derived from the heteronuclear ^{13}C–^{1}H coupling between C7 and the protons H21, H61 and H62. Since