Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

5-Chloro-1*H*-indole-3-carboxylic acid

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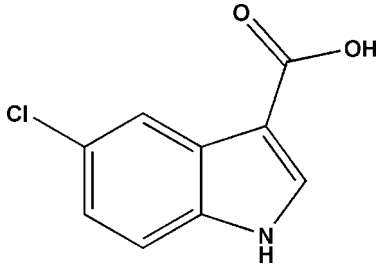
Received 5 December 2011; accepted 12 December 2011

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.037; wR factor = 0.104; data-to-parameter ratio = 15.6.

In the title compound, $C_9H_6ClNO_2$, the carboxyl group is twisted from the indole ring system by 9.00 (8)°. In the crystal, inversion dimers linked by pairs of O—H···O hydrogen bonds generate $R_2^2(8)$ loops and N—H···O hydrogen bonds link the dimers into (001) sheets. Aromatic π–π stacking interactions [centroid–centroid distance = 3.7185 (12) A °] are also observed.

Related literature

For background to indole derivatives as pharmaceuticals, see: Kunzer & Wendt (2011); Woodward & Bartel (2005).



Experimental

Crystal data

$C_9H_6ClNO_2$ M_r = 195.60
Orthorhombic, *Pbca*
a = 7.2934 (15) Å
b = 13.065 (3) Å
c = 17.902 (4) Å
V = 1705.9 (6) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.41 mm^{-1}
T = 293 K
0.30 × 0.20 × 0.15 mm

Data collection

Rigaku SCXmini CCD diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.907, T_{max} = 0.941
16050 measured reflections
1919 independent reflections
1590 reflections with $I > 2\sigma(I)$
R_{int} = 0.050

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.104
S = 1.04
1919 reflections
123 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2^{i}	0.82	1.86	2.6558 (17)	164
N1—H1B···O2^{ii}	0.86 (2)	2.26 (2)	3.005 (2)	144.3 (19)

Symmetry codes: (i) $-x, -y+2, -z+1$; (ii) $-x+\frac{1}{2}, y-\frac{1}{2}, z$.

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2005); software used to prepare material for publication: *SHELXL97*.

We thank Southeast University for support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6559).

References

Brandenburg, K. & Putz, H. (2005). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Kunzer, A. R. & Wendt, M. D. (2011). *Tetrahedron*, **52**, 1815–1818.
Rigaku. (2005). *CrystalClear*. Rigaku Corporation, Tokyo, Japan.
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Woodward, A. W. & Bartel, B. (2005). *Ann. Bot. (London)*, **95**, 707–735.

supporting information

Acta Cryst. (2012). E**68**, o145 [doi:10.1107/S1600536811053384]

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S1. Comment

Indole-3-carboxylic acid and its derivatives are important chemical materials, because they are excellent auxins for plants and drug intermediates for many pharmaceutical products (Woodward, *et al.*,2005, Kunzer, *et al.*,2011). As part of our interest in these materials, we report here the crystal structure of the title compound.

The molecular structure of the title compound is shown in Fig. 1. All the non-H atoms are approximately coplanar: the carboxy O atoms deviating by 0.124 and -0.223 Å from the indole plane.

In the crystal, O—H···O hydrogen bonds linked the molecules into dimers and the dimers packed *via* N—H···O hydrogen bonds and π–π interactions [centroid–centroid distance = 3.7185 (12) A °] (Fig. 2).

S2. Experimental

The title compound was purchased from ChemFuture PharmaTech, Ltd (Nanjing, Jiangsu). Colourless blocks were obstained by slow evaporation of a methanol solution.

S3. Refinement

All H atoms attached to C, N and O atoms were fixed geometrically and treated as riding with C—H = 0.93 Å (CH), O—H = 0.82 Å and N—H = 0.86 Å with U_{iso}(H) = 1.2U_{eq}(CH), U_{iso}(H) = 1.35U_{eq}(N) and U_{iso}(H) = 1.5U_{eq}(O).

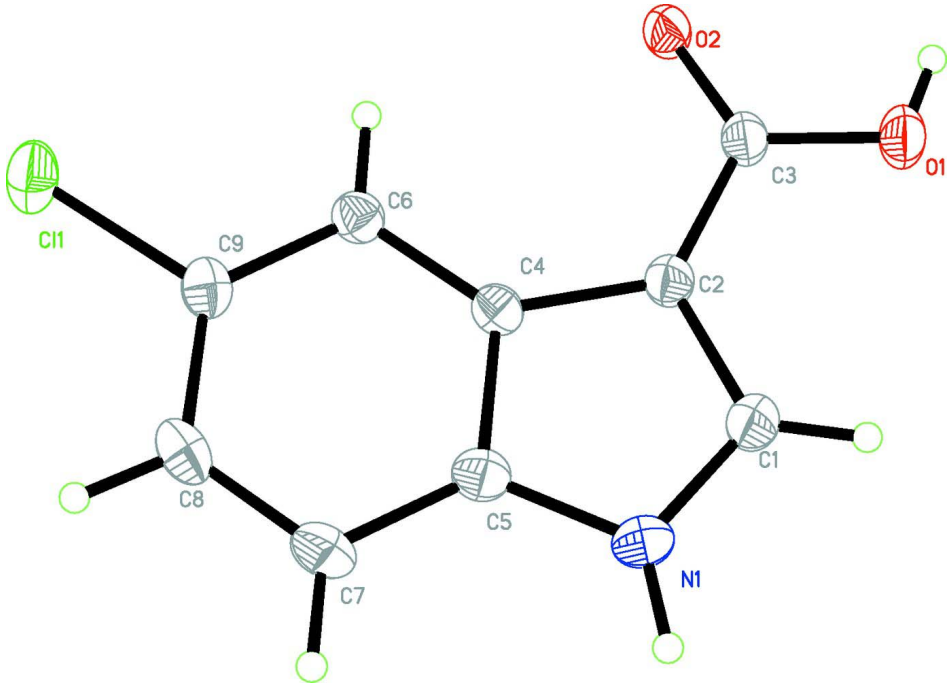

Figure 1

The molecular structure of the title compound with displacement ellipsoids drawn at the 30% probability level.

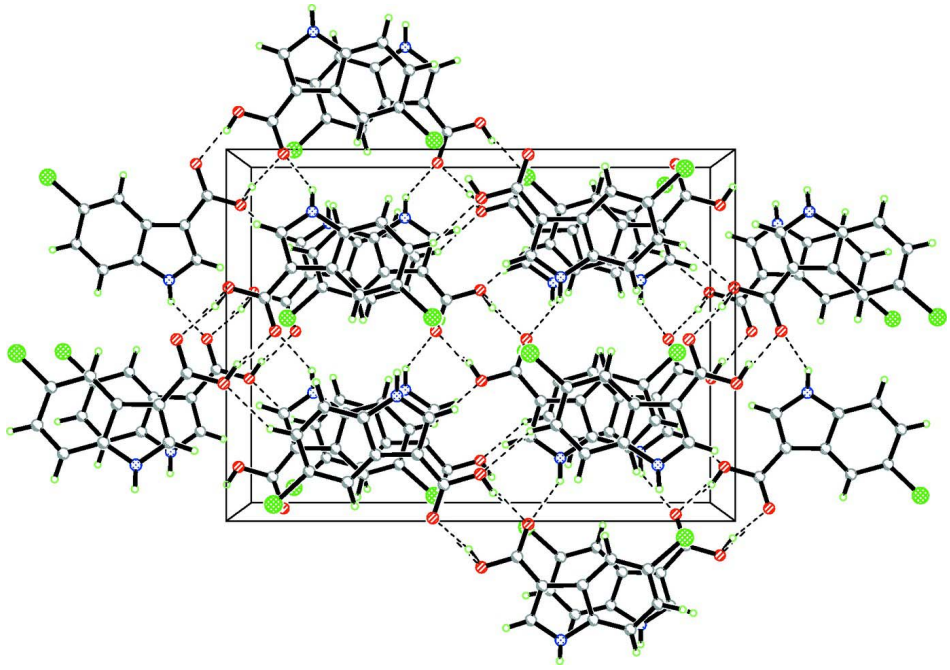

Figure 2

A packing view down the *a* axis showing hydrogen bonds as dashed lines.

5-Chloro-1*H*-indole-3-carboxylic acid

Crystal data

$C_9H_6ClNO_2$
$M_r = 195.60$
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
$a = 7.2934$ (15) Å
$b = 13.065$ (3) Å
$c = 17.902$ (4) Å
$V = 1705.9$ (6) Å^3
$Z = 8$
$F(000) = 800$
$D_x = 1.523$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1919 reflections
$\theta = 3.1$–$27.4°$
$\mu = 0.41$ mm^{-1}
$T = 293$ K
Block, colourless
0.30 × 0.20 × 0.15 mm

Data collection

Rigaku SCXmini CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 13.6612 pixels mm^{-1}
CCD_Profile_fitting scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
$T_{min} = 0.907$, $T_{max} = 0.941$
16050 measured reflections
1919 independent reflections
1590 reflections with $I > 2\sigma(I)$
$R_{int} = 0.050$
$\theta_{max} = 27.4°$, $\theta_{min} = 3.1°$
$h = -9 \rightarrow 9$
$k = -16 \rightarrow 16$
$l = -23 \rightarrow 23$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.104$
$S = 1.04$
1919 reflections
123 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.052P)^2 + 0.5573P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.17$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.07292 (7)	0.95139 (4)	0.09595 (2)	0.05145 (18)
O2	0.05698 (16)	0.98847 (9)	0.40623 (6)	0.0402 (3)
C3	0.0835 (2)	0.89840 (12)	0.42814 (9)	0.0343 (3)

N1	0.2225 (2)	0.65957 (11)	0.33911 (8)	0.0428 (4)
C4	0.1415 (2)	0.81729 (11)	0.29670 (8)	0.0301 (3)
C6	0.1018 (2)	0.89102 (11)	0.24148 (8)	0.0329 (3)
H6	0.0636	0.9566	0.2542	0.040*
O1	0.0656 (2)	0.87130 (10)	0.49869 (6)	0.0568 (4)
H1	0.0422	0.9220	0.5240	0.085*
C5	0.1970 (2)	0.71749 (12)	0.27449 (9)	0.0350 (4)
C2	0.1353 (2)	0.81582 (11)	0.37768 (9)	0.0322 (3)
C9	0.1218 (2)	0.86192 (13)	0.16732 (9)	0.0365 (4)
C7	0.2150 (2)	0.68926 (13)	0.19938 (10)	0.0429 (4)
H7	0.2508	0.6235	0.1860	0.052*
C1	0.1853 (2)	0.71802 (13)	0.39985 (9)	0.0403 (4)
H1A	0.1924	0.6958	0.4491	0.048*
C8	0.1775 (2)	0.76284 (14)	0.14570 (9)	0.0434 (4)
H8	0.1890	0.7468	0.0953	0.052*
H1B	0.265 (3)	0.5983 (18)	0.3404 (11)	0.058 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0648 (3)	0.0559 (3)	0.0337 (2)	−0.0029 (2)	−0.00505 (19)	0.00701 (18)
O2	0.0590 (8)	0.0296 (6)	0.0320 (6)	0.0048 (5)	0.0087 (5)	0.0028 (4)
C3	0.0418 (9)	0.0338 (8)	0.0274 (7)	0.0000 (6)	0.0024 (6)	0.0008 (6)
N1	0.0569 (9)	0.0271 (7)	0.0443 (8)	0.0067 (6)	0.0023 (7)	0.0004 (6)
C4	0.0302 (7)	0.0293 (7)	0.0307 (8)	−0.0027 (6)	0.0013 (6)	−0.0031 (6)
C6	0.0350 (8)	0.0308 (7)	0.0331 (8)	−0.0013 (6)	0.0009 (6)	−0.0020 (6)
O1	0.1032 (12)	0.0388 (7)	0.0285 (6)	0.0165 (7)	0.0126 (7)	0.0045 (5)
C5	0.0353 (8)	0.0309 (8)	0.0387 (8)	−0.0004 (6)	0.0011 (6)	−0.0030 (6)
C2	0.0370 (8)	0.0302 (8)	0.0295 (8)	0.0001 (6)	0.0013 (6)	0.0011 (6)
C9	0.0369 (8)	0.0419 (9)	0.0306 (8)	−0.0046 (7)	−0.0015 (6)	−0.0005 (7)
C7	0.0498 (10)	0.0358 (9)	0.0432 (9)	0.0022 (7)	0.0038 (8)	−0.0122 (7)
C1	0.0526 (10)	0.0346 (8)	0.0338 (8)	0.0037 (7)	0.0013 (7)	0.0027 (6)
C8	0.0493 (10)	0.0496 (10)	0.0314 (8)	−0.0036 (8)	0.0023 (7)	−0.0119 (7)

Geometric parameters (Å, º)

Cl1—C9	1.7681 (17)	C6—C9	1.389 (2)
O2—C3	1.256 (2)	C6—H6	0.9300
C3—O1	1.3181 (19)	O1—H1	0.8200
C3—C2	1.457 (2)	C5—C7	1.400 (2)
N1—C1	1.356 (2)	C2—C1	1.387 (2)
N1—C5	1.395 (2)	C9—C8	1.411 (2)
N1—H1B	0.86 (2)	C7—C8	1.387 (3)
C4—C6	1.410 (2)	C7—H7	0.9300
C4—C5	1.422 (2)	C1—H1A	0.9300
C4—C2	1.451 (2)	C8—H8	0.9300
O2—C3—O1	122.39 (14)	C1—C2—C4	106.86 (13)

O2—C3—C2	122.71 (14)	C1—C2—C3	124.96 (14)
O1—C3—C2	114.89 (14)	C4—C2—C3	128.16 (14)
C1—N1—C5	109.45 (14)	C6—C9—C8	122.94 (15)
C1—N1—H1B	125.1 (14)	C6—C9—Cl1	119.25 (13)
C5—N1—H1B	125.2 (14)	C8—C9—Cl1	117.81 (12)
C6—C4—C5	119.26 (14)	C8—C7—C5	117.67 (15)
C6—C4—C2	134.69 (14)	C8—C7—H7	121.2
C5—C4—C2	106.02 (13)	C5—C7—H7	121.2
C9—C6—C4	117.48 (14)	N1—C1—C2	109.99 (15)
C9—C6—H6	121.3	N1—C1—H1A	125.0
C4—C6—H6	121.3	C2—C1—H1A	125.0
C3—O1—H1	109.5	C7—C8—C9	120.19 (15)
N1—C5—C7	129.87 (15)	C7—C8—H8	119.9
N1—C5—C4	107.67 (14)	C9—C8—H8	119.9
C7—C5—C4	122.45 (15)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2^{i}	0.82	1.86	2.6558 (17)	164
N1—H1*B*···O2^{ii}	0.86 (2)	2.26 (2)	3.005 (2)	144.3 (19)

Symmetry codes: (i) −*x*, −*y*+2, −*z*+1; (ii) −*x*+1/2, *y*−1/2, *z*.