Methyl 3-(1*H*-indol-3-yl)propanoate

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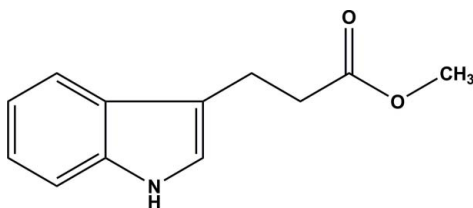Correspondence e-mail: lidongfeng@mail.ccut.edu.cn

The molecule of the title compound, $C_{12}H_{13}NO_2$, adopts an essentially planar conformation (r.m.s. deviation = 0.057 Å). In the crystal, the molecules are linked by intermolecular N—H···O hydrogen bonds, generating chains along [201].

Related literature

For the biological activity of indole derivatives, see: Zeynep *et al.* (2005); Seefeld *et al.* (2003). For details of the synthesis, see: Pedras & Soledade (2006).

Experimental

Crystal data

$C_{12}H_{13}NO_2$	$V = 1060.5$ (13) Å^3
$M_r = 203.23$	$Z = 4$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 6.893$ (5) Å	$\mu = 0.09$ mm^{-1}
$b = 9.146$ (8) Å	$T = 296$ K
$c = 18.052$ (10) Å	$0.46 \times 0.19 \times 0.18$ mm
$\beta = 111.27$ (3)°	

Data collection

Rigaku R-AXIS RAPID diffractometer	10015 measured reflections
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)	2414 independent reflections
$T_{min} = 0.961$, $T_{max} = 0.984$	1509 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.048$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.052$	1 restraint
$wR(F^2) = 0.138$	H-atom parameters constrained
$S = 1.04$	$\Delta\rho_{max} = 0.16$ e Å^{-3}
2414 reflections	$\Delta\rho_{min} = -0.16$ e Å^{-3}
138 parameters	

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^i	0.95	2.08	2.972 (3)	157

Symmetry code: (i) $x + 1, -y + \frac{3}{2}, z + \frac{1}{2}$.

Data collection: *RAPID-AUTO* (Rigaku Corporation, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (MSC & Rigaku, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008) and *DIAMOND* (Brandenburg & Berndt, 2001); software used to prepare material for publication: *SHELXL97*.

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supplementary materials

Methyl 3-(1*H*-indol-3-yl)propanoate

R.-B. Hou and D.-F. Li

Comment

Indole derivatives constitute an important class of therapeutic agents in medicinal chemistry including anticancer, antioxidant, antirheumatoidal and anti-HIV (Zeynep *et al.*, 2005; Seefeld *et al.*, 2003). We have recently synthesized some indole derivatives as histone deacetylase (HDAC) inhibitors with the precursor. In this paper, we report the crystal structure of the title compound.

The molecular structure of tiltle compound, $C_{12}H_{13}O_2N$, as shown in Fig. 1, all bond lengths and angles are in the normal ranges. All non-hydrogen atoms except for O1 are nearly coplanar. In the crystal, the intermolecular N—H⋯O hydrogen bonds link the molecules into chains along the [201] direction.

Experimental

The title compound was prepared according to the literature (Pedras & Soledade, 2006). Single crystals suitable for X-ray diffraction were prepared by slow evaporation method from a solution in dichloromethane/ petroleum (60–90 °C) at room temperature.

Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.93 and 0.97 Å) and were included in the refinement in the riding model with $U_{iso}(H) = 1.5$ or $1.2\ U_{eq}(C)$. The N-bound H atom was located from a difference map and refined with the distance restraint N—H = 0.90 Å and $U_{iso}(H) = 1.5\ U_{eq}(N)$.
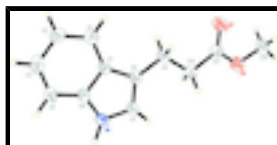
Figures

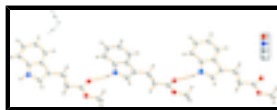Fig. 1. Molecular structure of the title compound with displacement ellipsoids drawn at the 30% probality level.

Fig. 2. Hydrogen-bonded chain in the title compound. Dashed lines indicate hydrogen bonds.

Methyl 3-(1*H*-indol-3-yl)propanoate

Crystal data

$C_{12}H_{13}NO_2$	$F(000) = 432$

supplementary materials

$M_r = 203.23$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 6.893$ (5) Å

$b = 9.146$ (8) Å

$c = 18.052$ (10) Å

$\beta = 111.27$ (3)°

$V = 1060.5$ (13) Å^3

$Z = 4$

$D_x = 1.273$ Mg m^{-3}

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 5849 reflections

$\theta = 3.2–27.4°$

$\mu = 0.09$ mm^{-1}

$T = 296$ K

Block, colorless

$0.46 \times 0.19 \times 0.18$ mm

Data collection

Rigaku R-AXIS RAPID diffractometer

Radiation source: fine-focus sealed tube

graphite

ω scans

Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)

$T_{min} = 0.961$, $T_{max} = 0.984$

10015 measured reflections

2414 independent reflections

1509 reflections with $I > 2\sigma(I)$

$R_{int} = 0.048$

$\theta_{max} = 27.5°$, $\theta_{min} = 3.2°$

$h = -8 \rightarrow 8$

$k = -11 \rightarrow 11$

$l = -23 \rightarrow 23$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.052$

$wR(F^2) = 0.138$

$S = 1.04$

2414 reflections

138 parameters

1 restraint

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0614P)^2 + 0.1289P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.16$ e Å^{-3}

$\Delta\rho_{min} = -0.16$ e Å^{-3}

Extinction correction: *SHELXL*, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.036 (5)

Special details

Experimental. (See detailed section in the paper)

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, convention-al 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}
O1	−0.1892 (2)	0.69967 (17)	0.43997 (8)	0.0762 (5)
O2	−0.3213 (2)	0.88674 (17)	0.48330 (8)	0.0760 (5)
N1	0.5979 (2)	0.80147 (17)	0.76433 (8)	0.0566 (4)
H1	0.6448	0.8259	0.8192	0.085*
C1	0.5726 (3)	0.65764 (19)	0.66104 (9)	0.0469 (4)
C2	0.6478 (3)	0.5573 (2)	0.61952 (11)	0.0585 (5)
H2	0.5628	0.5253	0.5693	0.070*
C3	0.8477 (3)	0.5065 (2)	0.65345 (13)	0.0668 (6)
H3	0.8984	0.4403	0.6258	0.080*
C4	0.9761 (3)	0.5526 (2)	0.72877 (13)	0.0671 (6)
H4	1.1109	0.5161	0.7507	0.080*
C5	0.9079 (3)	0.6506 (2)	0.77134 (11)	0.0598 (5)
H5	0.9941	0.6810	0.8217	0.072*
C6	0.7059 (3)	0.70276 (19)	0.73679 (10)	0.0481 (4)
C7	0.4000 (3)	0.8150 (2)	0.70877 (10)	0.0546 (5)
H7	0.2961	0.8736	0.7145	0.066*
C8	0.3779 (3)	0.7306 (2)	0.64415 (9)	0.0491 (5)
C9	0.1912 (3)	0.7142 (2)	0.56905 (10)	0.0606 (5)
H9A	0.1467	0.6129	0.5639	0.073*
H9B	0.2316	0.7372	0.5242	0.073*
C10	0.0095 (3)	0.8096 (2)	0.56507 (10)	0.0558 (5)
H10A	−0.0319	0.7868	0.6097	0.067*
H10B	0.0529	0.9111	0.5699	0.067*
C11	−0.1734 (3)	0.7903 (2)	0.48976 (10)	0.0532 (5)
C12	−0.5084 (3)	0.8803 (3)	0.41298 (12)	0.0820 (7)
H12A	−0.4753	0.9036	0.3671	0.123*
H12B	−0.6080	0.9494	0.4178	0.123*
H12C	−0.5661	0.7836	0.4074	0.123*

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0724 (10)	0.0813 (11)	0.0522 (7)	0.0196 (8)	−0.0047 (7)	−0.0124 (7)
O2	0.0590 (9)	0.0844 (11)	0.0673 (8)	0.0192 (8)	0.0020 (7)	−0.0161 (7)
N1	0.0537 (9)	0.0640 (10)	0.0445 (8)	−0.0005 (8)	0.0087 (7)	−0.0074 (7)
C1	0.0477 (10)	0.0480 (10)	0.0424 (8)	−0.0038 (8)	0.0132 (8)	0.0026 (7)
C2	0.0677 (13)	0.0552 (12)	0.0516 (10)	−0.0013 (10)	0.0204 (9)	−0.0030 (8)
C3	0.0675 (14)	0.0577 (13)	0.0807 (14)	0.0070 (10)	0.0338 (11)	−0.0018 (10)
C4	0.0520 (12)	0.0571 (13)	0.0878 (14)	0.0035 (10)	0.0202 (11)	0.0053 (11)

C5	0.0475 (11)	0.0566 (12)	0.0635 (11)	−0.0053 (9)	0.0060 (9)	0.0001 (9)
C6	0.0458 (10)	0.0459 (10)	0.0486 (9)	−0.0054 (8)	0.0124 (8)	0.0030 (8)
C7	0.0476 (11)	0.0620 (12)	0.0485 (9)	0.0046 (9)	0.0107 (8)	−0.0029 (8)
C8	0.0487 (10)	0.0542 (11)	0.0401 (9)	−0.0019 (8)	0.0109 (8)	−0.0001 (7)
C9	0.0529 (11)	0.0744 (14)	0.0440 (9)	0.0054 (10)	0.0050 (8)	−0.0041 (9)
C10	0.0574 (12)	0.0549 (11)	0.0464 (9)	−0.0020 (9)	0.0084 (8)	0.0004 (8)
C11	0.0542 (11)	0.0548 (11)	0.0460 (9)	0.0027 (9)	0.0126 (8)	0.0045 (8)
C12	0.0555 (13)	0.0987 (19)	0.0722 (13)	0.0191 (12)	−0.0002 (11)	−0.0103 (12)

Geometric parameters (Å, °)

O1—C11	1.198 (2)	C5—C6	1.388 (3)
O2—C11	1.321 (2)	C5—H5	0.9300
O2—C12	1.446 (2)	C7—C8	1.361 (3)
N1—C6	1.373 (2)	C7—H7	0.9300
N1—C7	1.375 (2)	C8—C9	1.500 (2)
N1—H1	0.9498	C9—C10	1.507 (3)
C1—C2	1.398 (3)	C9—H9A	0.9700
C1—C6	1.404 (2)	C9—H9B	0.9700
C1—C8	1.429 (3)	C10—C11	1.491 (2)
C2—C3	1.371 (3)	C10—H10A	0.9700
C2—H2	0.9300	C10—H10B	0.9700
C3—C4	1.392 (3)	C12—H12A	0.9600
C3—H3	0.9300	C12—H12B	0.9600
C4—C5	1.370 (3)	C12—H12C	0.9600
C4—H4	0.9300		
C11—O2—C12	117.67 (16)	C7—C8—C1	106.12 (15)
C6—N1—C7	108.66 (15)	C7—C8—C9	128.62 (17)
C6—N1—H1	120.5	C1—C8—C9	125.26 (16)
C7—N1—H1	127.7	C8—C9—C10	114.27 (16)
C2—C1—C6	118.42 (17)	C8—C9—H9A	108.7
C2—C1—C8	133.97 (16)	C10—C9—H9A	108.7
C6—C1—C8	107.61 (16)	C8—C9—H9B	108.7
C3—C2—C1	119.42 (18)	C10—C9—H9B	108.7
C3—C2—H2	120.3	H9A—C9—H9B	107.6
C1—C2—H2	120.3	C11—C10—C9	112.84 (16)
C2—C3—C4	120.9 (2)	C11—C10—H10A	109.0
C2—C3—H3	119.5	C9—C10—H10A	109.0
C4—C3—H3	119.5	C11—C10—H10B	109.0
C5—C4—C3	121.4 (2)	C9—C10—H10B	109.0
C5—C4—H4	119.3	H10A—C10—H10B	107.8
C3—C4—H4	119.3	O1—C11—O2	122.57 (17)
C4—C5—C6	117.73 (18)	O1—C11—C10	125.61 (18)
C4—C5—H5	121.1	O2—C11—C10	111.82 (16)
C6—C5—H5	121.1	O2—C12—H12A	109.5
N1—C6—C5	130.60 (17)	O2—C12—H12B	109.5
N1—C6—C1	107.26 (15)	H12A—C12—H12B	109.5
C5—C6—C1	122.14 (17)	O2—C12—H12C	109.5
C8—C7—N1	110.32 (16)	H12A—C12—H12C	109.5

C8—C7—H7	124.8	H12B—C12—H12C	109.5
N1—C7—H7	124.8		
C6—C1—C2—C3	−0.2 (3)	N1—C7—C8—C1	1.2 (2)
C8—C1—C2—C3	−179.77 (19)	N1—C7—C8—C9	−178.59 (18)
C1—C2—C3—C4	0.5 (3)	C2—C1—C8—C7	179.6 (2)
C2—C3—C4—C5	−0.4 (3)	C6—C1—C8—C7	−0.1 (2)
C3—C4—C5—C6	−0.1 (3)	C2—C1—C8—C9	−0.6 (3)
C7—N1—C6—C5	−178.76 (18)	C6—C1—C8—C9	179.73 (17)
C7—N1—C6—C1	1.8 (2)	C7—C8—C9—C10	2.6 (3)
C4—C5—C6—N1	−179.00 (18)	C1—C8—C9—C10	−177.13 (17)
C4—C5—C6—C1	0.4 (3)	C8—C9—C10—C11	−179.92 (16)
C2—C1—C6—N1	179.23 (15)	C12—O2—C11—O1	0.1 (3)
C8—C1—C6—N1	−1.06 (19)	C12—O2—C11—C10	−179.83 (17)
C2—C1—C6—C5	−0.3 (3)	C9—C10—C11—O1	7.5 (3)
C8—C1—C6—C5	179.44 (16)	C9—C10—C11—O2	−172.55 (16)
C6—N1—C7—C8	−1.9 (2)		

Hydrogen-bond geometry (Å, °)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1—H1⋯O1[i]	0.95	2.08	2.972 (3)	157

Symmetry codes: (i) $x+1$, $−y+3/2$, $z+1/2$.

Fig. 1

Fig. 2