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3-Hydroxy-3-nitromethylindolin-2-one

Ying Tang, Gang Chen,* Jie Zhang and Shijun Chen

Xi'an Shiyou University, College of Chemistry and Chemical Engineering, Dianzi'er Road No. 18 Xi'an 710065, Xi'an, People's Republic of China
Correspondence e-mail: tangying78@xsyu.edu.cn

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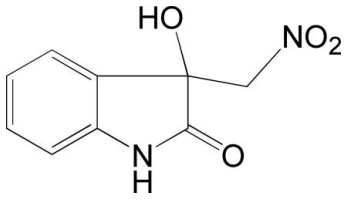
Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.002 Å; R factor = 0.045; wR factor = 0.135; data-to-parameter ratio = 28.0.

In the title compound, $C_9H_8N_2O_4$, the indolin-2-one ring system is substantially planar [maximum deviation = 0.0353 (15) Å]. In the crystal structure, intermolecular N—H···O and O—H···O hydrogen bonds are responsible for the formation of a three-dimensional network.

Related literature

For the synthesis of the title compound, see: Imre *et al.* (2001); Long *et al.* (1978).



Experimental

Crystal data

$C_9H_8N_2O_4$
M_r = 208.17
Orthorhombic, *Pbca*
a = 10.515 (2) Å
b = 7.3736 (14) Å
c = 23.261 (4) Å
V = 1803.6 (6) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 293 K
0.21 × 0.18 × 0.15 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.941, T_{max} = 0.961
16322 measured reflections
3807 independent reflections
2098 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.135
S = 1.03
3807 reflections
136 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O2^{i}	0.86	2.13	2.9849 (14)	171
O2—H2A···O1^{ii}	0.82	1.93	2.7408 (13)	171

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

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *DIAMOND* Brandenburg (1999); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

This work was supported financially by two grants from the Natural Science Research Plan Projects of Shaanxi Science and Technology Department (SJ08B20) and the Scientific Research Plan Projects of Shaanxi Education Department (08JK413).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2364).

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supporting information

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3-Hydroxy-3-nitromethylindolin-2-one

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S1. Comment

3-Hydroxy-3-nitromethyl-1,3-dihydro-indolin-2-one, an important intermediate for the synthesis of natural products, has been synthesized by Henry reaction (Imre *et al.*,2001; Long *et al.*, 1978). Dehydration of this compound as well as its derivatives provides 3-nitromethylene-1,3-dihydro-indolin-2-one, which is used as a dipolarophile in 1,3-dipolar cyclo-additon reactions to synthesize spiro-oxindole compounds. In this paper we report the X-ray crystal structure of the title compound.

The X-ray structural analysis confirmed the assignment of the structure of the title compound from spectroscopic data. The molecular structure is depicted in Fig. 1, and a packing diagram of is depicted in Fig. 2. Geometric parameters of the title compound are in the usual ranges. The indolin-2-one ring system is substantially planar, with a maximum deviation of 0.0353 (15) Å for atom C4. In the crystal structure, intermolecular N–H···O and O–H···O hydrogen bonds (Table 1) are effective in the stabilization of the structure and are responsible for the formation of a three-dimensional network. The O atoms of nitro group are not involved in any hydrogen bond.

S2. Experimental

Isatin (1 mmol) was dissolved in nitromethane (20 ml), catalyzed by DBU, until the disappearance of the starting material, as evidenced by thin-layer chromatography. The solvent was removed *in vacuo* and the residue was separated by column chromatography (silica gel, petroleum ether/ethyl acetate = 5:1 v/v), giving the title compound. ^{1}H-NMR (D_6—DMSO, 400 MHz): 10.56 (1*H*, s), 7.39 (1*H*, d, J = 7.2 Hz), 7.26 (1*H*, td, J = 7.6, 1.2 Hz), 6.98 (1*H*, t, J = 7.6 Hz), 6.85 (1*H*, d, J = 7.6 Hz), 6.75 (1*H*, s), 4.99 (2*H*, dd, J = 12.8, 8.0 Hz); ^{13}C-NMR ($CDCl_3$, 100 MHz): 176.0, 142.6, 130.3, 127.9, 124.7, 121.9, 110.1, 78.5, 72.8; MS (EI) m/*z*: 208 (M^+). 30 mg of the solid compound was dissolved in methanol (30 ml) and the solution was kept at room temperature for 4 d. Slow evaporation of the solvent gave colourless single crystals suitable for X-ray analysis.

S3. Refinement

All H atoms were positioned geometrically, with C–H = 0.93–0.97 Å, O–H = 0.82 Å, N–H = 0.86 Å, and refined using riding model, with U_{iso}(H) = 1.2U_{eq}(C, N) or 1.5U_{eq}(O).

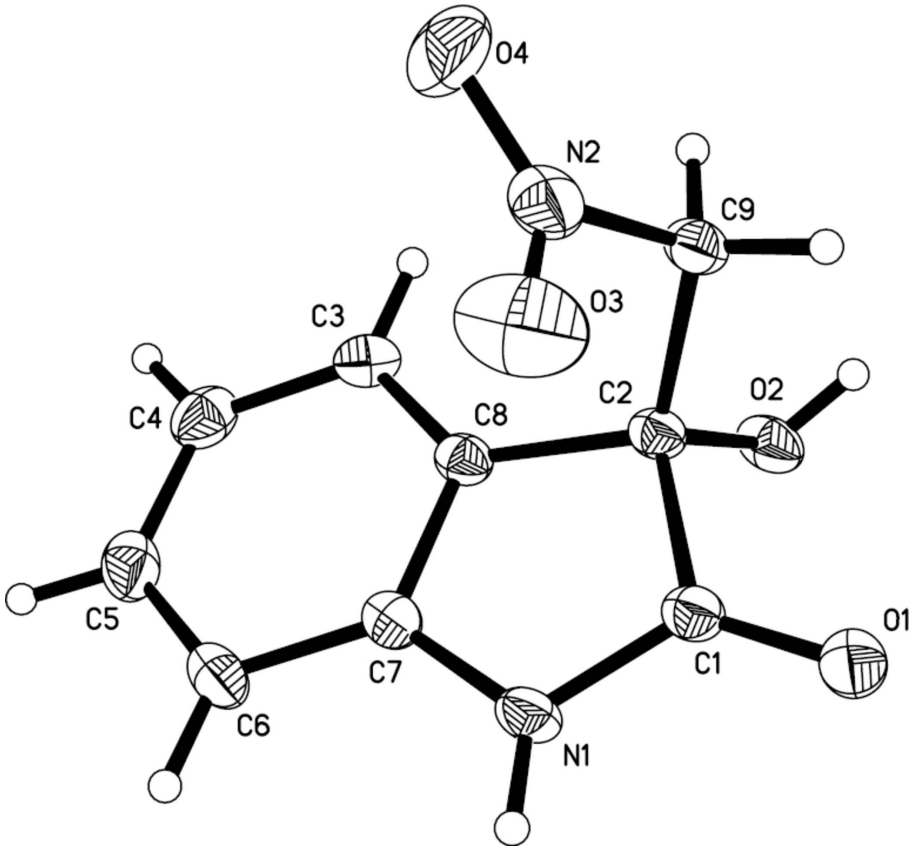

Figure 1

An *ORTEP-3* drawing of the title compound, with the atom-numbering scheme and 30% probability displacement ellipsoids.

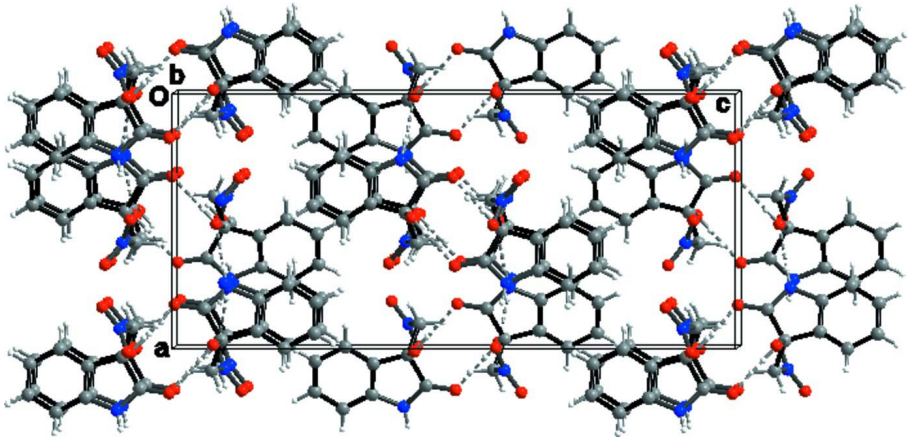

Figure 2

Packing diagram of the title compound viewed approximately along the *b* axis. Dashed lines indicate hydrogen bonds.

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Crystal data

$C_9H_8N_2O_4$
$M_r = 208.17$
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
$a = 10.515$ (2) Å
$b = 7.3736$ (14) Å
$c = 23.261$ (4) Å
$V = 1803.6$ (6) Å^3
$Z = 8$
$F(000) = 864$
$D_x = 1.533$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3122 reflections
$\theta = 1.8$–$34.3°$
$\mu = 0.12$ mm^{-1}
$T = 293$ K
Block, colourless
0.21 × 0.18 × 0.15 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{min} = 0.941$, $T_{max} = 0.961$
16322 measured reflections
3807 independent reflections
2098 reflections with $I > 2\sigma(I)$
$R_{int} = 0.026$
$\theta_{max} = 34.3°$, $\theta_{min} = 1.8°$
$h = -12 \rightarrow 12$
$k = -9 \rightarrow 11$
$l = -28 \rightarrow 36$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.135$
$S = 1.03$
3807 reflections
136 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0699P)^2 + 0.2564P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.31$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ 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}
N1	0.73793 (10)	0.22536 (14)	0.59575 (4)	0.0345 (2)
H1A	0.8121	0.2684	0.5882	0.041*
C7	0.68870 (11)	0.20320 (15)	0.65183 (5)	0.0313 (3)
C8	0.56744 (11)	0.12898 (14)	0.64887 (5)	0.0294 (2)
C2	0.53211 (11)	0.10262 (14)	0.58677 (5)	0.0287 (2)

O1	0.67140 (9)	0.16574 (14)	0.50346 (4)	0.0452 (3)
C3	0.50087 (12)	0.08802 (17)	0.69844 (5)	0.0374 (3)
H3A	0.4196	0.0386	0.6966	0.045*
C9	0.41221 (12)	0.20580 (16)	0.56828 (5)	0.0354 (3)
H9A	0.4059	0.2048	0.5267	0.043*
H9B	0.3377	0.1453	0.5837	0.043*
C6	0.74609 (13)	0.24268 (17)	0.70380 (5)	0.0396 (3)
H6A	0.8264	0.2951	0.7055	0.048*
N2	0.41524 (12)	0.39586 (15)	0.58894 (5)	0.0470 (3)
C5	0.67832 (15)	0.20049 (18)	0.75336 (6)	0.0450 (3)
H5A	0.7145	0.2253	0.7890	0.054*
C4	0.55872 (14)	0.1227 (2)	0.75123 (5)	0.0448 (3)
H4A	0.5167	0.0934	0.7852	0.054*
O3	0.50508 (14)	0.48843 (17)	0.57450 (9)	0.0904 (5)
O4	0.32881 (14)	0.44827 (19)	0.61941 (6)	0.0776 (4)
C1	0.65394 (11)	0.17044 (15)	0.55545 (5)	0.0317 (3)
O2	0.51621 (8)	−0.08414 (11)	0.57414 (4)	0.0391 (2)
H2A	0.4586	−0.0966	0.5507	0.059*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0244 (5)	0.0384 (5)	0.0408 (5)	−0.0081 (4)	−0.0017 (4)	0.0000 (4)
C7	0.0299 (6)	0.0267 (5)	0.0372 (6)	0.0003 (4)	−0.0040 (4)	0.0000 (4)
C8	0.0259 (6)	0.0268 (5)	0.0353 (5)	0.0019 (4)	−0.0030 (4)	0.0005 (4)
C2	0.0237 (6)	0.0254 (5)	0.0370 (5)	−0.0008 (4)	−0.0022 (4)	−0.0024 (4)
O1	0.0378 (5)	0.0603 (6)	0.0374 (5)	−0.0092 (4)	0.0023 (4)	−0.0040 (4)
C3	0.0303 (6)	0.0389 (6)	0.0431 (6)	0.0043 (5)	0.0031 (5)	0.0048 (5)
C9	0.0276 (6)	0.0352 (6)	0.0435 (6)	0.0037 (5)	−0.0069 (5)	−0.0055 (5)
C6	0.0370 (7)	0.0356 (6)	0.0463 (6)	−0.0006 (5)	−0.0139 (5)	−0.0037 (5)
N2	0.0474 (7)	0.0366 (6)	0.0569 (7)	0.0135 (5)	−0.0168 (5)	−0.0048 (5)
C5	0.0520 (8)	0.0454 (7)	0.0377 (6)	0.0136 (6)	−0.0120 (5)	−0.0052 (5)
C4	0.0476 (8)	0.0503 (7)	0.0364 (6)	0.0154 (6)	0.0037 (5)	0.0044 (5)
O3	0.0736 (9)	0.0354 (6)	0.1620 (17)	−0.0061 (6)	−0.0100 (9)	0.0024 (7)
O4	0.0866 (10)	0.0763 (8)	0.0700 (8)	0.0389 (7)	−0.0037 (7)	−0.0255 (7)
C1	0.0262 (6)	0.0314 (5)	0.0374 (6)	−0.0017 (4)	−0.0008 (4)	−0.0019 (4)
O2	0.0317 (5)	0.0265 (4)	0.0591 (5)	−0.0010 (3)	−0.0098 (4)	−0.0076 (3)

Geometric parameters (Å, º)

N1—C1	1.3500 (16)	C3—H3A	0.9300
N1—C7	1.4130 (16)	C9—N2	1.4819 (17)
N1—H1A	0.8600	C9—H9A	0.9700
C7—C6	1.3822 (17)	C9—H9B	0.9700
C7—C8	1.3893 (17)	C6—C5	1.390 (2)
C8—C3	1.3823 (17)	C6—H6A	0.9300
C8—C2	1.5042 (15)	N2—O3	1.2129 (19)
C2—O2	1.4180 (13)	N2—O4	1.2155 (18)

C2—C9	1.5340 (16)	C5—C4	1.383 (2)
C2—C1	1.5563 (16)	C5—H5A	0.9300
O1—C1	1.2237 (14)	C4—H4A	0.9300
C3—C4	1.3940 (19)	O2—H2A	0.8200
C1—N1—C7	111.51 (10)	C2—C9—H9A	109.4
C1—N1—H1A	124.2	N2—C9—H9B	109.4
C7—N1—H1A	124.2	C2—C9—H9B	109.4
C6—C7—C8	121.81 (11)	H9A—C9—H9B	108.0
C6—C7—N1	128.55 (11)	C7—C6—C5	117.02 (12)
C8—C7—N1	109.63 (10)	C7—C6—H6A	121.5
C3—C8—C7	120.63 (11)	C5—C6—H6A	121.5
C3—C8—C2	130.37 (11)	O3—N2—O4	124.39 (14)
C7—C8—C2	108.98 (10)	O3—N2—C9	117.33 (13)
O2—C2—C8	110.71 (9)	O4—N2—C9	118.28 (14)
O2—C2—C9	109.07 (9)	C4—C5—C6	121.94 (12)
C8—C2—C9	114.09 (9)	C4—C5—H5A	119.0
O2—C2—C1	108.19 (9)	C6—C5—H5A	119.0
C8—C2—C1	101.81 (9)	C5—C4—C3	120.29 (12)
C9—C2—C1	112.70 (9)	C5—C4—H4A	119.9
C8—C3—C4	118.28 (12)	C3—C4—H4A	119.9
C8—C3—H3A	120.9	O1—C1—N1	126.62 (11)
C4—C3—H3A	120.9	O1—C1—C2	125.24 (10)
N2—C9—C2	111.13 (9)	N1—C1—C2	108.05 (10)
N2—C9—H9A	109.4	C2—O2—H2A	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O2^{i}	0.86	2.13	2.9849 (14)	171
O2—H2*A*···O1^{ii}	0.82	1.93	2.7408 (13)	171

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