Crystal structure of (*E*)-3-(*N*-oxide-benzylimino)indolin-2-one, $C_{15}H_{12}N_2O_2$

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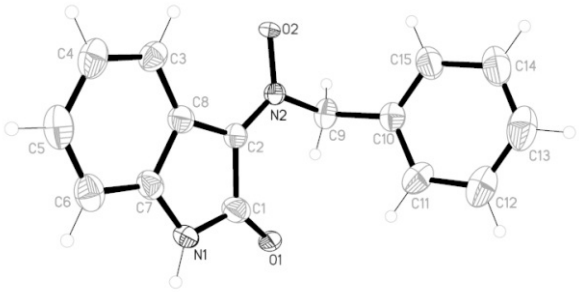

Abstract

$C_{15}H_{12}N_2O_2$, monoclinic, $P2_1/n$ (no. 14), a = 12.343(4) Å, b = 8.172(3) Å, c = 13.375(5) Å, β = 114.466(4)°, V = 1228.0 Å^3, Z = 4, $R_{gt}(F)$ = 0.048, $wR_{ref}(F^2)$ = 0.127, T = 296 K.

Source of material

Isatin (1 mmol) was dissolved in methanol (20 ml), 10 ml methanol solution of 1.2 mmol N-benzyl-hydroxylamine was added dropwise. The mixture was refluxed for 1 h, and the solution was poured into a beaker, which was kept at room temperature for 7 d. Natural evaporation gave yellow single crystals of the title compound suitable for X-ray analysis.

Experimental details

H atoms bonded to N atoms were located in a Fourier difference map and refined isotropically without restraints. H atoms bonded to C atoms were positioned geometrically and allowed to ride on their parent atoms, with d(C–H)=0.93 Å and U_{iso}(H)=1.2 U_{eq}(C).

Discussion

3-Imine-indole-2-one derivatives have driven much attention for their bioactivities on anti-bacterial, anti-virus and neuroprotection [1], [2]. 1*H*-indole-2,3-dione-3-oxime has been synthesized by condensation reaction of isatin and hydroxyl- amine. But its N-oxide has scarcely been studied [3]. In this paper we report the synthesis and the X-ray crystal structure of (*E*)-3-(*N*-oxide-benzylimino)indolin-2-one. The values of the geometric parameters of the title compound are within normal ranges and experimental errors. Geometric parameters of the title compound are in the usual ranges. It crystallizes in the monoclinic space group, $P2_1/n$, with unit cell dimensions a = 12.343(4) Å, b = 8.172(3) Å, c = 13.375(5) Å. The indol-2-one ring system is substantially planar with the O2–C9–C8–N1 torsion angle of -1.7(4)°. The length of N1–O1 is 1.281(2) Å, and the length of N1–C8 is 1.307(2) Å. The angle of C8–N1–O1 is 121.1(2)°, and the angle of N1–C7–C6 is 111.1(2)°. The dihedral angle of indol-2-one and phenyl proup is 70.46(8)°. There is a kind of typical hydrogen bond, N2–H2A···O1 with the D···A distance of 2.808(2) Å and D–H···A angle of 157°, which is responsible for the crystal's 1-dimensional supramolecular structure.

Table 1. Data collection and handling.

Crystal:	yellow blocks, size 0.23×0.28×0.35 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.93 cm^{-1}
Diffractometer, scan mode:	Bruker Smart Apex II, φ and ω
$2\theta_{max}$:	50.24°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6166, 2184
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1534
$N(param)_{refined}$:	172
Programs:	SHELXS-97, SHELXL-97 [4], WinGX [5], ORTEP-3 [6]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1A)	4*e*	0.2141	0.8437	0.7935	0.07
H(2B)	4*e*	0.3540	0.6822	0.7698	0.093
H(3A)	4*e*	0.5286	0.6249	0.9137	0.099
H(4A)	4*e*	0.5690	0.7299	1.0845	0.095
H(5A)	4*e*	0.4287	0.8955	1.1101	0.07
H(7A)	4*e*	0.2514	1.0810	1.0166	0.053
H(7B)	4*e*	0.1607	1.0343	0.8973	0.053
H(11A)	4*e*	–0.2868	0.5567	0.8421	0.063
H(12A)	4*e*	–0.2656	0.4683	1.0141	0.071
H(13A)	4*e*	–0.1016	0.5374	1.1691	0.067
H(14A)	4*e*	0.0472	0.7003	1.1589	0.054
H(2A)	4*e*	–0.1766	0.7322	0.7412	0.058

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Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	4*e*	0.2857(2)	0.8214(3)	0.8531(2)	0.068(2)	0.060(2)	0.055(2)	0.006(1)	0.033(1)	–0.002(1)
C(2)	4e	0.3692(3)	0.7247(3)	0.8388(3)	0.105(2)	0.069(2)	0.083(2)	0.014(2)	0.064(2)	–0.000(2)
C(3)	4*e*	0.4728(3)	0.6911(4)	0.9241(3)	0.092(2)	0.075(2)	0.115(3)	0.029(2)	0.077(2)	0.028(2)
C(4)	4*e*	0.4970(2)	0.7532(4)	1.0258(3)	0.055(2)	0.089(2)	0.097(2)	0.024(2)	0.037(2)	0.038(2)
C(5)	4*e*	0.4127(2)	0.8522(3)	1.0410(2)	0.048(1)	0.069(2)	0.061(2)	0.002(1)	0.025(1)	0.010(1)
C(6)	4*e*	0.3068(2)	0.8856(2)	0.9548(2)	0.044(1)	0.035(1)	0.052(1)	0.000(1)	0.027(1)	0.005(1)
C(7)	4*e*	0.2134(2)	0.9900(3)	0.9681(2)	0.045(1)	0.038(1)	0.051(1)	–0.000(1)	0.022(1)	–0.002(1)
C(8)	4*e*	0.0401(2)	0.8252(2)	0.9546(2)	0.034(1)	0.039(1)	0.031(1)	0.0068(9)	0.0120(9)	–0.0023(9)
C(9)	4*e*	–0.0249(2)	0.8393(3)	0.8321(2)	0.040(1)	0.050(1)	0.036(1)	0.009(1)	0.014(1)	0.001(1)
C(10)	4*e*	–0.1315(2)	0.6805(3)	0.8997(2)	0.037(1)	0.038(1)	0.046(1)	0.005(1)	0.017(1)	–0.004(1)
C(11)	4*e*	–0.2202(2)	0.5852(3)	0.9050(2)	0.044(1)	0.044(1)	0.068(2)	0.001(1)	0.022(1)	–0.010(1)
C(12)	4*e*	–0.2068(2)	0.5331(3)	1.0078(2)	0.053(2)	0.041(1)	0.098(2)	0.002(1)	0.044(2)	0.003(1)
C(13)	4*e*	–0.1083(2)	0.5750(3)	1.1011(2)	0.064(2)	0.049(2)	0.072(2)	0.015(1)	0.045(1)	0.014(1)
C(14)	4*e*	–0.0190(2)	0.6720(3)	1.0957(2)	0.049(1)	0.045(1)	0.043(1)	0.011(1)	0.023(1)	0.001(1)
C(15)	4*e*	–0.0311(2)	0.7258(2)	0.9933(2)	0.036(1)	0.036(1)	0.040(1)	0.0073(9)	0.0171(9)	–0.0007(9)
N(1)	4*e*	0.1425(1)	0.8932(2)	1.0146(1)	0.0353(9)	0.041(1)	0.0334(9)	0.0065(8)	0.0128(8)	–0.0026(7)
N(2)	4*e*	–0.1240(2)	0.7475(2)	0.8071(1)	0.042(1)	0.061(1)	0.033(1)	–0.0007(9)	0.0056(8)	–0.0057(8)
O(1)	4*e*	0.1893(1)	0.8787(2)	1.1194(1)	0.0442(9)	0.073(1)	0.0291(8)	0.0032(8)	0.0077(6)	–0.0034(7)
O(2)	1*e*	0.0013(1)	0.9153(2)	0.7671(1)	0.058(1)	0.084(1)	0.0385(9)	0.0006(9)	0.0191(8)	0.0107(8)

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