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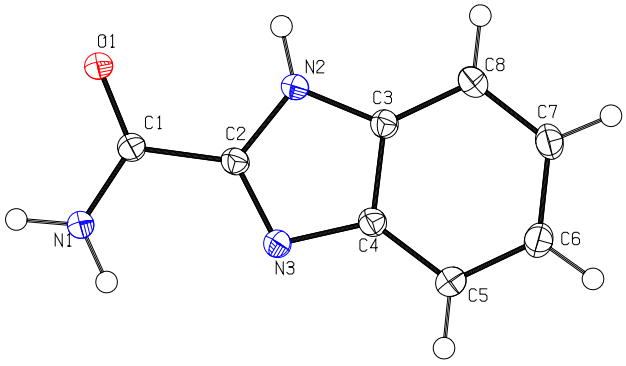
The crystal structure of 1*H*-benzimidazole-2-carboxamide, $C_8H_7N_3O$



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Abstract

$C_8H_7N_3O$, orthorhombic, *Pccn* (no. 56), a = 9.9071(14) Å, b = 11.1950(19) Å, c = 13.315(2) Å, V = 1476.8(4) Å^3, Z = 8, $R_{gt}(F)$ = 0.0356, $wR_{ref}(F^2)$ = 0.1034, T = 100(2) K, β = 90°.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.12 × 0.10 × 0.10 mm
Wavelength:	Synchrotron radiation (1.34139 Å)
μ:	0.53 mm^{-1}
Diffractometer, scan mode:	Bruker Photon III, Profile data from θ/2θ
θ_{max}, completeness:	61.7°, 99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	8081, 1691, 0.036
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 1509
$N(param)_{refined}$:	118
Programs:	Bruker [1], SHELX [2–4], Diamond [5], Olex2 [6], PLATON [7]

Source of material

All the reagents and solvents were used as obtained without further purification. The preparation of 1*H*-benzimidazole-2-carboxamide were performed according to the below procedures. Firstly, 1,2-diaminobenzene (50.0 mmol, 5.4 g) and glycolic acid (50.0 mmol, 3.8 g) were mixed in a hydrochloric aqueous solution (6.0 mol/L, 50.0 mL). The solution was stirred at 90 °C for 7 h. To the cooled solution, a sodium hydroxide water solution (3.0 mol/L) was added gradually until no more white solid was precipitated. The white solid, 1*H*-benzimidazole-2-methanol, was filtered and dried (yield: 90%, 6.72 g). Secondly, 1*H*-benzimidazole-2-methanol (37.0 mmol, 6.0 g), sodium carbonate (5.0 g, 47.2 mmol) and a potassium permanganate (44.0 mmol, 7.0 g) water solution was refluxed for 2 h. Then the solution was adjusted to be pH = 4.0 and some white solid powder was obtained, filtered and dried (5.1 g, yield: 85%). Lastly, the white powder (1*H*-benzimidazole-2-carboxylic acid, 5.0 g, 31.0 mmol) produced in the second step was added to 50.0 mL thionyl chloride and stirred at 70 °C for 5 h. The obtained solution was cooled to ambient temperature and concentrated *in vacuo*, to which ammonia water (50.0 mL) was added. The solution was further stirred at 70 °C for 5 h and cooled to ambient temperature. A pale yellow solid of the titled compound was obtained (3.93 g, 78%). Crystals were obtained by recrystallization of the title compound (1.0 g) in a methanol solution (10.0 mL) at the ambient condition after a week.

Experimental details

H atoms bonded to C atoms were located in difference maps and subsequently treated as riding, with C–H distances of 0.93 Å(aromatic C) U_{iso}(H) = 1.2U_{eq}(C atoms). Hydrogen atoms bonded to nitrogen were located from the Fourier difference maps with their displacement

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.50766 (9)	0.82548 (9)	0.45016 (7)	0.0183 (2)
C2	0.50008 (8)	0.69893 (9)	0.41662 (7)	0.0173 (2)
C3	0.57099 (9)	0.51930 (8)	0.37837 (7)	0.0175 (2)
C4	0.43181 (9)	0.53106 (8)	0.35948 (7)	0.0179 (2)
C5	0.35760 (10)	0.43400 (9)	0.32256 (8)	0.0213 (2)
H5	0.263559	0.440423	0.309687	0.026*
C6	0.42643 (11)	0.32863 (9)	0.30559 (8)	0.0231 (2)
H6	0.378376	0.261133	0.281264	0.028*
C7	0.56638 (11)	0.31875 (9)	0.32350 (8)	0.0236 (2)
H7	0.610441	0.245159	0.309799	0.028*
C8	0.64090 (10)	0.41319 (9)	0.36040 (8)	0.0219 (2)
H8	0.734978	0.406334	0.372937	0.026*
N1	0.39305 (8)	0.88550 (8)	0.44704 (7)	0.0220 (2)
H1A	0.3935 (12)	0.9645 (12)	0.4656 (10)	0.026*
H1B	0.3168 (13)	0.8534 (11)	0.4233 (10)	0.026*
N2	0.61178 (8)	0.62859 (7)	0.41480 (7)	0.0180 (2)
H2	0.6937 (12)	0.6446 (10)	0.4385 (9)	0.022*
N3	0.38985 (8)	0.64568 (7)	0.38417 (7)	0.0185 (2)
O1	0.61756 (6)	0.86763 (6)	0.47894 (6)	0.0214 (2)

parameters being set 1.2 times of the parent nitrogen atoms and the coordinates refined freely.

Comment

The 1*H*-benzimidazole-2-carboxamide (BC), is often used as a chemical and biological material in the organic synthesis [8,9]. For instance, it has been applied as the initial precursors in the design of potential inhibitors of protein interacting with never in mitosis A-1 (Pin1) [10]. Due to the presence of one imine and one amide groups, it can be easily interacting with other H-bonding acceptors which may be influenced and directed in some organic preparation [10]. In order to confirm its crystallographic characteristics, especially its crystal packing tendency, its crystal structure was determined.

The title compound crystallized in the orthorhombic, space group *Pccn* with one molecule in its asymmetric unit. The C2–N2 bond (1.3585(12) Å) is about 0.04 Å longer than that the C2–N3 bond (1.3171(12) Å), indicating the benzimidazole nitrogen-bonded hydrogen atom locating around N2 atoms [10,11]. All the non-hydrogen atoms in the molecule is almost completely coplanar with the least square plane equation of $-1.9482x - 3.3203y + 12.4434z = 1.8732$ and the largest deviation away from the plane comes from the N1 and C2 atoms with a distance of *ca.* 0.016(1) Å. The N1–C1–C2–N3 torsional angle of only –178.64(9)° is comparable to some analogs (176.55(1)°, CSD Refcode: USAVAN) [12].

Its crystal packing, the molecules are linked together by two intermolecular N–H···O (N2–H2···O1 ($d_{N2\cdots O1}$ = 2.814(2) Å, 3/2 – *x*, 3/2 – *y*, *z*) and N1–H1A···O1($d_{N1\cdots O1}$ = 2.936(1) Å, 1 – *x*, 2 – *y*, 1 – *z*)) and one N–H···N ($d_{N1\cdots N3}$ = 2.946(1) Å, 1/2 – *x*, 3/2 – *y*, *z*) hydrogen bonds into a two-dimensional network parallel to the (001) plane. An analysis indicates there is also a $\pi\cdots\pi$ stacking interaction between two neighbouring imidazole and benzo moieties with the centroid-to-centroid distance of 3.579(1) Å(7), which further consolidated the crystal structure.

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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