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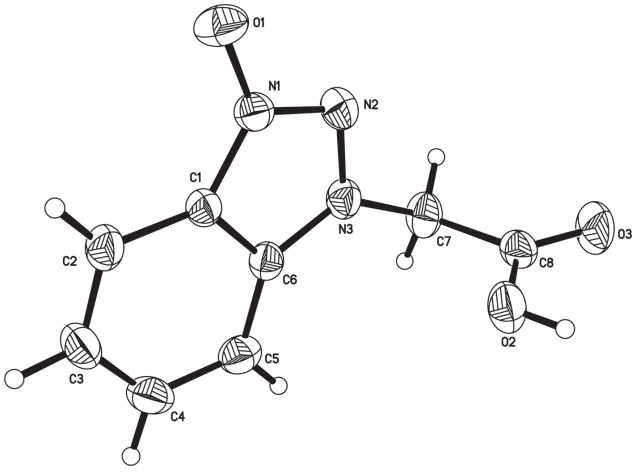
Crystal structure of 1-(carboxymethyl)-1*H*-benzo[*d*][1,2,3]triazole 3-oxide, $C_8H_7N_3O_3$



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Abstract

$C_8H_7N_3O_3$, monoclinic, $P2_1/c$ (no. 14), $a = 7.2526(12)$ Å, $b = 10.0770(18)$ Å, $c = 11.906(2)$ Å, $\beta = 104.393(3)°$, $V = 842.8(2)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0375$, $wR_{ref}(F^2) = 0.0766$, $T = 293$ K.

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The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

A mixture of 1*H*-benzo[*d*][1,2,3]triazol-1-ol (1 mmol, 135 mg), 2-chloroacetic acid (1 mmol, 94 mg) and sodium hydroxide (1 mmol, 40 mg) in ethanol (30 mL) under reflux conditions (351 K) for 2 h. The compound formed was filtered off, and dried. The solid product was recrystallized from dichloromethane. After three days colourless, block-shaped crystals were obtained.

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Table 1: Data collection and handling.

Crystal:	Block, colorless
Size:	0.25 × 0.20 × 0.15 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.12 mm^{-1}
Diffractometer, scan mode:	Bruker APEX2, φ and ω-scans
$2\theta_{max}$, completeness:	25.1°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	4506, 1486, 0.027
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1244
$N(param)_{refined}$:	128
Programs:	Bruker programs [1], SHELX [2]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.7606(2)	0.00246(17)	0.86168(14)	0.0311(4)
C2	0.7214(3)	−0.12802(19)	0.88667(17)	0.0399(5)
H2	0.7050	−0.1953	0.8316	0.048*
C3	0.7088(3)	−0.1504(2)	0.99762(18)	0.0468(5)
H3	0.6822	−0.2357	1.0189	0.056*
C4	0.7348(3)	−0.0484(2)	1.08055(18)	0.0485(5)
H4	0.7245	−0.0687	1.1550	0.058*
C5	0.7745(3)	0.0796(2)	1.05629(16)	0.0418(5)
H5	0.7913	0.1466	1.1117	0.050*
C6	0.7883(2)	0.10338(17)	0.94307(15)	0.0318(4)
C7	0.8661(3)	0.34894(18)	0.92805(18)	0.0417(5)
H7A	0.9641	0.3868	0.8953	0.050*
H7B	0.9138	0.3486	1.0117	0.050*
C8	0.6915(3)	0.43563(18)	0.89612(15)	0.0372(4)
N1	0.7873(2)	0.06056(15)	0.76254(12)	0.0334(4)
N2	0.8291(2)	0.18682(15)	0.77669(13)	0.0371(4)
N3	0.8311(2)	0.21316(14)	0.88787(13)	0.0350(4)
O1	0.77648(19)	0.00258(14)	0.66317(11)	0.0459(4)
O2	0.53514(19)	0.37066(13)	0.89260(13)	0.0493(4)
H2A	0.4447	0.4221	0.8777	0.074*
O3	0.7021(2)	0.55186(14)	0.87779(14)	0.0595(4)

Experimental details

All H atoms were placed in idealized positions (C—H = 0.93 Å (aromatic), C—H = 0.97 Å (methylene), O—H = 0.82 Å) and refined using a riding model. The U_{iso} values were constrained to be $1.2U_{eq}$ of the carrier atom for aromatic H atoms and methylene H atoms, $1.5U_{eq}$ for the carboxyl H atom.

Discussion

Benzotriazole and its derivatives have attracted a great interest because of their biological activities such as anti-inflammatory, anti-viral, diuretic and anti-hypertensive [3–6]. In order to search for new benzotriazoles with higher bioactivity, the title compound was synthesized and its crystal structure is reported here.

The asymmetric unit of the title compound contains one molecule of the title compound Bond lengths and angles are in the expected ranges [7–10]. The benzotriazole ring is essentially planar with the maximum deviation from planarity being 0.024(15) Å for atom C1. In the crystal structure, the intermolecular O—H. . . O hydrogen bonds link the molecules into zigzag chains along the *b* axis. The adjacent infinite chains are further connected by $\pi\cdots\pi$ interactions between the benzene rings with the centroid-centroid distance of 3.578(13) Å [symmetry code: 2-x, $-y$, $2-z$] into a two dimensional network.

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