Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1*H*-Benzimidazole-1-carbohydrazonamide

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.046
wR factor = 0.143
Data-to-parameter ratio = 15.2

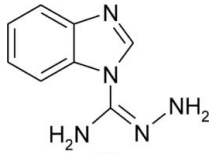
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 7 June 2006
Accepted 28 June 2006

The title compound, $C_8H_9N_5$, was prepared by the reaction of 1-cyanobenzimidazole with a twofold excess of hydrazine. The crystal packing is stabilized by weak intermolecular N—H···N hydrogen bonds and π–π interactions.

Comment

N-Substituted benzimidazole (BzIm) derivatives are widely used in preparative organic syntheses (Staab, 1962). Usually, the BzIm derivatives are not stable in solution (Staab, 1962). Recent studies of *N*-benzimidazole carbohydrazonamides (Pan'kov *et al.*, 2001*a*,*b*) have shown their stability under normal atmospheric pressure in solution and in the solid state for a period of one month and more. The title compound, (I), was prepared by the reaction of 1-cyanobenzimidazole with hydrazine with a twofold excess of hydrazine. Here we present its crystal structure.



(I)

The bond lengths and angles in (I) (Fig. 1) are normal (Allen *et al.*, 1987). In the crystal structure, the N atoms of the carbohydrazonamide group form four weak intermolecular N—H···N hydrogen bonds with three neighboring molecules

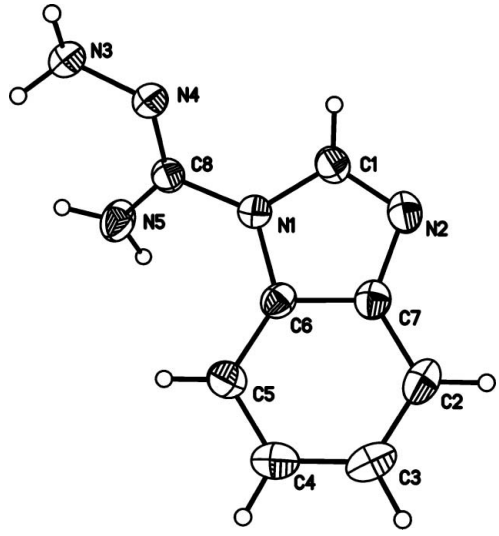


Figure 1
A view of (I) with the atom numbering. Displacement ellipsoids are drawn at the 50% probability level.

(Table 1), leading to the formation of a three-dimensional hydrogen-bonded network. The short intermolecular contact C2···C6^{iv} of 3.445 (8) Å indicates the presence of stacking interactions [symmetry code: (iv) $1-x, \frac{3}{2}+y, \frac{5}{2}-z$], which contribute to the stabilization of the crystal structure (Fig. 2) along with the N—H···N hydrogen bonds.

Experimental

1*H*-Benzimidazole-1-carbohydrazonamide was prepared according to the known procedure (Pan'kov *et al.*, 2001*a*). A portion of hydrazine hydrate (0.2 ml, 4.1 mmol) was added to a solution of 1-cyanobenzimidazole (0.3 g, 2.1 mmol) in 3 ml of 2-propanol. The reaction mixture was stirred and the temperature increased to 313–323 K. After 15 min it was cooled to room temperature and filtered. The product was washed with 2-propanol and recrystallized from ethanol. Yield 91%, m.p. 436–437 K.

Crystal data

$C_8H_9N_5$
M_r = 175.20
Monoclinic, $P2_1/c$
a = 7.389 (2) Å
b = 8.152 (2) Å
c = 13.660 (3) Å
β = 94.25 (3)°
V = 820.5 (3) Å^3
Z = 4
D_x = 1.418 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 293 (2) K
Prism, colorless
0.60 × 0.20 × 0.20 mm

Data collection

Siemens P3/PC diffractometer
$\omega/2\theta$ scans
Absorption correction: none
1929 measured reflections
1795 independent reflections
1448 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 27.1°
2 standard reflections
every 98 reflections
intensity decay: 2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.143
S = 1.10
1795 reflections
118 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0798P)^2 + 0.29P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.011
$\Delta\rho_{max}$ = 0.35 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
N3—H6···N2^{i}	0.90	2.30	3.181 (2)	165
N3—H7···N4^{ii}	0.90	2.37	3.224 (2)	158
N5—H9···N3^{iii}	0.90	2.21	3.069 (2)	160
N5—H8···N2^{i}	0.90	2.23	3.083 (2)	158

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

All H atoms were located in a difference map and refined as riding with C—H = 0.93 Å, N—H = 0.90 Å, and U_{iso}(H) = 1.2U_{eq}(parent atom).

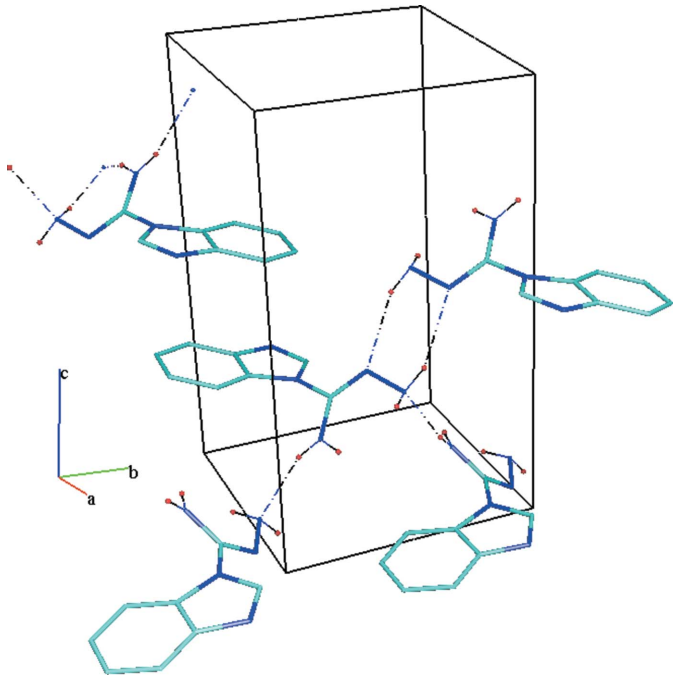


Figure 2
The crystal packing in (I). Hydrogen bonds are shown as dashed lines. The H atoms not participating in hydrogen bonds have been omitted for clarity.

Data collection: *P3*/*PC* (Siemens, 1989); cell refinement: *P3*/*PC*; data reduction: *XDISK* (Siemens, 1989); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL* and *TOPOS* (Blatov *et al.*, 1999); software used to prepare material for publication: *SHELXTL*.

The authors are grateful to the Laboratory of X-ray Diffraction Studies of the A. N. Nesmeyanov Institute of Organoelement Compounds RAS for collecting the X-ray data.

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