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6-Bromopyridine-2-carboxamide

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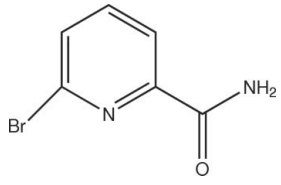
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.012 Å; R factor = 0.063; wR factor = 0.172; data-to-parameter ratio = 14.2.

In the the title compound, $C_6H_5BrN_2O$, an intramolecular N—H···N hydrogen bond generates an S(5) ring. In the crystal structure, intermolecular bifurcated N—H···(O,O) hydrogen bonds link the molecules, leading to sheets propagating in (100).

Related literature

For medicinal background to inhibitors of the cysteine protease cathepsin K, see: Altmann & Aichholz (2007).



Experimental

Crystal data

$C_6H_5BrN_2O$
M_r = 201.03
Monoclinic, $P2_1/c$
a = 13.034 (3) Å
b = 6.4050 (13) Å
c = 8.5540 (17) Å
β = 94.85 (3)°
V = 711.6 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 5.70 mm^{-1}
T = 293 K
0.20 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.395, T_{max} = 0.599
1354 measured reflections
1296 independent reflections
756 reflections with $I > 2\sigma(I)$
R_{int} = 0.063
3 standard reflections
every 200 reflections
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.172
S = 1.01
1296 reflections
91 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.57 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2B···N1	0.86	2.41	2.730 (10)	102
N2—H2A···O^{i}	0.86	1.99	2.849 (9)	176
N2—H2B···O^{ii}	0.86	2.22	3.002 (9)	151

Symmetry codes: (i) $-x+1, y+\frac{1}{2}, -z+\frac{5}{2}$; (ii) $x, -y+\frac{1}{2}, z-\frac{1}{2}$.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *PLATON* (Spek, 2009).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5211).

References

Altmann, E. & Aichholz, R. (2007). *J. Med. Chem.* **50**, 591–594.
Enraf–Nonius (1994). *CAD-4 EXPRESS*. Enraf–Nonius, Delft. The Netherlands.
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North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

supporting information

Acta Cryst. (2009). E**65**, o3161 [doi:10.1107/S1600536809047114]

6-Bromopyridine-2-carboxamide

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S1. Experimental

A mixture of 30 g of 6-bromopyridine-2-carboxylic acid (0.1485 mol) in 300 ml of thionyl chloide was refluxed for twenty hours. Excess thionyl chloride was removed in vacuo. The residue was added as a slurry in dioxane or benene to 75 ml cold, stirred concentrated ammonium hydroxide. The mixture was stored overnight and the filtered to give 25 g of the title compound (yield 83.4%, m.p. 417 K). Colourless blocks of (I) were obtained by the slow evaporation of an ethyl acetate solution.

S2. Refinement

H atoms were positioned geometrically, with C-H = 0.93–0.97Å, and refined as riding with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

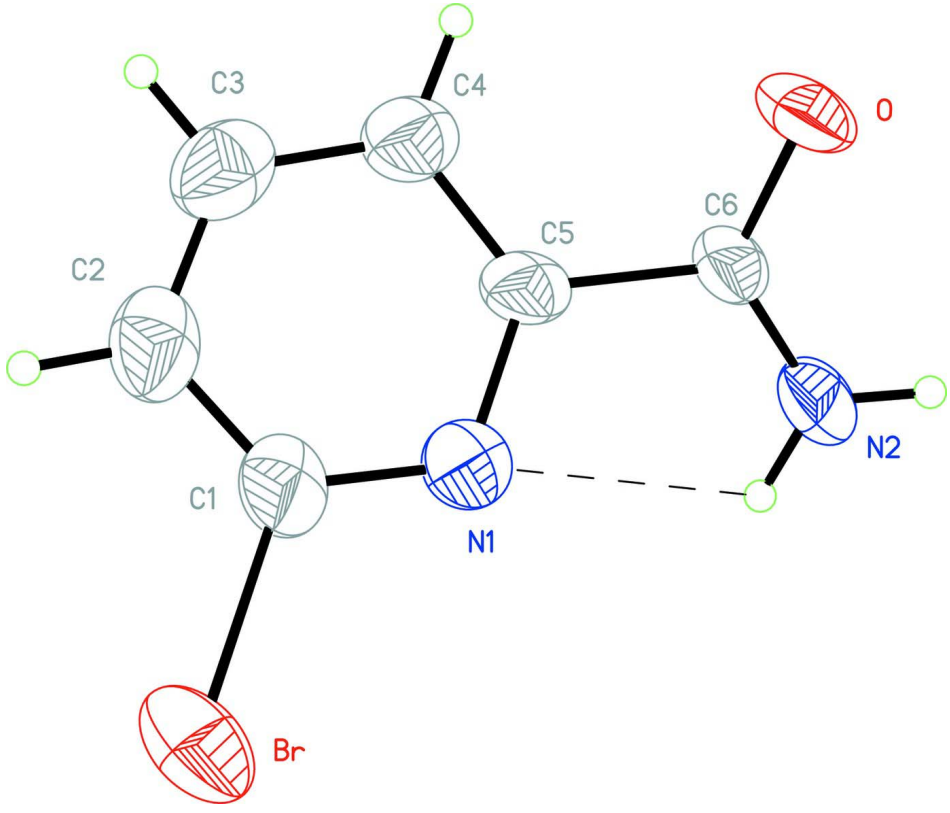

Figure 1

The molecular structure of (I) showing 50% displacement ellipsoids.

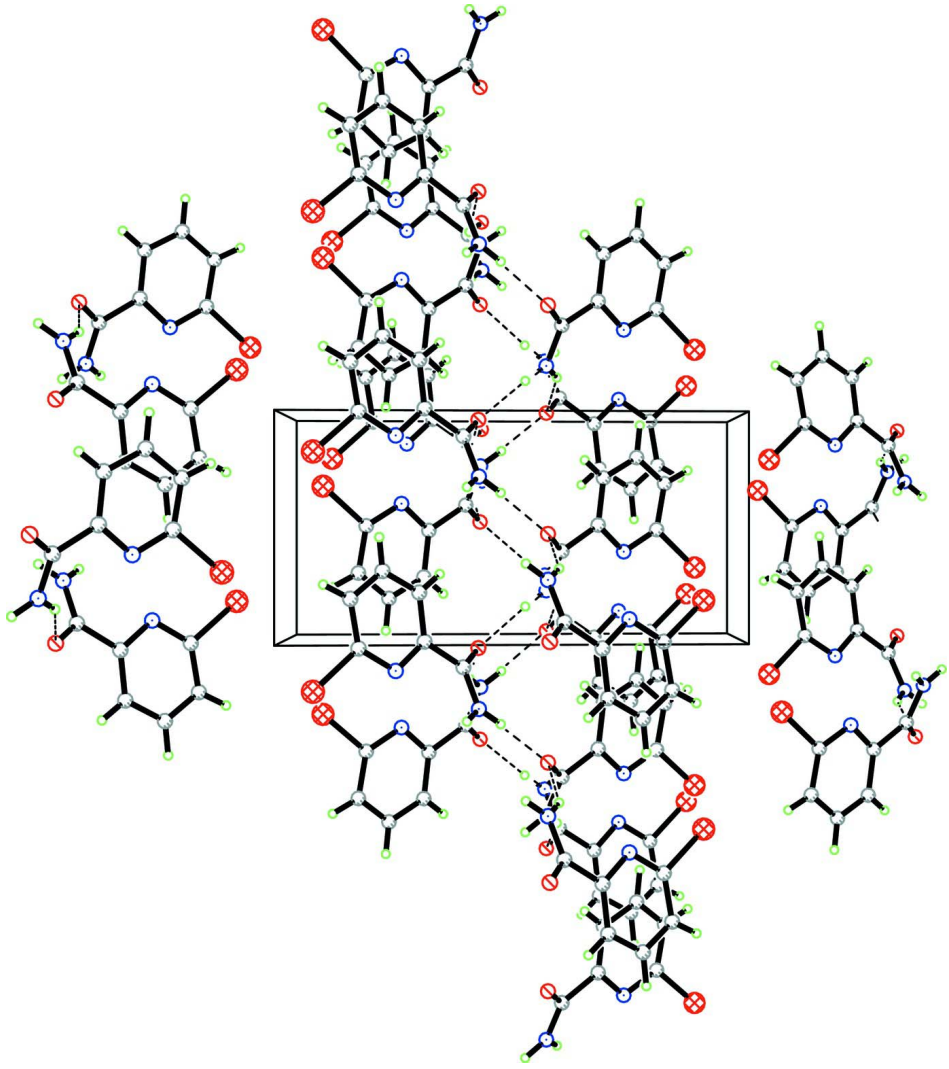

Figure 2

A partial packing diagram of (I). Hydrogen bonds are shown as dashed lines.

6-Bromopyridine-2-carboxamide

Crystal data

$C_6H_5BrN_2O$
$M_r = 201.03$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 13.034\ (3)$ Å
$b = 6.4050\ (13)$ Å
$c = 8.5540\ (17)$ Å
$\beta = 94.85\ (3)°$
$V = 711.6\ (2)$ Å^3
$Z = 4$

$F(000) = 392$
$D_x = 1.877$ Mg m^{-3}
Melting point: 417 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9–12°$
$\mu = 5.70$ mm^{-1}
$T = 293$ K
Block, colourless
0.20 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.395, T_{max} = 0.599
1354 measured reflections
1296 independent reflections
756 reflections with $I > 2\sigma(I)$
R_{int} = 0.063
θ_{max} = 25.3°, θ_{min} = 1.6°
$h = -15\rightarrow0$
$k = 0\rightarrow7$
$l = -10\rightarrow10$
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.172
S = 1.01
1296 reflections
91 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
$w = 1/[\sigma^2(F_o^2) + (0.095P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.57 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2$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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br	0.91125 (7)	0.18241 (18)	0.81361 (11)	0.0756 (5)
O	0.5752 (4)	0.0268 (9)	1.2691 (6)	0.0584 (15)
N1	0.7461 (5)	0.1078 (10)	0.9836 (6)	0.0440 (16)
N2	0.5728 (6)	0.2794 (11)	1.0883 (8)	0.061 (2)
H2A	0.5265	0.3491	1.1317	0.073*
H2B	0.5970	0.3260	1.0045	0.073*
C1	0.8216 (6)	0.0086 (14)	0.9207 (8)	0.049 (2)
C2	0.8389 (7)	−0.2007 (15)	0.9279 (9)	0.057 (2)
H2C	0.8929	−0.2615	0.8798	0.068*
C3	0.7743 (7)	−0.3160 (14)	1.0077 (10)	0.059 (2)
H3A	0.7813	−0.4605	1.0110	0.071*
C4	0.6990 (7)	−0.2223 (12)	1.0837 (9)	0.052 (2)
H4A	0.6566	−0.2999	1.1439	0.062*
C5	0.6875 (6)	−0.0119 (11)	1.0690 (7)	0.0408 (18)
C6	0.6063 (6)	0.1033 (13)	1.1493 (8)	0.0417 (18)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br	0.0750 (7)	0.0972 (9)	0.0596 (7)	−0.0039 (6)	0.0341 (5)	0.0056 (6)
O	0.084 (4)	0.056 (3)	0.039 (3)	−0.012 (3)	0.031 (3)	0.006 (3)
N1	0.063 (4)	0.046 (4)	0.023 (3)	0.000 (3)	0.007 (3)	−0.002 (3)
N2	0.088 (5)	0.057 (4)	0.044 (4)	0.014 (4)	0.039 (4)	0.011 (4)
C1	0.065 (5)	0.058 (6)	0.025 (4)	0.012 (5)	0.010 (3)	0.001 (4)
C2	0.068 (5)	0.063 (6)	0.038 (5)	0.015 (5)	0.008 (4)	−0.008 (4)
C3	0.089 (6)	0.045 (5)	0.044 (5)	0.006 (5)	0.005 (5)	−0.004 (4)
C4	0.077 (5)	0.042 (5)	0.037 (4)	−0.002 (5)	0.012 (4)	0.000 (4)
C5	0.061 (5)	0.038 (4)	0.024 (4)	−0.005 (4)	0.009 (3)	0.003 (3)
C6	0.057 (5)	0.038 (4)	0.032 (4)	−0.007 (4)	0.020 (3)	−0.006 (4)

Geometric parameters (Å, º)

Br—C1	1.905 (8)	C2—C3	1.349 (12)
O—C6	1.235 (8)	C2—H2C	0.9300
N1—C1	1.322 (9)	C3—C4	1.362 (12)
N1—C5	1.342 (9)	C3—H3A	0.9300
N2—C6	1.303 (10)	C4—C5	1.361 (10)
N2—H2A	0.8600	C4—H4A	0.9300
N2—H2B	0.8600	C5—C6	1.503 (10)
C1—C2	1.360 (11)		
C1—N1—C5	115.1 (6)	C2—C3—H3A	119.8
C6—N2—H2A	120.0	C4—C3—H3A	119.8
C6—N2—H2B	120.0	C5—C4—C3	118.1 (8)
H2A—N2—H2B	120.0	C5—C4—H4A	121.0
N1—C1—C2	125.6 (8)	C3—C4—H4A	121.0
N1—C1—Br	115.0 (6)	N1—C5—C4	123.6 (7)
C2—C1—Br	119.4 (6)	N1—C5—C6	115.1 (6)
C3—C2—C1	117.0 (8)	C4—C5—C6	121.4 (7)
C3—C2—H2C	121.5	O—C6—N2	123.6 (7)
C1—C2—H2C	121.5	O—C6—C5	118.5 (7)
C2—C3—C4	120.4 (8)	N2—C6—C5	117.8 (6)
C5—N1—C1—C2	4.5 (11)	C1—N1—C5—C6	176.1 (6)
C5—N1—C1—Br	−175.2 (5)	C3—C4—C5—N1	0.2 (12)
N1—C1—C2—C3	−0.9 (13)	C3—C4—C5—C6	−179.9 (7)
Br—C1—C2—C3	178.7 (6)	N1—C5—C6—O	−154.5 (7)
C1—C2—C3—C4	−3.3 (13)	C4—C5—C6—O	25.5 (11)
C2—C3—C4—C5	3.6 (13)	N1—C5—C6—N2	25.3 (10)
C1—N1—C5—C4	−4.0 (10)	C4—C5—C6—N2	−154.6 (8)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*B*···N1	0.86	2.41	2.730 (10)	102
N2—H2*A*···O^{i}	0.86	1.99	2.849 (9)	176
N2—H2*B*···O^{ii}	0.86	2.22	3.002 (9)	151

Symmetry codes: (i) −*x*+1, *y*+1/2, −*z*+5/2; (ii) *x*, −*y*+1/2, *z*−1/2.