2-Chloropyridine-3-carboxamide

Yu-Peng Hua, Ying Xu, Xue-Hong Wei and Hong-Bo Tong*

Institute of Applied Chemistry, Shanxi University, Taiyuan 030006, People's Republic of China
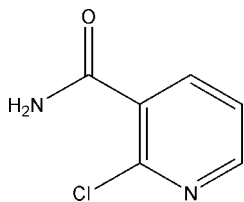
Correspondence e-mail: tong@sxu.edu.cn

In the crystal structure of the title compound, $C_6H_5ClN_2O$, the dihedral angle between the pyridine ring and the carboxamine group is 63.88 (8)°. Intermolecular N—H⋯N and N—H⋯O hydrogen bonds link the molecules into a two-dimensional network.

Related literature

Details of applications of the title compound can be found in: Oda *et al.* (1993); Qin *et al.* (2001).

Experimental

Crystal data

$C_6H_5ClN_2O$	$V = 675.8$ (8) Å³
$M_r = 156.57$	$Z = 4$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
$a = 6.980$ (5) Å	$\mu = 0.49$ mm⁻¹
$b = 13.627$ (9) Å	$T = 293$ (2) K
$c = 7.108$ (5) Å	$0.30 \times 0.20 \times 0.20$ mm
$\beta = 91.82$ (5)°	

Data collection

Siemens SMART CCD area-detector diffractometer	2716 measured reflections
	1188 independent reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)	1083 reflections with $I > 2\sigma(I)$
$T_{min} = 0.868$, $T_{max} = 0.909$	$R_{int} = 0.021$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.034$	92 parameters
$wR(F^2) = 0.096$	H-atom parameters constrained
$S = 1.11$	$\Delta\rho_{max} = 0.18$ e Å⁻³
1188 reflections	$\Delta\rho_{min} = -0.23$ e Å⁻³

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N2—H2A⋯N1[i]	0.86	2.21	3.003 (3)	154
N2—H2B⋯O[ii]	0.86	2.17	3.015 (3)	168

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; 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: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NC2131).

References

Oda, M., Sakaki, T., Sasaki, N., Nonaka, H., Yamagishi, K. & Tomita, H. (1993). *J. Pestic. Sci.* **18**, 49–57.
Qin, Z., Jennings, M. C. & Puddephatt, R. J. (2001). *Inorg. Chem.* **40**, 6220–6228.
Sheldrick, G. M. (1997). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison Wisconsin, USA.

supporting information

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S1. Comment

The structure of 2-chloropyridine-3-carboxamide has attracted us owing to its fungicidal activities (Oda *et al.*, 1993) and its application in coordination chemistry (Qin *et al.*, 2001). The dihedral angles formed by the pyridine ring and the carboxamine group amount to 63.88 (8)° (Fig. 1). The molecules are connected via intermolecular N—H···N and N—H···O hydrogen bonding into layers, with H···N distances of 2.21 and O···H distances of 2.17 Å (Fig. 2 and Tab. 1).

S2. Experimental

Ammonia (10 ml, 66 mmol, 25%) was added slowly to a solution of 2-chloropyridine-3-carbonyl chloride (4.0 g, 22 mmol) in THF (20 ml) at 0°C. The reaction mixture was allowed to warm up to room temperature and stirred for 1.5 h. The resulting mixture was dried under vacuum and washed with two 20 ml portions of THF. Then the solution was dried over anhydrous magnesium sulfate. The solvent was removed by vacuum, and the product was collected, yield: 1.93 g, 56%; m.p. 162.5°C. The crystal suitable for X-ray analysis was grown by slow evaporation of the solvent from a diethyl ether solution at 20°. Anal. Calcd for $C_6H_5ClN_2O$: C, 45.97; H, 3.14; N, 17.82%. Found: C, 46.03; H, 3.22; N, 17.89%.

S3. Refinement

All H atoms were positioned with idealized geometry, with C—H = 0.96 and N—H = 0.86 Å, and were refined with U_{iso}(H) values set to 1.2 U_{eq}(C,N).
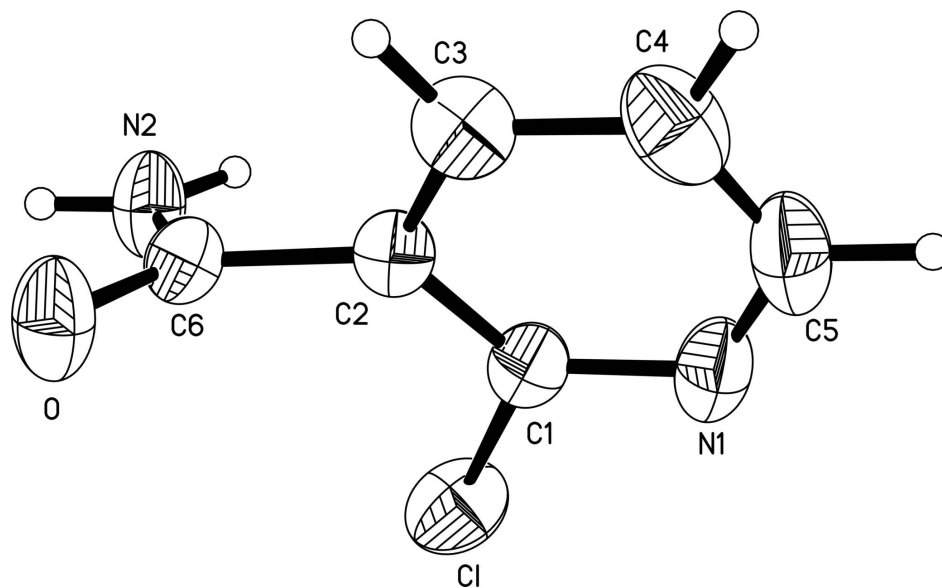
Figure 1

View of the molecule of the title compound showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.
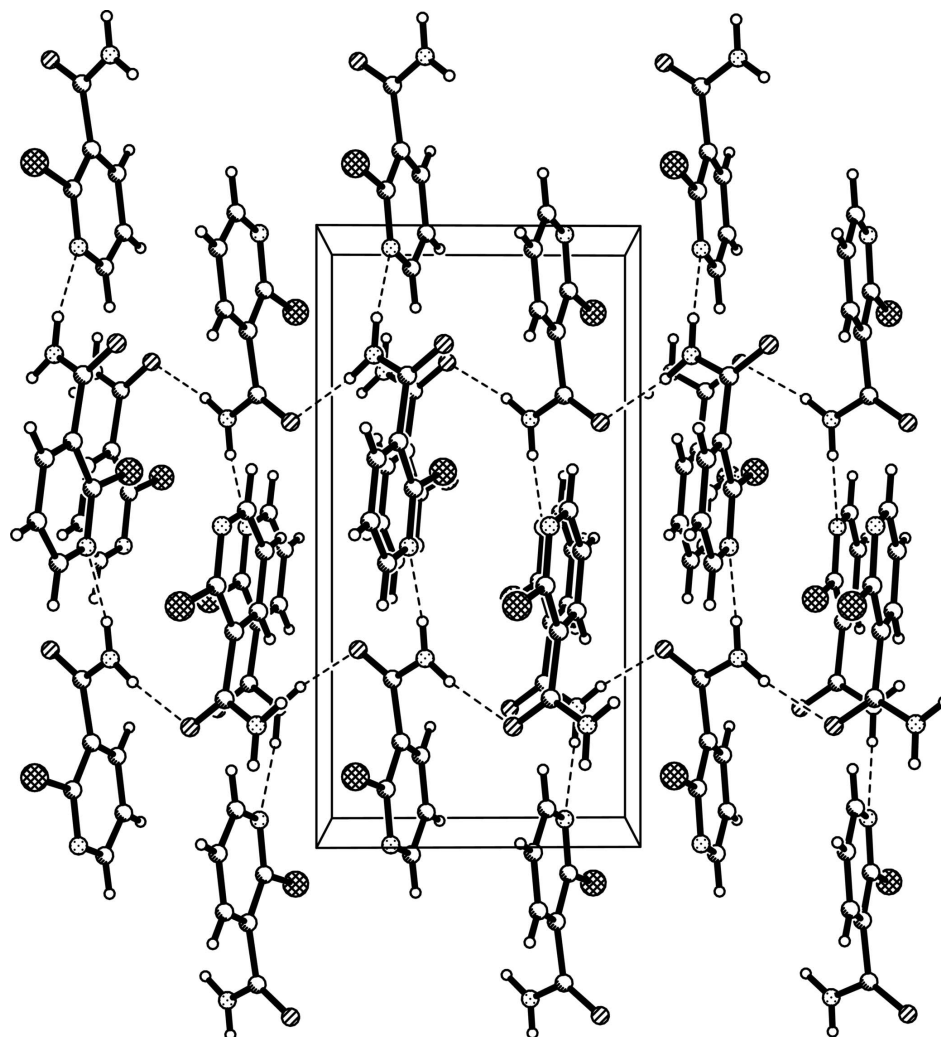
Figure 2

Crystal structure of the title compound along [100] with intermolecular N—H···N and N—H···O hydrogen bonding shown as dashed lines.

2-Chloropyridine-3-carboxamide

Crystal data

$C_6H_5ClN_2O$	$F(000) = 320$
$M_r = 156.57$	$D_x = 1.539$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 6.980\ (5)$ Å	Cell parameters from 1764 reflections
$b = 13.627\ (9)$ Å	$\theta = 2.9–26.9°$
$c = 7.108\ (5)$ Å	$\mu = 0.49$ mm^{-1}
$\beta = 91.82\ (5)°$	$T = 293$ K
$V = 675.8\ (8)$ Å^3	Plate, yellow
$Z = 4$	$0.30 \times 0.20 \times 0.20$ mm

Data collection

Siemens SMART CCD area-detector diffractometer	2716 measured reflections
Radiation source: fine-focus sealed tube	1188 independent reflections
Graphite monochromator	1083 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.021$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)	$\theta_{max} = 25.0°$, $\theta_{min} = 3.0°$
	$h = -8\rightarrow6$
	$k = -15\rightarrow16$
$T_{min} = 0.868$, $T_{max} = 0.909$	$l = -6\rightarrow8$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.034$	H-atom parameters constrained
$wR(F^2) = 0.096$	$w = 1/[\sigma^2(F_o^2) + (0.0551P)^2 + 0.1035P]$
$S = 1.11$	where $P = (F_o^2 + 2F_c^2)/3$
1188 reflections	$(\Delta/\sigma)_{max} < 0.001$
92 parameters	$\Delta\rho_{max} = 0.18$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.23$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: *SHELXL97* (Sheldrick, 2008), Fc*=kFc[1+0.001xFc$^2\lambda^3$/sin(2θ)]$^{-1/4}$
Secondary atom site location: difference Fourier map	Extinction coefficient: 0.051 (8)

Special details

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

	x	y	z	U_{iso}*/U_{eq}
Cl	0.14993 (7)	0.10791 (4)	0.88571 (7)	0.0533 (2)
C1	0.3716 (2)	0.07827 (13)	0.7965 (2)	0.0349 (4)
N1	0.4064 (2)	−0.01655 (11)	0.7824 (2)	0.0446 (4)
O	0.5546 (2)	0.30761 (9)	0.8932 (2)	0.0507 (4)
C6	0.4574 (2)	0.25974 (12)	0.7786 (2)	0.0351 (4)
N2	0.3180 (2)	0.29749 (11)	0.6708 (2)	0.0446 (4)
H2A	0.2909	0.3589	0.6786	0.054*
H2B	0.2545	0.2606	0.5930	0.054*
C3	0.6760 (3)	0.12278 (14)	0.6910 (3)	0.0430 (5)
H3	0.7681	0.1692	0.6616	0.052*
C4	0.7153 (3)	0.02376 (16)	0.6735 (3)	0.0505 (5)
H4	0.8331	0.0026	0.6312	0.061*
C2	0.4989 (2)	0.15240 (12)	0.7524 (2)	0.0325 (4)
C5	0.5774 (3)	−0.04231 (14)	0.7198 (3)	0.0506 (6)
H5	0.6042	−0.1088	0.7071	0.061*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl	0.0357 (3)	0.0599 (4)	0.0647 (4)	−0.0034 (2)	0.0066 (2)	0.0116 (2)
C1	0.0347 (9)	0.0346 (9)	0.0350 (9)	−0.0023 (7)	−0.0060 (7)	0.0017 (7)
N1	0.0541 (10)	0.0302 (8)	0.0485 (9)	−0.0029 (7)	−0.0125 (8)	0.0009 (6)
O	0.0548 (9)	0.0356 (7)	0.0603 (9)	−0.0041 (6)	−0.0181 (7)	−0.0060 (6)
C6	0.0333 (9)	0.0319 (9)	0.0399 (9)	−0.0025 (7)	−0.0003 (7)	0.0020 (7)
N2	0.0456 (9)	0.0300 (8)	0.0573 (10)	0.0039 (6)	−0.0124 (8)	−0.0020 (7)
C3	0.0336 (10)	0.0493 (12)	0.0460 (10)	0.0009 (8)	−0.0022 (8)	−0.0017 (8)
C4	0.0431 (11)	0.0566 (13)	0.0512 (12)	0.0162 (9)	−0.0069 (9)	−0.0109 (9)
C2	0.0298 (9)	0.0335 (9)	0.0337 (9)	0.0002 (7)	−0.0051 (7)	−0.0002 (7)
C5	0.0649 (14)	0.0346 (10)	0.0509 (11)	0.0130 (9)	−0.0191 (10)	−0.0081 (8)

Geometric parameters (Å, º)

Cl—C1	1.738 (2)	N2—H2B	0.8600
C1—N1	1.319 (2)	C3—C4	1.383 (3)
C1—C2	1.388 (3)	C3—C2	1.385 (3)
N1—C5	1.334 (3)	C3—H3	0.9300
O—C6	1.230 (2)	C4—C5	1.366 (3)
C6—N2	1.324 (2)	C4—H4	0.9300
C6—C2	1.504 (3)	C5—H5	0.9300
N2—H2A	0.8600		
N1—C1—C2	125.08 (18)	C4—C3—H3	120.2
N1—C1—Cl	115.07 (14)	C2—C3—H3	120.2
C2—C1—Cl	119.80 (14)	C5—C4—C3	118.6 (2)
C1—N1—C5	116.88 (16)	C5—C4—H4	120.7
O—C6—N2	123.86 (17)	C3—C4—H4	120.7
O—C6—C2	119.55 (15)	C3—C2—C1	116.34 (17)
N2—C6—C2	116.57 (15)	C3—C2—C6	120.00 (16)
C6—N2—H2A	120.0	C1—C2—C6	123.56 (16)
C6—N2—H2B	120.0	N1—C5—C4	123.49 (18)
H2A—N2—H2B	120.0	N1—C5—H5	118.3
C4—C3—C2	119.63 (19)	C4—C5—H5	118.3

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···N1[i]	0.86	2.21	3.003 (3)	154
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