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2-Chloropyridine-3-carboxamide

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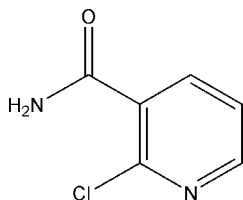
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.034; wR factor = 0.096; data-to-parameter ratio = 12.9.

In the crystal structure of the title compound, $\text{C}_6\text{H}_5\text{ClN}_2\text{O}$, the dihedral angle between the pyridine ring and the carboxamide group is $63.88(8)^\circ$. Intermolecular $\text{N}-\text{H}\cdots\text{N}$ and $\text{N}-\text{H}\cdots\text{O}$ 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

$\text{C}_6\text{H}_5\text{ClN}_2\text{O}$
 $M_r = 156.57$
 Monoclinic, $P2_1/n$
 $a = 6.980(5)$ Å
 $b = 13.627(9)$ Å
 $c = 7.108(5)$ Å
 $\beta = 91.82(5)^\circ$

$V = 675.8(8)$ Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.49$ mm⁻¹
 $T = 293(2)$ K
 $0.30 \times 0.20 \times 0.20$ mm

Data collection

Siemens SMART CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1997)
 $T_{\min} = 0.868$, $T_{\max} = 0.909$

2716 measured reflections
 1188 independent reflections
 1083 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.021$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.034$
 $wR(F^2) = 0.096$
 $S = 1.11$
 1188 reflections

92 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.18$ e Å⁻³
 $\Delta\rho_{\min} = -0.23$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N2}-\text{H2A}\cdots\text{N1}^{\text{i}}$	0.86	2.21	3.003 (3)	154
$\text{N2}-\text{H2B}\cdots\text{O}^{\text{ii}}$	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.

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

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supplementary materials

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2-Chloropyridine-3-carboxamide

Y.-P. Hua, Y. Xu, X.-H. Wei and H.-B. Tong

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 carboxamide 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).

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 C₆H₅ClN₂O: C, 45.97; H, 3.14; N, 17.82%. Found: C, 46.03; H, 3.22; N, 17.89%.

Refinement

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

Figures

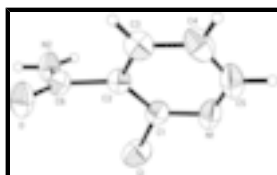


Fig. 1. View of the molecule of the title compound showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

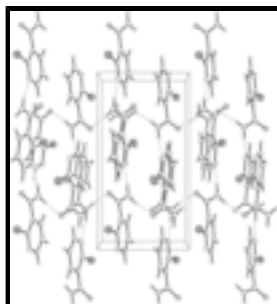


Fig. 2. Crystal structure of the title compound along [100] with intermolecular N—H···N and N—H···O hydrogen bonding shown as dashed lines.

2-Chloropyridine-3-carboxamide

Crystal data

$C_6H_5ClN_2O$

$M_r = 156.57$

Monoclinic, $P2_1/n$

$a = 6.980$ (5) Å

$b = 13.627$ (9) Å

$c = 7.108$ (5) Å

$\beta = 91.82$ (5)°

$V = 675.8$ (8) Å³

$Z = 4$

$F_{000} = 320$

$D_x = 1.539$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 1764 reflections

$\theta = 2.9$ – 26.9 °

$\mu = 0.49$ mm⁻¹

$T = 293$ (2) K

Plate, yellow

$0.30 \times 0.20 \times 0.20$ mm

Data collection

Siemens SMART CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 293$ (2) K

ω scans

Absorption correction: multi-scan (SADABS; Sheldrick, 1997)

$T_{\min} = 0.868$, $T_{\max} = 0.909$

2716 measured reflections

1188 independent reflections

1083 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.021$

$\theta_{\text{max}} = 25.0$ °

$\theta_{\text{min}} = 3.0$ °

$h = -8$ → 6

$k = -15$ → 16

$l = -6$ → 8

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.034$

$wR(F^2) = 0.096$

$S = 1.11$

1188 reflections

92 parameters

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

$$w = 1/[\sigma^2(F_o^2) + (0.0551P)^2 + 0.1035P]$$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} < 0.001$

$\Delta\rho_{\text{max}} = 0.18$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.23$ e Å⁻³

Extinction correction: SHELXL97 (Sheldrick, 2008),

$$F_c^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$$

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	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 (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	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 (\AA , $^\circ$)

Cl—C1	1.738 (2)	N2—H2B	0.8600
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supplementary materials

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—C1	115.07 (14)	C2—C3—H3	120.2
C2—C1—C1	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 (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N2—H2A \cdots N1 ⁱ	0.86	2.21	3.003 (3)	154
N2—H2B \cdots O ⁱⁱ	0.86	2.17	3.015 (3)	168

Symmetry codes: (i) $-x+1/2, y+1/2, -z+3/2$; (ii) $x-1/2, -y+1/2, z-1/2$.

Fig. 1

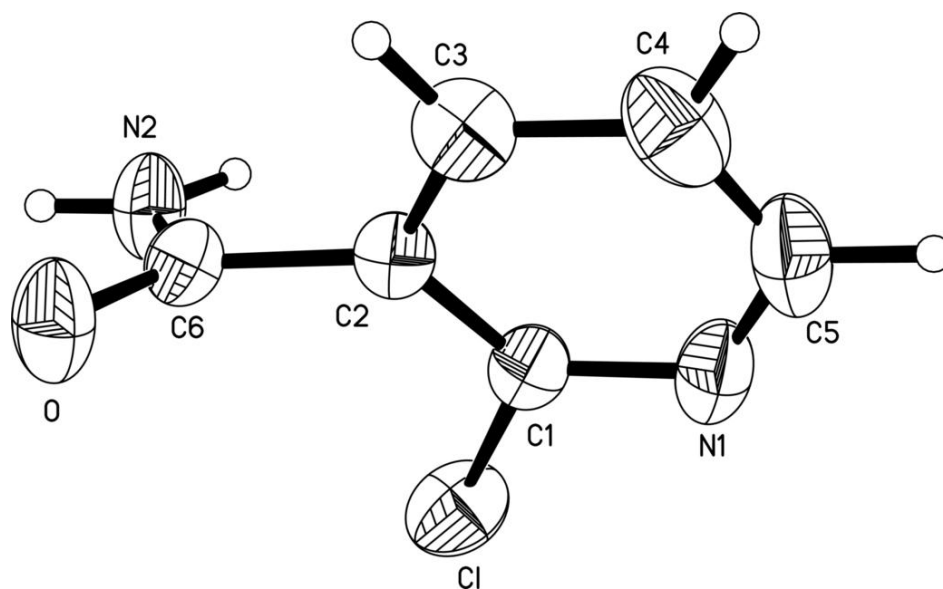


Fig. 2

