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3-Amino-6-chloropyridazine at 125 K

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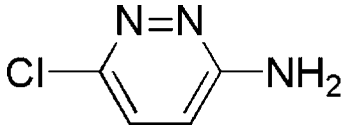
Key indicators: single-crystal X-ray study; T = 125 K; mean σ(C–C) = 0.002 Å; R factor = 0.033; wR factor = 0.088; data-to-parameter ratio = 16.8.

Molecules of the title compound, $C_4H_4ClN_3$, are essentially planar. N—H···N hydrogen bonds link the molecules into a two-dimensional network parallel to the (100) plane. The pyridazine rings of inversion-related molecules are stacked with their centroids separated by a distance of 3.7597 (10) Å, indicating π–π interactions.

Related literature

For synthesis, see: Steck *et al.* (1954). For general background, see: Ishida *et al.* (1994); Pitarch *et al.* (1974); Herter *et al.* (1989); Guery *et al.* (2001). For related structures, see: Gong & Krische (2005). For the Cambridge Structural Database (CSD), see: Allen (2002).



Experimental

Crystal data

$C_4H_4ClN_3$
M_r = 129.55
Monoclinic, $P2_1/c$
a = 7.5235 (14) Å
b = 6.5887 (11) Å
c = 11.1748 (19) Å
β = 105.045 (10)°
V = 534.95 (16) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.59 mm^{-1}
T = 125 (2) K
0.50 × 0.50 × 0.40 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{\min}$ = 0.756, $T_{\max}$ = 0.794
5258 measured reflections
1227 independent reflections
1153 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.015

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.088
S = 1.12
1227 reflections
73 parameters
H-atom parameters constrained
$\Delta\rho_{\max}$ = 0.25 e Å^{-3}
$\Delta\rho_{\min}$ = −0.35 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3A···N2$^{\rm i}$	0.86	2.26	3.1038 (18)	168
N3—H3B···N1$^{\rm ii}$	0.86	2.31	3.1372 (17)	162

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

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2002); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2455).

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supplementary materials

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3-Amino-6-chloropyridazine at 125 K

B. Gao

Comment

Pyridazines have demonstrated versatile biological activities, for example, antibacterial (Ishida *et al.*, 1994), antidepressant (Pitarch *et al.*, 1974) and antihypertensive (Herter *et al.*, 1989) activities. A search of the Cambridge Structural Database (CSD, 2007 Release, Version 5.28; Allen, 2002) reveals that there are 639 crystal structures containing the pyridazine moiety. In the design of functional synthetic oligomers and ploymers, 3,6-diaminopyridazine is applied to the design of related monomeric, dimeric and trimeric duplex molecular strands (Gong *et al.*, 2005). And in the preparation of various pyridazines, 3-amino-6-chloropyridazine is an important intermediate (Guery *et al.*, 2001). We report here the crystal structure of the title compound.

In the title molecule (Fig. 1), the chloro and amino groups are coplanar with the pyridazine ring. Atoms N3 and Cl1 deviate from the pyridazine plane by 0.030 (1) and 0.019 (1) Å, respectively. The N1—N2 distance of 1.3548 (17) Å is slightly shorter than the N—N distance of 1.379 (2)Å reported by Gong *et al.* (2005).

The intermolecular N—H···N hydrogen bonds (Table 1) link the molecules into a two-dimensional network parallel to the (100) plane (Fig.2). The pyridazine rings of the inversion-related molecules at (x, y, z) and $(-x, 1 - y, -z)$ are stacked with their centroids separated by a distance of 3.7597 (10) Å, indicating π-π interactions. In addition, C—Cl···π [Cl···pyridazine ring centroid = 3.6065 (10) Å] interactions also contribute to the stabilization of the structure.

Experimental

The title compound was prepared according to the literature method (Steck *et al.*, 1954). Crystals suitable for X-ray analysis were obtained by slow evaporation of a isoproanol solution at room temperature (m.p. 483–485 K).

Refinement

H atoms were positioned geometrically (N—H = 0.86 Å and C—H = 0.93 Å) and refined using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2\text{–}1.5U_{\text{eq}}(\text{C})$.

Figures

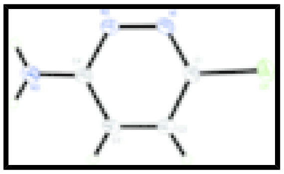

Fig. 1. Molecular structure of the title compound, showing 50% probability displacement ellipsoids and the atomic numbering.

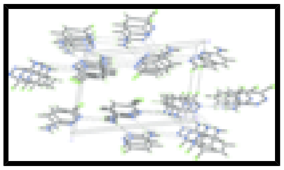

Fig. 2. The crystal packing of the title compound, viewed down the *b* axis. Dashed lines indicate intermolecular hydrogen bonds.

3-Amino-6-chloropyridazine

Crystal data

$C_4H_4ClN_3$

$M_r = 129.55$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 7.5235$ (14) Å

$b = 6.5887$ (11) Å

$c = 11.1748$ (19) Å

$\beta = 105.045$ (10)°

$V = 534.95$ (16) Å^3

$Z = 4$

$F_{000} = 264$

$D_x = 1.609$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å

Cell parameters from 1227 reflections

$\theta = 2.8$–27.6°

$\mu = 0.59$ mm^{-1}

$T = 125$ (2) K

Block, colourless

0.50 × 0.50 × 0.40 mm

Data collection

Bruker SMART CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 125(2)$ K

φ and ω scans

Absorption correction: multi-scan (SADABS; Bruker, 2002)

$T_{min} = 0.756$, $T_{max} = 0.794$

5258 measured reflections

1227 independent reflections

1153 reflections with $I > 2\sigma(I)$

$R_{int} = 0.015$

$\theta_{max} = 27.6$°

$\theta_{min} = 2.8$°

$h = -8 \rightarrow 9$

$k = -8 \rightarrow 8$

$l = -14 \rightarrow 12$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.033$

$wR(F^2) = 0.088$

$S = 1.12$

1227 reflections

73 parameters

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.047P)^2 + 0.167P]$
where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{max} = 0.25$ e Å^{-3}

$\Delta\rho_{min} = -0.35$ e Å^{-3}

Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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}
Cl1	0.34908 (6)	0.22748 (6)	0.47446 (3)	0.04506 (16)
N1	0.19326 (16)	0.58112 (18)	0.46416 (10)	0.0322 (3)
C4	0.17199 (19)	0.7781 (2)	0.63301 (12)	0.0303 (3)
N3	0.11738 (19)	0.9525 (2)	0.67524 (11)	0.0439 (3)
H3A	0.0622	1.0436	0.6238	0.053*
H3B	0.1377	0.9727	0.7537	0.053*
C2	0.3197 (2)	0.4557 (2)	0.67021 (12)	0.0353 (3)
H2	0.3804	0.3520	0.7212	0.042*
C3	0.26466 (19)	0.6271 (2)	0.71699 (11)	0.0341 (3)
H3	0.2870	0.6455	0.8021	0.041*
C1	0.28026 (18)	0.4428 (2)	0.54031 (12)	0.0301 (3)
N2	0.13712 (17)	0.75340 (18)	0.50956 (11)	0.0330 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0612 (3)	0.0370 (2)	0.0391 (2)	0.00197 (15)	0.01685 (18)	−0.00858 (13)
N1	0.0413 (6)	0.0333 (6)	0.0217 (5)	−0.0046 (5)	0.0078 (4)	−0.0009 (4)
C4	0.0343 (6)	0.0328 (7)	0.0236 (6)	−0.0024 (5)	0.0068 (5)	0.0002 (5)
N3	0.0626 (8)	0.0405 (7)	0.0262 (6)	0.0139 (6)	0.0073 (5)	−0.0010 (5)
C2	0.0442 (7)	0.0359 (7)	0.0244 (6)	0.0042 (6)	0.0064 (5)	0.0048 (5)
C3	0.0428 (7)	0.0395 (7)	0.0187 (5)	0.0015 (6)	0.0056 (5)	0.0018 (5)
C1	0.0360 (6)	0.0301 (6)	0.0251 (6)	−0.0046 (5)	0.0097 (5)	−0.0026 (5)
N2	0.0429 (6)	0.0327 (6)	0.0218 (5)	0.0001 (4)	0.0057 (5)	0.0023 (4)

Geometric parameters (Å, °)

Cl1—C1	1.7378 (14)	N3—H3A	0.86
N1—C1	1.3015 (18)	N3—H3B	0.86
N1—N2	1.3548 (17)	C2—C3	1.354 (2)
C4—N2	1.3456 (17)	C2—C1	1.4067 (18)

C4—N3	1.3460 (18)	C2—H2	0.93
C4—C3	1.4204 (19)	C3—H3	0.93
C1—N1—N2	119.64 (11)	C1—C2—H2	121.8
N2—C4—N3	117.82 (12)	C2—C3—C4	118.44 (12)
N2—C4—C3	121.61 (12)	C2—C3—H3	120.8
N3—C4—C3	120.57 (12)	C4—C3—H3	120.8
C4—N3—H3A	120.0	N1—C1—C2	124.61 (13)
C4—N3—H3B	120.0	N1—C1—Cl1	116.70 (10)
H3A—N3—H3B	120.0	C2—C1—Cl1	118.69 (11)
C3—C2—C1	116.44 (12)	C4—N2—N1	119.25 (11)
C3—C2—H2	121.8		
C1—C2—C3—C4	0.3 (2)	C3—C2—C1—N1	−1.2 (2)
N2—C4—C3—C2	0.6 (2)	C3—C2—C1—Cl1	179.46 (11)
N3—C4—C3—C2	−178.90 (14)	N3—C4—N2—N1	178.83 (12)
N2—N1—C1—C2	1.2 (2)	C3—C4—N2—N1	−0.6 (2)
N2—N1—C1—Cl1	−179.49 (9)	C1—N1—N2—C4	−0.20 (19)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3A···N2^{i}	0.86	2.26	3.1038 (18)	168
N3—H3B···N1^{ii}	0.86	2.31	3.1372 (17)	162

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

Fig. 1

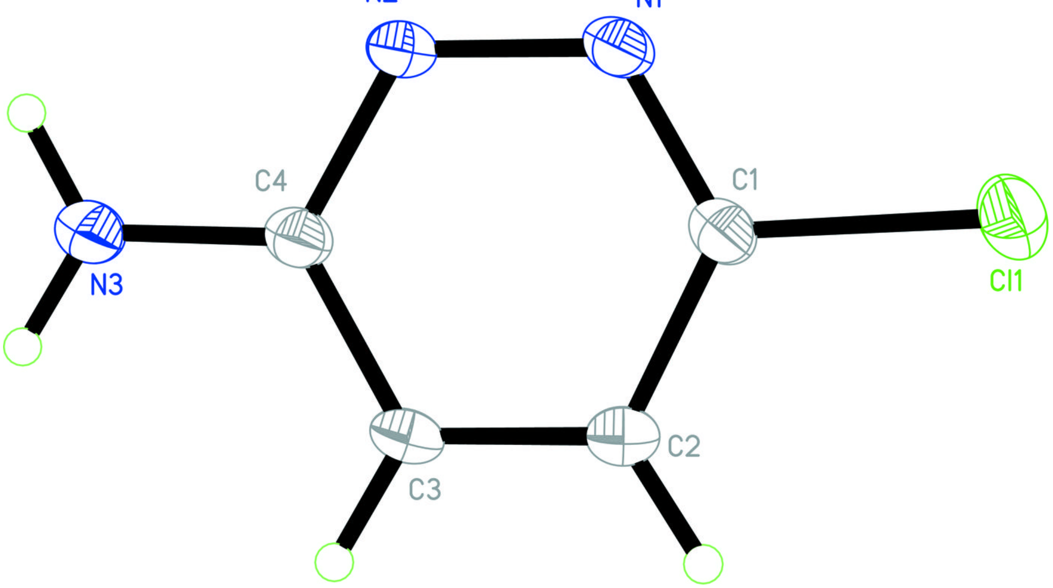

Fig. 2