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A redetermination of 2-amino-5-chloropyridine at 100 K

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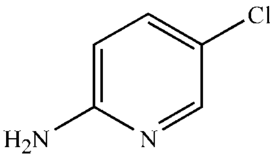
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.027; wR factor = 0.071; data-to-parameter ratio = 13.8.

X-ray and neutron diffraction studies of 2-amino-5-chloropyridine, $C_5H_5ClN_2$, were previously carried out at room temperature [Kvick & Backéus (1974). *Acta Cryst.* B**30**, 474–480; Kvick, Thomas & Koetzle (1976). *Acta Cryst.* B**32**, 224–231]. This report is a redetermination of the crystal structure at 100 K. As previously observed, molecules form centrosymmetric dimers *via* two N—H···N hydrogen bonds. In addition, C—H···π interactions are generated from molecules related by c-glide transformations, which form extended two-dimensional aggregation in the bc plane.

Related literature

For related literature, see: Almlöf *et al.* (1971); Averbuch-Pouchot *et al.* (1988); Barlow *et al.* (1989); Iitaka (1961); Kvick & Backéus (1974); Kvick *et al.* (1976); Kvick & Booles (1972); Kvick & Olovsson (1968); Pourayoubi & Mahjoub (2007); Ravikumar *et al.* (2001); Zakaria *et al.* (2002); Zou *et al.* (2003); Simpson & Marsh (1966).



Experimental

Crystal data

$C_5H_5ClN_2$
M_r = 128.56
Monoclinic, $P2_1/c$
a = 13.352 (4) Å
b = 5.7576 (16) Å
c = 7.266 (2) Å
β = 104.787 (6)°
V = 540.1 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.58 mm^{-1}
T = 100 (2) K
0.45 × 0.30 × 0.28 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: none
4557 measured reflections
1285 independent reflections
1120 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.071
S = 0.99
1285 reflections
93 parameters
All H-atom parameters refined
$\Delta\rho_{\rm max}$ = 0.35 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.26 e Å^{-3}

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

Cg is the centroid of the benzene ring.

D—H···A	D—H	H···A	D···A	D—H···A
N2—H5···N1$^{\rm i}$	0.84 (2)	2.19 (3)	3.031 (2)	175
C2—H3···$Cg^{\rm ii}$	0.95 (2)	2.71	3.414	134
C5—H1···$Cg^{\rm iii}$	0.96 (2)	2.76	3.506	131

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

We thank Professor Kvick for useful help in preparing the discussion. Support of this investigation by Imam Hossein University is gratefully acknowledged.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2530).

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supplementary materials

Acta Cryst. (2007). E**63**, o4631 [**doi:10.1107/S1600536807055328**]

A redetermination of 2-amino-5-chloropyridine at 100 K

M. Pourayoubi, S. Ghadimi and **A. A. Ebrahimi Valmoozi**

Comment

This work is a continuation of the previous studies on the influence of hydrogen bonds (Ravikumar *et al.* 2001; Zakaria *et al.* 2002; Zou *et al.* 2003) and other non-covalent forces (Pourayoubi & Mahjoub 2007) on crystal stabilization. Some investigations of crystal structures of organic molecules which can play a role as model substances for biologically systems have been reported (Simpson & Marsh 1966; Averbuch-Pouchot *et al.* 1988; Iitaka 1961; Barlow *et al.* 1989). The crystal structures of 6-chloro-2-hyroxypyridine, the addition compound 6-chloro-2-hydroxypyridine-2-pyridone and 5-chloro-2-pyridone have been reported by Kvick & Olovsson (1968), Almlöf *et al.* (1971) and Kvick & Booles (1972). Furthermore, the X-ray crystal structure and neutron diffraction study of 2-amino-5-chloropyridine was previously studied at room temperature (Kvick & Backéus 1974; Kvick *et al.* 1976). Here, we report the structure of 2-amino-5-chloropyridine (Fig. 1), $C_5H_3N(NH_2)Cl$, at 100 K. *a*, *b*, and *c* and the volume of the unit cell are 13.352 (4) Å, 5.7576 (16) Å, 7.266 (2) Å and 540.1 (3) Å^3 compared to 13.4370 (6) Å, 5.7963 (5) Å, 7.5123 (6) Å and 563.78 Å^3 at room temperature (Kvick & Backéus 1974)]. In the crystal structure, molecules exist as a centrosymmtric dimers produced *via* two N2—H1N···N1^i hydrogen bonds, Table 1. The donor···acceptor distance at 100 K is similar to the one reported in the previous work at room temperature [N···N = 3.058 (2)Å with an almost linear bond angle, N2—H1···N1=179 (2)°, (Kvick & Backéus 1974)]. The C—H..π interactions (between hydrogen atoms of the C2 and C5 and the phenyl groups) are generated from molecules related by c-glide transformations which form extended 2-D aggregation along the *bc* plane (Table 1 and Fig. 2). An alternate view of part the crystal structure (along *c* crystal axes) is shown in Fig. 3.

Experimental

Single crystal of the title compound was grown in $CHCl_3$ at room temperature.

Refinement

All hydrogen atoms were found in difference Fourier synthesis and refined with isotropic displacement parameters.

Figures

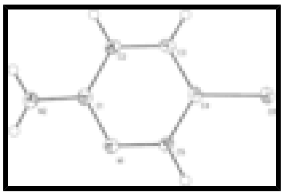


Fig. 1. Molecular structure (50% probability level) of the title compound.

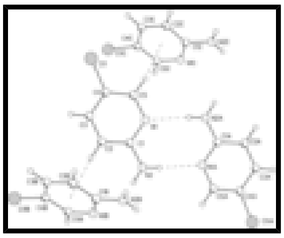


Fig. 2. The N—H···N hydrogen bonds and C—H···π interations (dotted lines) in the title compound [symmetry codes: (A) 1 − *x*, −*y*, 1 − *z*, (B) *x*, −*y* − 1/2, *z* + 1/2, (C) *x*, −*y* + 1/2, *z* − 1/2].

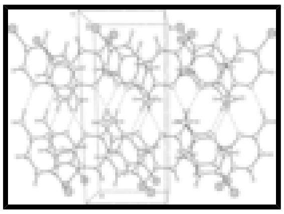


Fig. 3. Crystal packing fragment (along *c* crystal axes). Hydrogen bonds are shown with dashed lines.

2-amino-5-chloropyridine

Crystal data

$C_5H_5ClN_2$

M_r = 128.56

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

a = 13.352 (4) Å

b = 5.7576 (16) Å

c = 7.266 (2) Å

β = 104.787 (6)°

V = 540.1 (3) Å^3

Z = 4

F_{000} = 264

D_x = 1.581 Mg m^{-3}

Mo *K*α radiation
λ = 0.71073 Å

Cell parameters from 2192 reflections

θ = 3.2–30.2°

μ = 0.58 mm^{-1}

T = 100 (2) K

Plate, colourless

0.45 × 0.30 × 0.28 mm

Data collection

Bruker APEXII CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

T = 100(2) K

phi and ω scans

Absorption correction: none

4557 measured reflections

1285 independent reflections

1120 reflections with $I > 2\sigma(I)$

$R_{\rm int}$ = 0.027

$\theta_{\rm max}$ = 28.0°

$\theta_{\rm min}$ = 3.2°

$h = -17 \rightarrow 16$

$k = -7 \rightarrow 7$

$l = -9 \rightarrow 9$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.027$

Secondary atom site location: difference Fourier map

Hydrogen site location: difference Fourier map

All H-atom parameters refined

$wR(F^2) = 0.071$

$S = 0.99$

1285 reflections

93 parameters

Primary atom site location: structure-invariant direct methods

$w = 1/[\sigma^2(F_o^2) + (0.0369P)^2 + 0.2721P]$
where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{max} = 0.35$ e Å^{-3}

$\Delta\rho_{min} = -0.26$ e Å^{-3}

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 $F^2 > 2\mathrm{sigma}(F^2)$ 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	0.07655 (3)	0.32409 (6)	0.36049 (5)	0.01544 (12)
N1	0.36278 (9)	0.0679 (2)	0.46525 (17)	0.0137 (3)
N2	0.43944 (11)	−0.2738 (2)	0.5964 (2)	0.0215 (3)
C1	0.35261 (11)	−0.1409 (2)	0.5433 (2)	0.0137 (3)
C2	0.25635 (11)	−0.2195 (2)	0.56561 (19)	0.0134 (3)
C3	0.17031 (11)	−0.0806 (2)	0.50649 (19)	0.0129 (3)
C4	0.18174 (11)	0.1365 (2)	0.42853 (19)	0.0120 (3)
C5	0.27789 (11)	0.2027 (2)	0.4098 (2)	0.0125 (3)
H1	0.2870 (14)	0.350 (3)	0.354 (2)	0.010 (4)*
H2	0.1058 (16)	−0.130 (3)	0.514 (3)	0.023 (5)*
H3	0.2503 (13)	−0.367 (3)	0.621 (2)	0.013 (4)*
H4	0.4369 (16)	−0.398 (4)	0.660 (3)	0.029 (5)*
H5	0.4960 (19)	−0.226 (4)	0.580 (3)	0.038 (6)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.01112 (18)	0.01549 (19)	0.01969 (19)	0.00267 (12)	0.00392 (13)	0.00262 (12)
N1	0.0130 (6)	0.0129 (6)	0.0151 (5)	−0.0003 (4)	0.0036 (5)	0.0008 (4)
N2	0.0162 (7)	0.0180 (7)	0.0317 (8)	0.0056 (5)	0.0089 (6)	0.0107 (6)
C1	0.0145 (7)	0.0134 (6)	0.0133 (6)	0.0011 (5)	0.0036 (5)	−0.0009 (5)
C2	0.0172 (7)	0.0109 (6)	0.0126 (6)	−0.0007 (5)	0.0050 (5)	0.0000 (5)
C3	0.0134 (7)	0.0140 (7)	0.0124 (6)	−0.0030 (5)	0.0052 (5)	−0.0019 (5)
C4	0.0109 (6)	0.0121 (6)	0.0122 (6)	0.0023 (5)	0.0016 (5)	−0.0013 (5)
C5	0.0137 (7)	0.0105 (6)	0.0131 (6)	−0.0009 (5)	0.0030 (5)	−0.0005 (5)

Geometric parameters (Å, °)

Cl1—C4	1.7404 (14)	C2—C3	1.375 (2)
N1—C5	1.3479 (19)	C2—H3	0.954 (17)
N1—C1	1.3506 (19)	C3—C4	1.3970 (19)
N2—C1	1.3602 (19)	C3—H2	0.92 (2)
N2—H4	0.86 (2)	C4—C5	1.379 (2)
N2—H5	0.84 (2)	C5—H1	0.961 (16)
C1—C2	1.410 (2)		

C5—N1—C1	118.11 (12)	C2—C3—C4	118.55 (13)
C1—N2—H4	118.4 (14)	C2—C3—H2	121.5 (12)
C1—N2—H5	120.5 (16)	C4—C3—H2	119.9 (12)
H4—N2—H5	121 (2)	C5—C4—C3	119.23 (13)
N1—C1—N2	116.76 (13)	C5—C4—Cl1	120.31 (11)
N1—C1—C2	121.73 (13)	C3—C4—Cl1	120.45 (11)
N2—C1—C2	121.50 (13)	N1—C5—C4	122.98 (13)
C3—C2—C1	119.39 (13)	N1—C5—H1	116.5 (10)
C3—C2—H3	119.8 (10)	C4—C5—H1	120.5 (10)
C1—C2—H3	120.8 (10)		

C5—N1—C1—N2	179.49 (13)	C2—C3—C4—C5	1.3 (2)
C5—N1—C1—C2	0.6 (2)	C2—C3—C4—Cl1	−177.34 (11)
N1—C1—C2—C3	0.0 (2)	C1—N1—C5—C4	−0.3 (2)
N2—C1—C2—C3	−178.79 (14)	C3—C4—C5—N1	−0.7 (2)
C1—C2—C3—C4	−1.0 (2)	Cl1—C4—C5—N1	177.96 (11)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H5···N1$^{\rm i}$	0.84 (2)	2.19 (3)	3.031 (2)	175
C2—H3···Cg$^{\rm ii}$	0.95 (2)	2.71	3.414	134
C5—H1···Cg$^{\rm iii}$	0.96 (2)	2.76	3.506	131

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

Fig. 1

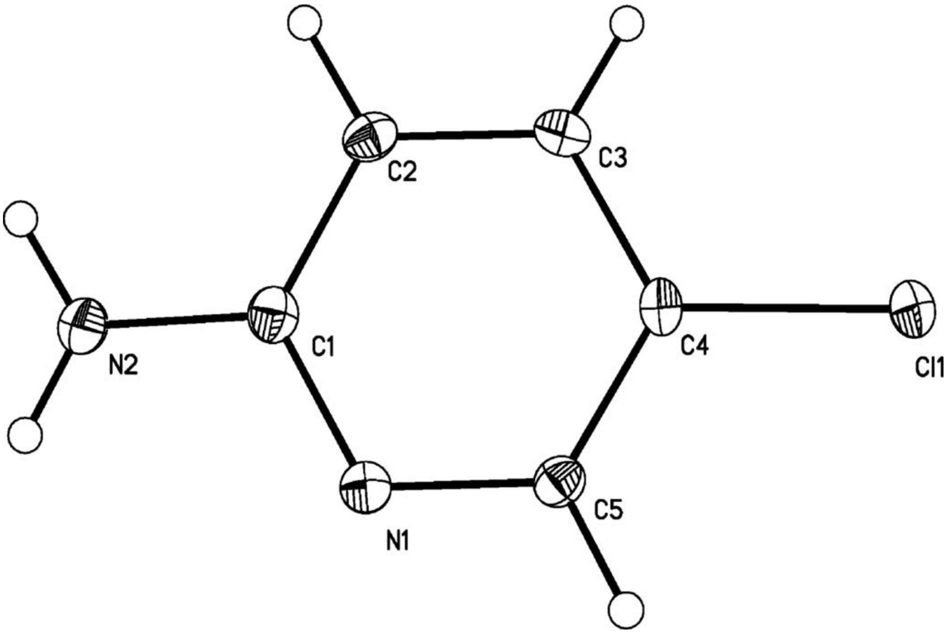
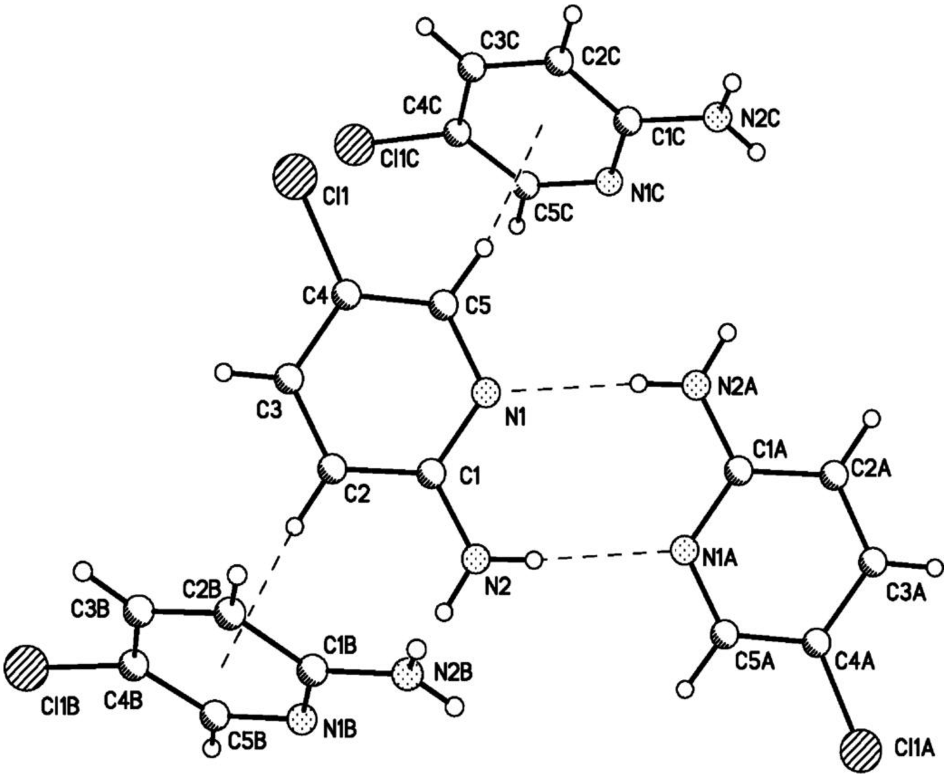


Fig. 2


C3C
C2C
C4C
N2C
C1C
Cl1C
Cl1
C5C
N1C
C4
C5
N2A
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N1
C1A
C2A
C2
C1
N1A
C2B
N2
C3A
C3B
C1B
N2B
C5A
C4A
Cl1B
C4B
C5B
N1B
Cl1A

Fig. 3