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2-Chloro-5-(chloromethyl)pyridine

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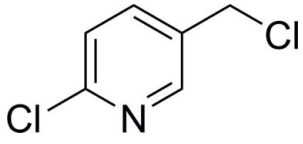
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.037; wR factor = 0.129; data-to-parameter ratio = 15.7.

The title compound, $C_6H_5Cl_2N$, is almost planar, with an r.m.s. deviation of 0.0146 Å for all atoms except for the 5-cholоromethyl Cl atom. The offset Cl atom lies above this plane with a Cl—C—C angle of 111.11 (17)°. In the crystal, molecules are connected *via* intermolecular C—H···N hydrogen bonds, forming dimers.

Related literature

For the synthetic procedure, see: Nishihara *et al.* (1993). For bond-length data, see: Allen *et al.* (1987). The title compound is an intermediate in the synthesis of imidacloprid [systematic name (*E*)-1-(6-chloro-3-pyridylmethyl)-*N*-nitroimidazolidin-2-ylideneamine], see: Shroff *et al.* (2007).



Experimental

Crystal data

$C_6H_5Cl_2N$
M_r = 162.01
Monoclinic, $P2_1/c$
a = 4.0770 (8) Å
b = 10.322 (2) Å
c = 16.891 (3) Å
β = 95.95 (3)°
V = 707.0 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.82 mm^{-1}
T = 293 K
0.30 × 0.20 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.791, T_{max} = 0.853
2886 measured reflections
1299 independent reflections
1028 reflections with $I > 2\sigma(I)$
R_{int} = 0.049
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.129
S = 1.00
1299 reflections
83 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6A···N^{i}	0.97	2.57	3.453 (3)	151

Symmetry code: (i) $-x, y-\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1985); cell refinement: *CAD-4 Software*; 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: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BQ2267).

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supporting information

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2-Chloro-5-(chloromethyl)pyridine

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S1. Comment

The tittle compound, 2-chloro-5-(chloromethyl)pyridine (I), is an important intermediate for the synthesis of imidacloprid (Shroff *et al.*, 2007) and we report here its crystal structure.

The molecular structure of (I) is shown in Fig. 1. The bond lengths and angles are within normal ranges (Allen *et al.*, 1987). In the crystal structure, the molecules were connected together *via* weak C—H···N intermolecular hydrogen bonds forming dimers, which seems to be effective in the stabilization of the crystal structure.

S2. Experimental

The title compound, (I) was prepared by a method reported in literature (Nishihara *et al.*, 1993). The crystals were obtained by dissolving (I) (0.2 g, 1.2 mmol) in ethanol (25 ml) and evaporating the solvent slowly at room temperature for about 5 d.

S3. Refinement

H atoms were positioned geometrically (C–H = 0.93–0.97 Å) and refined using a riding model with U_{iso}(H) = xU_{eq}(C, O), where x = 1.5 for methyl and oxygen H-atoms and x = 1.2 for all other H-atoms.

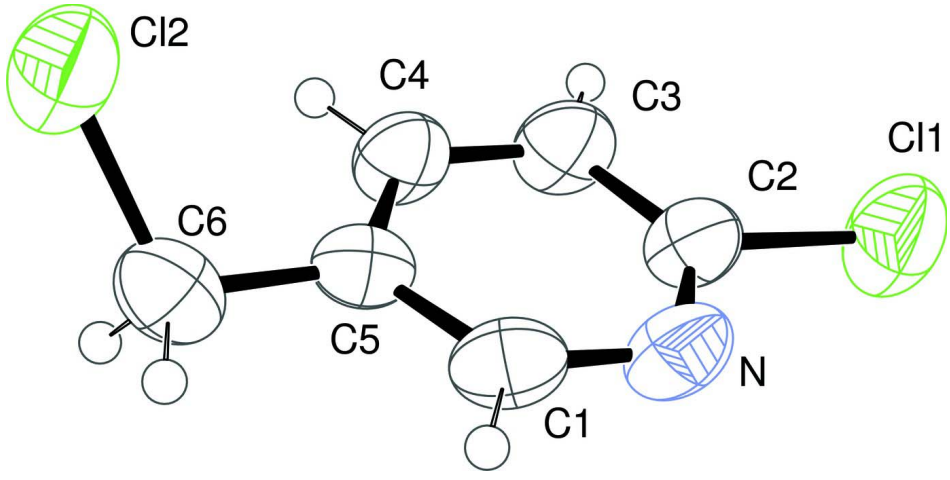

Figure 1

The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

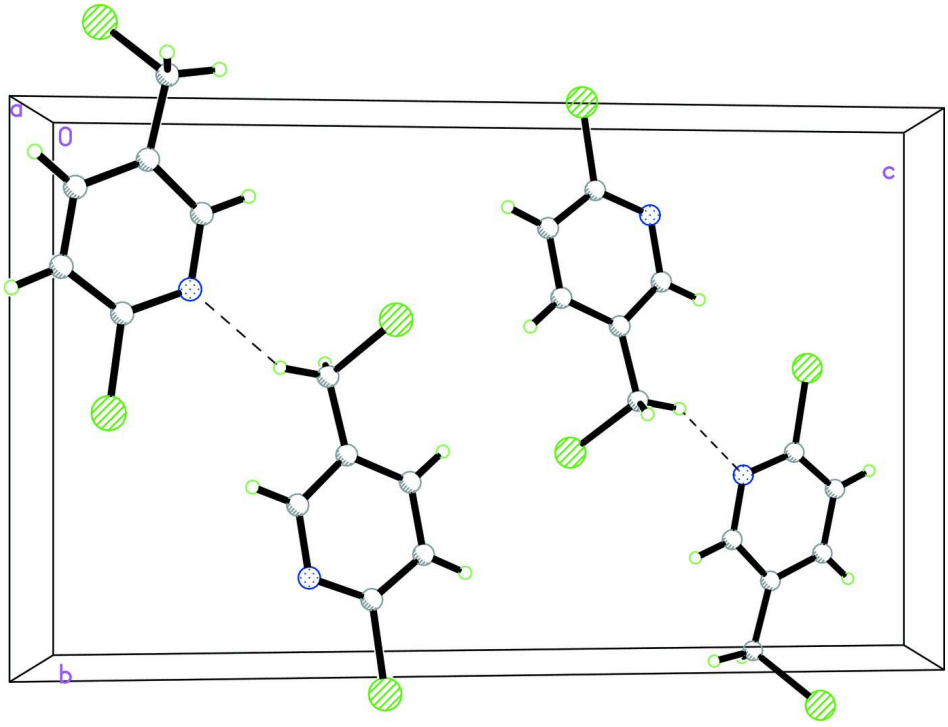

Figure 2

A packing diagram for (I). C—H···N hydrogen bonds are shown by dashed lines.

2-Chloro-5-(chloromethyl)pyridine

Crystal data

$C_6H_5Cl_2N$
$M_r = 162.01$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 4.0770\ (8)$ Å
$b = 10.322\ (2)$ Å
$c = 16.891\ (3)$ Å
$\beta = 95.95\ (3)°$
$V = 707.0\ (2)$ Å^3
$Z = 4$
$F(000) = 328$
$D_x = 1.522$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10–14°$
$\mu = 0.82$ mm^{-1}
$T = 293$ K
Block, colorless
0.30 × 0.20 × 0.20 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)
$T_{min} = 0.791$, $T_{max} = 0.853$
2886 measured reflections
1299 independent reflections
1028 reflections with $I > 2\sigma(I)$
$R_{int} = 0.049$
$\theta_{max} = 25.4°$, $\theta_{min} = 2.3°$
$h = 0\rightarrow4$
$k = -12\rightarrow12$
$l = -20\rightarrow20$
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.129$
$S = 1.00$
1299 reflections
83 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
$w = 1/[\sigma^2(F_o^2) + (0.090P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.19$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.025 (5)

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
N	0.1376 (5)	0.3311 (2)	0.19100 (11)	0.0614 (5)
Cl1	0.2101 (2)	0.54073 (7)	0.11039 (4)	0.0867 (4)
C1	0.1642 (6)	0.2038 (2)	0.20387 (12)	0.0573 (6)
H1A	0.0900	0.1710	0.2501	0.069*
Cl2	0.11280 (19)	−0.12135 (6)	0.09764 (4)	0.0757 (3)
C2	0.2433 (6)	0.3739 (2)	0.12537 (14)	0.0559 (6)
C3	0.3774 (7)	0.2985 (2)	0.06990 (14)	0.0598 (6)
H3A	0.4474	0.3344	0.0240	0.072*
C4	0.4038 (6)	0.1686 (2)	0.08483 (13)	0.0558 (6)
H4A	0.4954	0.1144	0.0491	0.067*
C5	0.2944 (5)	0.1178 (2)	0.15315 (12)	0.0490 (5)
C6	0.3244 (6)	−0.0224 (2)	0.17406 (15)	0.0621 (6)
H6A	0.2322	−0.0375	0.2239	0.075*
H6B	0.5556	−0.0464	0.1812	0.075*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N	0.0734 (14)	0.0648 (12)	0.0481 (10)	−0.0050 (10)	0.0169 (9)	−0.0118 (9)
Cl1	0.1250 (8)	0.0567 (4)	0.0825 (6)	0.0007 (4)	0.0292 (5)	−0.0030 (3)
C1	0.0626 (14)	0.0737 (14)	0.0374 (11)	−0.0081 (12)	0.0137 (10)	−0.0006 (9)
Cl2	0.0946 (6)	0.0576 (4)	0.0772 (5)	−0.0046 (3)	0.0203 (4)	−0.0059 (3)
C2	0.0645 (14)	0.0562 (13)	0.0477 (11)	−0.0062 (10)	0.0089 (11)	−0.0047 (9)

C3	0.0761 (16)	0.0632 (13)	0.0426 (11)	−0.0057 (12)	0.0187 (11)	0.0020 (10)
C4	0.0640 (14)	0.0614 (13)	0.0444 (12)	0.0020 (11)	0.0166 (10)	−0.0048 (9)
C5	0.0445 (11)	0.0614 (12)	0.0410 (10)	−0.0022 (10)	0.0044 (9)	0.0011 (9)
C6	0.0638 (15)	0.0686 (14)	0.0545 (13)	0.0052 (12)	0.0088 (11)	0.0120 (11)

Geometric parameters (Å, º)

N—C2	1.307 (3)	C3—C4	1.367 (3)
N—C1	1.334 (3)	C3—H3A	0.9300
Cl1—C2	1.743 (3)	C4—C5	1.383 (3)
C1—C5	1.378 (3)	C4—H4A	0.9300
C1—H1A	0.9300	C5—C6	1.491 (3)
Cl2—C6	1.795 (3)	C6—H6A	0.9700
C2—C3	1.375 (3)	C6—H6B	0.9700
C2—N—C1	116.3 (2)	C3—C4—H4A	120.0
N—C1—C5	124.3 (2)	C5—C4—H4A	120.0
N—C1—H1A	117.9	C1—C5—C4	116.9 (2)
C5—C1—H1A	117.9	C1—C5—C6	120.3 (2)
N—C2—C3	125.1 (2)	C4—C5—C6	122.7 (2)
N—C2—Cl1	115.46 (18)	C5—C6—Cl2	111.11 (17)
C3—C2—Cl1	119.40 (18)	C5—C6—H6A	109.4
C4—C3—C2	117.3 (2)	Cl2—C6—H6A	109.4
C4—C3—H3A	121.3	C5—C6—H6B	109.4
C2—C3—H3A	121.3	Cl2—C6—H6B	109.4
C3—C4—C5	120.0 (2)	H6A—C6—H6B	108.0
C2—N—C1—C5	0.2 (4)	N—C1—C5—C4	0.1 (4)
C1—N—C2—C3	−0.1 (4)	N—C1—C5—C6	177.9 (2)
C1—N—C2—Cl1	−179.33 (18)	C3—C4—C5—C1	−0.6 (3)
N—C2—C3—C4	−0.4 (4)	C3—C4—C5—C6	−178.3 (2)
Cl1—C2—C3—C4	178.9 (2)	C1—C5—C6—Cl2	123.5 (2)
C2—C3—C4—C5	0.7 (4)	C4—C5—C6—Cl2	−58.9 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6*A*···N^{i}	0.97	2.57	3.453 (3)	151

Symmetry code: (i) −*x*, *y*−1/2, −*z*+1/2.