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1,4-Bis(6-chloropyridin-3-ylmethyl)piperazine

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The complete molecule of the title compound, $C_{16}H_{18}Cl_2N_4$, is generated by inversion symmetry. The piperazine ring displays a normal chair conformation. Weak C—H···N interactions between neighbouring pyridine rings help to stabilize the crystal structure.

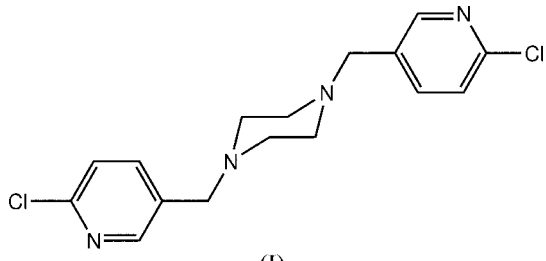
Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.004 Å
R factor = 0.041
wR factor = 0.144
Data-to-parameter ratio = 14.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

As part of our ongoing investigation of anticancer compounds, the title compound, (I), has been prepared and its structure is presented here.



(I)

The molecular structure of (I) is shown in Fig. 1. The molecule is centrosymmetric; the piperazine ring is located on the inversion centre and displays a normal chair conformation. Methylene atom C6 occupies an equatorial position with respect to the piperazine ring, the C6—N2—C7—C8 torsion angle being −178.5 (2)°.

Intermolecular C—H···N hydrogen bonding is observed between neighbouring translation-related pyridine rings (Table 1 and Fig. 2), forming infinite chains of molecules propagating along [010].

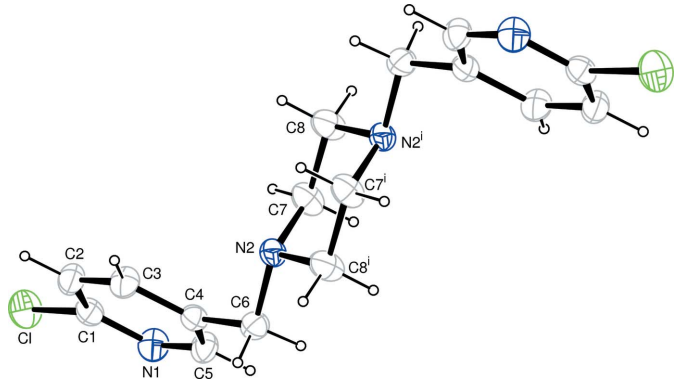


Figure 1
The molecular structure of (I), with 40% probability displacement ellipsoids (arbitrary spheres for H atoms). [Symmetry code: (i) $-x, 2-y, 1-z$.]

Experimental

An aqueous solution (15 ml) of piperazine (4.3 g, 50 mmol) and KOH (5.6 g, 100 mmol) was added dropwise to a benzene solution (150 ml) of 2-chloro-5-chloromethylpyridine (16.2 g, 100 mmol) with continuous stirring. The mixture was then refluxed for 3 h. After cooling to room temperature, the water layer was separated and the oil layer was washed twice with cold water. After removing the solvent under vacuum, a colourless solid appeared. Recrystallization was performed twice from absolute ethanol to obtain single crystals of (I).

Crystal data

$C_{16}H_{18}Cl_2N_4$
M_r = 337.24
Triclinic, $P\overline{1}$
a = 5.802 (5) Å
b = 6.144 (6) Å
c = 12.473 (7) Å
α = 81.72 (4)°
β = 83.78 (4)°
γ = 70.31 (4)°
V = 413.4 (6) Å^3
Z = 1
D_x = 1.355 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.39 mm^{-1}
T = 295 (2) K
Block, colourless
0.32 × 0.28 × 0.20 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.880, T_{max} = 0.930
3363 measured reflections
1488 independent reflections
1230 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 25.2°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.144
S = 1.21
1488 reflections
100 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0782P)^2 + 0.0716P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···N1^{i}	0.93	2.58	3.452 (3)	157

Symmetry code: (i) $x, y+1, z$.

H atoms were placed in calculated positions, with C—H = 0.93 (aromatic) or 0.97 Å (methylene), and refined in riding mode, with U_{iso}(H) = 1.2U_{eq}(C).

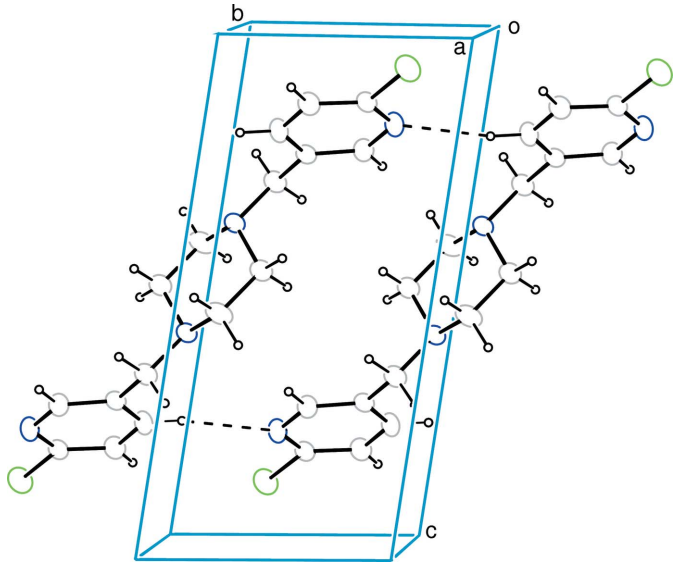


Figure 2
A unit-cell packing diagram, with dashed lines indicating the intermolecular C—H···N interactions.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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