Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

6,6′-Bis(chloromethyl)-2,2′-bipyridine

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.002 Å
R factor = 0.025
wR factor = 0.069
Data-to-parameter ratio = 18.3

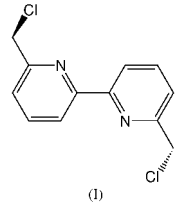
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 31 March 2004
Accepted 14 April 2004
Online 30 April 2004

In the crystalline state, the two pyridyl groups of the centrosymmetric title molecule, $C_{12}H_{10}Cl_2N_2$, are coplanar, with the N atoms *trans* to each other and with the Cl atoms of the chloromethyl groups protruding on opposite sides of the bipyridyl plane. In the crystal structure, the molecules are arranged in discrete layers propagated by edge-to-face and offset face-to-face aryl–aryl interactions. The interlayer spaces are occupied by chloromethyl groups.

Comment

The title compound, (I), has been used as a synthetic intermediate in the preparation of extended open-chain (Newkome *et al.*, 1985, 1986) and macrocyclic ligands (Newkome *et al.*, 1989; Bottino *et al.*, 1988). We are currently investigating macrocycle formation using this species and the structure reported here, (I), was obtained while pursuing those investigations.



(I)

The molecule of (I) has crystallographic inversion symmetry. The pyridyl rings (Fig. 1) are coplanar, the N atoms are *trans* with respect to each other and the Cl atoms of the chloromethyl groups protrude on opposite sides of the plane of the bipyridine moiety.

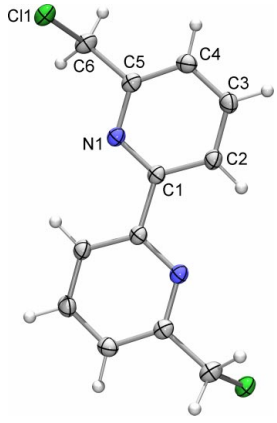


Figure 1
The molecular structure of (I). The molecule is centrosymmetric (only symmetry-independent atoms are labelled). Displacement ellipsoids are drawn at the 50% probability level.

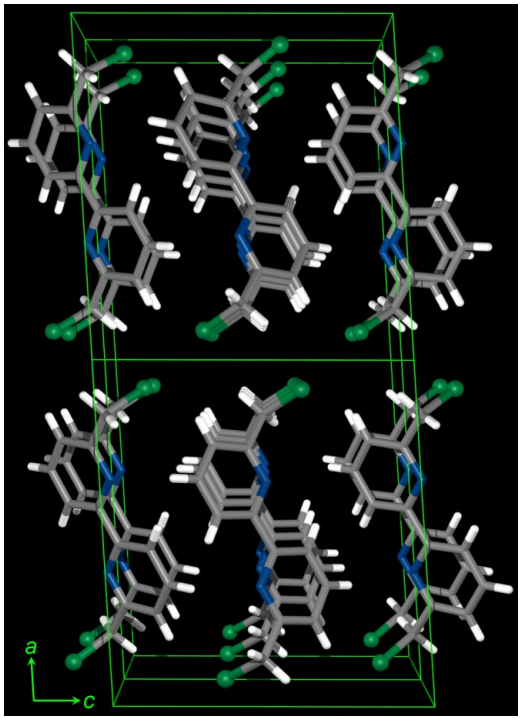


Figure 2
Crystal packing diagram of (I) (C grey, H white, Cl green and N blue). The molecules are arranged in layers parallel to the *bc* plane. Chloromethyl groups protrude into the space between adjacent layers.

In the crystal structure (Fig. 2), the molecules are arranged in discrete two-dimensional layers that lie parallel to the crystallographic *bc* plane. The chloromethyl groups project into the interlayer region. The layers propagate by a combination of offset face-to-face and edge-to-face aryl-aryl interactions, as illustrated in Fig. 3.

Experimental

6,6′–Bis(chloromethyl)-2,2′-bipyridine was synthesized according to the method of Newkome *et al.* (1982) and purified by flash chromatography on silica gel 60 (0.063–0.200 mm) using chloroform as eluant. Crystals were obtained by slow evaporation of a chloroform solution of (I).

Crystal data

$C_{12}H_{10}Cl_2N_2$
$M_r = 253.12$
Monoclinic, $P2_1/c$
$a = 11.7692\ (11)$ Å
$b = 4.3678\ (4)$ Å
$c = 10.8889\ (10)$ Å
$\beta = 93.588\ (1)^\circ$
$V = 558.65\ (9)$ Å^3
$Z = 2$
$D_x = 1.505$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4044 reflections
$\theta = 2.5$–28.4°
$\mu = 0.55$ mm^{-1}
$T = 150\ (2)$ K
Prism, colourless
0.54 × 0.40 × 0.30 mm

Data collection

Bruker SMART 1000 CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1999)
$T_{min} = 0.755$, $T_{max} = 0.850$
5097 measured reflections
1337 independent reflections
1269 reflections with $I > 2\sigma(I)$
$R_{int} = 0.013$
$\theta_{max} = 28.4^\circ$
$h = -15 \rightarrow 15$
$k = -5 \rightarrow 5$
$l = -14 \rightarrow 14$

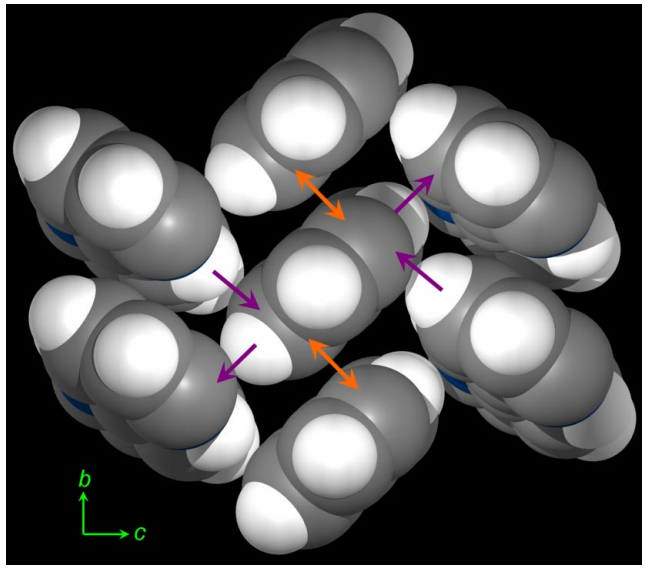


Figure 3
CPK depiction of a section of one layer viewed normal to the *bc* plane (C grey, H white and N blue). Purple arrows indicate the edge-to-face and orange arrows indicate offset face-to-face aryl-aryl interactions between adjacent molecules. The chloromethyl groups have been omitted for clarity.

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.025$
$wR(F^2) = 0.069$
$S = 1.05$
1337 reflections
73 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0366P)^2 + 0.216P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.38$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}

C-bound H atoms were included in idealized positions and refined using a riding model, with methylene and aromatic C—H bond lengths fixed at 0.99 and 0.95 Å, respectively. U_{iso}(H) values were fixed at $1.2U_{eq}$ of the parent C atoms.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SAINT* (Siemens, 1995); data reduction: *SAINT* and *XPREP* (Siemens, 1995); program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *enCIFer* (Allen *et al.*, 2004) and *WinGX*-32 (Farrugia, 1999).

We gratefully acknowledge the Australian Research Council for support.

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