Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2,6-Bis[bis(2-pyridyl)hydroxymethyl]pyridine

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Key indicators

Single-crystal X-ray study
T = 180 K
Mean σ(C–C) = 0.002 Å
R factor = 0.036
wR factor = 0.095
Data-to-parameter ratio = 13.5

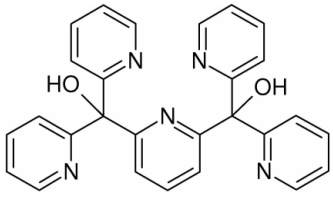
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 1 July 2004
Accepted 5 July 2004
Online 9 July 2004

The crystal structure of the title compound, 2,6-bis[bis(2-pyridyl)hydroxymethyl]pyridine, $C_{27}H_{21}N_5O_2$, at 180 K contains intramolecular O—H···N contacts. In space group *C*2/*c*, the molecule is sited on a twofold axis.

Comment

The methoxy derivative, *viz*. 2,6-bis[bis(2-pyridyl)methoxymethyl]pyridine, of the title compound, (I), has been employed as a neutral pentadentate ligand for the preparation of mononuclear biomimetic coordination complexes (de Vries *et al.*, 1997; Goldsmith, Jonas, Cole & Stack, 2002). The crystal structure of the free ligand has also been reported (Goldsmith, Jonas & Stack, 2002). In the crystal structure of the title compound at 180 K, the H atoms of the hydroxyl groups lie approximately in the same plane as one of the pyridyl rings, forming intramolecular O—H···N contacts with H···N = 1.90 (2) Å and O—H···N = 128 (2)° (Fig. 1). The molecule is twofold symmetric, with atoms N1, C1 and H1*A* lying on the twofold axis.



(I)

Experimental

Under an argon atmosphere, a solution of 2-bromopyridine [6.1 ml in 250 ml dry tetrahydrofuran (THF)] was cooled in an acetone/dry ice bath and *n*-BuLi (25 ml, 2.5 *M* in hexane) was added dropwise, maintaining the temperature below 203 K. A solution of 2,6-pyridinedicarbonyl dichloride (3.13 g in 25 ml dry THF) was added, followed by 25 ml of MeOH. The mixture was stirred overnight and allowed to warm to room temperature. After addition of 25 ml water and 50 ml aqueous HCl (10%), the organic layer was separated and made basic with NaOH. The product was extracted with dichloromethane and evaporation of the solvent left a crude brown oil, which was purified by flash column chromatography (DCM/3% MeOH). Single crystals suitable for X-ray diffraction analysis were grown from a chloroform solution layered with hexane. ^{1}H NMR ($CDCl_3$): δ 8.49 (*d*, *J* = 4.8 Hz, 6-C_6H_4N, 4H), 7.73 (*s*, C—OH, 2H), 7.53 (*t* of *d*, J_1 = 7.9 Hz, J_2 = 1.9 Hz, 4-C_6H_4N, 4H), 7.44 (*d*, *J* = 8.2 Hz, 3-C_6H_4N, 4H), 7.18–7.13 (*m*, 3-C_5H_3N, 4-C_5H_3N, 5-C_6H_4N, 7H). MALDI–TOF mass spectrometry: *m*/*z* = 448 (M^+, 100%).

Crystal data

$C_{27}H_{21}N_5O_2$
$M_r = 447.49$
Monoclinic, $C2/c$
$a = 15.0847\ (12)$ Å
$b = 7.6587\ (6)$ Å
$c = 18.4907\ (15)$ Å
$\beta = 92.216\ (1)^\circ$
$V = 2134.6\ (3)$ Å^3
$Z = 4$
$D_x = 1.392$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2609 reflections
$\theta = 2.7$–26.4°
$\mu = 0.09$ mm^{-1}
$T = 180\ (2)$ K
Block, yellow
$0.25 \times 0.15 \times 0.12$ mm

Data collection

Bruker–Nonius X8APEX-II CCD diffractometer
Thin-slice ω and φ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{min} = 0.877$, $T_{max} = 0.989$
6383 measured reflections
2142 independent reflections
1754 reflections with $I > 2\sigma(I)$
$R_{int} = 0.022$
$\theta_{max} = 26.4^\circ$
$h = -18 \rightarrow 18$
$k = -9 \rightarrow 9$
$l = -23 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.095$
$S = 1.04$
2142 reflections
159 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0435P)^2 + 1.2461P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.15$ e Å^{-3}

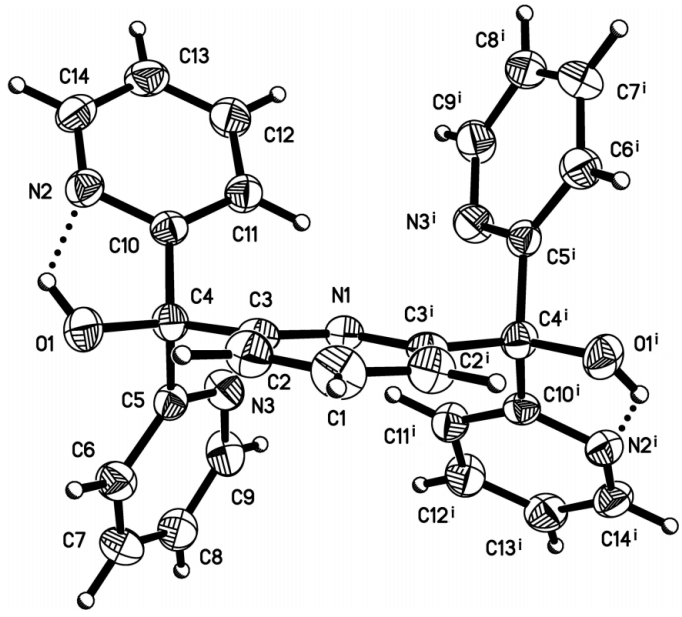


Figure 1
The molecular structure of (I), showing displacement ellipsoids at the 50% probability level. H atoms are shown as spheres of arbitrary radius [symmetry code: (i) $1 - x, y, \frac{1}{2} - z$]. Intramolecular O—H···N contacts are shown by dotted lines.

H atoms bound to carbon were positioned geometrically and allowed to ride during subsequent refinement, with C—H = 0.95 Å and $U_{iso}(H) = 1.2U_{eq}(C)$. Atom H1, associated with the hydroxyl group, was located in a difference Fourier map and allowed to refine freely with an isotropic displacement parameter.

Data collection: *APEX*2 (Bruker–Nonius, 2003); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

We are grateful to the Danish Natural Science Research Council (SNF) and Calsbergfondet for provision of the X-ray equipment.

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