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meso-1,2-Bis(pyridin-2-yl)ethane-1,2-diol

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Key indicators

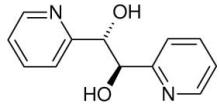
Single-crystal X-ray study
T = 180 K
Mean σ(C–C) = 0.004 Å
R factor = 0.059
wR factor = 0.147
Data-to-parameter ratio = 16.5

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

In the crystal structure of *meso*-1,2-bis(pyridin-2-yl)ethane-1,2-diol, $C_{12}H_{12}N_2O_2$, at 180 K, the molecules lie on inversion centres and are linked into ribbons by complementary O—H···N hydrogen bonds.

Comment

The title compound (I), $C_{12}H_{12}N_2O_2$ (Fig. 1), was obtained as a byproduct from the synthesis of the tetrapodal N_4 ligand *N,N*-bis[2-(pyridin-2-yl)ethyl]-*N*-[(pyridin-2-yl)methyl]amine (II), $N(CH_2C_5H_4N)(CH_2CH_2C_5H_4N)_2$, by reductive alkylation. Its formation appears to be the result of a rare example of a pinacol coupling induced by sodium trihydrocyanoborate (Smith & March, 2001). In the crystal structure of (I) at 180 K, the *meso* molecules are sited on inversion centres. The molecular conformation is such that the C6—H6*a* bond lies close to the plane of the pyridyl ring and the C6—O1 bond lies approximately perpendicular to this plane. Adjacent molecules are linked by complementary O—H···N hydrogen bonds [H1···N1^{i} = 1.97 Å, O1···N1^{i} = 2.797 (3) Å and O—H···N = 169°; symmetry code (i): $1-x, -y, 1-z$] into ribbons running along [100] (Fig. 2).



(I)

In the closely comparable bis-methylated analogue of (I), 2,3-bis(pyridin-2-yl)butane-2,3-diol (Brown *et al.*, 1972), the C—CH_3 bonds lie approximately perpendicular to the planes of the pyridyl rings. In this conformation – apparently driven by the steric influence of the methyl groups – the C—O(H) bonds lie approximately in the planes of the pyridyl rings, and intramolecular O—H···N hydrogen bonds exist.

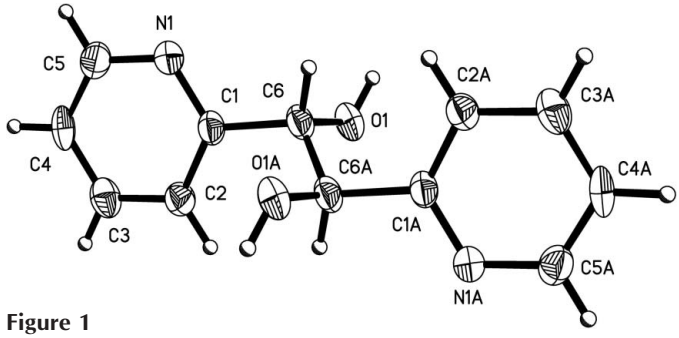


Figure 1
The molecular structure, showing displacement ellipsoids drawn at the 50% probability level. H atoms are shown as spheres of arbitrary radius. Suffix A denotes symmetry operator $-x, -y, 1-z$.

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Experimental

For the preparation of the title compound, *N,N*-bis[2-(pyridin-2-yl)ethyl]amine hemihydrate (1.464 g, 6.195 mmol) (Leaver *et al.*, 2003) and pyridine-2-carboxaldehyde (4.36 g, 40.1 mmol) were dissolved in ethanol over 4 Å molecular sieves. The solution was stirred for 30 min at 323 K and excess sodium trihydrocyanoborate was added slowly. Stirring was continued for 1 h and the solution was allowed to cool and then filtered. Excess trihydrocyanoborate was destroyed by the addition of hydrochloric acid (6 *M*). The solution was made alkaline [2 *M* NaOH(aq)] and extracted with CH_2Cl_2. The organic phase was dried ($MgSO_4$) and the solvent was removed by rotary evaporation to give 3.3 g impure *N,N*-bis[2-(pyridin-2-yl)ethyl]-*N*-[(pyridin-2-yl)-methyl]amine, (II). The mass spectrum showed primarily (II)Na^+ (*m*/*z* 341.4, 100%). The crude product was left to stand in a freezer (255 K) for several months, during which time crystals of the title compound were formed. At this point, there was still no indication of (I) in the mass spectrum of the bulk product. Careful selection of a few crystals of (I) for ESI–MS showed some contamination with (II), but a main peak due to (I)Na^+ (*m*/*z* 239.1). Under more gentle ionization conditions, peaks due to (I)H^+ (*m*/*z* 217.2, 20%) were also evident.

Crystal data

$C_{12}H_{12}N_2O_2$	D_x = 1.420 Mg m^{-3}
M_r = 216.24	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 1853 reflections
a = 5.3155 (14) Å	θ = 3.8–25.1°
b = 8.108 (2) Å	μ = 0.10 mm^{-1}
c = 11.737 (3) Å	T = 180 (2) K
β = 90.196 (10)°	Needle, pale orange
V = 505.8 (2) Å^3	0.30 × 0.08 × 0.08 mm
Z = 2	

Data collection

Bruker–Nonius X8APEX–II CCD diffractometer	1236 independent reflections
	1026 reflections with $I > 2\sigma(I)$
Thin-slice ω and φ scans	R_{int} = 0.043
Absorption correction: multi-scan *SADABS* (Sheldrick, 2003)	θ_{max} = 28.3°
	$h = -7 \rightarrow 6$
T_{min} = 0.588, T_{max} = 0.992	$k = -10 \rightarrow 8$
6075 measured reflections	$l = -15 \rightarrow 12$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0396P)^2 + 0.2558P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.059	
$wR(F^2)$ = 0.147	
S = 1.11	$(\Delta/\sigma)_{max} < 0.001$
1236 reflections	$\Delta\rho_{max}$ = 0.32 e Å^{-3}
75 parameters	$\Delta\rho_{min}$ = −0.25 e Å^{-3}
H-atom parameters constrained	

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1^{i}	0.84	1.97	2.797 (3)	169

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

The crystal is monoclinic with β close to 90 ° and was twinned: twin law (100,0$\bar{1}$0,00$\bar{1}$), refined twin ratio 0.614:0.386 (3). The wide range of transmission factors suggests that the multi-scan correction treats systematic effects other than absorption by the crystal itself. H atoms bound to C atoms were positioned geometrically and allowed to ride during subsequent refinement with C—H = 0.95 Å, U_{iso}(H) = 1.2 U_{eq}(C) for C(sp^2) and C—H = 1.00 Å, U_{iso}(H) = 1.2 U_{eq}(C) for C(sp^3). The H atom of the hydroxyl group was not evident in a difference Fourier map; it was placed geometrically with O—H = 0.84 Å and allowed to rotate around the C6—O1 bond during subsequent refinement, with U_{iso}(H) = 1.5 U_{eq}(O).

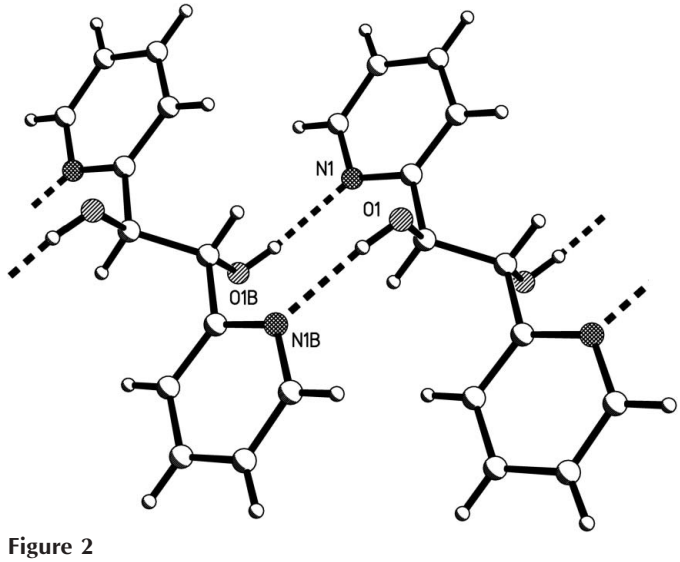

Figure 2
Complementary O—H···N hydrogen bonds (dashed lines) between adjacent molecules of (I). Suffix B denotes symmetry operator $1-x, -y, 1-z$.

Data collection: *APEX2* (Bruker–Nonius, 2004); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT* (Bruker, 2003); 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* .

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