Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

N,*N*′-Bis[2-(2-pyridyl)methyl]pyridine-2,6-dicarboxamide hemihydrate

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Received 18 September 2002
Accepted 4 October 2002
Online 18 October 2002

Key indicators

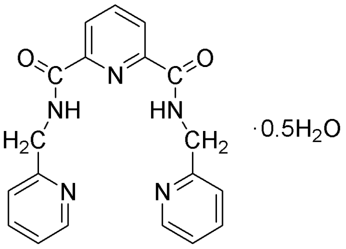
Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.005 Å
Disorder in solvent or counterion
R factor = 0.055
wR factor = 0.137
Data-to-parameter ratio = 16.9

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

The structure of the title pentadentate ligand as its hemihydrate, $C_{19}H_{17}N_5O_2\cdot0.5H_2O$, is different from the structure of a similar tridentate ligand reported previously [Qi *et al.* (2001). *Acta Cryst*. E**57**, o749–o750; Yang *et al.* (2001). *Acta Cryst*. E**57**, o1161–o1162]. The two pyridine side arms are not in the same plane, owing to their rotation around an axis between the pyridine ring and the methylene group. There is an intermolecular hydrogen bond between the ligand and the disordered lattice water molecule.

Comment

There are two coordination modes when the title ligand, $MePy_3P$, (I), is coordinated to metal centers in its deprotonated form. One is the pentadentate form involving the N atoms of two deprotonated carboxamide groups and the N atoms of the three pyridine rings, for example, in the binuclear structure $[Cu_2(MePy_3P)_2]$ (Marlin *et al.*, 1999). The other is the tridentate form involving the N atoms of two deprotonated carboxamide groups and the N atom of the middle pyridine ring, for example, in the complex $[Fe(MePy_3P)_2]$ (Marlin *et al.*, 2001). Therefore, the coordination behavior of the ligand is very interesting as a result flexibility about of the two methylene groups.



(I)

The three pyridine rings in (I) (Fig. 1) are not coplanar. The terminal pyridine rings are rotated out of the plane of the central pyridine ring, forming dihedral angles of 86.0 (2) and 81.0 (2)° with it. The dihedral angle between the terminal pyridine rings is 122.7 (2)°. There is an intermolecular hydrogen bond involving a carbonyl O atom of the molecule and an H atom of the disordered water molecule.

Experimental

The title compound was synthesized from 2,6-pyridinedicarboxylic acid and 2-(aminomethyl)pyridine according to the general procedure of Johnson *et al.* (1960). The crystal used for data collection was obtained by slow evaporation from a saturated EtOH–H_2O solution at room temperature.

Crystal data

$C_{19}H_{17}N_5O_2 \cdot 0.5H_2O$
$M_r = 356.38$
Monoclinic, $C2/c$
$a = 17.784\ (3)$ Å
$b = 17.611\ (3)$ Å
$c = 14.217\ (2)$ Å
$\beta = 126.310\ (3)^\circ$
$V = 3588.2\ (9)$ Å^3
$Z = 8$
$D_x = 1.319$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2885 reflections
$\theta = 1\text{–}27.5^\circ$
$\mu = 0.09$ mm^{-1}
$T = 294\ (2)$ K
Prism, colorless
$0.34 \times 0.28 \times 0.24$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\rm min} = 0.970$, $T_{\rm max} = 0.978$
12125 measured reflections
4127 independent reflections
1561 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.053$
$\theta_{\rm max} = 27.6^\circ$
$h = -12 \rightarrow 23$
$k = -22 \rightarrow 22$
$l = -18 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.137$
$S = 1.05$
4127 reflections
244 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.05P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.64$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.23$ e Å^{-3}

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N2—H2$A\cdots$N3$^{\rm i}$	0.86	2.45	3.207 (3)	147
N4—H4$B\cdots$N3$^{\rm i}$	0.86	2.38	3.130 (3)	146
O1W—H1$WA\cdots$O1$W^{\rm ii}$	0.85	1.71	2.435 (9)	142
O1W—H1$WA\cdots$O2	0.85	2.22	2.762 (5)	121

Symmetry codes: (i) $-x, y, \frac{1}{2} - z$; (ii) $\frac{1}{2} - x, \frac{1}{2} - y, 1 - z$.

All H atoms were placed in geometrically calculated positions and included in the final refinement in the riding-model approximation. Half-occupancy of the water molecule, located in a difference map, was assumed on the basis of the observed electron density, reasonable values for refined displacement parameters, and an unacceptably short distance between symmetry-related pairs of these molecules.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SMART*; data reduction: *SAINT-Plus* in *SHELXTL-NT* (Bruker, 1995); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL-NT; software used to prepare material for publication: SHELXTL-NT.

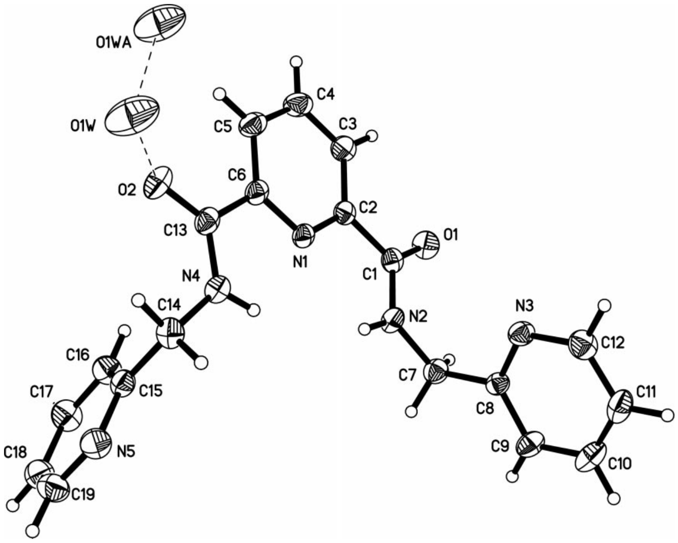


Figure 1
The molecular structure of (I), showing ellipsoids at the 30% probability level (Siemens, 1995). The two sites shown for the water molecule (H atoms omitted) are symmetry-related alternatives, each with half-occupancy.

The Hong Kong Polytechnic University ASD fund and the National Natural Science Foundation of China (D20063002) are thanked for their financial support of this study.

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