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2-(4-Pyridyl)-1*H*-benzimidazole trihydrate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.050
wR factor = 0.142
Data-to-parameter ratio = 15.3

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

The benzimidazole and pyridyl portions of the molecule of the title compound, $C_{12}H_9N_3\cdot 3H_2O$ are essentially co-planar; the three N atoms interact with the water molecules, forming a three-dimensional hydrogen-bonded network structure.

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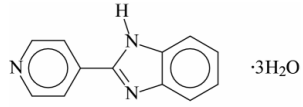
Comment

2-Phenyl-1*H*-benzimidazole is a heterocyclic ligand that can bind to metal atoms through the deprotonated and tertiary N atoms (Huang *et al.*, 2003, 2004); since the N atoms are on opposite sides of the five-membered imidazole ring, the ligand can function as a spacer in the formation of chain-type coordination polymers. A third Lewis basic site in the 2-aromatic substituent should ensure that the present ligand (Fig. 1) will bind to three metal atoms simultaneously.



(I)

The benzimidazole and pyridyl portions of the title molecule, (I), are coplanar [dihedral angle = 2.8 (1)°] The N atoms of the benzimidazole portion of the molecule interact with water molecules to form a linear chain (Fig. 2), as does the pyridyl N atom (Table 2), forming a hydrogen-bonded three-dimensional network structure.

Experimental

Isonicotinic acid (1.25 g, 10.0 mmol) and 1,2-diaminobenzene (1.08 g, 10.0 mmol) were added to polyphosphoric acid (14 g). The mixture was heated under nitrogen at 433 K for 8 h. The resulting viscous syrup was poured into 500 ml water. The tan solid that separated was collected and then suspended in 500 ml 0.5 *M* sodium carbonate. The solid was broken up to give a yellow powder. Recrystallization of the powder from methanol/water yielded the pure compound as very

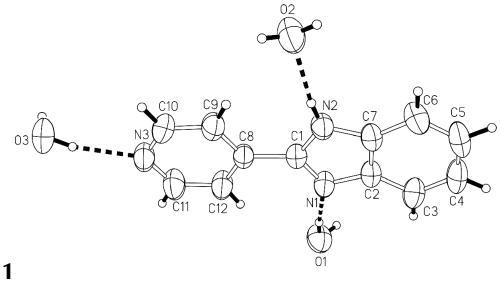


Figure 1
An *ORTEP*II (Johnson, 1976) plot of (I), with displacement ellipsoids drawn at the 50% probability level. H atoms are shown as spheres of arbitrary radii. The dashed lines indicate hydrogen bonds.

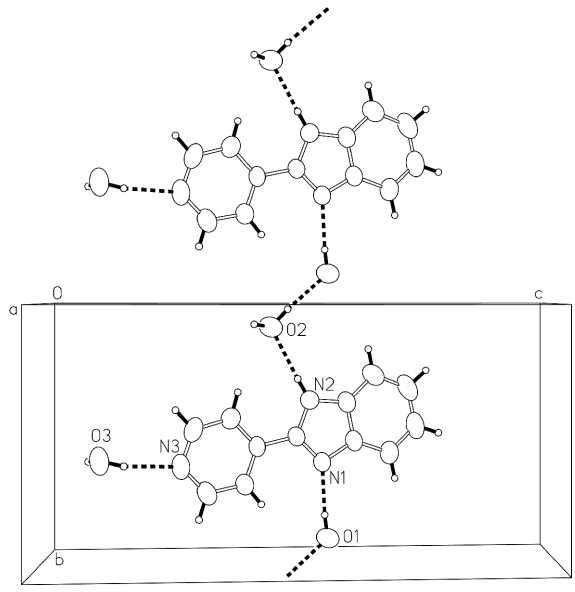


Figure 2
An *ORTEP*II (Johnson, 1976) plot of the chain propagating along the *a* axis in (I), with displacement ellipsoids drawn at the 50% probability level. H atoms are shown as spheres of arbitrary radii. Adjacent chains are linked by hydrogen bonds (shown as dashed lines) into a three-dimensional network.

faint yellow crystals (1.30 g, 67% yield). Elemental analysis calculated for $C_{12}H_{15}N_3O_3$: C 57.82, H 6.07, N 16.86%; found C 57.93, H 5.89, N 16.91%. IR (KBr, cm^{-1}): 3420 (*br*), 3052 (*br*), 1601 (*m*), 1463 (*m*), 1443 (*vs*), 1403 (*vs*), 1316 (*vs*), 1280 (*vs*), 1150 (*m*), 1122 (*s*), 991 (*m*), 974 (*m*).

Crystal data

$C_{12}H_9N_3\cdot 3H_2O$
M_r = 249.27
Monoclinic, $P2_1/c$
a = 7.3913 (6) Å
b = 9.3377 (8) Å
c = 18.643 (2) Å
β = 94.209 (2)°
V = 1283.3 (2) Å^3
Z = 4
D_x = 1.290 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1887 reflections
θ = 2.4–23.9°
μ = 0.10 mm^{-1}
T = 293 (2) K
Block, colorless
0.20 × 0.16 × 0.13 mm

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: none
10725 measured reflections
2925 independent reflections
1910 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 27.5°
$h = -9 \rightarrow 9$
$k = -12 \rightarrow 12$
$l = -24 \rightarrow 24$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.142
S = 1.01
2925 reflections
191 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0733P)^2 + 0.0813P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C1	1.316 (2)	C3—C4	1.363 (3)
N1—C2	1.388 (2)	C4—C5	1.385 (3)
N2—C1	1.354 (2)	C5—C6	1.367 (3)
N2—C7	1.376 (2)	C6—C7	1.387 (2)
N3—C11	1.323 (2)	C8—C12	1.376 (2)
N3—C10	1.324 (2)	C8—C9	1.382 (2)
C1—C8	1.470 (2)	C9—C10	1.374 (3)
C2—C7	1.393 (2)	C11—C12	1.377 (2)
C2—C3	1.396 (2)		
C1—N1—C2	105.0 (1)	C5—C6—C7	116.6 (2)
C1—N2—C7	107.2 (1)	N2—C7—C6	132.8 (2)
C10—N3—C11	116.0 (2)	N2—C7—C2	105.3 (1)
N1—C1—N2	112.8 (1)	C2—C7—C6	121.9 (2)
N1—C1—C8	123.8 (1)	C9—C8—C12	116.9 (2)
N2—C1—C8	123.5 (1)	C1—C8—C9	122.0 (2)
N1—C2—C7	109.7 (1)	C1—C8—C12	121.1 (1)
N1—C2—C3	130.2 (2)	C8—C9—C10	119.1 (2)
C3—C2—C7	120.1 (2)	N3—C10—C9	124.4 (2)
C2—C3—C4	117.7 (2)	N3—C11—C12	124.0 (2)
C3—C4—C5	121.5 (2)	C8—C12—C11	119.6 (2)
C4—C5—C6	122.2 (2)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—HO11···N1	0.86 (1)	1.91 (1)	2.764 (2)	175 (2)
O1—HO12···O2^{i}	0.86 (1)	2.07 (2)	2.868 (2)	155 (3)
O2—HO21···O1^{ii}	0.85 (1)	1.94 (1)	2.782 (2)	167 (2)
O2—HO22···O3^{iii}	0.86 (1)	1.87 (1)	2.719 (2)	170 (2)
O3—HO31···N3	0.85 (1)	2.01 (1)	2.841 (2)	167 (2)
O3—HO32···O1^{iv}	0.86 (1)	2.01 (1)	2.862 (2)	174 (2)
N2—H2···O2	0.86 (1)	2.05 (1)	2.892 (2)	169 (2)

Symmetry codes: (i) $1-x, 1-y, 1-z$; (ii) $x, y-1, z$; (iii) $2-x, y-\frac{1}{2}, \frac{1}{2}-z$; (iv) $x, \frac{3}{2}-y, z-\frac{1}{2}$.

The aromatic H atoms were placed at calculated positions in the riding-model approximation [C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C)]. The water and amino H atoms were located and refined with a distance restraint [O—H = N—H = 0.85 (1) Å].

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

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