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NOTE

Solvent Evaporation Method and Crystal Structure of 2-[5-(1H-benzo[d]imidazol-2-yl)pyridin-3-yl]-1*H*-benzo[d]imidazole

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One new benzimidazole compound *i.e.*, 2-[5-(1H-benzo[d]imidazol-2-yl)pyridin-3-yl]-1H-benzo[d]imidazole with m.f. $C_{22}H_{17}N_6O_2$ was synthesized with pyridine-3,5-dicarboxylic acid, benzene-1,2-diamine and polyphosphoric acid. The compound has been characterized by X-ray single-crystal diffraction. The compound shows a one-dimensional framework. The 3D supramolecular structure is formed *via* hydrogen bonding connection.

Keywords: Solvent evaporation, Crystal structure, Benzimidazole.

In this report, the synthesis and crystal structure of 2-[5-(1H-benzo[d]imidazol-2-yl)pyridin-3-yl]-1H-benzo[d]-imidazole are reported.

All reagent and solvents employed were commercially available and used as received without further purification.

A mixture of benzene-1,2-diamine (0.20 mmol), pyridine-3,5-dicarboxylic acid (0.10 mmol) and polyphosphoric acid (0.05 mmol) and distilled water (10 mL) was heated in a 25 mL stainless steel reactor with a teflon liner 453 K for 12 h, followed by slow cooling to room temperature. Colourless crystals of the compound formed (Fig. 1).

Detection method: Diffraction intensity data of the single crystal of the compound was collected on a Bruker SMART APEX-II CCD diffractometer equipped with a graphite monochromated MoK_α radiation (λ = 0.71073 Å) by using a ω-scan mode. All the structures were solved by direct methods and refined by full-matrix least-squares methods on F^2 using the program SHELXTL97[1]. All non-hydrogen atoms were refined anisotropically. The hydrogen atoms were located by geometrically calculations and their positions and thermal parameters were fixed during the structure refinement. The crystallographic data and experimental details of structural analyses for coordination polymers are summarized in Table-1. Selected bond and angle parameters are listed in Table-2.

X-ray diffraction analysis revealed that the fundamental building unit consists of pyridine ring and 2-methyl-1H-benzoimidazole as bridging ligands to construct a new coordination polymer. The asymmetric unit of the title structure contains

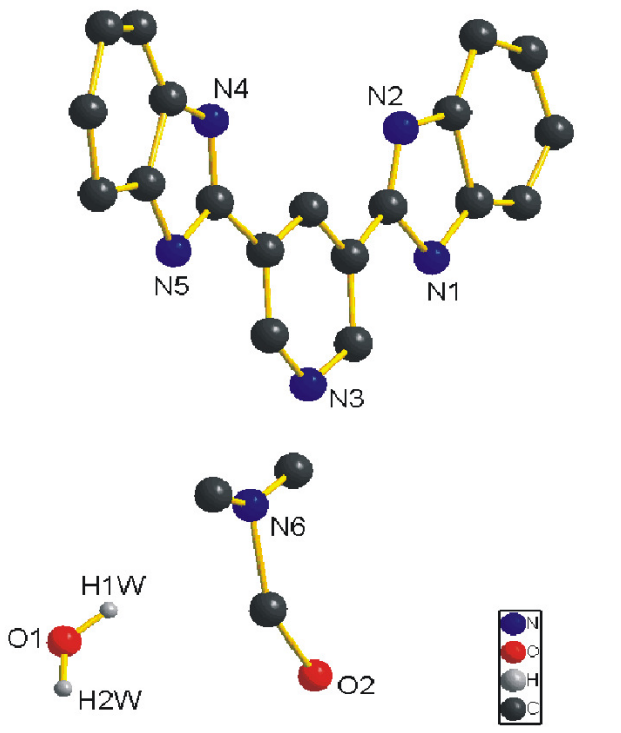

Fig. 1. Molecular structure of the title compound at 30 % probability displacement ellipsoids

one pyridine ring, one DMF and two 2-methyl-1H-benzoimidazole molecules. On the Pyridine ring, the hydrogen atoms

TABLE-1
CRYSTALLOGRAPHIC DATA AND STRUCTURE REFINEMENT SUMMARY FOR BENZOIMIDAZOLE

Empirical formula	$C_{22}H_{17}O_2N_6$
Formula weight	397.42
Crystal system space group	Triclinic, P-1
Unit cell dimensions	a = 7.836(9)Å; b = 11.623(13)Å; c = 14.224(16)Å
Volume (Å^3)	1211(2)
θ range for data collection	2.25-25.50
Final R indices [I>2σ(I)]	R_1= 0.1769; wR_2=0.4324
Z, calculated density (mg/m^3)	2, 1.089
Absorption coefficient (mm^{-1})	0.074
F(000)	414
Limiting indices	-9 ≤ h ≤ 9; -14 ≤ k ≤ 14; 0 ≤ l ≤ 17
Largest diff. peak and hole (e/Å^3)	0.442 and -0.444
Goodness-of-fit on F^2	0.969
R indices (all data)	R_1=0.3374, wR_2=0.4823

TABLE-2
SELECTED BOND LENGTHS (Å) AND ANGLES (°) FOR BENZOIMIDAZOLE

N(1)-C(18)	1.449(15)	N(3)-C(4)	1.340(13)
N(1)-C(16)	1.547(15)	N(3)-C(8)	1.379(13)
N(2)-C(16)	1.273(12)	N(4)-C(9)	1.310(12)
N(2)-C(17)	1.34(2)	N(4)-C(11)	1.448(13)
N(5)-C(10)	1.465(19)	N(6)-C(1)	1.19(3)
N(5)-C(9)	1.469(14)	N(6)-C(2)	1.34(3)
C(18)-N(1)-C(16)	110.6(14)	C(16)-N(2)-C(17)	112.6(11)
C(4)-N(3)-C(8)	115.9(11)	C(9)-N(4)-C(11)	110.1(9)
C(10)-N(5)-C(9)	102.5(11)	C(1)-N(6)-C(2)	148(2)
C(1)-N(6)-C(3)	113(2)	C(2)-N(6)-C(3)	99(2)

were assigned with Uiso(H) = 1.2 Ueq(C) and included in the final refinement by using geometrical restraints, with d(C-H) = 0.93 Å. And four nitrogen atoms (N1, N2, N4 and N5) from two 2-methyl-1H-benzoimidazole ligands. The N(1)-C(18), N(2)-C(16), N(4)-C(9), N(5)-C(10) bond lengths are 1.449 (15) Å, 1.273 (12) Å, 1.310(12) Å, 1.465 (19) Å, respectively. In addition, the carboxylic and the carboxylate group as well as the water molecule. Some are listed as follows: O(1)-H(2W)···N(3)#2, [O···O = 2.851 (13)\%A, O-H...O = 170.4\%]. Symmetry codes: #2 -x+1, -y+1, -z+1. The chains are further assembled by the intermolecular hydrogen bonding interaction leading to the formation of a 3D framework (Fig. 2).

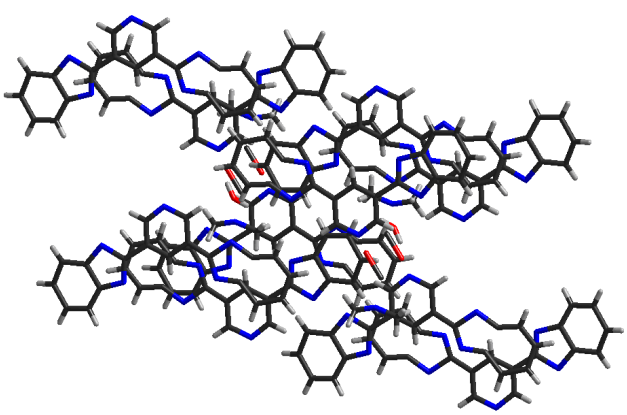

Fig. 2. 3D structure formed *via* hydrogen bonding interactions

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