NOTE

Synthesis and Structural Characterization of N-(4-Pyridyl)benzamide

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Determination of the structure of N-(4-pyridyl)benzamide with the molecular formula $C_{12}H_{10}N_2O$ that the conformations of the N-H and C=O bonds are *anti* to each other, The -NH-C=O- group makes dihedral angles of $33.5(2)^\circ$ and $17.1(2)^\circ$ with the benzoyl and pyridyl rings, respectively, while the angle between the benzoyl and pyridine rings is $49.9(6)^\circ$. In the crystal structure, N-H...N hydrogen bonds link the molecules into a chain along the b axis. In addition, the structure is stabilized by π - π stacking interactions involving the benzene rings [centroid-to-centroid distance = $2.257(3)$ Å].

Key Words: N-(4-Pyridyl)benzamide, Synthesis, Crystal structure.

Benzanilide and its N-substituted derivatives have been considered to be a class of privileged structural compounds, which usually have excellent biological activities^{1,2}. However, the literatures are full of the function of the 2-chloro-4-nitrophenyl¹, 3,5-dichlorophenyl² and 3-chlorophenyl^{3,4} and also structures of benzamide and related compounds. The aim of the present work was to combine benzamide and 4-pyridyl group in a single structure which is not well known in the literature.

Polyethylene glycol-400 and 4-aminopyridine was purchased from Alfa Aesar and used without further purification. The other reagents and solvents were of analytical reagent grade and were used without further purification. C, H and N analyses were obtained using a GmbH VarioEL V3.00 automatic elemental analysis instrument. X-Ray single crystal structure determination was carried out on a Bruker Smart 1000 CCD diffractometer. Melting points were obtained by use of a microscopic melting point apparatus made in Beijing Taike Instrument Limited Company and were uncorrected.

General procedure: N-(4-Pyridyl)benzamide was synthesized according to an analogous method reported earlier⁵⁻⁹. Benzoyl chloride (1.40 g, 0.01 mol) was reacted with ammonium thiocyanate (1.15 g, 0.015 mol) in CH_2Cl_2 solution (25 mL) under solid-liquid phase transfer catalysis, using 3 % polyethylene glycol-400 (0.18 g) as the catalyst and then continuing stirring for 3 h at room temperature, to give the corresponding benzoyl isothiocyanate, which was added 4-aminopyridine (1.42 g, 0.015 mol). After stirring for 20 h at room temperature, the

precipitate was filtered, washed successively with water, acetone and diethyl ether. The product was dried under reduced pressure and obtain the title compound. Yield, 29.02 % m.p. 486-487 K. Anal. calcd. (%) for $C_{12}H_{10}N_2O$: C, 72.71; H, 5.08; N, 14.13. Found (%): C, 72.54; H, 5.28; N, 13.85.

Colourless prismatic single crystals suitable for X-ray diffraction studies were obtained after two weeks by slow evaporation from a mixture of ethyl acetate/acetone (1:1) of N-(4-pyridyl)benzamide at room temperature.

X-Ray structure determination: The crystal data and structure refinement for $C_{12}H_{10}N_2O$ is given in Table-1. The single crystal of $C_{12}H_{10}N_2O$ with approximate dimension of $0.48 \text{ mm} \times 0.45 \text{ mm} \times 0.40 \text{ mm}$ was placed on a Bruker Smart 1000 CCD area detector. The diffraction were collected using a graphite monochromated MoK_α radiation ($\lambda = 0.71073$ Å) at 298(2) K. The structure was solved by using the program SHELXL-97 and Fourier difference technique and refined by full-matrix least-square method on F^2 . All hydrogen atoms were added theoretically. CCDC: 877598.

X-Ray crystallographic analysis revealed the crystal structure of title compound. And the structure is shown in Fig. 1. Selected bond distances and angles are listed in Table-2. In the molecule of the title compound, the conformations of the N-H and C=O bonds are anti to each other, which is similar to those observed in the structures of N-(3,5-dichlorophenyl) benzamide² and N-(3-chlorophenyl)-2-methylbenzamide³. The -NH-C=O- group makes dihedral angles of $33.5(2)^\circ$ and $17.1(2)^\circ$ with the benzoyl and pyridyl rings, respectively, while

TABLE-2
 SELECTED BOND DISTANCES (Å) AND ANGLES (°) OF N-(4-PYRIDYL)BENZAMIDE

Bond	Lengths	Bond	Lengths	Bond	Lengths
N(1)-C(8)	1.333(2)	C(2)-C(3)	1.387(2)	C(8)-C(9)	1.380(2)
N(1)-C(12)	1.335(2)	C(2)-C(7)	1.389(2)	C(9)-C(10)	1.382(2)
N(2)-C(1)	1.363(2)	C(3)-C(4)	1.383(2)	C(10)-C(11)	1.391(2)
N(2)-C(10)	1.400(2)	C(4)-C(5)	1.378(3)	C(11)-C(12)	1.377(2)
O(1)-C(1)	1.222(2)	C(5)-C(6)	1.375(3)	—	—
C(1)-C(2)	1.496(2)	C(6)-C(7)	1.382(3)	—	—
Bond	Angles	Bond	Angles	Bond	Angles
C(8)-N(1)-C(12)	115.27(15)	C(7)-C(2)-C(1)	122.26(16)	C(8)-C(9)-C(10)	118.66(16)
C(1)-N(2)-C(10)	127.24(14)	C(4)-C(3)-C(2)	119.93(17)	C(9)-C(10)-C(11)	117.51(15)
O(1)-C(1)-N(2)	123.80(15)	C(5)-C(4)-C(3)	120.07(18)	C(9)-C(10)-N(2)	124.36(15)
O(1)-C(1)-C(2)	121.16(15)	C(6)-C(5)-C(4)	120.15(17)	C(11)-C(10)-N(2)	118.12(14)
N(2)-C(1)-C(2)	115.02(14)	C(5)-C(6)-C(7)	120.44(18)	C(12)-C(11)-C(10)	118.94(16)
C(3)-C(2)-C(7)	119.82(16)	C(6)-C(7)-C(2)	119.58(18)	N(1)-C(12)-C(11)	124.59(16)
C(3)-C(2)-C(1)	117.89(15)	N(1)-C(8)-C(9)	125.03(16)	—	—

 TABLE-1
 CRYSTAL DATA AND STRUCTURE REFINEMENT
 FOR THE N-(4-PYRIDYL)BENZAMIDE

Empirical formula	C ₁₂ H ₁₀ N ₂ O
Formula weight	198.22
Temperature	298(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	P2(1)/c
Cell dimensions	a = 5.7119(12) Å, b = 11.2316(14) Å, c = 15.2446(18) Å, β = 95.2800(10)
Volume	973.8(3) Å ³
Z	4
Density (calculated)	1.352 mg/m ³
Absorption coefficient	0.089 mm ⁻¹
F ₍₀₀₀₎	416
Index ranges	-6 ≤ h ≤ 6, -11 ≤ k ≤ 13, -18 ≤ l ≤ 16
Reflections collected/unique	4856/1701 [R _(int) = 0.0431]
Independent reflections	2248
Data/restraints/parameters	1701/0/137
Goodness of fit indicator	1.072
R [I > 2σ(I)]	R ₁ = 0.0407, wR ₂ = 0.0996
Largest diff. peak and hole	0.203 and -0.196 e. Å

Fig. 1. Molecule structure of N-(4-pyridyl)benzamide with atom numbering scheme. Displacement ellipsoids for non-hydrogen atoms are drawn at the 30 % probability level

the angle between the benzoyl and pyridine rings is 49.9(6)°. In the crystal structure of N-(4-pyridyl)benzamide, intermolecular hydrogen bonds N2-H2...N1 (symmetry code: $-x + 1, y - 1/2, -z + 1/2$) link molecules into infinite chain superamolecular structure along the b axis (Fig. 2.)^{10,11}.

 Fig. 2. View of the 1D chain supramolecular structure of N-(4-pyridyl)benzamide along b axis. Intermolecular hydrogen bonds are shown as dashed lines. [Symmetry codes: $-x + 1, y - 1/2, -z + 1/2$]

Supplement data: Further details of the crystal structure investigation(s) may be obtained from the Cambridge Crystallographic Data Centre, Postal Address: CCDC, 12 Union Road, CAMBRIDGE CB2 1EZ, UK. Telephone: (44) 01223 762910; Facsimile: (44) 01223 336033; E-mail: deposit@ccdc.cam.ac.uk, on quoting the depository number CCDC 877598.

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