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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)º and 17.1(2)º with the benzoyl and pyridyl rings, respectively, while the angle between the benzoyl and pyridine rings is 49.9(6)º. 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-nitrophenyl[1], 3,5-dichlorophenyl[2] 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 earlier[5-9]. 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 soild-liquid phase transfer catalysis, using 3 % polyethylene glycol-400 (0.18 g) as the catalyst and then continuring 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 temperture.

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 mm × 0.45 mm × 0.40 mm was placed on a Bruker Smart 1000 CCD area detector. The diffraction were collected using a graphite monochromated MoK_α radition (λ = 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) benzamide[2] and N-(3-chlorophenyl)-2-methylbenzamide[3]. The -NH-C=O- group makes dihedral angles of 33.5(2)º and 17.1(2)º 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	$C_{12}H_{10}N_2O$
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	$-6 \le h \le 6$, $-11 \le k \le 13$, $-18 \le l \le 16$
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

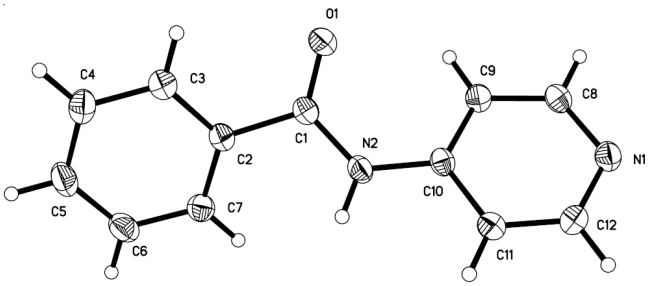

Fig. 1. Molecule structure of N-(4-pyridyl)benzamide with atom numbering scheme. Displacement ellipsoids for non-hydrogen atoms are drawn at the 30 % probability level

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].

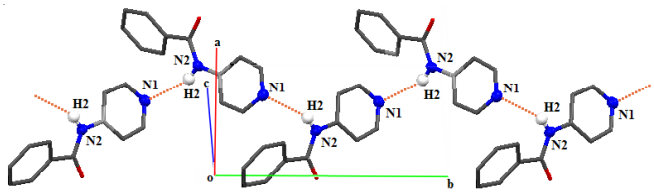

Fig. 2. View of the 1D chain supramolecular structure of N-(4-pyridyl)-benzamide along b axis. Intermolecular hydrogen bonds are shown as dashed lines. [Symmetry codes: -x + 1, y - 1/2, -z + 1/2]

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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REFERENCES

1. A. Saeed, S. Hussain and U. Flörke, *Acta Cryst.*, **E64**, o705 (2008).
2. B.T. Gowda, S. Foro, B.P. Sowmya and H. Fuess, *Acta Cryst.*, **E64**, o1243 (2008).
3. B.T. Gowda, S. Foro, B.P. Sowmya and H. Fuess, *Acta Cryst.*, **E64**, o861 (2008).
4. B.T. Gowda, S. Foro, B.P. Sowmya and H. Fuess, *Acta Cryst.*, **E64**, o1300 (2008).
5. W.K. Dong, X.Q. Yang and J.H. Feng, *Acta Cryst.*, **E62**, o3459 (2006).
6. W.K. Dong, X.Q. Yang, L. Xu, L. Wang, G.L. Liu and J.H. Feng, *Z. Kristallogr. NCS*, **222**, 79 (2007).
7. W.K. Dong, X.Q. Yang, L.Q. Chai, Y.Q. Tian and J.H. Feng, *Phosphorus Sulfur Silicon Rel Elem.*, **183**, 1181 (2008).
8. W.K. Dong, H.B. Yan, L.Q. Chai, Z.W. Lv and C.Y. Zhao, *Acta Cryst.*, **E64**, o1097 (2008).
9. Y.J. Ding, X.B. Chang, X.Q. Yang and W.K. Dong, *Acta Cryst.*, **E64**, o658 (2008).
10. W.K. Dong, G. Wang, Y.X. Sun, X.Y. Dong and X.H. Gao, *Z. Naturforsch.*, **67b**, 17 (2012).
11. W.K. Dong, Y.X. Sun, Y.P. Zhang, L. Li, X.N. He and X.L. Tang, *Inorg. Chim. Acta*, **362**, 117 (2009).