Asian Journal of Chemistry; Vol. 24, No. 7 (2012), 3253-3254

ASIAN JOURNAL OF CHEMISTRY

www.asianjournalofchemistry.co.in

NOTE

Hydrothermal Synthesis and Crystal Structure Based on Benzophenone-2,4'-carboxylate and 1,2-*Bis*(4-pyridyl)ethane

XIAOSHUANG ZHOU

Department of Mathematics, Dezhou University, Shandong 253023, P.R. China

*Corresponding author: E-mail: jixiangtian115@163.com

(*Received*: 30 June 2011; *Accepted*: 24 February 2012) AJC-11117

Co-crystallization of benzophenone-2,4'-carboxylate and 1,2-*bis*(4-pyridyl)ethane was synthesized and characterized by X-ray diffraction at the triclinic, space group P-1 with a = 7.443(5), b = 9.293(10), c = 13.131(5) Å, β = 79.276(5)°, M_r = 363.36, V = 843.1(8) Å^3, D_c = 1.431 g/cm^3, F(000) = 380 and Z = 2. There are one molecule of benzopenone-2,4'-carboxylate and 1,2-*bis*(4-pyridyl)ethane in the asymmetric unit. The intermolecular hydrogen bonds are observed in the compound.

Key Words: Hydrothermal, Synthesis, Crystal structure, Benzophenone-2,4'-carboxylate, 1,2-*Bis*(4-pyridyl)ethane.

A bidentate 1,2-*bis*(4-pyridyl)ethane ligand has been widely used as building blocks for constructing a functional supramolecular architecture in the crystal engineering[1-7]. Molecular components can be connected by various types of the bipyridine ligands *via* metal-coordination bonds and/or hydrogen bonds. The 2D layer structure was stabilized by intermolecular C-H···O interactions. In the course of this work, we have prepared a new 1:1 co-crystal of benzopenone-2,4'-carboxylate with 1,2-*bis*(4-pyridyl)ethane.

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

Preparation of compound: A mixture of benzopenone-2,4'-carboxylate (1 mmol), 1,2-*bis*(4-pyridyl)ethane (1 mmol) and distilled water (15 mL) was heated in a 25 mL stainless steel reactor with a Teflon liner 160 ºC for 96 h, followed by slow cooling to room temperature. Yellow crystals of the compound formed.

X-crystallography: Suitable single crystals were selected under a polarizing microscope and fixed with epoxy cement on fine glass fibers which were mounted on a Bruker Smart 1000 CCD diffractometer with a MoK$_\alpha$ radiation (λ = 0.71073 Å) at 293(2) K. The hydrogen atoms bound to carbon were located by geometrically calculations. All non-hydrogen atoms were refined by full-matrix least-squares techniques. All calculations were performed by the SHELXTL 97 program[8].

Structure description: The molecular structure of the co-crystal with the atom labels in shown in Fig. 1. Bond lengths and angles are listed in Table-1. The co-crystal crystallizes in the P-1 space group with one molecule of benzopenone-2,4'-carboxylate and one molecule of 1,2-*bis*(4-pyridyl)ethane in the asymmetric unit. The carboxylate groups are twisted with respect to correspondingly linking phenyl rings with the dihedral angles 15.17° and 7.11°.

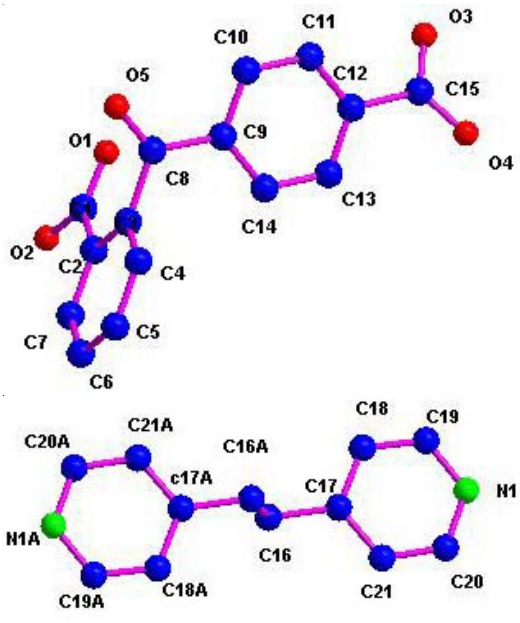

Fig. 1. Molecular structure of the title compound at 30 % probability displacement ellipsoids

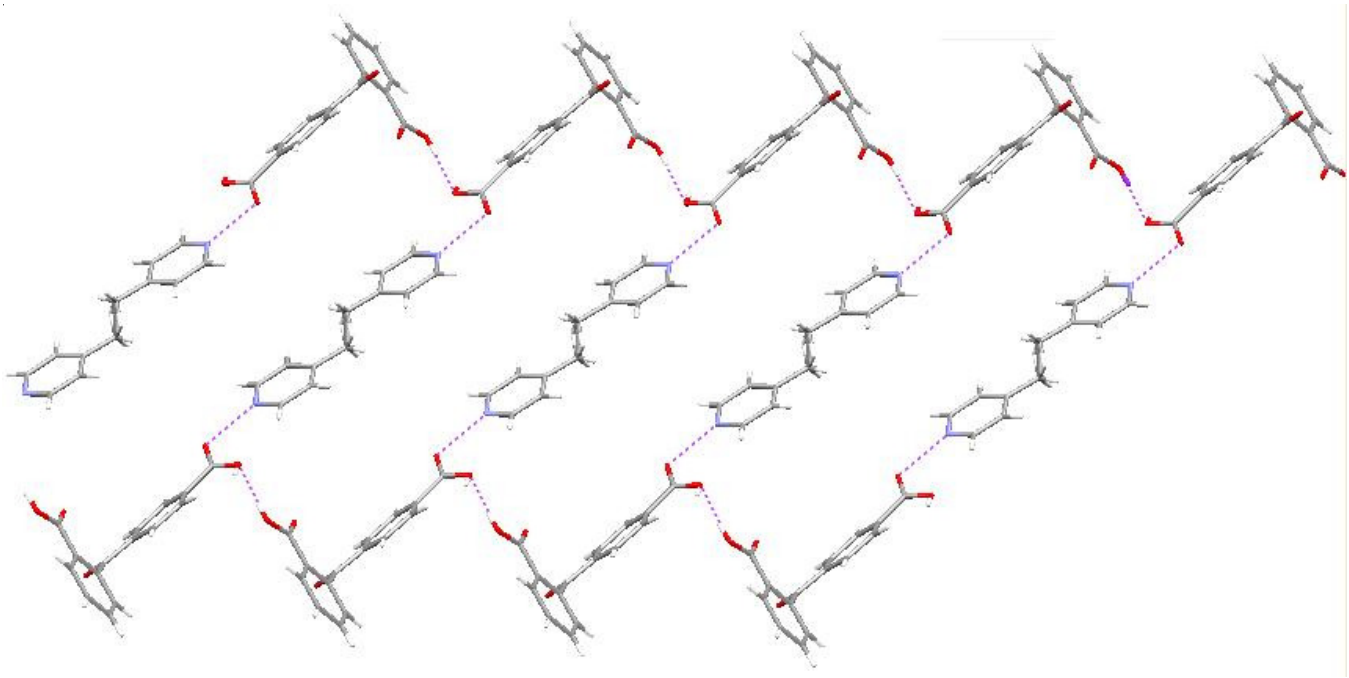

Fig. 2. View of the 2D layer structure

TABLE-1
SELECTED BOND LENGTHS AND ANGLES

N(1)-C(20)	1.333(3)	N(1)-C(19)	1.334(3)
C(15)-O(4)	1.244(2)	C(15)-O(3)	1.259(2)
C(15)-C(12)	1.513(3)	C(16)-C(17)	1.504(3)
C(20)-C(21)-C(17)	119.74(18)	O(1)-C(1)-O(2)	123.63(18)
O(1)-C(1)-C(2)	122.20(19)	O(2)-C(1)-C(2)	114.17(16)
C(7)-C(2)-C(3)	19.27(18)	C(7)-C(2)-C(1)	121.14(18)

In the compound, there exist intermolecular hydrogen bonds between the O-H groups of benzopenone-2,4'-carboxylate and N atoms of 1,2-*bis*(4-pyridyl)ethane (Fig. 2).The 1,2-*bis*(4-pyridyl)ethane molecules are linked to benzopenone-2,4'-carboxylate molecules *via* intermolecular O2-H2···N1 [2.677(4); 160.4; symmetry operators: -1/2 + x, -y, z] and O4-H4···N1A [2.667(4); 164.0; symmetry operators: x, -1 + y, z], which indicated that there are strong hydrogen bonds in the compound.

REFERENCES

1. H.W. Roesky and M. Andruh, *Coord. Chem. Rev.*, **236**, 91 (2001).
2. N.F. Zheng, J. Zhang, X.H. Bu and P.Y. Feng, *Cryst. Growth Des.*, **7**, 2576 (2007).
3. M. Du, Z.H. Zhang and Y.P. You, *Acta Cryst.*, **C61**, 574 (2005).
4. N.J. Babu and A. Nangia, *Cryst. Growth Des.*, **6**, 1995 (2006).
5. J.W. Zhao, J. Zhang, S.T. Zheng and G.Y. Yang, *Chin. J. Struct. Chem.*, **27**, 933 (2008).
6. Y.Q. Yang, C.H. Li, W. Li and Y.F. Kuang, *Chin. J. Struct. Chem.*, **27**, 404 (2008).
7. X.L. Zhang, B.Y. Zhu and F. Guo, *Asian J. Chem.*, **21**, 7072 (2009).
8. G.M. Sheldrick, SHELXTL97, Program for the Refinement of Crystal Structure, University of Gottingen, Germany (1997).