

NOTE**Hydrothermal Synthesis and Crystal Structure of
A One-dimensional Zigzag Chain Based on
2,4'-Biphenyl-dicarboxylic Acid and 4,4'-Bipyridine**

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Co-crystallization of 2,4'-biphenyl-dicarboxylic acid (2,4'-dpa) and 4,4'-bipyridine (4,4'-bpy) was synthesized and characterized by X-ray diffraction at the orthorhombic, space group *Pbca* with $a = 24.421(5)$, $b = 9.055(10)$, $c = 17.843(2)$ Å, $\alpha = \beta = \gamma = 90^\circ$, $M_r = 398.40$, $V = 3945.4(7)$ Å³, $D_c = 1.341$ g/cm³, $F(000) = 1664$ and $Z = 8$. There are one molecule of 2,4'-biphenyl-dicarboxylic acid and 4,4'-bipyridine in the asymmetric unit. The dihedral angle between the pyridine ring planes is 2.40° . The intermolecular hydrogen bonds are observed in the compound.

Key Words: Hydrothermal Synthesis, Crystal structure, One-dimensional, 2,4'-Biphenyl-dicarboxylic acid, 4,4'-Bipyridine.

A bidentate 4,4'-bipyridine ligand has been widely used as building blocks for constructing a functional supramolecular architecture in the crystal engineering¹⁻⁶. Molecular components can be connected by various types of the bipyridine ligands *via* metal-coordination bonds and/or hydrogen bonds. We have recently reported that a robust and a predictable zigzag chain structure can be constructed from phthalic acid with 4,4'-bipyridine⁷. The zigzag chains were stabilized by intermolecular C-H...O interactions. In the course of this work, we have prepared a new 1:1 co-crystal of 2,4'-biphenyl-dicarboxylic acid and 4,4'-bipyridine.

All reagent and solvents employed were commercially available and used as received without further purification. Elemental analysis was carried out on a Carlo Erba 1106 full-automatic trace organic element analyzer.

Synthesis of the title compound: A mixture of 2,4'-biphenyl-dicarboxylic acid (1 mmol), 4,4'-bipyridine (1 mmol) and distilled water (15 mL) was heated in a 25 mL stainless steel reactor with a Teflon liner 160 °C for 96 h, followed by slow cooling to room temperature. Yellow crystals of the compound formed. Anal. calcd. (%) for C₂₄H₁₈N₂O₄: C, 72.35; H, 4.55; N, 7.03. Found. (%): C, 71.84; H, 4.54, N, 7.12.

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X-Ray crystallography: A suitable yellow block crystal with dimensions of 0.32 mm × 0.26 mm × 0.21 mm was mounted on a glass fiber and data were collected on a Bruker Smart 1000 CCD diffractometer with a MoK α radiation ($\lambda = 0.71073 \text{ \AA}$) at 293(2) K by using an ω scan mode in the range of $1.67 < \theta < 27.47$. All non-hydrogen atoms were located by geometrically calculations and their positions and thermal parameters were fixed during the structure refinement. The highest and lowest residual peaks in the final difference Fourier map are 0.367 and $-0.199 e/\text{\AA}^3$, respectively. All calculations were performed by the SHELXTL 97 program⁸. The selected bond lengths and bond angles are listed in Table-1. CCDC:759390.

TABLE-1
SELECTED BOND LENGTHS (\AA) AND ANGLES ($^\circ$) FOR COMPOUND

C35-O6	1.196(5)	C48-O7	1.206(4)
C35-O5	1.312(4)	C48-O8	1.306(4)
C29-N3	1.326(5)	C32-N4	1.301(5)
C33-N4	1.307(6)	C25-N3	1.324(5)
C25-N3-C29	116.7(3)	O7-C48-O8	123.4(3)
C33-N4-C32	117.9(3)	O6-C35-O5	123.2(3)

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 Pbc a space group with one molecular of 2,4'-biphenyl-dicarboxylic acid and one molecule of bipyridine in the asymmetric unit. Within each 4,4'-bipyridine subunit, the dihedral angle between the rings is 2.40° . The carboxylate groups are twisted with respect to correspondingly linking phenyl rings with the dihedral angles 33.87° and 6.88° and two phenyl rings about the central bond have a twisting with a dihedral angle 56.91° .

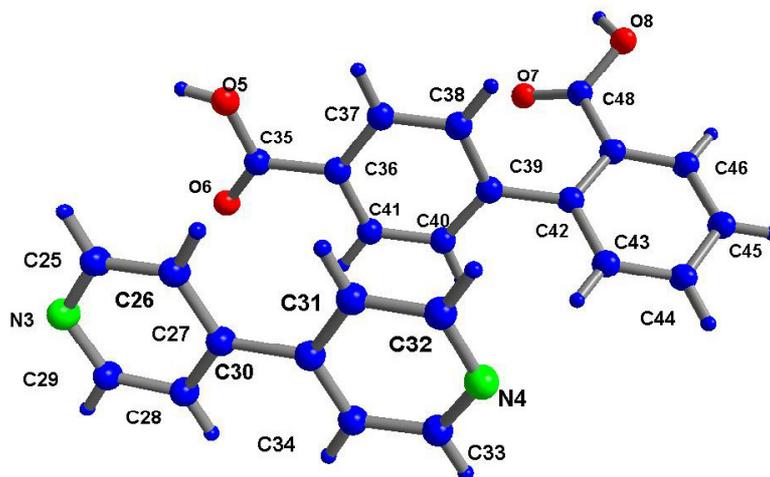


Fig. 1. Molecular structure of the title compound at 30 % probability displacement ellipsoids

In the compound, there exist intermolecular hydrogen bonds between the O-H groups of 2,4'-biphenyl-dicarboxylic acid and N atoms of 4,4'-bipyridine. (Fig. 2). The 4,4'-bipyridine molecules are linked to 2,4'-biphenyl-dicarboxylic acid molecules *via* intermolecular O8-H8...N3 [2.677(4); 160.4; symmetry operators: $-1/2 + x, -y, z$] and O5-H5...N4 [2.667(4); 164.0; symmetry operators: $x, -1 + y, z$], which indicated that there are strong hydrogen bonds in the compound.

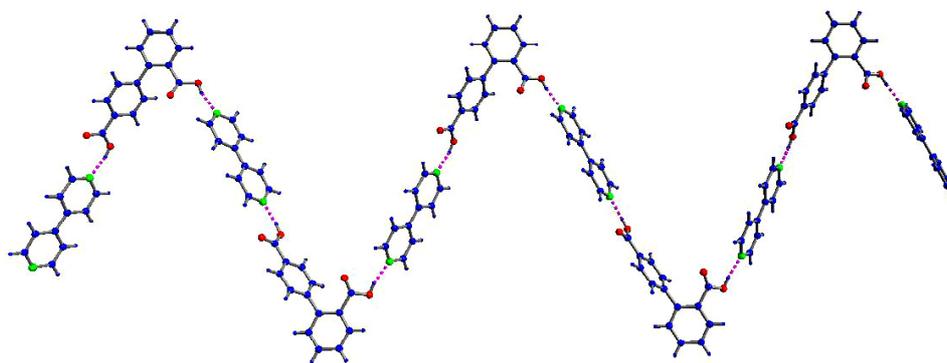


Fig. 2. View of the 1-D zigzag chain along the c-axis

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