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Synthesis, Crystal Structure of 3-Nitro-2-pyridinecarboxaldehyde-furan-2-carbohydrazone

Lihua Wang*, Dandan Lin

College of Information and Control Engineering, Weifang University, Weifang 261061, China

*Corresponding author e-mail: taixs@wfu.edu.cn

Abstract. A new hydrazone compound, 3-nitro-2-pyridinecarboxaldehyde-furan-2-carbohydrazone (**1**) was synthesized by the reaction of furan-2-carbohydrazide and 3-nitro-2-pyridinecarboxaldehyde in ethanol solution. The structure of **1** has been characterized by single crystal X-ray diffraction analysis. The results show that the **1** belongs to monoclinic, space group $P2_1/c$ with a = 17.3723(6) Å, b = 9.1481(3) Å, c = 15.5841(5) Å, β = 104.538(4)º, V = 2397.38(14) Å^3, Z = 4, Dc = 1.608 mg·m^{-3}, μ = 0.167 mm^{-1}, F(000) = 1161, and final R_1 = 0.0702, ωR_2 = 0.1852, S = 1.090. The 3-nitro-2-pyridinecarboxaldehyde-furan-2-carbohydrazone (**1**) molecules form a 1D chain structure by N-H···O hydrogen bond. And the 1D chains form 3D supramolecular network structure by the interaction of N-H···O hydrogen bonds.

1. Introduction

During the past decades, many researches on the synthesis and property of hydrazone derivates and their metal complexes have been carried out. Because they have exhibited potential use in many aspects such as antibacterial activity [1, 2], antituberculosis activity [3, 4], antioxidant activity [5], antitumor activity [6, 7], luminescent and photochemical property [8], and electrochemical property [9].

In this paper, we synthesized a new hydrazone compound, 3-nitro-2-pyridinecarboxaldehyde-furan-2-carbohydrazone (**1**) by the reaction of furan-2-carbohydrazide and 3-nitro-2-pyridinecarboxaldehyde in ethanol solution. The structure of **1** has been determined by single crystal X-ray diffraction analysis. The scheme of 3-nitro-2-pyridinecarboxaldehyde-furan-2-carbohydrazone (**1**) is shown in Figure 1.

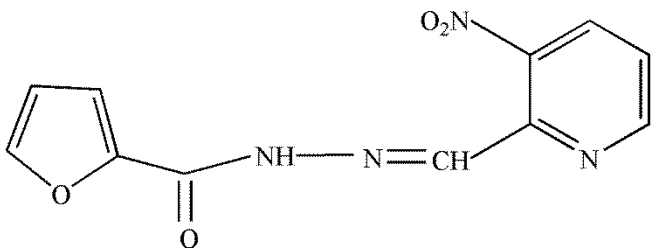

Figure 1. The scheme of 3-nitro-2-pyridinecarboxaldehyde-furan-2-carbohydrazone (**1**)

1.1. Materials and Instrumentation

Furan-2-carbohydrazide, 3-nitro-2-pyridinecarboxaldehyde, and ethanol were used as received. The structure of 3-nitro-2-pyridinecarboxaldehyde-furan-2-carbohydrazone (**1**) was determined by a Bruker Smart CCD diffractometer.

1.2. Synthesis of 3-Nitro-2-pyridinecarboxaldehyde-furan-2-carbohydrazone (1)

The mixture of furan-2-carbohydrazide (0.2522 g, 2.0 mmol) and 3-nitro-2-pyridinecarboxaldehyde (0.3040 g, 2.0 mmol) were added to a 100 mL round flask, then 20 mL mixed solvents of CH_3CH_2OH: H_2O (v:v = 3:1) were added. The mixture was heated to 85 60 °C and kept this temperature for 6 h with stirring. The products were obtained by filtering, and the colorless block crystals were obtained by evaporating filtrate at room temperature.

1.3. Data Collection, Structural Determination, and Refinement

The crystal data of 3-nitro-2-pyridinecarboxaldehyde-furan-2-carbohydrazone (**1**) were collected with a Bruker Smart CCD diffractometer at 293(2) K using a graphite-monochromatized MoKα (λ = 0.71073 Å) radiation. The structure was solved by direct methods with SHELXL-97 [9] and refined on F^2 by full-matrix least-squares procedures with SHELXTL-97 [9].

2. Results and Discussion

2.1. Structural Description of 3-Nitro-2-pyridinecarboxaldehyde-furan-2-carbohydrazone (1)

The molecular structure is shown in Figure 2. The selected bond lengths (Å) and angles (º) for **1** are given in Table 2. The results of structural analysis show that **1** belongs to monoclinic, space group $P2_1/c$ with a = 17.3723(6) Å, b = 9.1481(3) Å, c = 15.5841 (5) Å, β = 104.538 (4)º, V = 2397.38(14) $Å^3$, Z = 4, Dc = 1.608 mg·m^{-3}, μ = 0.167 mm^{-1}, F (000) = 1161, and final R_1 = 0.0702, ωR_2 = 0.1852, S = 1.090. From Figure 2, it can be seen that the crystal structure of 3-nitro-2-pyridinecarboxaldehyde-furan-2-carbohydrazone (**1**) is built up by 3-nitro-2-pyridinecarboxaldehyde-furan-2-carbohydrazone molecules. From Table 2, it can be seen that the bond lengths of C7-O1, C18-O5, C6-N3 and C17-N7 are 1.223(4) Å, 1.232(4) Å, 1.267(5) Å and 1.265(5) Å, respectively. Which are much shorter than those of C-O and C-N bonds in this crystal structure, indicating that the bonds of C7-O1, C18-O5, C6-N3 and C17-N7 are double bonds. The dihedral angles of pyridine ring 1 (C1-C2-C3-C4-C5-N1) and furan ring 1 (C8-C9-C10-C11-O2), pyridine ring 2 (C12-C13-C14-C15-C16-N5) and furan ring 2 (C19-C20-C21-C22-O6) are 35.6º and 26.3º, respectively. The 3-nitro-2-pyridinecarboxaldehyde-furan-2-carbohydrazone (**1**) molecules form a 1D chain structure by N-H···O hydrogen bond. The 1D chained structure is shown in Figure 3. And the 1D chains further form 3D network supramolecular structure by the π-π stacking of furan rings and pyridine rings. The 3D network supramolecular structure is shown in Figure 4.

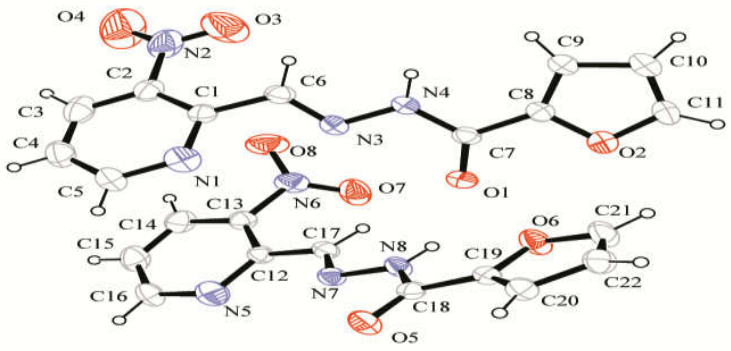

Figure 2. The molecular structure of 3-nitro-2-pyridinecarboxaldehyde-furan-2-carbohydrazone (**1**).

Table 1. Selected bond lengths (Å) and angles (º) for **1**

Bond	**Distance**
C19-O6	1.354(4)
C21-O6	1.365(5)
C18-O5	1.232(4)
C7-O1	1.223(4)
C18-N8	1.342(5)
N8-N7	1.388(4)
N4-C7	1.355(5)
N4-N3	1.381(4)
C17-N7	1.265(5)
C8-O2	1.356(5)
C11-O2	1.361(5)
C6-N3	1.267(5)
N6-O7	1.228(5)
N6-O8	1.221(5)
C16-N5	1.386(5)
C5-N1	1.390(6)
N2-O4	1.174(6)
N2-O3	1.180(6)
C6-N3	1.267(5)
C12-N5	1.404(5)
C16-N5	1.386(5)
Angle	°
C19-O6-C21	106.2(3)
C18-N8-N7	120.0(3)
C7-N4-N3	118.6(3)
C17-N7-N8	113.8(3)
C8-O2-C11	106.8(4)
C6-N3-N4	115.2(3)
O1-C7-N4	123.8(3)
O1-C7-C8	121.7(3)
O8-N6-O7	124.2(4)
O8-N6-C13	118.0(4)
O7-N6-C13	117.7(3)
O5-C18-N8	124.5(3)
O5-C18-C19	120.6(3)
C16-N5-C12	119.4(4)
O2-C8-C7	116.4(3)
O4-N2-O3	119.3(6)
O4-N2-C2	118.1(6)
O3-N2-C2	121.2(4)
C4-C5-N1	121.1(5)
C10-C11-O2	110.4(4)
C2-C1-N1	116.8(4)
N1-C1-C6	118.1(3)
C5-N1-C1	120.3(4)

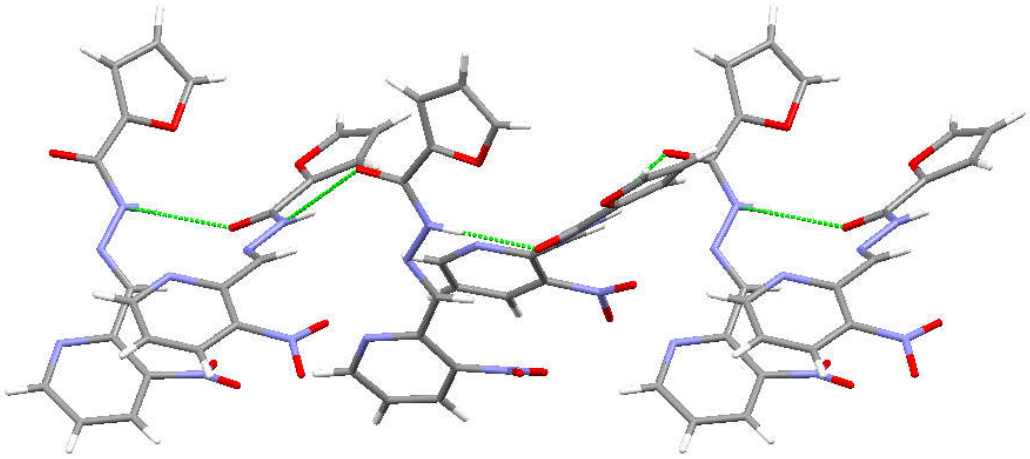

Figure 3. The 1D chained structure of **1**

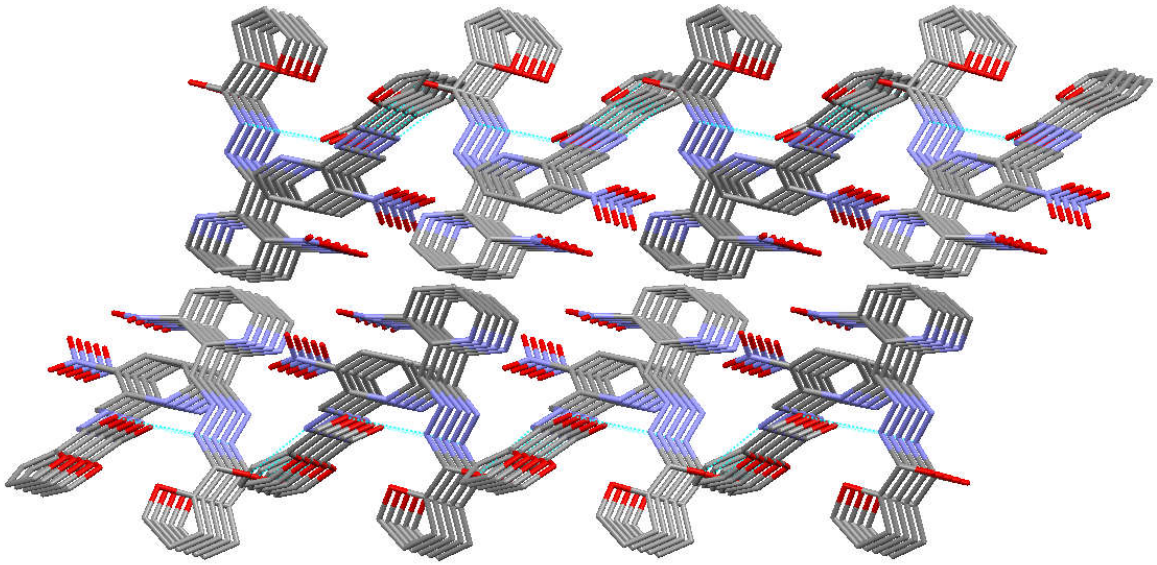

Figure 4. The 3D network supramolecular structure of **1**

3. Conclusion

In this paper, we synthesized a new hydrazone compound, 3-nitro-2-pyridinecarboxaldehyde-furan-2-carbohydrazone (**1**) by the reaction of furan-2-carbohydrazide and 3-nitro-2-pyridinecarboxaldehyde in ethanol solution. The structure of **1** has been determined by single crystal X-ray diffraction analysis. The 3-nitro-2-pyridinecarboxaldehyde-furan-2-carbohydrazone (**1**) molecules form a 1D chain structure by N-H···O hydrogen bond.

Acknowledgments
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