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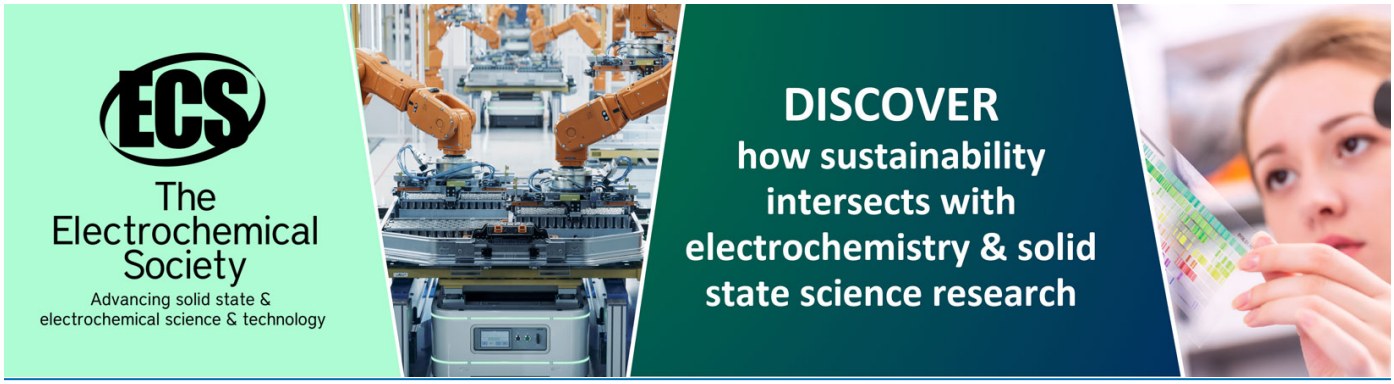

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Synthesis and Crystal Structure of 4-Hydroxy-benzaldehyd-thiosemicarbazide

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Abstract. A Schiff base compound, 4-hydroxy-benzaldehyd-thiosemicarbazide ($C_8H_9N_3OS$), has been prepared by the reaction of 4-hydroxy-benzaldehyd with thiosemicarbazide in ethanol-water (v: v = 3:1) solution. The structure of 4-hydroxy-benzaldehyd-thiosemicarbazide was determined by single crystal X-ray diffraction. The result gives that the 4-hydroxy-benzaldehyd-thiosemicarbazide belongs to triclinic, space group *P*-1 with a = 4.0285(8) Å, b = 10.754(2) Å, c = 21.229(4) Å, α = 92.50(3)º, β = 90.11(3)º, γ = 99.36(3)º, V= 906.5(3) Å^3, Z = 4, Dc = 1.431 mg·m^{-3}, μ = 0.318 mm^{-1}, F (000) = 408, and final R_1 = 0.1508, ωR_2 = 0.3641. The 4-hydroxy-benzaldehyd-thiosemicarbazide molecules form 1D chain structure by the O-H·S intermolecular hydrogen bond interaction.

1. Introduction

Schiff base compounds and their metal complexes have been the focus of chemists and materials scientists [1 – 3]. Because they exhibit many good properties in many areas: antimicrobial activity, selective detection, and liquid crystalline [4 – 6]. Based on the above comments, we have synthesized and characterized some Schiff base compounds and their metal complexes [7 – 9]. In this paper, a new Schiff base compound, 4-hydroxy-benzaldehyd-thiosemicarbazide ($C_8H_9N_3OS$), has been prepared by the reaction of 4-hydroxy-benzaldehyd with thiosemicarbazide in ethanol-water (v: v = 5:1) solution.

2. Experimental Section

2.1. Materials and Instrumentation

4-Hydroxy-benzaldehyd (A. R.), thiosemicarbazide (A. R.), and solvents were purchased from Innochem reagent.

The crystal data was collected on a Bruker Smart CCD Area Detector.

2.2. Synthesis of 4-Hydroxy-benzaldehyd-thiosemicarbazide

0.1221 g 4-hydroxy-benzaldehyd (1.0 mmol) was dissolved in15 mL ethanol solution with stirring. 5 mL water solution of 0.0910 g thiosemicarbazide (1.0 mmol) was added dropwise to the above solution. Then the mixture was heated for 5 h at 70ºC with stirring. After cooled to room temperature, the solution was filtrated, and the crystals were collected from the filtrate after 30 days.

2.3. Data Collection, Structural Determination, and Refinement

Diffraction data were collected on a Bruker Smart Apex CCD diffractometer using a φ~ω scan mode with a graphite-monochromatic Mo Kα radiation (λ = 0.71073 Å) at 293 (2) K. Crystal size: 0.19 Å × 0.18 Å × 0.17 Å; Empirical formula: $C_8H_9N_3OS$; Formula weight: 195.24; Crystal system: triclinic; Space group: P-1; a: 4.0285(8) Å; b: 10.754(2) Å; c: 21.229(4) Å; α: 92.50(3)º, β: 90.11(3)º, γ: 99.36(3)º; V: 906.5(3) $Å^3$; Z; 4; Dc: 1.431 $mg·m^{-3}$; Parameters: 236; Restraints: 0; μ: 0.318 mm^{-1}; F(000): 408; Refl'ns collected: 6950; Independent refl'ns [R(int)]: 3165 [0.1149]; Refl'ns observed (>2δ): 2258; Refinement method: Full-matrix least-squares on F^2; Goodness-of-fit: 1.023; Final R indices [I > 2δ(I)]: 0.1508, 0.3641; R indices (all data): 0.1843, 0.3889; Limiting indices: $-4 \le h \le 4$, $-12 \le k \le 12$, $-25 \le l \le 25$; S: 1.023; Min. and max. resd. Dens. ($e/Å^3$): 0.532, -0.554; SHELXL and OLEX2 were used to refine structure [10, 11]. DIAMOND was used to draw the molecular structure [12].

The hydrogen atoms were positioned geometrically (C-H = 0.93 Å, O-H = 0.82 Å and N-H = 0.86 - 0.89 Å). Their U_{iso} values were set to $1.2U_{eq}$ or $1.5U_{eq}$ of the parent atoms.

3. Results and Discussion

The molecular structure of 4-hydroxy-benzaldehyd-thiosemicarbazide is shown in Figure 1. One dimensional chain structure formed by hydrogen bonds is shown in Figure 2. Three dimensional network structure is shown in Figure 3. As shown in Figure 1, the crystal structure of 4-hydroxy-benzaldehyd-thiosemicarbazide is built up by 4-hydroxy-benzaldehyd-thiosemicarbazide molecule. The C7=N1 and C15=N4 bond lengths are 1.32(2) Å and 1.264(18) Å, respectively, suggesting that the two above C-N bonds are double bond. The 4-hydroxy-benzaldehyd-thiosemicarbazide molecules are interacted by O-H···S intermolecular hydrogen bond interaction to form 1D chain structure (Figure 2). The 1D chains are interacted by intermolecular hydrogen bonds and π-π stacking interaction of benzene rings to form three dimensional network structure (Figure 3).

Selected bonds: S2-C16 1.701(13) Å; O2-C11 1.372(15) Å; C16-N5 1.341(16) Å; N5-N4 1.406(14) Å; C15-N4 1.264(18) Å; C16-N6 1.307(17) Å; C13-C12 1.377(18) Å; C13-C14 1.397(19) Å; C15-C14 1.445(17) Å; C9-C14 1.424(18) Å; C9-C10 1.34(2) Å; C10-C11 1.37(2) Å; C11-C12 1.39(2) Å; S1-C8 1.684(13) Å; O1-C1 1.355(15) Å; N1-C7 1.32(2) Å; N1-N2 1.383(15) Å; N3-C8 1.355(18) Å; N2-C8 1.315(18) Å; C5-C6 1.374(17) Å; C5-C4 1.403(19) Å; C1-C6 1.403(19) Å; C1-C2 1.404(18) Å; C3-C4 1.394(19) Å; C2-C3 1.409(19) Å; C7-C4 1.432(19) Å.

Selected angles: C16-N5-N4 119.5(10)°; C15-N4-N5 116.0(11)°; C12-C13-C14 118.9(13)°; N4-C15-C14 123.9(13)°; C9-C14-C13 118.7(11)°; C13-C14-C15 120.8(12)°; C9-C14-C15 120.5(12)°; C10-C9-C14 120.1(12)°; C9-C10-C11 122.3(13)°; C10-C11-O2 120.3(12)°; C10-C11-C12 118.0(11)°; O2-C11-C12 121.7(12)°; N6-C16-N5 117.9(12)°; N6-C16-S2 123.8(10)°; N5-C16-S2 118.3(9)°; C11-C12-C13 122.1(12)°; C7-N1-N2 113.3(13)°; C8-N2-N1 121.6(13) °; C4-C5-C6 122.1(12) °; N2-C8-N3 116.1(12) °; N2-C8-S1 121.6(11)°; N3-C8-S1 122.1(11)°; C5-C6-C1 119.8(12)°; O1-C1-C2 116.9(12)°; O1-C1-C6 122.7(12)°; C2-C1-C6 120.4(12)°; C2-C3-C4 123.6(13)°; C1-C2-C3 117.2(12)°; N1-C7-C4 121.8(14)°; C3-C4-C5 116.9(12)°; C3-C4-C7 119.9(13)°; C5-C4-C7 123.2(13) °.

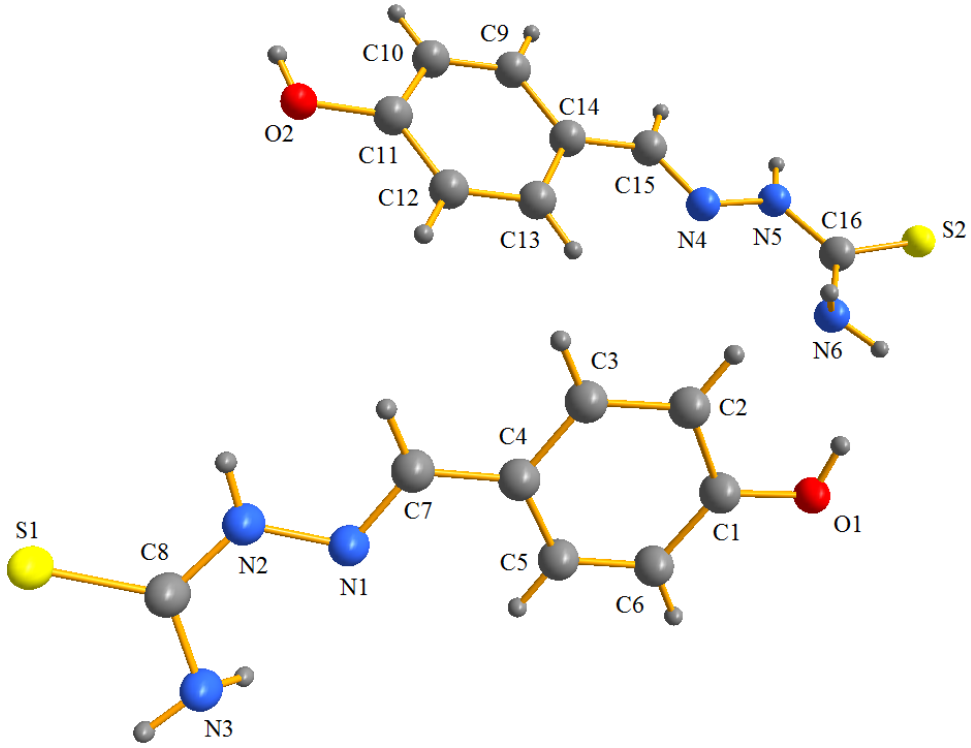

Figure 1. Molecular structure of the title compound, where the thermal ellipsoids were draw at 30% possibility.

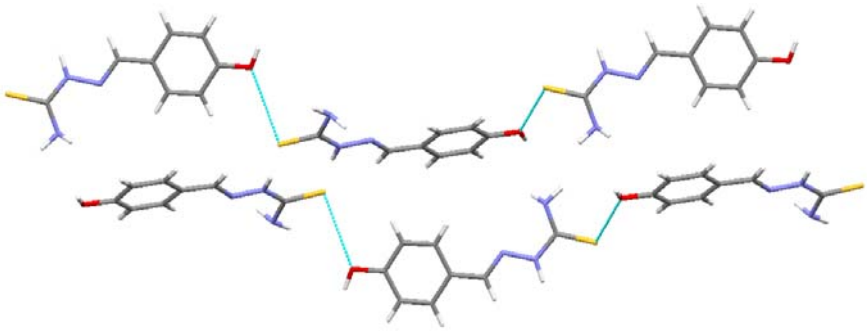

Figure 2. One dimensional chain structure formed by hydrogen bonds

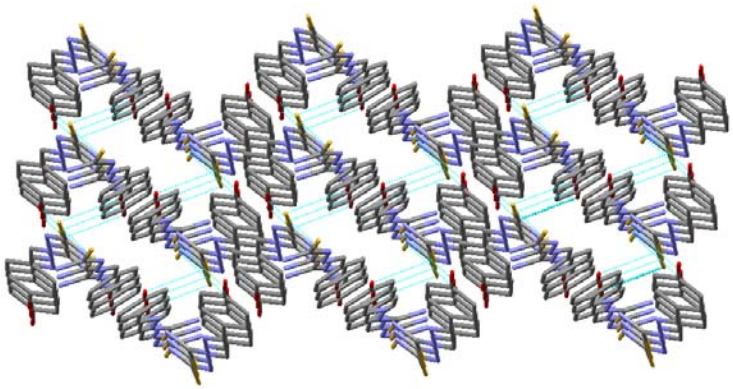

Figure 3. Three dimensional network structure

4. Conclusion

A Schiff base compound, 4-hydroxy-benzaldehyd-thiosemicarbazide, has been prepared by the reaction of 4-hydroxy-benzaldehyd with thiosemicarbazide in ethanol-water (v: v = 3:1) solution. Its structure was determined by single crystal X-ray diffraction. The 4-hydroxy-benzaldehyd-thiosemicarbazide molecules form 1D chain structure by the O-H···S interaction.

Acknowledgments

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