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NOTE

Synthesis and Crystal Structure of 4-Formaldehyde Benzaldehyde Methylthiosemicarbazone

WEI SU[1,2,*], QUANQUAN QIAN[1,2], CHUNLING HE[3], LIFENG LIU[1,2], QUAN ZHOU[1,2] and PEIYUAN LI[3,*]

[1]Key Laboratory of Beibu Gulf Environment Change and Resources Utilization, Ministry of Education, Guangxi Teachers Education University, Nanning 530001, P.R. China
[2]College of Chemistry and Life Science, Guangxi Teachers Education University, Nanning 530001, P.R. China
[3]College of Pharmacy, Guangxi University of Chinese Medicine, Nanning 530001, P.R. China

*Corresponding author: E-mail: lipearpear@yahoo.cn; aaasuwei@yahoo.com.cn

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A thiosemicarbazone single crystal synthesized with molecular formula as $C_{10}H_{11}N_3OS$, was synthesized by 1,4-phthalaldehyde and 4-methylthiosemicarbazide. The crystal is monoclinic, space group $P2_1/c$ with unit cell parameters: a = 10.1467(3) Å, b = 8.5255(2) Å, c = 13.1531(3) Å, α = 90.00º, β = 103.417(2)º, γ = 90.00º, V =1106.75(5) Å^3, Z = 4, M_r = 322.23, D_c = 1.934 g/cm^3, μ = 1.682 mm^{-1}, $F_{(000)}$ = 635, R = 0.0342, wR = 0.0807 for 11143 reflections with I > 2σ(I).

Key Words: Thiosemicarbazone, Terephthalaldehyde, Single crystal.

Thiosemicarbazones has attracted great investigative efforts because its pharmacological properties[1-3]. Here we report a novel 4-formaldehyde benzaldehyde methylthiosemicarbazone single crystal synthesized with a formula as $C_{10}H_{11}N_3OS$.

All reagents for synthesis were commercially available and employed as received or purified by standard methods prior to use. Analyses for carbon, hydrogen and nitrogen were performed on a Perkin-Elmer 1400C analyzer.

Synthesis: For the synthesis of present compoud, 0.134 g 1,4-phthalaldehyde (1 mmol) and 0.212 g 4-methylthiosemicarbazide were diluted using 10 mL mixture solution of ethane and water (1:1). The solution was refluxed and then cooled to room temperature and filtered. Single crystals suitable for X-ray analysis were grown from the filtrate by slow evaporation at room temperature in air.

Crystal structure determination: A single crystal of compound with dimensions of 0.21 mm × 0.14 mm × 0.10 mm was selected for crystallographic data collection at 293(2) K and structure determination on a Bruker SMART CCD-4K diffractometer employing graphite-monochromated MoK_α radiation (λ = 0.71073 Å). A total of 4031 reflections were collected in the range of 5.66º ≤ θ ≤ 50.5º, of which 1902 reflections were unique with R_{int} = 0.0414. The data were collected using SMART and reduced by the program SAINT. All the structures were solved by direct methods and refined by full-matrix least squares method on 'F^2_{obs}' by using SHELXTL-PC software package. Non-hydrogen atoms were placed in geometrically calculated positions. Hydrogen atoms were added according to theoretical model. The final full-matrix least-squares refinement including 126 variable parameters for 1902 reflections with I > 2σ(I) and converged with unweighted and weighted agreement factors of

$$R_1 = \frac{\Sigma(||F_0| - |F_C||)}{\Sigma|F_0|} = 0.0844 \quad (1)$$

TABLE-1
SELECTED BOND DISTANCES (Å) AND ANGLES (º)

S1-C2	1.6882(16)	C3-C4	1.461(2)	C3-N3-N2	116.13(15)	C9-C4-C5	118.85(16)
O1-C10	1.202(3)	C4-C5	1.396(3)	N1-C2-S1	124.57(14)	C6-C5-C4	120.67(18)
N1-C1	1.448(2)	C4-C9	1.389(3)	N1-C2-N2	117.13(15)	C5-C6-C7	120.03(19)
N1-C2	1.319(2)	C5-C6	1.380(2)	N2-C2-S1	118.29(13)	C6-C7-C10	120.54(19)
N2-N3	1.3780(18)	C6-C7	1.391(3)	N3-C3-C4	120.87(17)	C8-C7-C10	119.58(16)
N2-C2	1.360(2)	C2-N1-C1	124.37(15)	C5-C4-C3	121.44(17)	C7-C8-C9	120.50(18)
N3-C3	1.275(2)	C2-N2-N3	120.06(15)	C9-C4-C3	119.69(17)	O1-C10-C7	125.0(2)

and $$wR_2 = \left\{\frac{\Sigma[w(F_0^2 - F_C^2)^2]}{\Sigma w(F_0^2)^2}\right\}^{1/2} = 0.2607 \quad (2)$$

where $w = 1/[\sigma^2(F_0^2) + (0.1014P)^2]$ and $P = (F_0^2 + 2F_C^2)/3$. The maximum and minimum peaks on the final difference Fourier map are corresponding to 0.766 and -0.525 e/Å^3, respectively.

The selected bond lengths and bond angles are listed in Table-1. Fig. 1 shows the molecular structure of the present compound. Fig. 2 shows the packing diagram of the present compound. The present compound crystallizes in the triclinic system of Pc space group. The average S-C bond distance in thiosemicarbazone moiety is 1.737 Å and the average C-N bond distance is 1.357 Å.

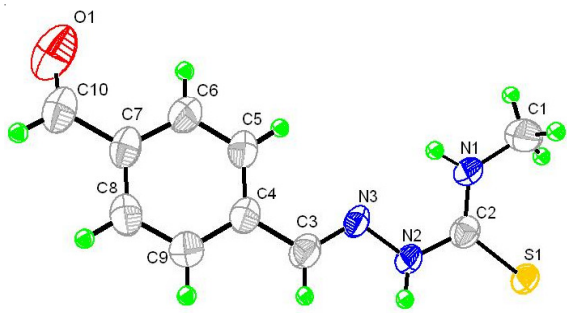

Fig. 1. Molecular structure of the 4-formaldehyde benzaldehyde methylthiosemicarbazone

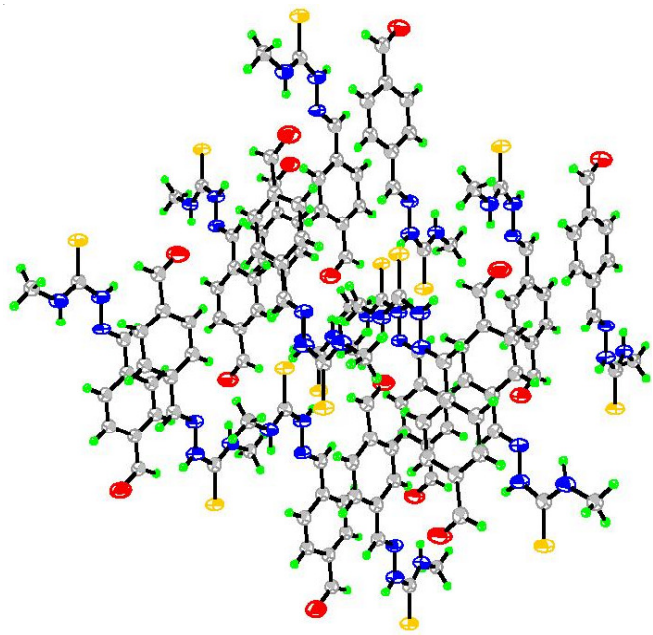

Fig. 2. View of a 3D supramolecular framework of the 4-formaldehyde benzaldehyde methylthiosemicarbazone

Conclusion

Crystal structure of a novel 4-formaldehyde benzaldehyde methylthiosemicarbazone has been synthesized and characterized by elemental analysis and X-ray diffraction analysis.

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