NOTE

Synthesis and Crystal Structure of 4-Formaldehyde Benzaldehyde Methylthiosemicarbazone

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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: $a = 10.1467(3) \text{ \AA}$, $b = 8.5255(2) \text{ \AA}$, $c = 13.1531(3) \text{ \AA}$, $\alpha = 90.00^\circ$, $\beta = 103.417(2)^\circ$, $\gamma = 90.00^\circ$, $V = 1106.75(5) \text{ \AA}^3$, $Z = 4$, $M_r = 322.23$, $D_c = 1.934 \text{ g/cm}^3$, $\mu = 1.682 \text{ mm}^{-1}$, $F_{(000)} = 635$, $R = 0.0342$, $wR = 0.0807$ for 11143 reflections with $I > 2\sigma(I)$.

Key Words: Thiosemicarbazone, Terephthalaldehyde, Single crystal.

Thiosemicarbazones has attracted great investigative efforts because its pharmacological properties¹⁻³. 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 compound, 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 $\times$ 0.14 mm $\times$ 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 ($\lambda = 0.71073 \text{ \AA}$). A total of 4031 reflections were collected in the range of $5.66^\circ \leq \theta \leq 50.5^\circ$, 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 $I > 2\sigma(I)$ and converged with unweighted and weighted agreement factors of

$$R_1 = \frac{\sum(|F_o| - |F_c|)}{\sum|F_o|} = 0.0844 \quad (1)$$

TABLE-1
SELECTED BOND DISTANCES ($\text{\AA}$) AND ANGLES ($^\circ$)

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)

$$\text{and } wR_2 = \left\{ \frac{\sum [w(F_o^2 - F_c^2)^2]}{\sum w(F_o^2)^2} \right\}^{1/2} = 0.2607 \quad (2)$$

where $w = 1/[\sigma^2(F_o^2) + (0.1014P)^2]$ and $P = (F_o^2 + 2F_c^2)/3$. The maximum and minimum peaks on the final difference Fourier map are corresponding to 0.766 and -0.525 e/Å³, 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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