NOTE

Synthesis and Crystal Structure of a Thiosemicarbazone

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A thiosemicarbazone single crystal synthesized with molecular formula as $C_{14}H_{12}N_4S$, was synthesized by 3-phthalaldehyde and 4-methylthiosemicarbazide. The crystal is monoclinic, space group Pc with unit cell parameters: $a = 11.3218(12) \text{ \AA}$, $b = 7.0361(5) \text{ \AA}$, $c = 8.5416(6) \text{ \AA}$, $\alpha = 90.00^\circ$, $\beta = 103.821(9)^\circ$, $\gamma = 90.00^\circ$, $V = 660.74(10) \text{ \AA}^3$, $Z = 2$, $M_r = 268.34$, $D_c = 1.349 \text{ g/cm}^3$, $\mu = 0.236 \text{ mm}^{-1}$, $F_{(000)} = 280$, $R = 0.0390$, $wR = 0.1016$ for 2130 reflections with $I > 2\sigma(I)$.

Key Words: Thiosemicarbazone, Terephthalaldehyde, Single crystal.

Recently, increasing attention has been given to thiosemicarbazones for its broad spectrum of pharmacological properties¹⁻³. Here we report a novel N^2, N^5 -bis(phenyl)-1,3,4-thiadiazole-2,5-diamine solvate single crystal synthesized with a formula as $C_{14}H_{12}N_4S$.

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 3-phthalaldehyde (1 mmol) and 0.324 g (2 mmol) 4-phenylthiosemicarbazide 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 $|F_o^2|$ 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-C7	1.737(3)	C1-C2	1.400(7)	C7-N2-N3	111.9(4)	C3-C4-C5	119.0(6)
S1-C8	1.740(5)	C1-C6	1.383(8)	C8-N3-N2	113.0(4)	C6-C5-C4	121.7(7)
N1-C1	1.398(6)	C2-C3	1.364(8)	C8-N4-C9	127.5(4)	C5-C6-C1	118.9(6)
N1-C7	1.354(5)	C3-C4	1.367(12)	N1-C1-C2	122.0(4)	N3-C8-S1	114.3(3)
N2-N3	1.390(5)	N4-C8	1.344(5)	C6-C1-C2	119.7(5)	N1-C7-S1	119.0(3)
N2-C7	1.313(5)	C7-S1-C8	86.7(2)	C3-C2-C1	119.5(6)	N2-C7-S1	114.1(3)
N3-C8	1.293(5)	C7-N1-C1	127.3(4)	C2-C3-C4	121.1(7)	N2-C7-N1	126.9(4)

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

where $w = 1/[s^2(F_o^2) + (0.1014P)^2]$ 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 structure of the N²,N⁵-bis(phenyl)-1,3,4-thiadiazole-2,5-diamine. The average S-C bond distance in thiosemicarbazone moiety is 1.739 Å.

Fig. 1. Molecular structure of the N²,N⁵-bis(phenyl)-1,3,4-thiadiazole-2,5-diamine

Fig. 2. View of a 3D supramolecular framework of the N²,N⁵-bis(phenyl)-1,3,4-thiadiazole-2,5-diamine

Conclusion

Crystal structure of a novel N²,N⁵-bis(phenyl)-1,3,4-thiadiazole-2,5-diamine has been synthesized and characterized by elemental analysis and X-ray diffraction analysis.

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