

NOTE

Synthesis and Crystal Structure of Terephthalaldehyde-*bis*-(thiosemicarbazone)-*bis*-(dimethyl sulfoxide) Solvate

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A thiosemicarbazone single crystal synthesized with a formula as $C_{10}H_{12}N_6S_2 \cdot 2C_2H_6OS$, was synthesized by 1,4-phthalaldehyde and 4-methylthiosemicarbazide. The crystal is triclinic, space group P-1 with unit cell parameters: $a = 5.9519(5)$ Å, $b = 7.4721(7)$ Å, $c = 12.5450(13)$ Å, $\alpha = 77.348(8)^\circ$, $\beta = 78.672(8)^\circ$, $\gamma = 79.193(7)^\circ$, $V = 527.64(8)$ Å³, $Z = 1$, $M_r = 436.63$, $D_c = 1.374$ g/cm³, $\mu = 0.471$ mm⁻¹, $F(000) = 230$, $R = 0.0844$, $wR = 0.2607$ for 1902 reflections with $I > 2\sigma(I)$.

Key Words: Thiosemicarbazone, Terephthalaldehyde, Single crystal, Dimethyl sulfoxide.

Thiosemicarbazones, which possess several donor atoms and can bind to a metal atom both in neutral as well as in anionic forms and have shown a number of bonding modes, has attracted intensive attentions due to its broad spectrum of pharmacological properties, including antitumor, antifungal, antibacterial, antiviral and antimalarial activities¹⁻⁴. Herein, we report a novel terephthalaldehyde *bis*-(thiosemicarbazone)-*bis*-(dimethyl sulfoxide) solvate single crystal synthesized with a formula $C_{10}H_{12}N_6S_2 \cdot 2C_2H_6OS$.

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 140 °C 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. The filtration residue was recrystallized using dimethyl sulfoxide/acetone (1:1). Single crystals suitable for X-ray analysis were grown from the dimethyl sulfoxide/ acetone solution 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-4 K diffractometer employing graphite-monochromated MoK α

radiation ($\lambda = 0.71073$ Å). 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_{obs}^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

$$R_1 = \Sigma(|F_o| - |F_c|) / \Sigma |F_o| = 0.0844 \quad (1)$$

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

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 P-1 space group. The structure of the terephthalaldehyde-*bis*-(thiosemi-carbazone)-*bis*-(dimethyl sulfoxide) solvate consists of the thiosemicarbazone and dimethyl sulfoxide. The average S-C bond distance in thiosemicarbazone

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

S(1A)-O(1A)	1.522(6)	S(1A)-C(6A)	1.769(9)	S(1A)-C(7A)	1.782(8)
S(2A)-C(1A)	1.700(7)	N(1A)-C(1A)	1.318(9)	N(2A)-N(3A)	1.365(8)
N(2A)-C(1A)	1.365(8)	N(3A)-C(2A)	1.278(9)	C(2A)-C(3A)	1.459(10)
C(3A)-C(4A)	1.391(10)	C(3A)-C(5N)	1.410(11)	C(4A)-C(5A)	1.379(10)
C(5A)-C(3N)	1.410(11)	O(1A)-S(1A)-C(6A)	106.2(4)	O(1A)-S(1A)-C(7A)	106.5(4)
C(6A)-S(1A)-C(7A)	97.8(4)	C(1A)-N(2A)-N(3A)	119.2(6)	C(1A)-N(3A)-N(2A)	116.6(6)
N(1A)-C(1A)-S(2A)	123.3(6)	N(1A)-C(1A)-N(2A)	117.3(6)	N(2A)-C(1A)-S(2A)	119.4(5)
N(3A)-C(2A)-C(3A)	122.1(7)	N(4A)-C(3A)-C(2A)	119.7(7)	N(4A)-C(3A)-C(5N)	119.0(7)
N(5N)-C(3A)-C(2A)	121.3(7)	N(5A)-C(4A)-C(3A)	121.3(7)	N(4A)-C(5A)-C(3N)	119.7(7)

moiety is 1.700 Å, while that in dimethyl sulfoxide is 1.769 Å. And the average C-N bond distance is 1.320 Å.

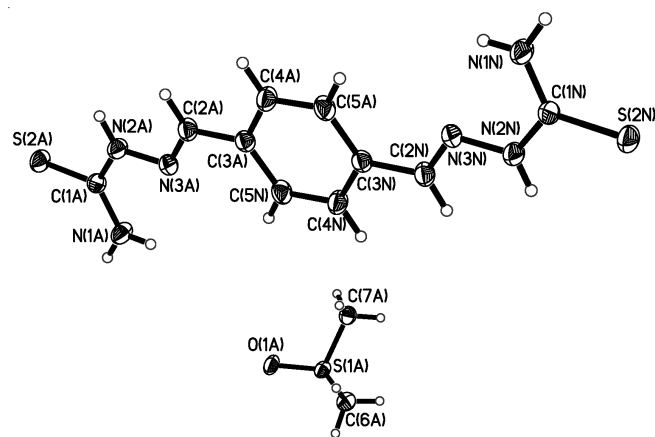


Fig. 1. Molecular structure of the terephthalaldehyde *bis*-(thiosemicarbazone)-*bis*-(dimethyl sulfoxide) solvate

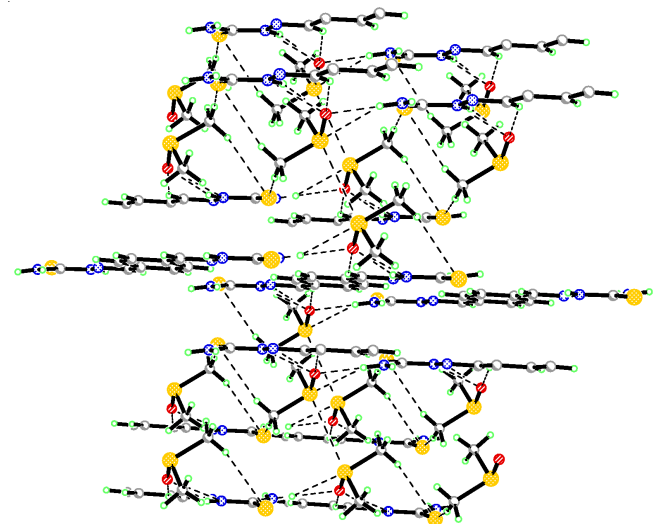


Fig. 2. View of a 3D supramolecular framework of the terephthalaldehyde *bis*-(thiosemicarbazone)-*bis*-(dimethyl sulfoxide) solvate

Conclusion

Crystal structure of a novel terephthalaldehyde *bis*-(thiosemicarbazone) *bis*-(dimethyl sulfoxide) solvate has been synthesized and characterized by elemental analysis and X-ray diffraction analysis.

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