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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), α = 77.348(8)º, β = 78.672(8)º, γ = 79.193(7)º, V = 527.64(8) Å^3, Z = 1, Mr = 436.63, Dc = 1.374 g/cm^3, μ = 0.471 mm^{-1}, F(000) = 230, R = 0.0844, wR = 0.2607 for 1902 reflections with I > 2σ (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 activities[1-4]. 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 ºC 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$_\alpha$ 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

$$R_1 = \Sigma(||F_0| - |F_C||)/ S |F_0| = 0.0844 \quad (1)$$

$$\text{and } wR_2 = \{S [w(F_0^2 - F_C^2)^2]/ Sw(F_0^2)^2\}^{1/2} = 0.2607 \quad (2)$$

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 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 (º)

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.

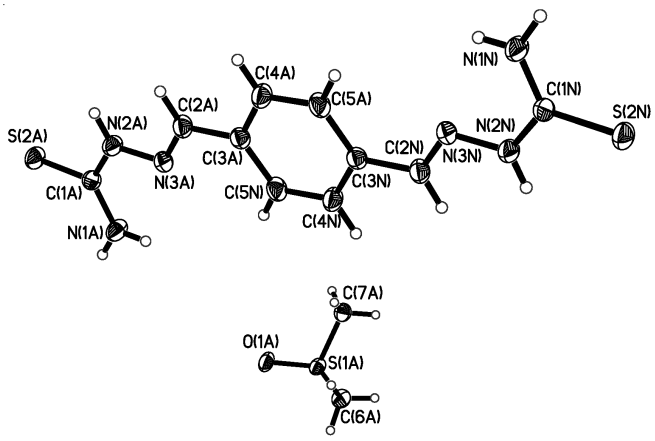

Fig. 1. Molecular structure of the terephthalaldehyde *bis*-(thiosemicarbazone)-*bis*-(dimethyl sulfoxide) solvate

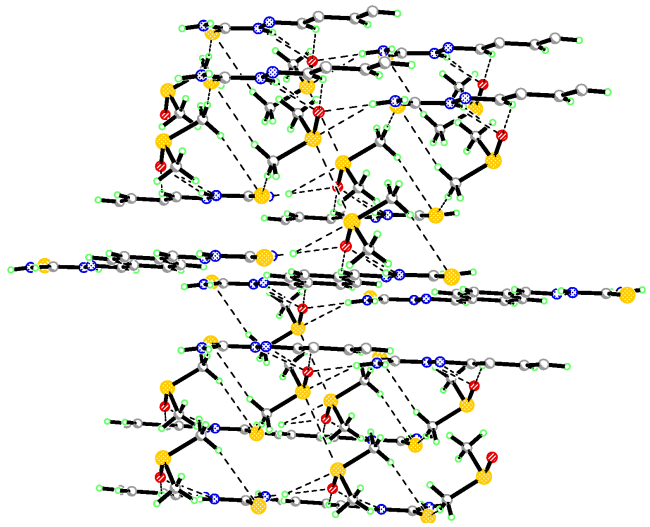

Fig. 2. View of a 3D supramolecular framework of the terephthalaldehyde *bis*-(thiosemi-carbazone)-*bis*-(dimethyl sulfoxide) solvate

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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