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# 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) Å, b = 7.0361(5) Å, c = 8.5416(6) Å, α = 90.00º, β = 103.821(9)º, γ = 90.00º, V = 660.74(10)Å3, Z = 2, $M_r$ = 268.34, $D_c$ = 1.349 g/cm$^3$, μ = 0.236 mm$^{-1}$, $F_{(000)}$ = 280, R = 0.0390, wR = 0.1016 for 2130 reflections with I > 2σ(I).

**Key Words: Thiosemicarbazone, Terephthalaldehyde, Single crystal.**

Recently, increasing attention has been given to thiosemicarbazones for its broad spectrum of pharmacological properties[1-3]. 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 compoud, 0.134 g 3-phthalaldehyde (1 mmol) and 0.324 g (2 mmol) 4-phenyl-thiosemicarbazide 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 $MoK_\alpha$ 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

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

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

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 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) |

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 $w = 1/[s^2(F_0^2) + (0.1014P)^2]$ 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 structure of the N$^2$,N$^5$-*bis*(phenyl)-1,3,4-thiadiazole-2,5-diamine. The average S-C bond distance in thiosemicarbazone moiety is 1.739 Å.

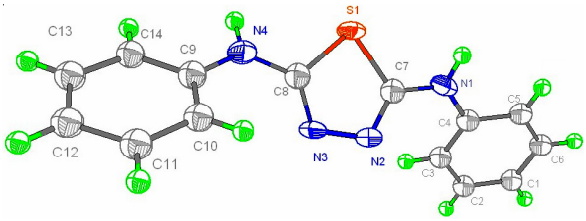


Fig. 1. Molecular structure of the N$^2$,N$^5$-*bis*(phenyl)-1,3,4-thiadiazole-2,5-diamine

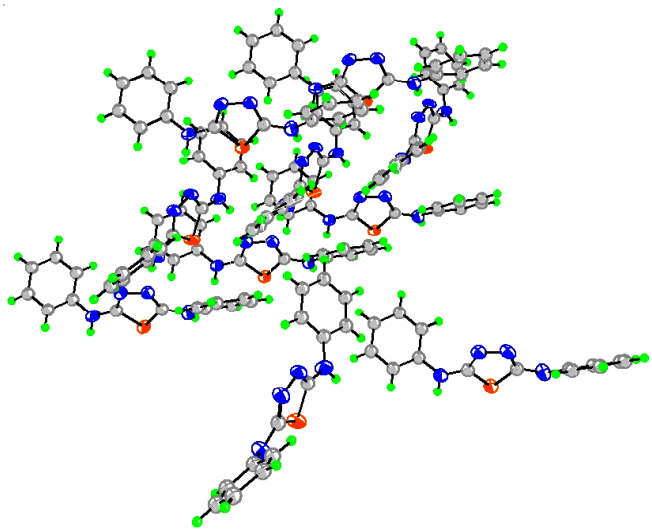

Fig. 2. View of a 3D supramolecular framework of the N$^2$,N$^5$-*bis*(phenyl)-1,3,4-thiadiazole-2,5-diamine

## Conclusion

Crystal structure of a novel N$^2$,N$^5$-*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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