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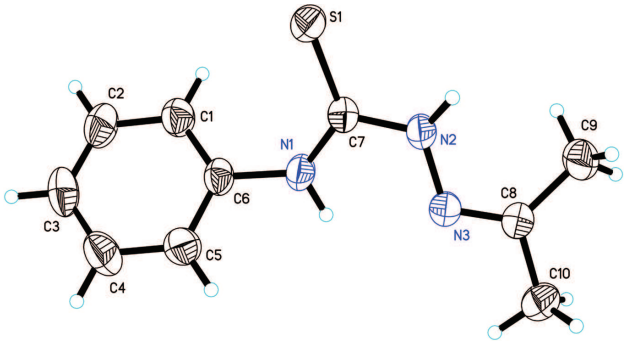
Crystal structure of *N*-phenyl-2-(propan-2-ylidene)hydrazine-1-carbothioamide, $C_{10}H_{13}N_3S$



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Abstract

$C_{10}H_{13}N_3S$, monoclinic, $P2_1/c$ (no. 14), $a = 12.2463(8)$ Å, $b = 7.6397(5)$ Å, $c = 11.6544(9)$ Å, $\beta = 102.684(2)°$, $V = 1060.72(11)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0448$, $wR_{ref}(F^2) = 0.1211$, $T = 301(2)$ K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions

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Table 1: Data collection and handling.

Crystal:	Size 0.60 × 0.40 × 0.32 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.27 mm^{-1}
Diffractometer, scan mode:	Bruker SMART, φ and ω-scans
θ_{max}, completeness:	28.4°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	37336, 2646, 0.039
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2126
$N(param)_{refined}$:	138
Programs:	Bruker programs [1], SHELX [2], PLATON [3]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
S1	0.16731(4)	0.89118(7)	0.53541(4)	0.04377(18)
N1	0.20128(12)	0.8324(2)	0.31739(13)	0.0407(4)
N2	0.02253(11)	0.8844(2)	0.33278(13)	0.0377(3)
N3	−0.00346(12)	0.8913(2)	0.21013(13)	0.0385(3)
C1	0.38114(15)	0.7416(3)	0.44260(18)	0.0452(4)
H1	0.3443	0.6781	0.4909	0.054*
C2	0.49744(16)	0.7471(3)	0.46641(19)	0.0531(5)
H2	0.5385	0.6882	0.5317	0.064*
C3	0.55231(16)	0.8385(3)	0.3947(2)	0.0540(5)
H3	0.6302	0.8420	0.4116	0.065*
C4	0.49191(17)	0.9249(3)	0.2977(2)	0.0570(6)
H4	0.5290	0.9863	0.2487	0.068*
C5	0.37579(16)	0.9206(3)	0.27298(18)	0.0477(5)
H5	0.3350	0.9777	0.2068	0.057*
C6	0.32076(13)	0.8312(2)	0.34670(15)	0.0360(4)
C7	0.13079(13)	0.8690(2)	0.38853(15)	0.0333(4)
C8	−0.10561(14)	0.8704(2)	0.15745(15)	0.0348(4)
C9	−0.20087(16)	0.8323(3)	0.21445(18)	0.0545(5)
H9A	−0.1736	0.7713	0.2872	0.082*
H9B	−0.2550	0.7606	0.1631	0.082*
H9C	−0.2354	0.9402	0.2298	0.082*
C10	−0.13165(17)	0.8822(3)	0.02644(16)	0.0488(5)
H10A	−0.1743	0.9865	0.0020	0.073*
H10B	−0.1744	0.7815	−0.0062	0.073*
H10C	−0.0630	0.8860	−0.0008	0.073*
H1A	0.1695(16)	0.837(3)	0.2429(9)	0.047(6)*
H2A	−0.0235(15)	0.924(3)	0.3739(16)	0.050(6)*

and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

A mixture of (1.36 g, 0.01 mol) of diethylcarbamoyl chloride and (0.76 g, 0.01 mol) of ammonium thiocyanate in 40 ml acetone was stirred and heated to reflux for 3 h [4]. The mixture was cooled, and then filtered off. A solution of 4-phenyl-3-thiosemicarbazide (1.67 g, 0.01 mol) in 20 ml of acetone was added to the filtrate. The reaction mixture was heated to reflux for 2 h, and then allowed to cool at room temperature to give the title compound as colorless crystals. Yield: 46%, mp.: 197–199 °C.

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions (SHELX System [2]) and constrained to ride on their parent atoms.

Discussion

The formation of Schiff bases, which are a remarkable species, has played a vital role in the development of organic and coordination chemistry [5, 6]. This is due to their unique properties and their wide range of applications [7–13].

N-phenyl-2-(propan-2-ylidene)hydrazine-1-carbothioamide, $C_{10}H_{13}N_3S$, crystalize as a monoclinic crystal system with space group of $P2_1/c$. The X-ray single-crystal investigation showed that, the formation of Schiff base has been achieved upon the reaction of the primary amine group of thiosemicarbazide with the carbonyl group of acetone solvent. The molecule of the compound *N*-phenyl-2-(propan-2-ylidene)hydrazine-1-carbothioamide is discrete and the asymmetric unit consist of one molecule (*cf*. the figure)). The central thiourea moiety [S1/N1/N2/C7] is essentially planar with a maximum deviation of 0.005(2) Å for atom C7 from least-squares plane. The mean plane of the central of thiourea moiety makes a dihedral angle of 43.98(12)° with the mean plane phenyl ring (C1—C6). The bond length of azomethine group C8=N3 is 1.275(2) Å. Analysis data indicated all bond lengths and angles of the molecule are unexceptional [14, 15].

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References

1. Bruker: SMART, SAINT and SADABS. Bruker AXS Inc., Madison, WI, USA (2009).
2. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112–122.
3. Spek, A. L.: Structure validation in chemical crystallography. Acta Crystallogr. **D65** (2009) 148–155.
4. Hussien, N. J.; Yusoff, S. F. M.; El-Hiti, G. A.; Farina, Y.; Al-Jeboori, M. J.; Yousif, E.: Crystal structure of 1-(4,4-dimethyl-2,6-dithioxo-1,3,5-triazinan-1-yl)-3-(diethylaminocarbonyl)thiourea, $C_{11}H_{20}N_6OS_3$. Z. Kristallogr. **232** (2017) 325–326.
5. Yinghua, J.; Youlong, Z.; Wei, Z.: Development of organic porous materials through Schiff-base chemistry. CrystEngComm. **15** (2013) 1484–1499.
6. Shiu, K. B.; Liu, S. A.; Lee, G. H.: One-pot conditional self-assembly of multicopper metallacycles. Inorg. Chem. **49** (2010) 9902–9908.
7. Enis, N. M. Y.; Thahira, B. S. A. R.; Edward, R. T. T.; Abhimanyu, V.; Karen, A. C.; Mohamed, I. M. T.; Haslina, A.: Synthesis, characterization and biological evaluation of transition metal complexes derived from N, S bidentate ligands. Int. J. Mol. Sci. **16** (2015) 11034–11054.
8. Gomathi, V.; Selvameena, R.: Synthesis, characterisation and biological studies of complexes of 3D transition metals and with Schiff base derived from sulfadiazine and 2-acetylnaphthalene. Int. J. Recent Sci. Res. **4** (2013) 94–97.
9. Shivakumar, K.; Shashidhar, P.; Vithal, R. P.; Halli, M. B.: Synthesis, spectral characterization and biological activity of benzofuran Schiff bases with Co(II), Ni(II), Cu(II), Zn(II), Cd(II) and Hg(II) complexes. J. Coord. Chem. **61** (2008) 2274–2287.
10. Hunashal, R. D.; Satyanarayana, D.: One pot synthesis of 3-(substituted phenoxymethyl)-6-phenyl/substituted phenoxymethyl-1,2,4-triazolo[3,4-b][1, 3, 4] thiadiazole derivatives as antimicrobial agents. Int. J. Pharm. Biol. Sci. **3** (2012) 183–192.
11. Tarafder, M. T. H.; Ali, M. A.; Wee, D. J.; Azahari, K.; Silong, S.; Crowse, K. A.: Complexes of a tridentate ONS Schiff base. Synthesis and biological properties. Transit. Metal Chem. **25** (2000) 456–460.
12. Dharamraj, N.; Viswanathanmurthi, P.; Natarajan, K.: Ruthenium (II) complexes containing bidentate Schiff bases and their antifungal activity. Transit. Metal Chem. **26** (2001) 105–109.
13. Ejiah, F. N.; Fasina, T. M.; Familoni, O. B.; Ogunsola, F. T.: Substituent effect on spectral and antimicrobial activity of Schiff bases derived from aminobenzoic acids. Adv. Biol. Chem. **3** (2013) 475–479.
14. Liu, C.: Crystal structure of (*E*)-2-(anthracen-9-ylmethylene) hydrazine-1-carbothioamide, $C_{16}H_{13}N_3S$. Z. Kristallogr. NCS **233** (2018) 315–316.
15. Oliveira, A. B.; Beck, J.; Landvogt, C.; Feitosa, B. R. S.; Rocha, F. V.: Crystal structure of (*E*)-2-[4-(4-hydroxyphenyl)butan-2-ylidene]hydrazine-1-carbothioamide. Acta Crystallogr. E **71** (2015), o33–o34.