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The crystal structure of *catena*-poly[(*E*)-2-(((5-((trimethylstannyl)thio)-1,3,4-thiadiazol-2-yl)imino)methyl)phenol], C₁₂H₁₅N₃OS₂Sn

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Abstract

C₁₂H₁₅N₃OS₂Sn, orthorhombic, *Pbca* (no. 61), $a = 13.101(6)$ Å, $b = 11.151(5)$ Å, $c = 20.855(10)$ Å, $V = 3047(2)$ Å³, $Z = 8$, $R_{\text{gt}}(F) = 0.0440$, $wR_{\text{ref}}(F^2) = 0.1560$, $T = 298(2)$ K.

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The crystal structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of materials

The title Schiff base ligand compound was synthesized by alicylaldehyde and 5-amino-1,3,4-thiadiazole-2-thiol in the refluxing in ethanol for 3 h. In this process, the orange yellow solid was precipitated and obtained by hot filtration. The solid powder was washed three times by ethanol. The yield is 81%. The title complex was obtained by the title ligand and trimethyltin chloride, in the presence of sodium ethoxide, as the dehydrogenation reagent, on heating for 3 h at 313 K. The mixture was filtered and the filtration was distilled. The product was recrystallized in hexane/dichloromethane (2/1). The colorless block crystals were obtained by evaporation in air. **Yield:** 73%.

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Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.33 × 0.29 × 0.27 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	1.95 mm ⁻¹
Diffractometer, scan mode:	Bruker SMART, φ and ω -scans
θ_{max} , completeness:	25°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	14581, 2685, 0.076
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1675
$N(\text{param})_{\text{refined}}$:	175
Programs:	Bruker programs [1], SHELX [2]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Sn1	0.06778(5)	1.06795(5)	0.26056(3)	0.0438(3)
N1	0.0516(6)	0.7902(6)	0.3248(4)	0.0480(19)
N2	0.0320(5)	0.6706(6)	0.3408(3)	0.0424(17)
N3	0.0745(6)	0.5207(6)	0.4151(4)	0.0462(18)
O1	−0.0027(6)	0.3007(7)	0.4230(4)	0.092(3)
H1	0.003703	0.369345	0.409436	0.138*
S1	0.16580(19)	0.7434(2)	0.42053(12)	0.0525(7)
S2	0.1605(2)	0.9854(2)	0.35983(13)	0.0587(7)
C1	0.1188(6)	0.8403(7)	0.3636(4)	0.040(2)
C2	0.0848(6)	0.6357(7)	0.3902(4)	0.039(2)
C3	0.1296(8)	0.4904(8)	0.4640(5)	0.054(2)
H3	0.173776	0.547572	0.480896	0.065*
C4	0.1270(8)	0.3738(9)	0.4941(5)	0.058(3)
C5	0.0631(9)	0.2830(10)	0.4728(5)	0.065(3)
C6	0.0671(11)	0.1719(10)	0.5022(7)	0.089(4)
H6	0.024759	0.110651	0.487744	0.107*
C7	0.1307(11)	0.1512(12)	0.5511(6)	0.084(4)
H7	0.132958	0.075471	0.569750	0.101*
C8	0.1920(11)	0.2395(14)	0.5739(6)	0.089(4)
H8	0.234260	0.224563	0.608797	0.106*
C9	0.1921(9)	0.3526(10)	0.5452(5)	0.071(3)
H9	0.235204	0.412737	0.560149	0.086*
C10	0.1246(8)	0.9441(8)	0.1928(5)	0.060(3)
H10A	0.128829	0.981869	0.151556	0.090*
H10B	0.079502	0.876365	0.190392	0.090*
H10C	0.191227	0.917706	0.205752	0.090*
C11	0.1463(7)	1.2330(8)	0.2661(5)	0.054(2)
H11A	0.218129	1.219432	0.260740	0.082*

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
H11B	0.134115	1.269110	0.307212	0.082*
H11C	0.122287	1.285617	0.232931	0.082*
C12	−0.0838(7)	1.0622(9)	0.2946(5)	0.062(3)
H12A	−0.098426	1.134451	0.317886	0.093*
H12B	−0.092033	0.994315	0.322390	0.093*
H12C	−0.129785	1.055329	0.258955	0.093*

Experimental details

All hydrogen atomic positions were taken from a difference Fourier map. Their *U*_{iso} values were assigned with common isotropic displacement factors *U*_{iso}(H) = 1.2 times *U*_{eq} (C, benzene ring and methylene) and 1.5 times *U*_{eq} (C, methyl and O, phenolic hydroxyl).

Comment

Organotin(IV) complexes have been causing more and more attention for their industrial applications and potential biological activities [3]. Especially many varieties of organotin(IV) complexes with heteroatoms groups have been synthesized and studied [4, 5]. Moreover, they have received much attention both in academic and applied research because of the ability of tin to afford stable bonds [6–11].

In an asymmetric unit of the title structure, the Sn atom centre exhibits a four-coordinated seriously distorted tetrahedral geometry with three methyl C and one S atom. The bond length of S–Sn is 2.571(3) Å, which is a little bit longer than the covalent radii of tin and sulfur atoms 2.42 Å. The distance of N···Sn is 2.736 Å, which is between the sum of the van der Waals and covalent radii of Sn and N (3.75 and 2.15 Å and can be considered as a weak coordination bond. Thus one-dimensional polymer chains are present. Taking the weak Sn–N coordination into account (see the figure), the Sn center shows a trigonal bipyramidal geometry. The three C atoms from three methyl groups occupy the equatorial plane and S and N atoms are in an apical position. The bond angle of S–Sn–N is 176.03°, showing the distortion. Importantly, there is an intramolecular OH···N hydrogen bond (see the figure) and other weak non-classical hydrogen bonds. Bond lengths and angles are all in the expected ranges [11–13].

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