Junshan Sun*

Crystal structure of (*E*)-1-(thiophen-2-yl)-*N*-(5-((triphenylstannyl)thio)-1,3,4-thiadiazol-2-yl)methanimine, C₂₅H₁₉N₃S₃Sn

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Abstract

C₂₅H₁₉N₃S₃Sn, monoclinic, *P*₂₁/*n* (no. 14), *a* = 14.347(4) Å, *b* = 9.908(3) Å, *c* = 17.834(5) Å, *V* = 2405.6(11) Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0287, *wR*_{ref}(*F*²) = 0.0777, *T* = 298(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 and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

The thio-ligand was prepared by thiophene-2-carbaldehyde and 5-amino-1,3,4-thiadiazole-2-thiol in the refluxing in 80 °C in solvent of ethanol for 2 h. The yellow precipitation was obtained by filtering. The title complex was synthesized by the thio-ligand and triphenyltin chloride, in the presence of sodium ethoxide, as the dehydrogenation reagent, heating for 4 h at 313 K. The mixture was filtered and the filtration was distilled by rotary evaporator. The product was recrystallized in hexane/dichloromethane with the ratio 1:1. The colorless block crystals were obtained by evaporation in air.

*Corresponding author: Junshan Sun, College of Chemistry and Chemical Engineering, Taishan University, Taian 271000, China, e-mail: sunjunshan79@163.com

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.68 × 0.52 × 0.49 mm
Wavelength:	Mo Kα radiation (0.71073 Å)
μ:	1.34 mm ⁻¹
Diffractometer, scan mode:	APEX2, φ and ω
θ _{max} , completeness:	25.0°, >99%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	12136, 4237, 0.041
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 3180
<i>N</i> (<i>param</i>) _{refined} :	289
Programs:	Bruker [1], SHELX [2]

Yield: 83% (based on Sn), and elemental analysis: calc. for C₂₅H₁₉N₃S₃Sn: C 52.10, H 3.32, N 7.29%; found: C 52.31, H 3.24, N 7.15%. The elemental analyses were performed with PERKIN ELMER MODEL 2400 SERIES II.

Experimental details

All hydrogen atomic positions were taken from a difference Fourier map. Hydrogen atoms were assigned with common isotropic displacement factors *U*_{iso}(H) = 1.2 times *U*_{eq}(C, phenyl ring and thiophene ring). All the H atoms were refined as riding on their parent atom.

Comment

Organotin(IV) complexes, the bioactive organometallics, have gained widely attention due to their structural diversity and effective anticancer activity [3–5]. Especially many varieties of organotin(IV) complexes with the heteroatoms of N, O and S are under investigation [6–8]. The heterocyclic thiones that contain at least one deprotonated heterocyclic thioamide group (N–C–S) have been subject for their versatile coordination modes and biological activities. Schiff base derived from various heterocycles were reported to possess biological activity [9]. So we are interested to assemble the Schiff base group and the heterocyclic thioamide group (N–C–S) to study the properties of a corresponding organotin(IV) complex.

The aforementioned thio-ligand was reacted with the triphenyltin chloride to give the title compound (see the figure). The bond length of S–Sn 2.4633(11) Å, which is close

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

Atom	x	y	z	U _{iso} */U _{eq}
Sn1	1.19065(2)	1.43448(2)	0.49628(2)	0.04420(10)
S1	1.54083(7)	1.34028(10)	0.57363(6)	0.0570(3)
S2	1.32957(7)	1.32324(11)	0.46818(6)	0.0623(3)
S3	1.84360(8)	1.52684(12)	0.84447(7)	0.0698(3)
N1	1.4027(2)	1.4777(3)	0.5971(2)	0.0541(8)
N2	1.4853(2)	1.5142(3)	0.65729(19)	0.0561(8)
N3	1.6518(2)	1.4678(3)	0.71334(19)	0.0568(8)
C1	1.4191(3)	1.3891(4)	0.5488(2)	0.0505(9)
C2	1.5627(3)	1.4492(4)	0.6549(2)	0.0520(9)
C3	1.7213(3)	1.3843(4)	0.7196(2)	0.0593(10)
H3	1.710315	1.313070	0.683939	0.071*
C4	1.8151(3)	1.3945(4)	0.7786(2)	0.0517(9)
C5	1.8925(3)	1.3085(4)	0.7921(2)	0.0586(10)
H5	1.891152	1.230356	0.762946	0.070*
C6	1.9736(3)	1.3492(5)	0.8537(2)	0.0643(11)
H6	2.032314	1.301361	0.870450	0.077*
C7	1.9582(3)	1.4649(5)	0.8866(2)	0.0678(12)
H7	2.005502	1.506906	0.928100	0.081*
C8	1.1875(2)	1.6483(4)	0.47801(19)	0.0435(8)
C9	1.2547(3)	1.7355(4)	0.5253(2)	0.0569(10)
H9	1.306351	1.701585	0.566858	0.068*
C10	1.2472(3)	1.8721(4)	0.5124(3)	0.0692(12)
H10	1.293617	1.929531	0.545208	0.083*
C11	1.1720(3)	1.9245(4)	0.4516(3)	0.0650(11)
H11	1.167569	2.017125	0.442771	0.078*
C12	1.1036(3)	1.8401(4)	0.4043(3)	0.0668(11)
H12	1.051776	1.875278	0.363343	0.080*
C13	1.1108(3)	1.7017(4)	0.4169(2)	0.0582(10)
H13	1.063994	1.644566	0.384114	0.070*
C14	1.0765(2)	1.3368(3)	0.40645(19)	0.0422(8)
C15	1.0647(3)	1.3528(4)	0.3265(2)	0.0579(10)
H15	1.106954	1.409494	0.310977	0.069*
C16	0.9913(3)	1.2858(4)	0.2699(2)	0.0651(11)
H16	0.984038	1.298456	0.216690	0.078*
C17	0.9292(3)	1.2008(4)	0.2915(2)	0.0613(11)
H17	0.880363	1.154679	0.253119	0.074*
C18	0.9392(3)	1.1842(4)	0.3694(2)	0.0614(10)
H18	0.896713	1.127050	0.384355	0.074*
C19	1.0121(3)	1.2518(4)	0.4262(2)	0.0517(9)
H19	1.017900	1.239642	0.479214	0.062*
C20	1.1837(2)	1.3584(4)	0.60551(19)	0.0423(8)
C21	1.1569(3)	1.4369(4)	0.6591(2)	0.0552(10)
H21	1.148131	1.529217	0.650398	0.066*
C22	1.1429(3)	1.3806(5)	0.7255(2)	0.0715(12)
H22	1.124899	1.434927	0.761169	0.086*
C23	1.1553(3)	1.2456(5)	0.7387(2)	0.0755(13)
H23	1.144914	1.207512	0.783082	0.091*
C24	1.1829(3)	1.1659(5)	0.6870(3)	0.0759(13)
H24	1.191897	1.073723	0.696256	0.091*
C25	1.1973(3)	1.2225(4)	0.6212(2)	0.0610(10)
H25	1.216663	1.167667	0.586433	0.073*

to the covalent radii of tin and sulfur atoms (2.42 Å) [10]. The environment of Sn atom is a distorted tetrahedron. The angles of C—Sn—S and C—Sn—C are from 97.07(9) to 119.10(13)°,

showing architecture of the Sn atom with surrounding atoms is seriously distorted from the tetrahedron geometry.

Geometric parameters in the thio-ligand are as expected [11, 12].

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