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Crystal structure of catena-poly[(1-(4-fluorophenyl)-*N*–(5-((trimethylstannyl)thio)-1,3,4-thiadiazol-2-yl)methanimine], $(C_{12}H_{14}FN_3S_2Sn)_n$

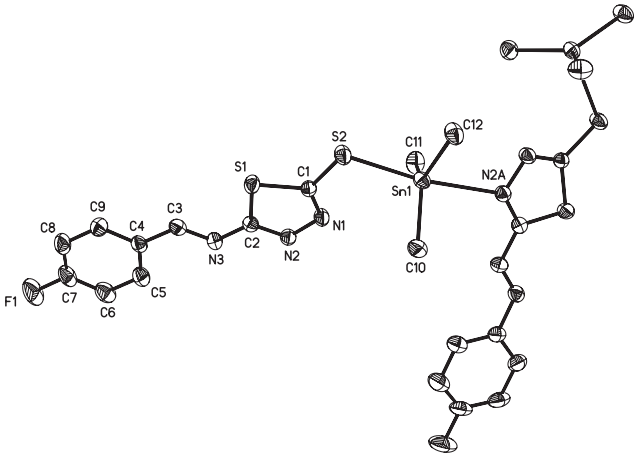

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.24 × 0.20 × 0.17 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	1.92 mm^{-1}
Diffractometer, scan mode:	Bruker SMART, φ and ω-scans
θ_{max}, completeness:	25°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	13303, 2730, 0.036
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2017
$N(param)_{refined}$:	172
Programs:	Bruker programs [1], SHELX [2]

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Abstract

$C_{12}H_{14}FN_3S_2Sn$, orthorhombic, *Pbca* (no. 61), $a = 12.954(3)$ Å, $b = 11.494(3)$ Å, $c = 20.825(5)$ Å, $V = 3100.7(13)$ Å^3, $Z = 8$, $R_{gt}(F) = 0.0274$, $wR_{ref}(F^2) = 0.0658$, $T = 298(2)$ K.

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The crystal structure is shown in the figure (The asymmetric unit is labelled plus the symmetry related N2a atom; a = $-x$, $-0.5 + y$, $0.5 - z$). 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 materials

The ligand was prepared by 4-fluorobenzaldehyde and 5-amino-1,3,4-thiadiazole-2-thiol in the refluxing in solvent of ethanol for 2 h. The yellow precipitation was obtained by the filtering. The title complex was synthesized by reacting the aforementioned ligand and trimethyltin chloride, in the presence of sodium ethoxide, as the dehydrogenation reagent, while heating for 4 h at 313 K. The mixture was filtered and

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Sn1	0.06879(2)	0.44181(2)	0.24525(2)	0.04387(10)
C10	0.1255(4)	0.5618(3)	0.31421(19)	0.0621(11)
H10A	0.195837	0.543236	0.324377	0.093*
H10B	0.121937	0.639195	0.297015	0.093*
H10C	0.084231	0.557255	0.352435	0.093*
S1	0.16267(8)	0.75847(8)	0.08153(5)	0.0506(3)
S2	0.16415(9)	0.52602(8)	0.14606(5)	0.0559(3)
N3	0.0709(2)	0.9768(2)	0.09061(14)	0.0451(7)
C2	0.0814(3)	0.8639(3)	0.11276(17)	0.0411(8)
N1	0.0505(2)	0.7144(2)	0.17868(14)	0.0480(8)
N2	0.0293(2)	0.8285(2)	0.16243(14)	0.0447(7)
C1	0.1175(3)	0.6669(3)	0.14093(16)	0.0410(8)
C11	−0.0837(3)	0.4526(4)	0.2100(2)	0.0664(12)
H11A	−0.095235	0.391449	0.179389	0.100*
H11B	−0.131473	0.444652	0.244936	0.100*
H11C	−0.093768	0.526592	0.189516	0.100*
C12	0.1509(3)	0.2830(3)	0.2351(2)	0.0611(11)
H12A	0.218592	0.291225	0.253209	0.092*
H12B	0.114463	0.222068	0.257005	0.092*
H12C	0.156666	0.263832	0.190325	0.092*
F1	0.1213(3)	1.4589(2)	−0.05275(16)	0.1110(11)
C3	0.1212(3)	1.0087(3)	0.04115(18)	0.0481(9)
H3	0.162038	0.954011	0.020228	0.058*
C4	0.1179(3)	1.1263(3)	0.01579(18)	0.0483(10)
C7	0.1195(4)	1.3486(4)	−0.0306(2)	0.0726(14)

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
C5	0.0610(3)	1.2126(3)	0.0462(2)	0.0607(11)
H5	0.022120	1.194576	0.082389	0.073*
C9	0.1745(4)	1.1541(4)	−0.03873(19)	0.0632(12)
H9	0.212256	1.096691	−0.059667	0.076*
C6	0.0619(4)	1.3252(4)	0.0228(2)	0.0754(14)
H6	0.024152	1.383544	0.042871	0.090*
C8	0.1748(4)	1.2668(4)	−0.0620(2)	0.0728(14)
H8	0.212346	1.285897	−0.098606	0.087*

the filtration was distilled. The product was recrystallized in hexane/dichloromethane with a ratio of 2:1. The colorless block crystals were obtained by evaporation. Yield: 69% (based on Sn element), and elemental analysis: calc. for $C_{12}H_{14}FN_3S_2Sn$: C 35.85, H 3.51, N 10.45; found: C 35.61, H 3.29, N 10.13. PERKIN ELMER MODEL 2400 SERIES II.

Experimental details

Hydrogen atoms were assigned with isotropic displacement factors $U_{iso}(H) = 1.5$ times U_{eq} (C, methyl) and $U_{iso}(H) = 1.2$ times U_{eq} (C, methyne and benzene ring). All the H atoms were refined as riding on their parent atom.

Comment

Organotin(IV) complexes have been causing more and more attention for their industrial applications and potential biological activities [3–5]. Especially many varieties of organotin(IV) complexes have been synthesized and studied in the context of their antitumour potential [6]. Heterocyclic thiones that contain at least one deprotonated heterocyclic thioamide group (N—C—S) have been studied for their versatile coordination modes and biological activities [7]. Also, Schiff base derived from various heterocycles were reported to possess cytotoxic, anticonvulsant, antiproliferative, anticancer and antifungal activities. So we are interesting to assemble the Schiff base group and the heterocyclic thioamide (N—C—S) to study the property of organotin(IV) complexes.

The title complex is shown in the figure. Each tin(IV) is coordinated by three methyl groups and two nitrogen atoms from two crystallographically dependent organic ligands. Thus the coordination is trigonal bipyramidal. The bond length of S-Sn 2.5942(11) Å, is a little bit longer than the sum of the covalent radii of tin and sulfur (2.42 Å). The Sn atom is 0.15 Å shifted out of the plane, formed by the three C methyl atoms (C10, C11, C12). All bond lengths are in the expected ranges.

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