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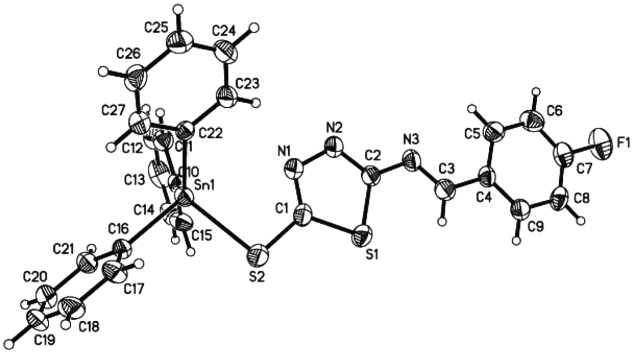
Crystal structure of 1-(4-fluorophenyl)-*N*-(5-((triphenylstannyl)thio)thiophen-2-yl) methanimine, $C_{27}H_{20}FN_3S_2Sn$



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Abstract

$C_{27}H_{20}FN_3S_2Sn$, monoclinic, $P2_1/n$ (no. 14), $a = 15.631(4)$ Å, $b = 9.865(3)$ Å, $c = 17.256(3)$ Å $\beta = 108.335(11)°$, $V = 2525.8(10)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0398$, $wR_{ref}(F^2) = 0.0917$, $T = 298$ 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 materials

The ligand: 5-((4-fluorobenzylidene)amino)thiophene-2-thiol was synthesized by 4-fluorobenzaldehyde and 5-aminothiophene-2-thiol by refluxing in ethanol for 2 h (yellow solid). The organic ligand and triphenyltin chloride were added to benzene and sodium ethoxide, refluxed for

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Table 1: Data collection and handling.

Crystal:	Block, colorless
Size:	0.53 × 0.40 × 0.22 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	1.21 mm^{-1}
Diffractometer, scan mode:	Bruker SMART, φ and ω-scans
$2\theta_{max}$, completeness:	25.1°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	12919, 4492, 0.050
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2906
$N(param)_{refined}$:	307
Programs:	Bruker programs [1], SHELX [2–4]

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

Atom	x	y	z	U_{iso}*/U_{eq}
Sn1	0.82025(2)	0.05801(3)	1.01054(2)	0.04710(13)
F1	−0.0347(2)	0.0918(4)	0.5827(2)	0.1067(12)
N1	0.6184(3)	0.0176(4)	0.9125(3)	0.0567(11)
N2	0.5385(3)	−0.0112(4)	0.8507(3)	0.0587(11)
N3	0.3837(3)	0.0447(4)	0.7970(3)	0.0579(11)
S1	0.49583(9)	0.16322(14)	0.94241(9)	0.0625(4)
S2	0.69500(9)	0.16156(15)	1.04966(9)	0.0673(4)
C1	0.6069(3)	0.1040(5)	0.9647(3)	0.0538(13)
C2	0.4691(3)	0.0567(5)	0.8562(3)	0.0526(12)
C3	0.3178(4)	0.1190(5)	0.8003(3)	0.0635(15)
H3	0.3286	0.1817	0.8425	0.076*
C4	0.2262(3)	0.1117(5)	0.7417(3)	0.0526(13)
C5	0.2038(4)	0.0220(5)	0.6771(4)	0.0681(16)
H5	0.2479	−0.0352	0.6697	0.082*
C6	0.1168(4)	0.0151(6)	0.6228(4)	0.0760(17)
H6	0.1024	−0.0455	0.5793	0.091*
C7	0.0524(4)	0.1001(6)	0.6350(4)	0.0688(16)
C8	0.0703(4)	0.1903(6)	0.6965(4)	0.0650(15)
H8	0.0258	0.2473	0.7032	0.078*
C9	0.1588(4)	0.1951(5)	0.7499(3)	0.0635(15)
H9	0.1726	0.2570	0.7927	0.076*
C10	0.8191(3)	0.1434(5)	0.8970(3)	0.0452(12)
C11	0.8398(3)	0.0702(5)	0.8372(3)	0.0578(13)
H11	0.8486	−0.0229	0.8435	0.069*
C12	0.8476(4)	0.1322(7)	0.7686(4)	0.0746(17)
H12	0.8623	0.0810	0.7293	0.090*
C13	0.8342(4)	0.2671(8)	0.7578(4)	0.0793(19)
H13	0.8394	0.3091	0.7112	0.095*
C14	0.8130(4)	0.3405(6)	0.8158(4)	0.0782(18)
H14	0.8036	0.4334	0.8086	0.094*

Table 2 (continued)

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C15	0.8051(4)	0.2804(5)	0.8847(3)	0.0625(15)
H15	0.7902	0.3325	0.9235	0.075*
C16	0.9315(3)	0.1498(5)	1.1032(3)	0.0464(12)
C17	0.9495(4)	0.1265(5)	1.1854(3)	0.0593(14)
H17	0.9127	0.0680	1.2031	0.071*
C18	1.0221(4)	0.1893(6)	1.2425(3)	0.0713(16)
H18	1.0335	0.1714	1.2978	0.086*
C19	1.0768(4)	0.2768(6)	1.2187(4)	0.0719(17)
H19	1.1249	0.3184	1.2574	0.086*
C20	1.0603(4)	0.3027(5)	1.1375(4)	0.0691(16)
H20	1.0969	0.3623	1.1205	0.083*
C21	0.9887(3)	0.2395(5)	1.0808(3)	0.0549(13)
H21	0.9782	0.2573	1.0257	0.066*
C22	0.8271(3)	−0.1574(4)	1.0250(3)	0.0389(11)
C23	0.7565(3)	−0.2417(5)	0.9883(3)	0.0580(14)
H23	0.7022	−0.2062	0.9551	0.070*
C24	0.7664(4)	−0.3796(5)	1.0008(4)	0.0716(16)
H24	0.7190	−0.4378	0.9754	0.086*
C25	0.8474(4)	−0.4313(5)	1.0517(4)	0.0692(15)
H25	0.8539	−0.5244	1.0602	0.083*
C26	0.9184(4)	−0.3465(6)	1.0897(3)	0.0642(15)
H26	0.9724	−0.3817	1.1236	0.077*
C27	0.9080(3)	−0.2097(5)	1.0767(3)	0.0558(13)
H27	0.9551	−0.1512	1.1024	0.067*

2 h and filtered off. The solution is evaporated to dryness and recrystallization was done in dichloromethane. The resulting colorless block-shaped crystals were obtained by slow cooling. The yield is 78% and elemental analysis: calc. for $C_{27}H_{20}FN_3S_2Sn$: C 55.12, H 3.44, N 7.14; found: C 55.31, H 3.69, N 6.53. The elemental analyses were performed with PERKIN ELMER 2400 SERIES II.

Experimental details

The U_{iso}(H) values were set at 1.2U_{eq}(C—H) for all the H atoms from benzene ring and the C atom of C=N moiety.

Comment

Metal thiolato complexes have been extensively studied because of their ability to adopt various nuclearities and their relevance in biology, since they form the inorganic part of the biologically active centers of some metalloproteins and enzymes [5, 6]. Recently, attention has been paid to the coordination chemistry of heterocyclic thiol/thione donors, which can give potential access to new complexes with unusual structures and reactivities [7]. The Schiff base ligand also exibits biological activity [8]. However, the organotin complexes also exhibit the biological and commercial applications [9]. To aid in understanding their biochemical action, it was felt that the coordination chemistry of tin(IV) toward S and N-containing ligands requires further exploration.

The title complex shows a four-coordinated Sn(IV) metal center. The distances of S—Sn is 2.4826(14) Å and N—Sn 3.102(4) Å, which is close to the sum of the covalent radii (2.42) [10] and similar to the parameters in $Ph_3Sn(SC_2H_2N_3)$ [11].

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