Crystal structure of diphenyl-(*N*-salicylideneacetylhydrazonato)tin(IV), $(C_6H_5)_2[OC(CH_3)NHNCHC_6H_4O]Sn$

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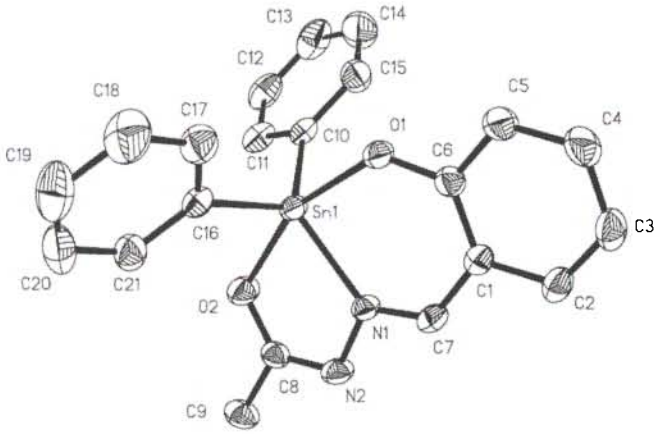

Abstract

$C_{21}H_{19}N_2O_2Sn$, triclinic, $P\bar{1}$ (No. 2), $a = 9.223(5)$ Å, $b = 10.059(5)$ Å, $c = 11.628(5)$ Å, $\alpha = 68.62(3)°$, $\beta = 80.52(4)°$, $\gamma = 71.74(3)°$, $V = 952.4$ Å^3, $Z = 2$, $R_{gt}(F) = 0.071$, $wR(F) = 0.071$, $T = 293$ K.

Source of material

The ligand salicylaldehydeacethylhydrazone was prepared by reacting stochiometric amounts of salicylaldehyde and acetylhydrazide in water-ethanol solution (50/50) at about 323 K. An ethanol solution of $SnPh_2Cl_2$ was then added to a 50% EtOH solution of the ligand with KOH. After heating and stirring for some hours the tin complex was obtained as a yellow precipitate. Crystals were grown by slow evaporation from a acetonitrile solution.

Discussion

Two phenyl carbon atoms and one nitrogen atom of the chelate ligand form the trigonal base of a distorted bipyramidal environment around the tin atom, with the oxygens in apical positions. The significant deviation of the O–Sn–O sequence from linearity is clearly due to the conformation of the tridentate ligand. Relevant bond distances are: Sn–C 2.12(1) Å and 2.13(1) Å, Sn–N 2.15(1) Å, Sn–O 2.07(1) Å and 2.13(1) Å. The N(1)–C(7) bond of 1.29(1) Å is essentially double in character.

Table 1. Data collection and handling.

Crystal:	yellow prism, max. dim. 0.2 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	15.0 cm^{-1}
Diffractometer, scan mode:	Philips PW1100 (FEBO SYSTEM), 2θ
$2\theta_{max}$:	60°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5423, 5127
Criterion for F_{obs}, $N(hkl)_{gt}$:	$F_{obs} > 4\,\sigma(F_{obs})$, 5063
$N(param)_{refined}$:	209
Program:	SHELX-76 [1]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(2)	2*i*	0.4028(5)	–0.1347(5)	0.7012(3)	0.063
H(3)	2*i*	0.6273(6)	–0.3253(5)	0.7164(3)	0.076
H(4)	2*i*	0.7256(5)	–0.4063(5)	0.5490(5)	0.085
H(5)	2*i*	0.5993(5)	–0.2966(6)	0.3665(4)	0.073
H(7)	2*i*	0.2062(5)	0.0463(5)	0.5935(4)	0.054
H(9A)	2*i*	–0.192(3)	0.490(1)	0.305(3)	0.074
H(9B)	2*i*	–0.211(3)	0.409(4)	0.448(2)	0.074
H(9C)	2*i*	–0.2951(9)	0.380(3)	0.357(5)	0.074
H(11)	2*i*	–0.1284(5)	0.0968(4)	0.1369(5)	0.070
H(12)	2*i*	–0.2390(5)	–0.0530(7)	0.0891(6)	0.087
H(13)	2*i*	–0.1193(8)	–0.3052(6)	0.1369(7)	0.105
H(14)	2*i*	0.1110(8)	–0.4076(3)	0.2326(7)	0.105
H(15)	2*i*	0.2217(5)	–0.2578(5)	0.2804(5)	0.073
H(17)	2*i*	0.4680(6)	0.0580(4)	0.1030(6)	0.081
H(18)	2*i*	0.5941(5)	0.2134(8)	–0.0497(7)	0.119
H(19)	2*i*	0.4743(8)	0.4655(7)	–0.1301(6)	0.120
H(20)	2*i*	0.2284(8)	0.5622(4)	–0.0578(6)	0.101
H(21)	2*i*	0.1023(5)	0.4069(5)	0.0949(6)	0.074
H(N2)	2*i*	–0.0001(5)	0.2297(6)	0.5239(6)	0.084

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Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Sn(1)	2*i*	0.16105(3)	0.07123(3)	0.25392(2)	0.0409(2)	0.0457(2)	0.0343(2)	–0.0059(1)	–0.0022(1)	–0.0186(1)
O(1)	2*i*	0.3590(4)	–0.0943(4)	0.3163(3)	0.052(2)	0.058(2)	0.042(2)	0.007(1)	–0.010(1)	–0.024(1)
O(2)	2*i*	–0.0472(5)	0.2392(5)	0.2605(4)	0.054(2)	0.066(2)	0.051(2)	0.007(2)	–0.008(1)	–0.028(2)
N(1)	2*i*	0.1402(4)	0.0919(4)	0.4336(3)	0.042(2)	0.046(2)	0.035(1)	–0.011(1)	0.004(1)	–0.019(1)
N(2)	2*i*	0.0173(5)	0.2060(5)	0.4543(4)	0.049(2)	0.057(2)	0.048(2)	–0.005(2)	0.005(1)	–0.030(2)
C(1)	2*i*	0.3665(3)	–0.1014(3)	0.5247(3)	0.046(2)	0.047(2)	0.037(2)	–0.018(2)	–0.003(1)	–0.013(1)
C(2)	2*i*	0.4422(4)	–0.1671(4)	0.6341(2)	0.061(3)	0.059(2)	0.042(2)	–0.022(2)	–0.012(2)	–0.013(2)
C(3)	2*i*	0.5767(4)	–0.2813(4)	0.6432(3)	0.070(3)	0.060(3)	0.060(3)	–0.017(2)	–0.028(3)	–0.009(2)
C(4)	2*i*	0.6356(4)	–0.3298(4)	0.5429(4)	0.062(3)	0.062(3)	0.083(4)	0.006(2)	–0.032(3)	–0.027(3)
C(5)	2*i*	0.5599(4)	–0.2641(4)	0.4336(3)	0.056(3)	0.062(3)	0.062(3)	0.005(2)	–0.018(2)	–0.028(2)
C(6)	2*i*	0.4253(3)	–0.1499(3)	0.4244(2)	0.045(2)	0.041(2)	0.046(2)	–0.009(1)	–0.008(2)	–0.015(2)
C(7)	2*i*	0.2310(5)	0.0186(5)	0.5231(4)	0.050(2)	0.054(2)	0.033(2)	–0.018(2)	0.004(1)	–0.018(2)
C(8)	2*i*	–0.0689(5)	0.2736(5)	0.3600(4)	0.044(2)	0.040(2)	0.050(2)	–0.007(1)	0.006(2)	–0.018(2)
C(9)	2*i*	–0.2038(7)	0.3995(6)	0.3684(6)	0.055(3)	0.052(2)	0.074(3)	–0.005(2)	0.011(2)	–0.030(2)
C(10)	2*i*	0.0576(4)	–0.0657(3)	0.2134(3)	0.049(2)	0.057(2)	0.032(2)	–0.017(2)	0.003(1)	–0.017(2)
C(11)	2*i*	–0.0804(4)	–0.0043(4)	0.1561(3)	0.053(2)	0.083(3)	0.046(2)	–0.021(2)	–0.000(2)	–0.027(2)
C(12)	2*i*	–0.1467(4)	–0.0941(5)	0.1274(4)	0.068(4)	0.110(5)	0.053(3)	–0.041(4)	–0.003(2)	–0.029(3)
C(13)	2*i*	–0.0750(6)	–0.2452(5)	0.1561(5)	0.123(6)	0.105(5)	0.062(4)	–0.073(5)	–0.010(4)	–0.024(4)
C(14)	2*i*	0.0630(6)	–0.3065(3)	0.2134(5)	0.126(7)	0.066(4)	0.085(5)	–0.034(4)	–0.024(5)	–0.026(3)
C(15)	2*i*	0.1293(4)	–0.2168(4)	0.2420(4)	0.071(3)	0.055(3)	0.059(3)	–0.016(2)	–0.017(2)	–0.018(2)
C(16)	2*i*	0.2727(4)	0.2170(3)	0.1141(3)	0.051(2)	0.055(2)	0.036(2)	–0.015(2)	–0.007(2)	–0.017(2)
C(17)	2*i*	0.4200(4)	0.1590(4)	0.0708(4)	0.064(3)	0.072(3)	0.059(3)	–0.019(3)	0.009(2)	–0.018(3)
C(18)	2*i*	0.4955(5)	0.2521(6)	–0.0207(5)	0.078(5)	0.115(7)	0.090(6)	–0.042(5)	0.027(4)	–0.018(5)
C(19)	2*i*	0.4237(6)	0.4032(6)	–0.0689(5)	0.107(6)	0.104(6)	0.078(5)	–0.061(5)	–0.006(4)	0.009(4)
C(20)	2*i*	0.2764(6)	0.4612(3)	–0.0256(4)	0.106(6)	0.064(3)	0.072(4)	–0.032(4)	–0.029(4)	0.007(3)
C(21)	2*i*	0.2009(4)	0.3681(4)	0.0659(4)	0.078(3)	0.058(3)	0.052(3)	–0.022(2)	–0.017(2)	–0.012(2)

Reference

1. Sheldrick, G. M.: SHELXL-76, Programs for Crystal Structures Determination. Cambridge 1976.