

Synthesis and Characterization of Diorganotin Compounds $\{[R_2Sn(ON=CHC_6H_5)]_2O\}_2$ and Crystal Structure of $\{[(C_6H_5CH_2)_2Sn(ON=CHC_6H_5)]_2O\}_2$

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Diorganotin compounds $\{[R_2Sn(ON=CHC_6H_5)]_2O\}_2$ [$R=C_6H_5CH_2$ (**1**), 2-FC₆H₄CH₂ (**2**), 4-FC₆H₄CH₂ (**3**), 2-ClC₆H₄CH₂ (**4**), 4-ClC₆H₄CH₂ (**5**)] were synthesized by the reaction of R₂SnO with HON=CHC₆H₅ in 1 : 1 molar ratio in refluxing anhydrous benzene or toluene. They were characterized by elemental analysis, IR, ¹H NMR and ¹¹⁹Sn NMR spectroscopy. And two sets of ¹¹⁹Sn chemical shifts were observed in the ¹¹⁹Sn NMR spectra of these compounds, indicating the presence of two types of environment around the tin atoms. The crystal structure of compound **1** was determined by X-ray single crystal diffraction analysis. The results showed that the crystal of compound **1** belongs to a monoclinic system with space group *P2₁/c* and unit cell dimensions: *a*=1.0718(9) nm, *b*=1.9666(17) nm, *c*=2.0480(17) nm, *β*=96.4371(14)°, *D_c*=1.450 g/cm³, *Z*=2, *F*(000)=1888, *V*=4.290(6) nm³, *μ*=1.206 mm⁻¹, *R*₁=0.0405, *wR*₂=0.0786. The compound **1** belongs to centrosymmetric dimer structure mode with a four-membered central *endo*-cyclic Sn₂O₂ unit in which the bridging oxygen atoms are tri-coordinated. Each bridging oxygen atom also connects with an *exo*-cyclic tin atom. The *endo*- and *exo*-cyclic tin atoms are both five-coordinated, and have coordination geometry of distorted trigonal bipyramid.

Keywords diorganotin compound, benzaldoxime, synthesis, crystal structure

Introduction

Dimeric tetraorganodistannoxanes are a kind of interesting organotin oxo clusters and have attracted considerable attention during the last several decades, in view of their unique structural features¹⁻⁵ as well as their applications as biocides^{6,7} and in homogenous catalysis.^{8,9} In the solid state, they contain characteristic Sn₄O₂X₂Y₂ structural motifs with staircase or ladder arrangements, a planar four-membered Sn₂O₂ ring and, generally, penta-coordination around the tin atom.^{10,11} Further, among main group organometallics, diorganotin compounds demonstrate considerably higher anti-cancer activity *in vitro* as well as lower toxicity than the known *cis*-platin.^{12,13} Of many multidentate organic ligands, oximes are of particular importance because of their remarkable structural diversity and biological implications.^{14,15} Recent research has been devoted to the influence of the nature and acidity of the entering nucleophile on the reactivity of distannoxanes and, more specifically, on the stability of Sn₂O₂ cluster network. Considerable effort has been made to determine whether the presence of an additional donor atom in the internally functionalized oxime ligand has any effect on the structure adopted by organotin oximates.^{5,11,16,17}

In order to investigate this type of ladder structure of

tetraorganodistannoxanes as well as coordination feature of oxime, we have synthesized and characterized five novel tetraorganodistannoxanes with benzaldoxime $\{[R_2Sn(ON=CHC_6H_5)]_2O\}_2$ [$R=C_6H_5CH_2$ (**1**), 2-FC₆H₄CH₂ (**2**), 4-FC₆H₄CH₂ (**3**), 2-ClC₆H₄CH₂ (**4**), 4-ClC₆H₄CH₂ (**5**)] and also determined the crystal structure of compound **1**.

Experimental

General procedure

All reagents were of analytical grade. The solvents used in this work were dried before use. The IR spectra were obtained on a Nicolet-460 spectrometer, as KBr discs. The ¹H and ¹¹⁹Sn NMR spectra were recorded on a Mercury Plus-400 NMR spectrometer in CDCl₃, and chemical shifts were given relative to Me₄Si and Me₄Sn, respectively. Elemental analyses were performed with a PE-2400 II elemental analyzer, and tin was gravimetrically estimated as SnO₂.

Preparation of title compounds

Benzaldoxime (4.0 mmol) was added to a benzene (or toluene) suspension (50 mL) of R₂SnO (4.0 mmol), then the mixture was stirred and heated at reflux for 4 h. The water formed during the reaction was fractionally

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Received January 5, 2004; revised and accepted May 20, 2004.

Project supported by the National Natural Science Foundation of China (No. 20271025) and the Natural Science Foundation of Shandong Province (No. L2003B01).

removed azeotropically with a Dean and Stark apparatus. The clear solution obtained after filtration was evaporated in vacuum to give a pinkish solid, which was recrystallized from hexane-benzene to give the product as colorless crystals. Their physical data are summarized as follows:

$\{[(C_6H_5CH_2)_2Sn(ON=CHC_6H_5)]_2O\}_2 \cdot 2C_6H_6$ (**1**): 1.40 g, yield 75%, m.p. 188 °C (dec.), 1H NMR δ : 2.97 (t, $J_{Sn-H}=75.6$ Hz, 16H, $PhCH_2Sn$), 7.15–7.45 (m, 72H, C_6H_5 , C_6H_6), 8.11 (br, 4H, $ON=CH$); ^{119}Sn NMR δ : -204.8, -223.4; IR ν : 612 (Sn—O—Sn), 545 (Sn—O), 465 (Sn—C) cm^{-1} . Anal. calcd for $C_{96}H_{92}N_4O_6 \cdot Sn_4$: C 61.58, H 4.95, N 2.99, Sn 25.35; found C 61.29, H 5.02, N 2.82, Sn 25.51.

$\{[(2-FC_6H_4CH_2)_2Sn(ON=CHC_6H_5)]_2O\}_2$ (**2**): 1.30 g, yield 70%, m.p. 165–167 °C, 1H NMR δ : 3.01 (t, $J_{Sn-H}=77.2$ Hz, 16H, $PhCH_2Sn$), 7.10–7.40 (m, 52H, $C_6H_5 + C_6H_4F$), 8.15 (br, 4H, $ON=CH$); ^{119}Sn NMR δ : -198.9, -220.1; IR ν : 608 (Sn—O—Sn), 553 (Sn—O), 460 (Sn—C) cm^{-1} . Anal. calcd for $C_{84}H_{72}F_8N_4O_6 \cdot Sn_4$: C 54.24, H 3.90, N 3.01, Sn 25.52; found C 54.57, H 4.12, N 3.09, Sn 25.44.

$\{[(4-FC_6H_4CH_2)_2Sn(ON=CHC_6H_5)]_2O\}_2$ (**3**): 1.38 g, yield 74%, m.p. 198–200 °C, 1H NMR δ : 2.99 (t, $J_{Sn-H}=76.9$ Hz, 16H, $PhCH_2Sn$), 7.13–7.41 (m, 52H, $C_6H_5 + C_6H_4F$), 8.08 (br, 4H, $ON=CH$); ^{119}Sn NMR δ : -201.6, -220.2; IR ν : 603 (Sn—O—Sn), 548 (Sn—O), 456 (Sn—C) cm^{-1} . Anal. calcd for $C_{84}H_{72}F_8N_4O_6 \cdot Sn_4$: C 54.24, H 3.90, N 3.01, Sn 25.52; found C 54.39, H 4.08, N 2.94, Sn 25.47.

$\{[(2-ClC_6H_4CH_2)_2Sn(ON=CHC_6H_5)]_2O\}_2$ (**4**): 1.34 g, yield 67%, m.p. 154–155 °C, 1H NMR δ : 2.98 (t, $J_{Sn-H}=76.2$ Hz, 16H, $PhCH_2Sn$), 7.01–7.42 (m, 52H, $C_6H_5 + C_6H_4Cl$), 8.12 (br, 4H, $ON=CH$); ^{119}Sn NMR δ : -195.7, -216.9; IR ν : 605 (Sn—O—Sn), 557 (Sn—O), 454 (Sn—C) cm^{-1} . Anal. calcd for $C_{84}H_{72}Cl_8N_4O_6 \cdot Sn_4$: C 50.58, H 3.64, N 2.81, Sn 23.80; found C 50.30, H 3.72, N 2.91, Sn 23.58.

$\{[(4-ClC_6H_4CH_2)_2Sn(ON=CHC_6H_5)]_2O\}_2$ (**5**): 1.42 g, yield 71%, m.p. 221–223 °C, 1H NMR δ : 3.00 (t, $J_{Sn-H}=76.6$ Hz, 16H, $PhCH_2Sn$), 7.06–7.39 (m, 52H, $C_6H_5 + C_6H_4Cl$), 8.06 (br, 4H, $ON=CH$); ^{119}Sn NMR δ : -202.5, -221.7; IR ν : 610 (Sn—O—Sn), 552 (Sn—O), 464 (Sn—C) cm^{-1} . Anal. calcd for $C_{84}H_{72}Cl_8N_4O_6 \cdot Sn_4$: C 50.58, H 3.64, N 2.81, Sn 23.80; found C 50.65, H 3.49, N 2.72, Sn 23.97.

Crystallographic measurement of compound 1

A colorless crystal with approximate dimensions of 0.34 mm \times 0.26 mm \times 0.10 mm was mounted on a glass capillary. All measurements were made on a Bruker Smart 1000 CCD diffractometer with graphite monochromated Mo $K\alpha$ (0.071073 nm) radiation at 298(2) K using the Φ - ω scan technique. A total of 22603 reflections were collected in the range of $1.91^\circ \leq \theta \leq 25.03^\circ$ and 7569 reflections were independent ($R_{int}=0.0554$). The crystal belongs to monoclinic system with space group $P2_1/c$, $a=1.0718(9)$ nm, $b=1.9666(17)$ nm, $c=2.0480(17)$ nm, $\beta=96.4371(14)^\circ$, $D_c=1.450$ g/cm³, $Z=$

2, $F(000)=1888$, $V=4.290(6)$ nm³, $\mu=1.206$ mm⁻¹, $R_1=0.0405$, $wR_2=0.0786$. The structure was solved by direct method and difference Fourier map using SHELXL-97 program, and refined by full-matrix least-squares on F^2 . All non-hydrogen atoms were refined anisotropically. Hydrogen atoms were located on calculated positions and refined isotropically. The largest difference peak and hole were 847 and -399 e/nm³ respectively.

Results and discussion

Synthesis and spectral analysis

Reaction of R_2SnO with benzaldoxime in 1 : 1 stoichiometry in refluxing anhydrous benzene readily gives $\{[R_2Sn(ON=CHC_6H_5)]_2O\}_2$ in 72%–80% yield after recrystallization.

All compounds are colorless crystalline solids and soluble in common organic solvents such as ethanol, benzene and dichloromethane. The crystal of compound **1** consists of the compound **1** and two uncoordinated benzene molecules.

The IR spectra of these compounds were interpreted by comparison with those of the free benzaldoxime and related diorganotin oxides. The free benzaldoxime shows an O—H absorption at 3310 cm^{-1} , which is absent from the spectra of the five title compounds, showing the deprotonation of the $HON=C$ group during the reactions. In compounds **1**–**5**, the Sn—C absorption band appears at 454–465 cm^{-1} region. The band in 545–557 cm^{-1} are assigned to the stretching mode of the Sn—O linkage and the strong band in the 610–603 cm^{-1} is attributed to $\nu(Sn-O-Sn)$ indicating a Sn—O—Sn link.^{1,11,18-20}

The 1H NMR spectra of these compounds exhibit characteristic signals and multiplicities for $ArCH_2Sn$ and ligand protons. The hydroxyl proton resonance that is present at the free oxime ligand is absent from the 1H NMR spectra of all corresponding compounds, indicating the deprotonation of the OH group and formation of the Sn—O bond. A significant shift in the position of the proton of $ON=C-H$ signal of the moiety further supports Sn—O bonding.

The ^{119}Sn NMR spectra of all of these compounds display two well separated resonances (δ -195.7 to -204.8 and -216.9 to -223.4), as is observed for tetraorganodistannoxane, indicating dimeric structures in solution.^{11,16-18} The low- and high-field resonances observed for these compounds are attributed to the *exo*-cyclic and *endo*-cyclic tin atoms, respectively.²¹ Of these two signals, the low field signal is broad which may be due to the 'Wagging motion' of the *exo*-cyclic tin atoms in solution.^{3,22} The values of the ^{119}Sn chemical shift (δ -195.7 to -223.4) for compounds **1**–**5** suggest that all the tin atoms exhibit penta-coordination.²³ The values of the ^{119}Sn chemical shift indicate that bonding should be similar to each other of the compounds **1**–**5**.

Crystal structure of compound 1

The molecular structure of the compound **1** is shown in Figure 1. And Figure 2 shows the packing of the molecules in the unit cell as seen in a projection on its

face. Table 1 gives the atomic coordinates and equivalent isotropic thermal parameters. The selected bond lengths and angles are listed in Table 2.

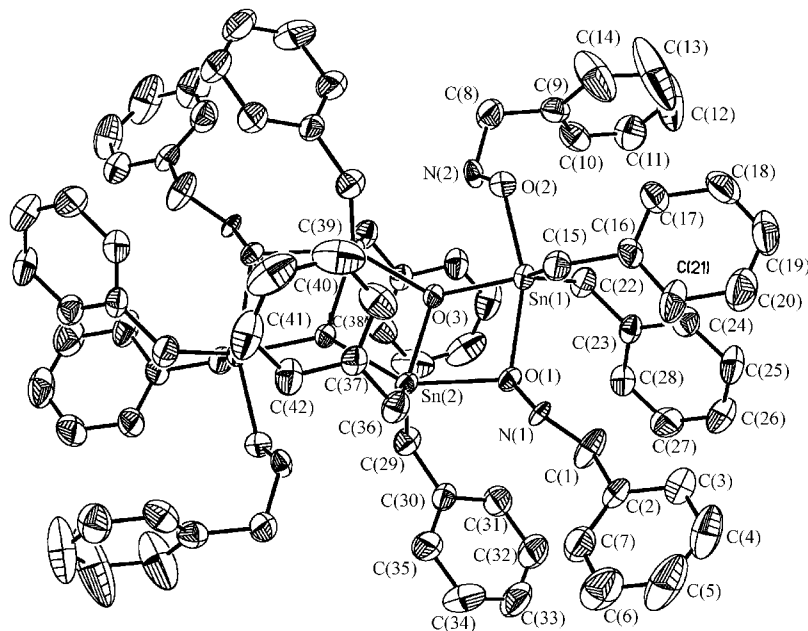


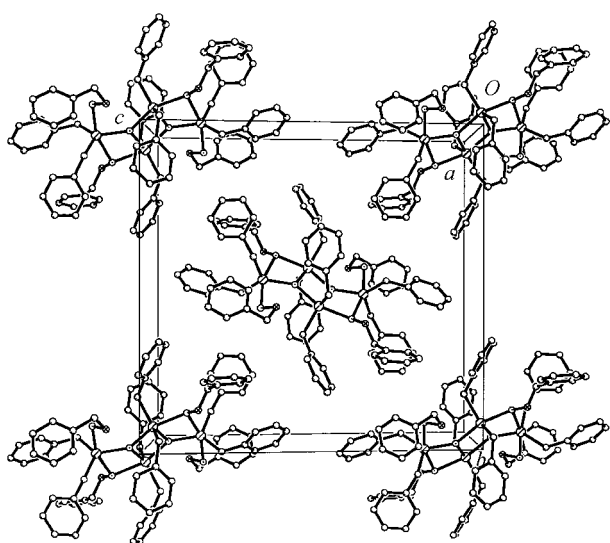
Figure 1 Molecular structure of compound 1.

Table 1 Atomic coordination ($\times 10^4$) and equivalent isotropic displacement parameters ($\times 10^5 \text{ nm}^2$) of compound 1

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq}	Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq}
Sn(1)	4625(1)	236(1)	1548(1)	49(1)	C(22)	3247(5)	948(3)	1806(3)	68(2)
Sn(2)	6047(1)	610(1)	170(1)	43(1)	C(23)	3723(5)	1580(3)	2146(3)	56(1)
N(1)	7063(3)	1160(2)	1561(2)	36(1)	C(24)	4117(6)	1584(3)	2804(3)	68(2)
N(2)	2525(4)	-623(2)	1111(2)	54(1)	C(25)	4558(7)	2163(4)	3132(3)	85(2)
O(1)	5869(3)	1025(2)	1161(1)	47(1)	C(26)	4605(7)	2752(4)	2792(4)	93(2)
O(2)	3637(4)	-665(2)	1588(2)	69(1)	C(27)	4218(6)	2765(3)	2141(4)	85(2)
O(3)	4671(3)	79(2)	566(1)	45(1)	C(28)	3772(6)	2188(3)	1817(3)	69(2)
C(1)	6974(6)	1806(3)	1841(3)	83(2)	C(29)	5452(6)	1526(3)	-319(2)	64(2)
C(2)	8238(6)	1987(3)	2157(3)	59(2)	C(30)	6157(6)	2144(3)	-65(2)	58(2)
C(3)	8468(7)	2043(3)	2830(3)	83(2)	C(31)	5612(7)	2616(3)	306(3)	87(2)
C(4)	9682(10)	2207(5)	3107(4)	122(3)	C(32)	6244(10)	3189(4)	550(4)	112(3)
C(5)	10596(9)	2298(5)	2718(6)	153(5)	C(33)	7417(10)	3301(4)	414(4)	108(3)
C(6)	10372(8)	2254(4)	2062(5)	135(4)	C(34)	7982(8)	2854(4)	60(4)	113(3)
C(7)	9204(7)	2100(3)	1793(3)	89(2)	C(35)	7360(7)	2270(3)	-184(3)	86(2)
C(8)	1605(6)	-1001(3)	1384(3)	93(2)	C(36)	7900(5)	254(3)	446(3)	63(2)
C(9)	1109(6)	-672(3)	1960(3)	66(2)	C(37)	8113(5)	-497(3)	367(2)	51(1)
C(10)	423(6)	-100(4)	1897(3)	78(2)	C(38)	7801(6)	-972(4)	811(3)	87(2)
C(11)	-38(7)	220(4)	2407(4)	97(2)	C(39)	8014(7)	-1651(4)	737(5)	110(3)
C(12)	161(11)	-44(6)	2985(5)	174(5)	C(40)	8587(8)	-1860(4)	209(5)	116(3)
C(13)	858(13)	-615(8)	3074(4)	256(9)	C(41)	8916(8)	-1407(4)	-241(4)	105(3)
C(14)	1327(11)	-940(5)	2558(4)	180(5)	C(42)	8681(6)	-732(3)	-158(3)	75(2)
C(15)	6090(5)	-193(3)	2226(2)	62(2)	C(43)	2730(17)	1310(6)	4737(12)	164(5)
C(16)	5896(6)	-3(3)	2923(2)	60(2)	C(44)	2740(15)	1282(6)	5369(13)	181(6)
C(17)	4901(7)	-251(3)	3213(3)	86(2)	C(45)	1760(20)	1090(7)	5663(6)	166(5)
C(18)	4716(9)	-45(4)	3849(4)	111(3)	C(46)	731(18)	902(8)	5286(13)	193(6)
C(19)	5531(11)	393(4)	4181(3)	116(3)	C(47)	676(17)	928(8)	4637(12)	200(6)
C(20)	6536(9)	632(4)	3896(4)	111(3)	C(48)	1730(20)	1124(8)	4371(7)	191(7)
C(21)	6713(7)	436(3)	3274(3)	89(2)					

Table 2 Selected interatomic bond distances (nm) and angles ($^{\circ}$) for compound **1**

Sn(1)—O(3)	0.2040(3)	Sn(2)—C(29)	0.2124(5)
Sn(1)—C(22)	0.2144(5)	Sn(2)—O(1)	0.2224(3)
Sn(1)—C(15)	0.2150(5)	Sn(2)—O(3)	0.2047(3)
Sn(1)—O(2)	0.2070(4)	N(1)—C(1)	0.1401(6)
Sn(1)—O(1)	0.2443(3)	N(2)—C(8)	0.1400(7)
Sn(2)—O(3)#1	0.2106(3)	N(1)—O(1)	0.1460(4)
Sn(2)—C(36)	0.2122(5)	N(2)—O(2)	0.1456(5)
O(3)-Sn(1)-O(2)	88.78(12)	O(3)-Sn(1)-C(22)	115.80(17)
O(2)-Sn(1)-C(22)	100.3(2)	O(3)-Sn(1)-C(15)	118.77(16)
O(2)-Sn(1)-C(15)	88.76(19)	C(22)-Sn(1)-C(15)	124.8(2)
O(3)-Sn(1)-O(1)	71.36(11)	O(2)-Sn(1)-O(1)	158.86(12)
C(22)-Sn(1)-O(1)	95.00(19)	C(15)-Sn(1)-O(1)	94.43(18)
O(3)-Sn(2)-O(3)#1	74.56(13)	O(3)-Sn(2)-C(36)	114.90(19)
O(3)#1-Sn(2)-C(36)	103.54(17)	O(3)-Sn(2)-C(29)	115.35(19)
O(3)#1-Sn(2)-C(29)	98.13(17)	C(36)-Sn(2)-C(29)	128.9(2)
O(3)-Sn(2)-O(1)	71.64(11)	O(3)#1-Sn(2)-O(1)	146.11(12)
C(36)-Sn(2)-O(1)	92.92(16)	C(29)-Sn(2)-O(1)	93.97(17)
C(1)-N(1)-O(1)	107.2(3)	C(8)-N(2)-O(2)	105.2(4)
N(1)-O(1)-Sn(1)	116.9(2)	N(2)-O(2)-Sn(1)	108.0(3)
Sn(2)-O(5)-Sn(1)	100.42(12)	N(1)-O(1)-Sn(2)	114.5(2)
Sn(1)-O(3)-Sn(2)	114.25(13)	Sn(1)-O(3)-Sn(2)#1	139.06(15)
Sn(2)-O(3)-Sn(2)#1	105.44(13)	C(16)-C(15)-Sn(1)	110.2(4)
C(30)-C(29)-Sn(2)	114.4(3)	C(3)-C(22)-Sn(1)	116.8(4)
C(37)-C(36)-Sn(2)	116.3(3)	N(1)-C(1)-C(2)	107.2(5)

**Figure 2** Projection of the unit cell of compound **1**.

It is interesting to mention here that the similar reactions of $\text{HON}=\text{CHC}_6\text{H}_4\text{OH}$ with R_2SnO have been studied in detail in previous literatures.^{4,24} In these cases,

a completely different structure crystallizes out and an entirely different cluster is formed from that reported here.

A comparison of the present results with those reported earlier yields some interesting variations. For example, the structure of $\{[(\text{C}_6\text{H}_5\text{CH}_2)_2\text{Sn}(\text{ON}=\text{CHC}_6\text{H}_5)]_2\text{O}\}_2$ (**1**) (seen in Figure 1) is similar to those of $\{[\text{R}_2\text{Sn}(\text{O}_2\text{CR}')]\}_2\text{O}\}_2$ reported previously.^{1,10,11,25}

As can be seen from Figure 1, compound **1** is dimeric with a tortuous ladder structure that contains two *endo*- and two *exo*-cyclic tin atoms. The two kinds of Sn atoms are both five-coordinate and have distorted trigonal bipyramidal geometries. The central part of the dimeric unit consists of a four-membered $(\text{SnO})_2$ ring, which is essentially planar. The centrosymmetric $\text{Bz}_4\text{Sn}_2\text{O}_2$ core is attached to two Bz_2Sn units, making the bridge-oxo atoms O(3) and O(3)#1 be three-coordinated. The four benzaldoxime ligands are divided into two different types according to their coordinating fashion. Two of them connect *exo*-cyclic tin atoms using the oxygen atom, whereas the others are bridging to each pair of *exo*- and *endo*-cyclic tin atoms by utilizing one oxygen atom only.

The distances of μ_3 -O to Sn atoms are extremely similar [Sn(1)—O(3) 0.2040(3) nm, Sn(2)—O(3) 0.2047(3) nm and Sn(2)—O(3)#1 0.2106(3) nm], reflecting the strong coordination of bridge-oxo with tin atoms in the dimer. There is another bridge in compound **1**, linking the crystallographic asymmetric unit and the inversion center, which is oxo-bridge Sn(1)—O(1) 0.2443(3) nm and Sn(2)—O(1) 0.2224(3) nm.

The *exo*-cyclic tin atom Sn(1) is five-coordinated [Sn(1)—O(3) 0.2040(3) nm, Sn(1)—C(22) 0.2144(5) nm, Sn(1)—C(15) 0.2150(5) nm, Sn(1)—O(2) 0.2070(4) nm, Sn(1)—O(1) 0.2443(3) nm], and has distorted trigonal bipyramidal geometry with atoms C(15), C(22) and O(3) occupying equatorial positions and O(2) and O(1) atoms from the benzaldoxime ligand occupying axial positions. The sum of equatorial angles [O(3)-Sn(1)-C(15) 118.77(16)°, C(22)-Sn(1)-C(15) 124.8(2)° and O(3)-Sn(1)-C(22) 115.80(17)°] is equal to 359.37°, showing that these atoms are co-planar. The axial bond angle, O(1)-Sn(1)-O(2), of 158.86(12)° deviates significantly from linearity owing to the tendency of O(1) to bridge the Sn(2) atom. The *endo*-cyclic tin atom Sn(2) is also five-coordinated [Sn(2)—O(1) 0.2224(3) nm, Sn(2)—O(3) 0.2047(3) nm, Sn(2)—O(3)#1 0.2106(3) nm, Sn(2)—C(29) 0.2124(5) nm and Sn(2)—C(36) 0.2122(5) nm], and its geometry is also distorted trigonal bipyramid, with atoms C(29), C(36) and O(3) occupying equatorial positions and the oxygen atoms O(1) and O(3)#1 occupying axial positions which subtend an angle of 146.11(12)° at the *endo*-cyclic tin atom Sn(2).

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