Synthesis and Crystal Structure of Drum Organooxotin Clusters from Heteroaromatic Carboxylic Acid $[PhCH_2Sn(O)(O_2CC_5H_4N)]_6$ and $[PhCH_2Sn(O)(O_2CC_4H_3O)]_6$

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Two novel drum organooxotin clusters $[PhCH_2Sn(O)(O_2CC_5H_4N)]_6$(1) and $[PhCH_2Sn(O)(O_2CC_4H_3O)]_6$(2) were synthesized by the reaction of $[(PhCH_2)_3Sn]_2O$ with 2-pyridinecarboxylic acid and 2-furancarboxylic acid in 1:2 molar ratio. Their structures were determined by X-ray single crystal diffraction analysis. The crystal 1 belongs to monoclinic with space group $P2(1)/c$, $a = 1.2194(6)$ nm, $b = 1.2378(2)$ nm, $c = 2.6084(4)$ nm, $\beta = 94.148(3)°$, $Z = 2$, $V = 3.9270(11)$ nm^3, $D_c = 1.765$ kg/m^3, $\mu = 1.951$ mm^{-1}, $F(000) = 2040$, $R = 0.0516$, $wR = 0.0989$. The crystal 2 belongs to orthorhombic with space group $Pccn$, $a = 2.0300(6)$ nm, $b = 2.2670(6)$ nm, $c = 1.6088(5)$ nm, $\beta = 90°$, $Z = 4$, $V = 7.404(4)$ nm^3, $D_c = 1.813$ g/cm^3, $\mu = 2.070$ mm^{-1}, $F(000) = 3936$, $R = 0.0459$, $wR = 0.0884$. Their structures show a distorted octahedral configuration with six-coordination for the central tin atom.

Keywords organooxotin cluster, 2-pyridinecarboxylic acid, 2-furancarboxylic acid, synthesis, crystal structure

Introduction

Recently we have investigated the structural chemistry of a number of di- or tri-organotin heteroaromatic carboxylates.[1-5] These studies have shown that the structure of organotin heteroaromatic carboxylates is dependent on both the nature of the alkyl or aryl substituent bound to the tin atom and the type of carboxylate ligand. In particular, major structural variations are observed when carboxylate ligand contains an additional donor atom, such as a pyridine N atom, available for coordination to the Sn atom.[1-3,5-8] We have now turned to the monoorganotin heteroaromatic carboxylates.

Two major prototypes have been structurally characterized for the monoorganooxotin carboxylates,[9-12] which are "drum" structure with general formula $[RSn(O)(O_2CR')]_6$ and "ladder" arrangement with the formula $\{[RSn(O)(O_2CR')]_2[RSn(O_2CR')_3]\}_2$. We report herein the synthesis and X-ray crystal structure of two novel drum organooxotin clusters $[PhCH_2Sn(O)(O_2CC_5H_4N)]_6$ (1) and $[PhCH_2Sn(O)(O_2CC_4H_3O)]_6$(2). The X-ray diffraction analysis shows that these two complexes appear as drum-shaped molecules containing six-coordinated tin atoms.

Experimental

General procedure

IR spectra were recorded with a Nicolet-460 spectrophotometer using KBr discs. ^{1}H NMR spectra was recorded on a Jeol-FX-90Q NMR spectrometer and referenced to Me_4Si in $CDCl_3$. Elemental analyses were performed in a PE-2400 II elemental analyzer and tin was estimated as SnO_2.

Synthesis of compounds

$[PhCH_2Sn(O)(O_2CC_5H_4N)]_6$(1) 2-Pyridinecarboxylcarboxylic acid (6.0 mmol) was added to a benzene solution (40 mL) of $[(PhCH_2)_3Sn]_2O$ (3.0 mmol). The mixture was heated under reflux with stirring for 12 h. The clear solution thus obtained was evaporated under vacuum to form a white solid and recrystallized in dichloromethane-hexane to give colorless crystals 1.21 g, yield 58%, m.p. 276—278 ℃ (dec.); ^{1}H NMR δ: 2.91 (t, J_{Sn-H} = 86.4 Hz, 12H), 7.13—7.37 (m, 36H, PhH, 3-pyridine-H), 7.75 (br, 6H, 4-pyridine-H), 8.12 (br, 6H, 5-pyridine-H), 8.82 (br, 6H, 6-pyridine-H); IR (KBr) ν: 3051, 3038 (Ar—H), 2925, 2864 (C—H), 1558, 1551 (COO^-), 601 (Sn—O—Sn), 559 (Sn—O) cm^{-1}. Anal. calcd for $C_{78}H_{66}N_6O_{18}Sn_6$: C 44.88, H 3.19, N 4.03, Sn 34.11; found C 44.56, H 3.21, N 3.87, Sn 34.43.

$[PhCH_2Sn(O)(O_2CC_4H_3O)]_6$(2) This compound was similarly prepared as compound 1 to give colorless crystals 1.11 g, yield 55%, m.p. 223—225 ℃ (dec.); ^{1}H NMR δ: 2.86 (t, J_{Sn-H} = 88.6 Hz, 12H, $PhCH_2Sn$), 6.52—7.31 (m, 48H, PhH, furan-H); IR (KBr) ν:

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3063, 3016 (Ar—H), 2922, 2852 (C—H), 1566, 1558 (COO^-), 626 (Sn—O—Sn), 552 (Sn—O) cm^{-1}. Anal. calcd for $C_{72}H_{60}O_{24}Sn_6$: C 42.78, H 2.99, Sn 35.23; found C 43.06, H 3.13, Sn 35.44.

Crystal structure determination

The colorless crystal with dimensions 0.10 mm × 0.10 mm × 0.05 mm (**1**) or 0.40 mm × 0.40 mm × 0.30 mm (**2**) was mounted in a fiber, respectively. All measurements were made on a Bruker Smart-1000 CCD diffractometer with graphite monochromated Mo Kα (λ = 0.071073 nm) radiation. The structures were 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 at calculated positions and refined isotropically.

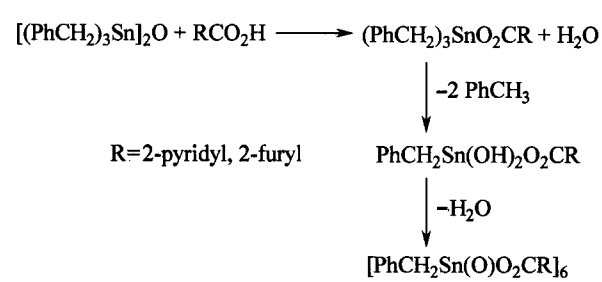
Result and discussion

Synthesis

Reaction of $[(PhCH_2)_3Sn]_2O$ with 2-pyridinecarboxylic acid and 2-furancarboxylic acid in 1:2 molar ratio gave hexameric benzyloxotin 2-pyridinecarboxylate (**1**) and hexameric benzyloxotin 2-furancarboxylate (**2**) respectively. The hexameric composition apparently formed as a result of slow hydrolysis of tribenzyltin heteroaromatic carboxylates, $(PhCH_2)_3SnO_2CR$, the major product of $[(PhCH_2)_3Sn]_2O$ and heteroaromatic carboxylic acid.

A possible mechanism is given in Scheme 1.

Scheme 1



IR *spectra*

The assignment of IR bands of these two complexes has been made by comparison with the IR spectra related to organotin compounds, carboxylates and $[(PhCH_2)_3Sn_2]O$. Infrared bands corresponding to the bridging carboxyl groups and Sn—O stretching vibration are useful in discriminating drum structure from the other form. For compounds **1** and **2**, the carboxyl absorptions, ν(COO), appear as a symmetrical doublet centered near 1555 cm^{-1} (1558, 1551 cm^{-1}) and 1560 cm^{-1} (1566, 1558 cm^{-1}) which indicate the presence of drum organooxotin cluster from carboxylic acid.[9] A very strong band at 601 or 626 cm^{-1}, the characteristic of the Sn—O—Sn linkage,[9] is assigned to ν_{Sn-O} for the drum form.

Crystal structure

The $[PhCH_2Sn(O)(O_2CC_5H_4N)]_6$ (**1**) and $[PhCH_2Sn(O)(O_2CC_4H_3O)]_6$ (**2**) compounds are hexameric and adopt the "drum" structure. Their molecular structures and molecular packing in the unit cell are shown in Fig. 1, Fig. 2, Fig. 3 and Fig. 4, respectively. Crystal data are listed in Table 1. Table 2 and Table 3 give the atomic coordinates and equivalent isotropic thermal parameters. The selected bond lengths and angles for **1** and **2** are listed in Table 4 and Table 5, respectively. The molecules are situated at about a crystallographic center of inversion so that half the molecule comprises the asymmetric unit and the unit cell contains one hexamer.

In compounds **1** and **2**, each of the lids of the "drum" comprises a hexagonal Sn_3O_3 ring of alternating Sn and O atoms. The top lid is twisted by approximately 60° relative to the lower one thereby enabling the formation of six Sn—O bonds which connect the lids; the rectangular sides of the drum thus formed may be considered as Sn_2O_2 stannoxane group. The hexagonal lids are not planar, however, a better description of their conformations is one based on a somewhat flattened chair conformation. In both compounds, each Sn atom bonds to three framework oxygen atoms, where the Sn—O bonds are all of comparable strength and have lengths ranging from 0.2068(6) nm to 0.2099(5) nm. The oxygen atoms of the framework are trivalence and have a distorted pyramidal geometry. For compounds **1** and **2**, the Sn atoms of each rectangular face are bridged by carboxylate ligands which form two somewhat different Sn—O bonds and have lengths ranging from 0.2124(7) nm to 0.2161(6) nm. As it has been observed in similar structures, the Sn—O bond lengths of the framework are significantly shorter than those involved in the carboxylate ligands.[9-11] The coordination geometry about each Sn atom is completed by a C atom of the $PhCH_2$ group which occupies a *trans* position to a framework O atom. Thus each Sn atom is coordinated by three "framework" O atoms, two carboxylate O atoms and one C atom so that the O_5C donor set defines a distorted octahedron.

Distortions from octahedron symmetry are reflected in the interatomic angles. For instance, around the Sn(2) atom of the compound **1**, the sum of equatorial angles O(2A)-Sn(2)-O(3) 103.5(2)°, O(3)-Sn(2)-O(5) 89.6(2)°, O(5)-Sn(2)-O(8A) 79.4(3)° and O(2A)-Sn(2)-O(8A) 84.2(3)° is equal to 356.7°, so the atoms O(2A), O(3), O(5), O(8A) and Sn(2) are almost in the same plane. The angles O(8A)-Sn(2)-C(26) 97.1(3)°, O(2A)-Sn(2)-C(26) 103.8(4)°, O(5)-Sn(2)-C(26) 93.6(4)° and O(3)-Sn(2)-C(26) 92.9(3)° are all greater than 90°. In contrast, the angles O(1)-Sn(2)-O(2A) 78.1(2)°, O(1)-Sn(2)-O(5) 86.9(3)° and O(1)-Sn(2)-O(3) 78.3(2)° are all less than 90°. Furthermore, the angle O(1)-Sn(2)-C(26) being in axial place is 171.2(3)°, which deviates from linear angle 180°. These data indicate that the Sn(2) atom of this compound is in distorted octahedron configura-

tion. The other tin atoms [Sn(1), Sn(3) and Sn(4)] of compound **1** and the four tin atoms of compound **2** are similar to the Sn(2), which are all in distorted octahedron configuration.

The other structure may be found for monoorganotin carboxylates based on the closely related formulation $\{[RSn(O)(O_2CR')]_2[RSn(O_2CR')_3]\}_2$.[12] Several crystal structures of them have shown that the structure is based on an Sn_4O_4 "ladder" with bridging and chelating carboxylate ligands. Our study provides another example of the "drum" hexameric form. We note that this structural type has now been observed

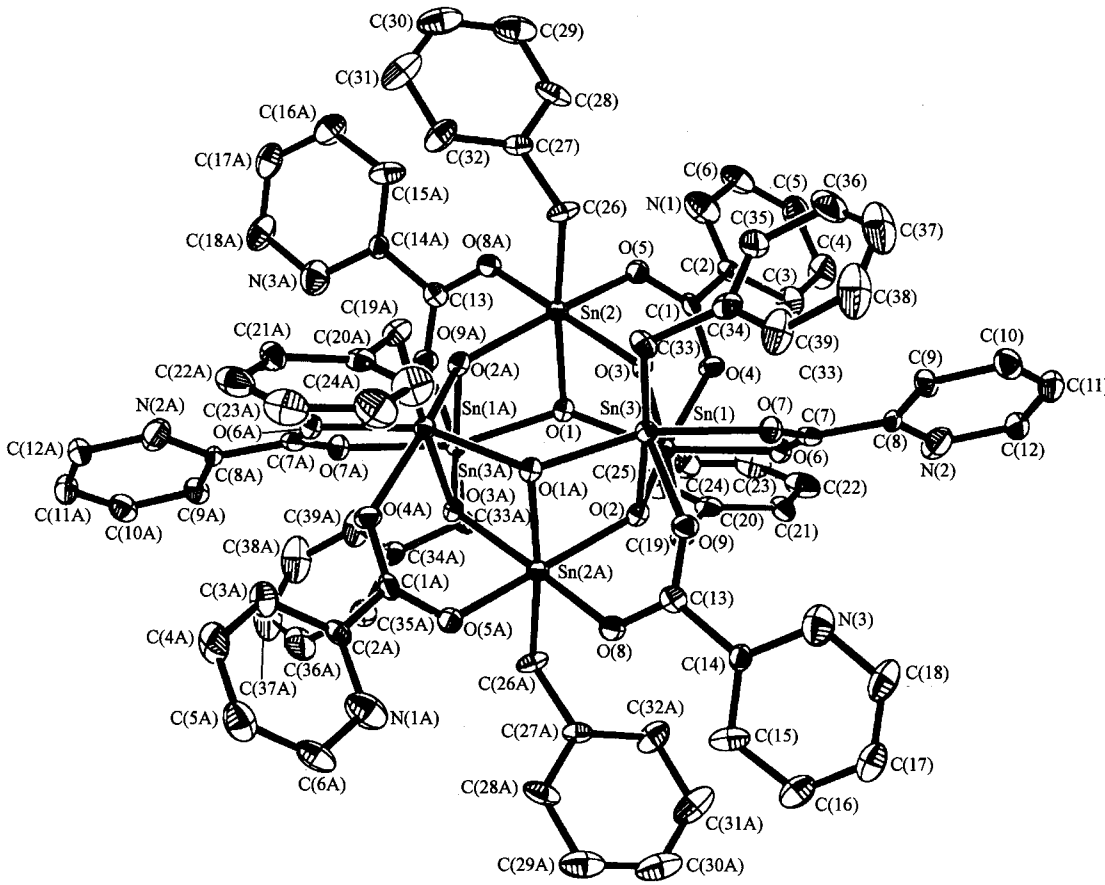


Fig. 1 Molecular structure of $[PhCH_2Sn(O)(O_2CC_5H_4N)]_6$.

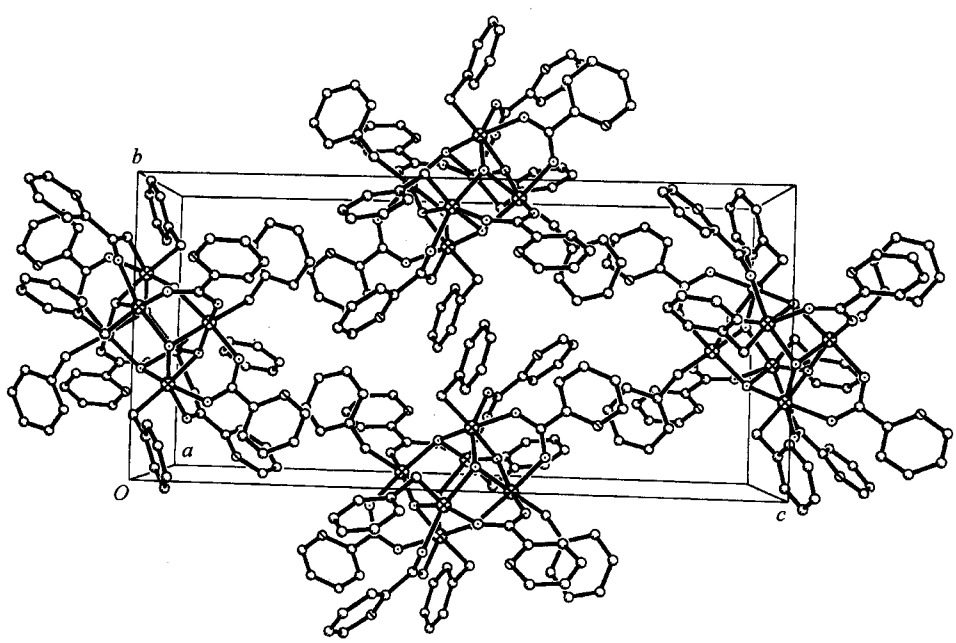


Fig. 2 Projection of the unit cell of $[PhCH_2Sn(O)(O_2CC_5H_4N)]_6$.

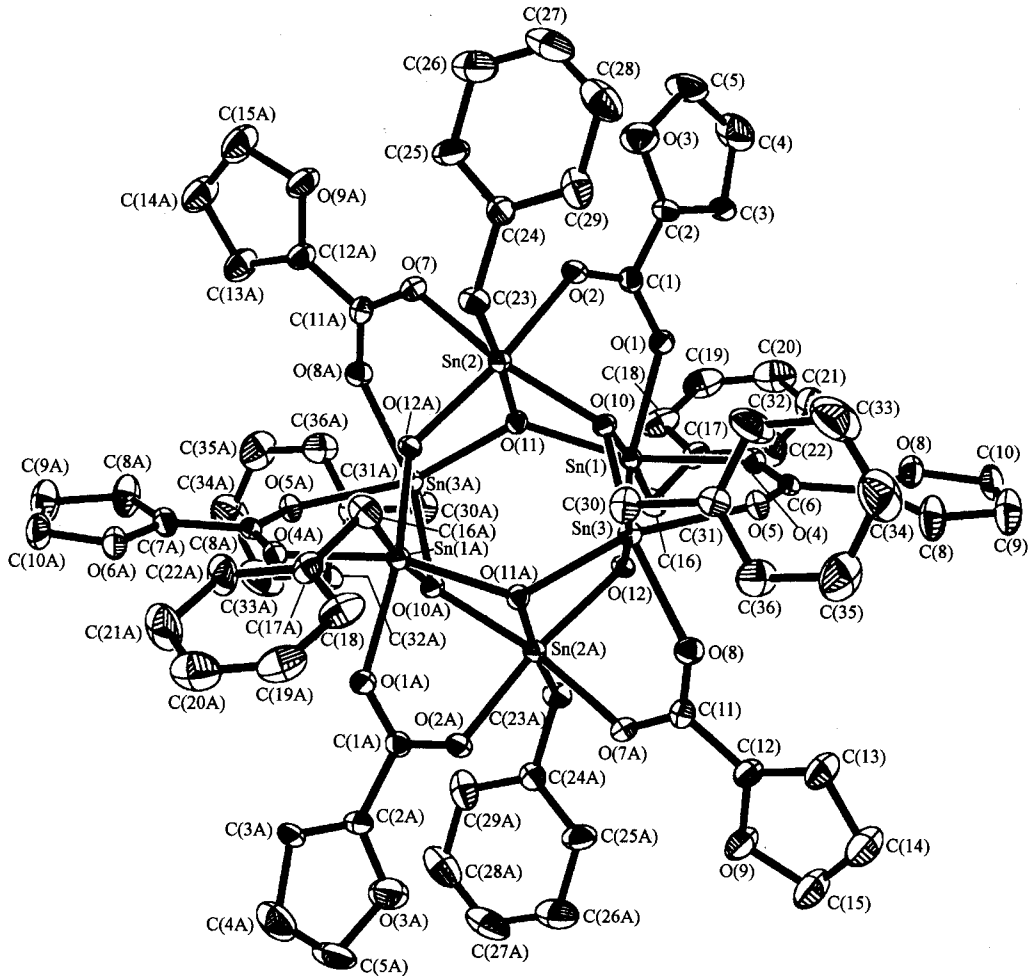


Fig. 3 Molecular structure of $[PhCH_2Sn(O)O_2C_4H_3O]_6$.

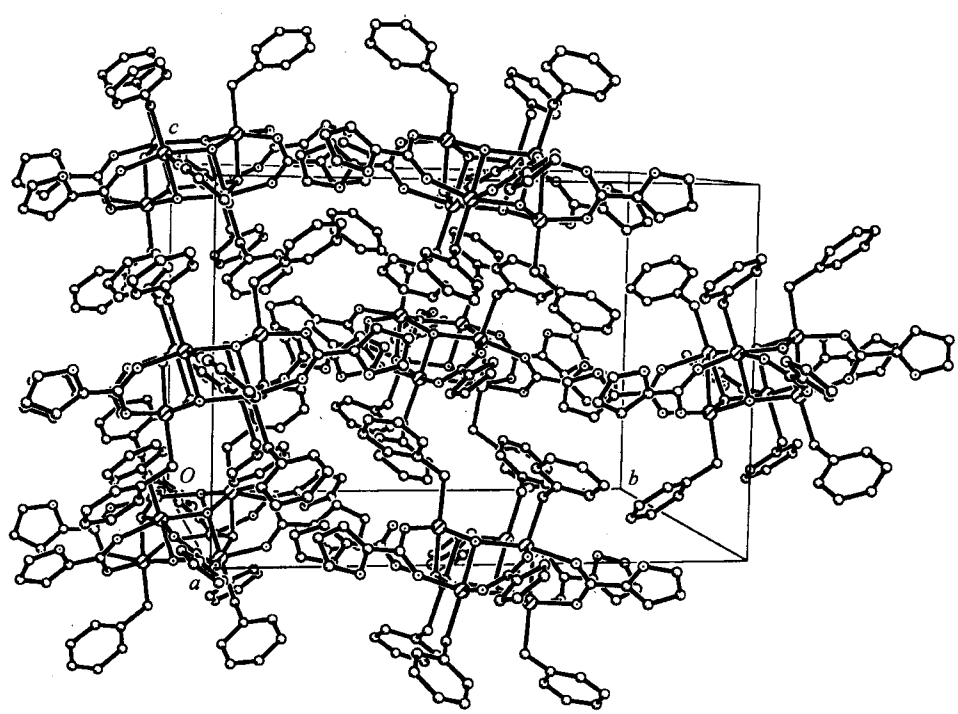


Fig. 4 Projection of the unit cell of $[PhCH_2Sn(O)O_2C_4H_3O]_6$.

in some of carboxylates bearing R′ groups such as CH_3,[10] CCl_3,[11] and c-C_6H_{11}[9], suggesting that the R′ group does not play a significant role in determining the structure. This observation may be rationalized in terms of the solid state structure which shows that the R′ groups are peripheral to the framework of the cluster.

It is noted to see from the molecular structures of compounds **1** and **2** that the hetero atom of carboxylate ligand does not coordinate to tin atom. As far as compound **1** is concerned, this result is different from that of the other type organotin compounds having 2-pyridinecarboxylate ligand[6-8] and may be connected with the special drum structure of this compound.

Table 1 Crystallographic data of compounds **1** and **2**

	1	**2**
Molecular formula	$C_{78}H_{66}N_6O_{18}Sn_6$	$C_{72}H_{60}O_{24}Sn_6$
Formular weigh	2087.55	2021.34
Crystal system	Monoclinic	Orthorhombic
Space group	*P*2(1)/*c*	*Pccn*
Unit cell dimensions		
a (nm)	1.2194(6)	2.0300(6)
b (nm)	1.2378(2)	2.2670(6)
c (nm)	2.6084(4)	1.6088(5)
β (°)	94.148(3)	90
Volume (nm^3)	3.9270(11)	7.404(4)
Z	2	4
D_{calcd} (g/cm^3)	1.765	1.813
F(000)	2040	3936
Scan range *θ* (°)	$1.57 \leqslant \theta \leqslant 25.03$	$1.80 \leqslant \theta \leqslant 25.03$
Total/unique/R_{int}	19809/6941/0.0927	36734/6497/0.0539
μ (mm^{-1})	1.951	2.070
R_1/wR_2	0.0516/0.0987	0.0459/0.0884
ρ_{max}/ρ_{min} (e/nm^3)	$8.53 \times 10^2/-7.71 \times 10^2$	$1.0348 \times 103/-7.76 \times 10^2$

Table 2 Atomic coordinates ($\times 10^4$) and thermal parameters ($nm^2 \times 10^5$) for compound **1**

Atom	*x*	*y*	*z*	U_{eq}	Atom	*x*	*y*	*z*	U_{eq}
Sn(1)	4465(1)	5246(1)	896(1)	34(1)	C(13)	6796(10)	2049(8)	640(4)	41(3)
Sn(2)	3085(1)	5683(1)	-170(1)	34(1)	C(14)	7280(9)	1298(9)	1054(4)	45(3)
Sn(3)	4622(1)	3093(1)	235(1)	35(1)	C(15)	8371(10)	1450(11)	1244(5)	74(4)
N(1)	396(10)	7300(9)	882(5)	92(4)	C(16)	8765(12)	836(11)	1675(6)	82(5)
N(2)	3205(9)	2853(10)	2225(4)	85(4)	C(17)	8066(15)	140(11)	1905(5)	80(5)
N(3)	6558(10)	550(9)	1285(5)	95(4)	C(18)	6985(15)	-37(11)	1714(6)	88(5)
O(1)	4332(5)	6398(5)	312(2)	36(2)	C(19)	5473(9)	6076(8)	1484(4)	46(3)
O(2)	5643(5)	4181(5)	657(2)	37(2)	C(20)	4830(9)	6906(10)	1775(4)	47(3)
O(3)	3579(5)	4392(5)	311(2)	33(2)	C(21)	4473(10)	6594(10)	2263(4)	60(4)
O(4)	2942(6)	5852(6)	1120(3)	47(2)	C(22)	3894(11)	7384(17)	2542(5)	92(5)
O(5)	1965(5)	6189(6)	376(3)	47(2)	C(23)	3720(13)	8435(14)	2344(7)	87(5)
O(6)	4018(5)	4016(6)	1425(2)	42(2)	C(24)	4098(13)	8695(12)	1864(7)	91(5)
O(7)	4013(6)	2532(5)	931(3)	41(2)	C(25)	4629(12)	7934(11)	1572(6)	83(5)
O(8)	7416(6)	2774(6)	485(3)	45(2)	C(26)	1866(9)	4719(8)	-605(4)	52(3)
O(9)	5810(6)	1896(5)	480(3)	45(2)	C(27)	1097(9)	5359(8)	-976(4)	44(3)
C(1)	2114(9)	6216(9)	865(4)	3(3)	C(28)	97(10)	5716(10)	-824(6)	73(4)
C(2)	1271(9)	6743(8)	1155(4)	41(3)	C(29)	-673(13)	6250(12)	-1176(8)	95(6)
C(3)	1360(11)	6678(11)	1690(5)	69(4)	C(30)	-356(15)	6401(13)	-1677(8)	100(7)
C(4)	589(13)	7196(12)	1961(5)	83(5)	C(31)	642(16)	6956(12)	-1836(6)	99(6)
C(5)	-264(12)	7795(11)	1711(6)	82(4)	C(32)	1400(12)	5513(10)	-1486(5)	73(4)

Continued

Atom	x	y	z	U_{eq}	Atom	x	y	z	U_{eq}
C(6)	-384(10)	7849(11)	1175(6)	76(4)	C(33)	3668(9)	2020(8)	-259(4)	45(3)
C(7)	3812(8)	3036(9)	1337(4)	41(3)	C(34)	2860(10)	1319(10)	20(4)	50(3)
C(8)	3234(8)	2415(9)	1725(4)	41(3)	C(35)	1821(12)	1756(12)	122(5)	77(4)
C(9)	2726(9)	1445(9)	1591(5)	53(3)	C(36)	1067(14)	1098(19)	359(6)	116(7)
C(10)	2123(10)	899(10)	1954(6)	66(4)	C(37)	1320(20)	60(20)	490(7)	130(10)
C(11)	2076(11)	1325(12)	2445(5)	67(4)	C(38)	2340(20)	-373(13)	388(7)	114(7)
C(12)	2614(11)	2259(12)	2588(5)	67(4)	C(39)	3123(13)	255(10)	160(5)	81(5)

Table 3 Atomic coordinates ($\times 10^4$) and thermal parameters ($nm^2 \times 10^5$) for compound **2**

Atom	x	y	z	U_{eq}	Atom	x	y	z	U_{eq}
Sn(1)	972(1)	4407(1)	-437(1)	37(1)	C(12)	-1326(5)	2648(4)	11(6)	56(2)
Sn(2)	903(1)	5578(1)	675(1)	37(1)	C(13)	-1178(6)	2202(5)	834(7)	81(3)
Sn(3)	-90(1)	4156(1)	996(1)	38(1)	C(14)	-1512(8)	1691(6)	512(9)	105(5)
O(1)	1990(3)	4581(2)	-173(4)	48(1)	C(15)	-1858(8)	1864(6)	-173(9)	108(5)
O(2)	1941(3)	5385(3)	645(4)	49(1)	C(16)	1083(5)	4121(4)	-1704(5)	59(3)
O(3)	3299(4)	5399(4)	753(5)	96(3)	C(17)	1767(5)	3968(4)	-1980(5)	53(2)
O(4)	1274(3)	3563(2)	47(4)	46(1)	C(18)	2092(6)	4339(5)	-2516(6)	81(3)
O(5)	573(3)	3422(2)	1107(3)	47(1)	C(19)	2738(7)	4167(7)	-2818(7)	91(4)
O(6)	1743(3)	2457(3)	215(4)	67(2)	C(20)	3020(7)	3661(8)	-2567(9)	108(5)
O(7)	1289(3)	6425(2)	299(4)	50(2)	C(21)	2697(7)	3282(6)	-2014(9)	111(5)
O(8)	-754(3)	3417(2)	901(3)	50(1)	C(22)	2056(6)	3441(5)	-1727(7)	81(3)
O(9)	-1739(4)	2455(3)	-307(5)	83(2)	C(23)	857(4)	5871(4)	1951(5)	51(2)
O(10)	749(2)	4674(2)	784(3)	37(1)	C(24)	1526(5)	5975(4)	2347(5)	52(2)
O(11)	870(2)	5313(2)	-574(3)	37(1)	C(25)	1871(5)	6498(5)	2178(7)	74(3)
O(12)	6(2)	4100(2)	-289(3)	36(1)	C(26)	2496(7)	6601(7)	2554(9)	105(4)
C(1)	2240(4)	4992(4)	254(5)	44(2)	C(27)	2740(7)	6156(9)	3066(10)	113(6)
C(2)	2962(4)	4989(4)	301(6)	59(3)	C(28)	2423(8)	5643(8)	3235(8)	108(5)
C(3)	3364(4)	4613(4)	-95(6)	58(3)	C(29)	1785(6)	5537(5)	2872(6)	82(4)
C(4)	4005(6)	4786(7)	119(9)	110(5)	C(30)	-288(5)	4268(4)	2308(5)	56(3)
C(5)	3973(5)	5252(7)	642(9)	101(4)	C(31)	-117(4)	3737(4)	2845(5)	52(2)
C(6)	1019(4)	3265(4)	616(5)	39(2)	C(32)	426(6)	3736(6)	3315(8)	103(4)
C(7)	1265(5)	2654(4)	739(6)	52(2)	C(33)	582(8)	3258(9)	3839(11)	145(7)
C(8)	1067(7)	2239(5)	1287(7)	87(4)	C(34)	168(10)	2778(8)	3848(11)	132(6)
C(9)	1477(8)	1740(5)	1086(8)	100(4)	C(35)	-405(9)	2774(6)	3391(9)	114(5)
C(10)	1880(6)	1890(5)	464(8)	83(4)	C(36)	-550(6)	3262(5)	2892(7)	83(3)
C(11)	-1103(4)	3254(4)	289(5)	43(2)					

Table 4 Selected bond distances (nm) and angles (°) for compound **1**

Sn(1)—O(2)	0.2078(6)	Sn(2)—C(26)	0.2163(9)
Sn(1)—O(3)	0.2091(6)	Sn(2)—O(5)	0.2139(7)
Sn(1)—O(1)	0.2083(6)	Sn(2)—O(8A)[a]	0.2150(7)
Sn(1)—O(4)	0.2124(7)	Sn(3)—C(33)	0.2135(9)
Sn(1)—O(6)	0.2150(7)	Sn(3)—O(1A)	0.2080(6)
Sn(1)—C(19)	0.2155(9)	Sn(3)—O(3)	0.2068(6)
Sn(2)—O(2A)	0.2081(7)	Sn(3)—O(2)	0.2094(6)
Sn(2)—O(1)	0.2098(6)	Sn(3)—O(7)	0.2129(7)
Sn(2)—O(3)	0.2095(6)	Sn(3)—O(9)	0.2138(7)
C(1)—C(2)	0.1472(14)	N(1)—C(2)	0.1419(14)

Continued

C(2)—C(3)	0.1392(14)	N(1)—C(6)	0.1433(15)
C(3)—C(4)	0.1376(15)	O(6)—C(7)	0.1256(11)
O(4)—C(1)	0.1252(12)	O(7)—C(7)	0.1268(11)
O(5)—C(1)	0.1275(11)	O(8)—C(13)	0.1258(12)
O(2)-Sn(1)-O(1)	103.5(2)	O(3)-Sn(3)-O(1A)	104.1(2)
O(2)-Sn(1)-O(3)	78.1(2)	O(3)-Sn(3)-O(2)	78.2(2)
O(3)-Sn(1)-O(1)	78.7(2)	O(1A)-Sn(3)-O(2)	78.2(2)
O(2)-Sn(1)-O(4)	160.7(3)	O(3)-Sn(3)-O(7)	85.2(2)
O(1)-Sn(1)-O(4)	86.3(3)	O(1A)-Sn(3)-O(7)	162.5(2)
O(3)-Sn(1)-O(4)	87.9(2)	O(2)-Sn(3)-O(7)	89.5(2)
O(2)-Sn(1)-O(6)	87.5(3)	O(3)-Sn(3)-O(9)	156.6(2)
O(1)-Sn(1)-O(6)	160.6(2)	O(1A)-Sn(3)-O(9)	88.8(3)
O(3)-Sn(1)-O(6)	88.2(2)	O(2)-Sn(3)-O(9)	85.6(2)
O(4)-Sn(1)-O(6)	78.8(3)	O(7)-Sn(3)-O(9)	77.8(3)
O(2)-Sn(1)-C(19)	98.3(3)	C(33)-Sn(3)-O(9)	94.4(3)
O(1)-Sn(1)-C(19)	101.8(3)	O(3)-Sn(3)-C(33)	103.3(3)
O(3)-Sn(1)-C(19)	176.3(3)	O(1A)-Sn(3)-C(33)	96.3(3)
O(4)-Sn(1)-C(19)	95.8(4)	O(2)-Sn(3)-C(33)	174.5(3)
O(6)-Sn(1)-C(19)	92.3(3)	O(7)-Sn(3)-C(33)	95.9(3)
O(2A)-Sn(2)-O(3)	103.5(2)	Sn(3A)-O(1)-Sn(1)	133.6(3)
O(2A)-Sn(2)-O(1)	78.1(2)	Sn(3A)-O(1)-Sn(2)	99.8(3)
O(3)-Sn(2)-O(1)	78.3(2)	Sn(1)-O(1)-Sn(2)	99.5(3)
O(2A)-Sn(2)-O(5)	157.5(3)	Sn(1)-O(2)-Sn(2A)	133.9(3)
O(3)-Sn(2)-O(5)	89.6(2)	Sn(1)-O(2)-Sn(3)	99.6(3)
O(1)-Sn(2)-O(5)	86.9(3)	Sn(2A)-O(2)-Sn(3)	99.9(3)
O(2A)-Sn(2)-O(8A)	84.2(3)	Sn(3)-O(3)-Sn(1)	100.0(2)
O(3)-Sn(2)-O(8A)	165.6(2)	Sn(3)-O(3)-Sn(2)	133.4(3)
O(1)-Sn(2)-O(8A)	91.6(2)	Sn(1)-O(3)-Sn(2)	99.3(2)
O(5)-Sn(2)-O(8A)	79.4(3)	C(1)-O(4)-Sn(1)	131.9(7)
O(2A)-Sn(2)-C(26)	103.8(4)	C(1)-O(5)-Sn(2)	128.5(7)
O(3)-Sn(2)-C(26)	92.9(3)	C(7)-O(6)-Sn(1)	128.6(7)
O(1)-Sn(2)-C(26)	171.2(3)	C(7)-O(7)-Sn(3)	130.7(7)
O(6)-Sn(2)-C(26)	93.6(4)	C(13)-O(8)-Sn(2A)	126.5(7)
O(8A)-Sn(2)-C(26)	97.1(3)	C(13)-O(8)-Sn(3)	127.2(7)
C(27)-C(26)-Sn(2)	114.5(7)	C(20)-C(19)-Sn(1)	112.8(7)
C(34)-C(33)-Sn(3)	114.0(7)	O(4)-C(11)-O(5)	126.0(6)

[a] Symmetry transformation used to generate equivalent atoms.

Table 5 Selected bond distances (nm) and angles (°) for compound **2**

Sn(1)—O(11)	0.2076(5)	Sn(2)-C(23)	0.2159(8)
Sn(1)—O(12)	0.2094(5)	Sn(2)—O(2)	0.2151(5)
Sn(1)—O(10)	0.2104(5)	Sn(2)—O(7)	0.2161(6)
Sn(1)—C(16)	0.2151(8)	Sn(3)—C(30)	0.2164(8)
Sn(1)—O(1)	0.2146(6)	Sn(3)—O(11A)[a]	0.2101(5)
Sn(1)—O(4)	0.2154(5)	Sn(3)—O(12)	0.2079(5)
Sn(2)—O(12A)	0.2081(5)	Sn(3)—O(10)	0.2096(5)
Sn(2)—O(10)	0.2081(5)	Sn(3)—O(5)	0.2147(5)
Sn(2)—O(11)	0.2099(5)	Sn(3)—O(8)	0.2155(6)
C(1)—C(2)	0.1467(12)	O(3)—C(2)	0.1364(11)
C(2)—C(3)	0.1340(12)	O(3)—C(5)	0.1419(13)
C(3)—C(4)	0.1403(15)	O(4)—C(6)	0.1250(9)

Continued

O(1)—C(1)	0.1265(10)	O(5)—C(6)	0.1253(9)
O(2)—C(1)	0.1249(10)	O(6)—C(7)	0.1361(10)
O(11)-Sn(1)-O(12)	104.36(19)	O(12)-Sn(3)-O(10)	78.22(19)
O(11)-Sn(1)-O(10)	78.12(19)	O(12)-Sn(3)-O(11A)	77.63(18)
O(12)-Sn(1)-O(10)	77.72(18)	O(10)-Sn(3)-O(11A)	103.9(2)
O(11)-Sn(1)-O(1)	86.3(2)	O(12)-Sn(3)-O(5)	86.4(2)
O(12)-Sn(1)-O(1)	160.1(2)	O(11A)-Sn(3)-O(5)	160.5(2)
O(10)-Sn(1)-O(1)	88.3(2)	O(12)-Sn(3)-O(8)	86.7(2)
O(11)-Sn(1)-C(16)	102.0(3)	O(10)-Sn(3)-O(8)	158.7(2)
O(12)-Sn(1)- C(16)	96.1(3)	O(11A)-Sn(3)-O(8)	87.2(2)
O(10)-Sn(1)- C(16)	173.5(3)	O(5)-Sn(3)-O(8)	78.2(2)
O(1)-Sn(1)- C(16)	98.1(3)	O(12)-Sn(3)-C(30)	173.8(3)
O(11)-Sn(1)-O(4)	161.0(2)	O(10)-Sn(3)-C(30)	104.1(3)
O(12)-Sn(1)-O(4)	86.0(2)	O(11A)-Sn(3)-C(30)	96.1(3)
O(10)-Sn(1)-O(4)	88.8(2)	O(5)-Sn(3)-C(30)	97.3(3)
O(1)-Sn(1)-O(4)	79.5(2)	O(8)-Sn(3)-C(30)	92.5(3)
C(16)-Sn(1)-O(4)	92.6(3)	Sn(2)-O(10)-Sn(3)	133.4(3)
O(12A)-Sn(2)-O(10)	103.73(19)	Sn(2)-O(10)-Sn(1)	99.9(2)
O(12A)-Sn(2)-O(11)	77.64(18)	Sn(3)-O(10)-Sn(1)	99.6(2)
O(10)-Sn(2)-O(11)	78.13(19)	Sn(1)-O(11)-Sn(2)	100.2(2)
O(12A)-Sn(2)-O(2)	159.0(2))	Sn(1)-O(11)-Sn(3A)	132.5(3)
O(10)-Sn(2)-O(2)	87.1(2)	Sn(2)-O(11)-Sn(3A)	99.7(2)
O(11)-Sn(2)-O(2)	87.2(2)	Sn(3)-O(12)-Sn(2A)	101.1(2)
O(12A)-Sn(2)-C(23)	97.9(3)	Sn(3)-O(12)-Sn(1)	100.4(2)
O(10)-Sn(2)- C(23)	102.6(3)	Sn(2A)-O(12)-Sn(1)	132.8(3)
O(11)-Sn(2)- C(23)	175.5(3)	C(17)-C(16)-Sn(1)	116.6(6)
O(2)-Sn(2)- C(23)	97.3(3)	C(24)-C(23)-Sn(2)	114.0(6)
O(12A)-Sn(2)-O(7)	85.7(2)	C(30)-C(30)-Sn(3)	114.7(6)
O(10)-Sn(2)-O(7)	162.5(2)	O(2)-C(1)-O(1)	127.2(8)
O(11)-Sn(2)-O(7)	89.8(2)	O(4)-C(6)-O(5)	127.3(8)
O(2)-Sn(2)-O(7)	79.6(2)	O(7A)-C(11)-O(8)	125.9(8)
C(23)-Sn(2)-O(7)	90.5(3)		

[a] Symmetry transformation used to generate equivalent atoms.

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