Crystal structure of bis(μ_3-oxo)-bis[μ_2-2-(2-formylphenoxy)acetato-*O*,*O'*]-bis[μ_2-2-(2-formylphenoxy)acetato-*O*,*O'*]-octakis(*n*-butyl)tetratin(IV), $Sn_4O_2(C_9H_7O_4)_4(C_4H_9)_8$

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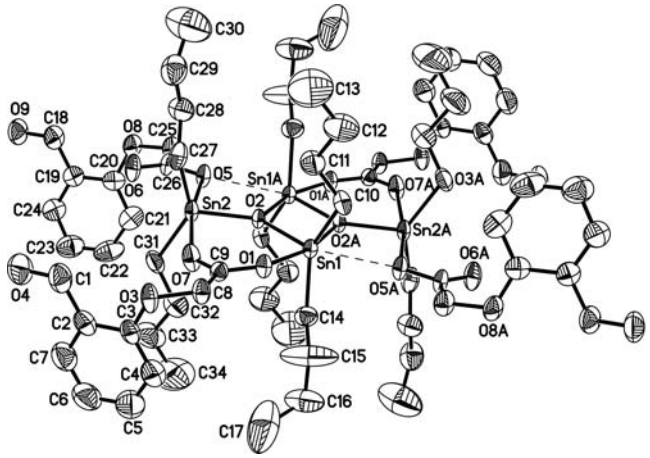

Abstract

$C_{68}H_{100}O_{18}Sn_4$, triclinic, $P\bar{1}$ (no. 2), a = 11.680(7) Å, b = 12.749(8) Å, c = 13.991(9) Å, α = 100.44(1)°, β = 105.20(1)°, γ = 98.87(1)°, V = 1932.0 Å^3, Z = 4, $R_{gt}(F)$ = 0.066, $wR_{ref}(F^2)$ = 0.200, T = 273 K.

Source of material

The chemicals and solvents were procured from commercial sources and used as received without further purification. The title compound was prepared by reaction of di-*n*-butyltin oxide (0.248 g, 1.0 mmol) and 2-(2-formylphenoxy)acetic acid (0.180 g, 1.0 mmol) in absolute benzene (30 ml) under reflux for 7 h, then the solvent was evaporated under vacuum. The residue was recrystallized from dichloromethane/hexane to give fine crystals suitable for X-ray diffraction measurement.

Discussion

The title crystal structure possesses tetranuclear centrosymmetric dimeric structure with a four-membered planar Sn_2O_2 ring where d(Sn—O) varies from 2.041 to 2.291 Å, which are similar to the corresponding distances in the compounds of di-*n*-butyltin(IV) complexes reported [1]. The four carboxylate ligands are divided into two different types according to their coordinating mode. Two of them are bidentate and connect with each of *exo*- and *endo*-cyclic tin atoms by using both oxygen atoms, whereas the other two are bidentate with one oxygen atom bridging to both of the *exo*- and *endo*-cyclic tin atoms.

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Table 1. Data collection and handling.

Crystal:	colorless block, size 0.15 × 0.21 × 0.25 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	13.39 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	50.1°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	9748, 6687
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 4634
$N(param)_{refined}$:	410
Programs:	SHELXS-97, SHELXL-97 [2]

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

Atom	Site	x	y	z	U_{iso}
H(1)	2*i*	0.6555	0.4489	0.5887	0.139
H(4)	2*i*	0.6058	0.1641	0.7450	0.147
H(5)	2*i*	0.6925	0.2461	0.9164	0.178
H(6)	2*i*	0.7655	0.4261	0.9711	0.181
H(7)	2*i*	0.7701	0.5308	0.8562	0.161
H(8A)	2*i*	0.5170	0.1317	0.4763	0.084
H(8B)	2*i*	0.5385	0.1019	0.5827	0.084
H(10A)	2*i*	0.7906	–0.1660	0.3152	0.097
H(10B)	2*i*	0.6736	–0.1517	0.3462	0.097
H(11A)	2*i*	0.8094	0.0097	0.2824	0.131
H(11B)	2*i*	0.6890	0.0203	0.3098	0.131
H(12A)	2*i*	0.7006	–0.1386	0.1421	0.157
H(12B)	2*i*	0.5801	–0.1315	0.1704	0.157
H(13A)	2*i*	0.6254	0.0582	0.1475	0.265
H(13B)	2*i*	0.5582	–0.0378	0.0520	0.265
H(13C)	2*i*	0.6982	0.0071	0.0783	0.265
H(14A)	2*i*	0.9333	–0.1132	0.6624	0.126
H(14B)	2*i*	0.9207	0.0067	0.6933	0.126
H(15A)	2*i*	0.7397	–0.1744	0.6531	0.222
H(15B)	2*i*	0.7213	–0.0546	0.6777	0.222
H(16A)	2*i*	0.7867	–0.1688	0.8143	0.208
H(16B)	2*i*	0.9068	–0.0851	0.8293	0.208
H(17A)	2*i*	0.8349	0.0546	0.8841	0.313
H(17B)	2*i*	0.7732	–0.0351	0.9287	0.313
H(17C)	2*i*	0.6996	–0.0062	0.8295	0.313
H(18)	2*i*	0.5402	0.3132	0.4000	0.121
H(21)	2*i*	0.6310	0.5993	0.2548	0.125
H(22)	2*i*	0.5824	0.5178	0.0837	0.167
H(23)	2*i*	0.5266	0.3299	0.0258	0.161
H(24)	2*i*	0.4854	0.2276	0.1361	0.124
H(25A)	2*i*	0.6540	0.6328	0.5127	0.106
H(25B)	2*i*	0.6252	0.6587	0.4045	0.106
H(27A)	2*i*	1.1623	0.6422	0.5760	0.104
H(27B)	2*i*	1.1982	0.7666	0.6292	0.104
H(28A)	2*i*	1.0150	0.7465	0.6790	0.134
H(28B)	2*i*	0.9897	0.6204	0.6331	0.134
H(29A)	2*i*	1.1598	0.6034	0.7507	0.154
H(29B)	2*i*	1.2032	0.7301	0.7893	0.154

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(30A)	2*i*	1.0908	0.7402	0.8870	0.249
H(30B)	2*i*	1.1050	0.6196	0.8846	0.249
H(30C)	2*i*	0.9878	0.6431	0.8143	0.249
H(31A)	2*i*	1.0203	0.6086	0.2896	0.125
H(31B)	2*i*	0.8902	0.6287	0.2832	0.125
H(32A)	2*i*	1.0696	0.7625	0.2370	0.162

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(32B)	2*i*	0.9463	0.7944	0.2409	0.162
H(33A)	2*i*	0.9719	0.6181	0.0977	0.179
H(33B)	2*i*	0.8442	0.6319	0.1074	0.179
H(34A)	2*i*	0.8713	0.7964	0.0583	0.282
H(34B)	2*i*	0.8756	0.6991	–0.0262	0.282
H(34C)	2*i*	0.9964	0.7783	0.0448	0.282

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*	0.85879(5)	–0.04856(4)	0.49928(4)	0.0442(4)	0.0377(3)	0.0647(4)	0.0087(2)	0.0169(3)	0.0068(2)
Sn(2)	2*i*	1.03455(5)	0.74259(4)	0.45621(5)	0.0504(4)	0.0349(3)	0.0888(5)	0.0110(3)	0.0251(3)	0.0031(3)
O(1)	2*i*	0.7013(5)	0.0372(4)	0.5069(5)	0.057(4)	0.042(3)	0.084(4)	0.015(3)	0.015(3)	0.007(3)
O(2)	2*i*	0.9737(5)	0.0964(4)	0.5151(4)	0.052(3)	0.031(3)	0.070(4)	0.008(2)	0.019(3)	0.004(2)
O(3)	2*i*	0.5733(6)	0.2609(5)	0.5922(6)	0.061(4)	0.057(4)	0.114(6)	0.026(3)	0.013(4)	–0.004(4)
O(4)	2*i*	0.721(1)	0.5818(8)	0.6813(8)	0.19(1)	0.071(6)	0.149(9)	–0.020(7)	0.063(8)	–0.017(6)
O(5)	2*i*	0.8514(5)	0.7310(4)	0.4735(5)	0.052(3)	0.038(3)	0.111(5)	0.011(3)	0.028(3)	0.010(3)
O(6)	2*i*	0.8465(6)	0.5543(5)	0.4372(6)	0.063(4)	0.055(4)	0.143(6)	0.026(3)	0.024(4)	0.012(4)
O(7)	2*i*	1.2203(6)	0.7859(5)	0.4306(6)	0.060(4)	0.056(4)	0.149(7)	–0.002(3)	0.055(4)	–0.011(4)
O(8)	2*i*	0.6000(6)	0.4995(6)	0.3994(6)	0.063(4)	0.062(4)	0.117(6)	–0.014(3)	0.029(4)	0.009(4)
O(9)	2*i*	0.508(1)	0.1802(7)	0.3083(8)	0.140(8)	0.072(6)	0.130(7)	–0.002(6)	0.025(6)	–0.003(5)
C(1)	2*i*	0.684(1)	0.482(1)	0.658(1)	0.11(1)	0.09(1)	0.14(1)	0.011(9)	0.043(9)	0.000(9)
C(2)	2*i*	0.680(1)	0.412(1)	0.7292(9)	0.074(7)	0.092(9)	0.087(8)	0.017(6)	0.032(6)	–0.005(7)
C(3)	2*i*	0.632(1)	0.3019(9)	0.6958(9)	0.065(6)	0.075(7)	0.088(7)	0.026(6)	0.032(6)	0.006(6)
C(4)	2*i*	0.637(2)	0.239(1)	0.766(1)	0.13(1)	0.11(1)	0.13(1)	0.04(1)	0.05(1)	0.02(1)
C(5)	2*i*	0.689(2)	0.289(2)	0.869(1)	0.19(2)	0.15(2)	0.10(1)	0.07(2)	0.02(1)	0.02(1)
C(6)	2*i*	0.734(2)	0.395(2)	0.902(1)	0.18(2)	0.14(2)	0.10(1)	0.01(1)	0.05(1)	–0.02(1)
C(7)	2*i*	0.734(2)	0.457(2)	0.834(1)	0.15(2)	0.12(1)	0.13(1)	0.01(1)	0.06(1)	–0.00(1)
C(8)	2*i*	0.5706(8)	0.1505(7)	0.5456(8)	0.049(5)	0.052(6)	0.107(8)	0.014(4)	0.028(5)	0.007(5)
C(9)	2*i*	0.6942(8)	0.1348(7)	0.5446(7)	0.055(5)	0.055(5)	0.072(6)	0.017(5)	0.025(4)	0.015(4)
C(10)	2*i*	0.752(1)	–0.1129(8)	0.3466(7)	0.077(5)	0.073(4)	0.088(4)	0.019(4)	0.025(4)	0.002(3)
C(11)	2*i*	0.732(1)	–0.031(1)	0.2817(8)	0.111(6)	0.103(5)	0.114(4)	0.030(4)	0.031(4)	0.024(4)
C(12)	2*i*	0.657(2)	–0.089(1)	0.1708(9)	0.133(6)	0.138(6)	0.114(4)	0.027(5)	0.025(4)	0.032(4)
C(13)	2*i*	0.633(2)	–0.008(1)	0.106(1)	0.190(8)	0.179(7)	0.161(6)	0.041(5)	0.033(4)	0.067(5)
C(14)	2*i*	0.878(1)	–0.064(1)	0.6499(9)	0.103(5)	0.098(5)	0.108(5)	0.035(4)	0.014(4)	0.023(4)
C(15)	2*i*	0.780(2)	–0.100(2)	0.690(1)	0.178(7)	0.205(8)	0.180(6)	0.024(5)	0.065(5)	0.064(5)
C(16)	2*i*	0.819(2)	–0.098(2)	0.805(1)	0.175(7)	0.190(7)	0.165(6)	0.031(5)	0.066(5)	0.049(5)
C(17)	2*i*	0.778(2)	–0.014(2)	0.867(1)	0.212(8)	0.207(8)	0.207(7)	0.052(5)	0.065(5)	0.037(4)
C(18)	2*i*	0.531(1)	0.280(1)	0.333(1)	0.084(8)	0.076(8)	0.12(1)	–0.012(7)	0.020(7)	–0.001(7)
C(19)	2*i*	0.5446(9)	0.348(1)	0.263(1)	0.054(6)	0.095(9)	0.099(9)	0.008(6)	0.021(6)	0.023(7)
C(20)	2*i*	0.5837(9)	0.461(1)	0.2989(9)	0.051(6)	0.105(9)	0.076(7)	0.006(6)	0.012(5)	0.023(7)
C(21)	2*i*	0.602(1)	0.524(1)	0.232(1)	0.069(8)	0.12(1)	0.12(1)	0.015(7)	0.011(8)	0.039(9)
C(22)	2*i*	0.575(1)	0.475(2)	0.130(1)	0.11(1)	0.20(2)	0.11(1)	0.01(1)	–0.002(9)	0.08(1)
C(23)	2*i*	0.538(2)	0.363(2)	0.094(1)	0.12(1)	0.16(2)	0.11(1)	0.01(1)	0.02(1)	0.03(1)
C(24)	2*i*	0.518(1)	0.302(1)	0.160(1)	0.091(9)	0.12(1)	0.094(9)	0.017(8)	0.016(8)	0.018(8)
C(25)	2*i*	0.6625(9)	0.6119(9)	0.445(1)	0.060(6)	0.070(7)	0.13(1)	0.001(5)	0.036(7)	0.009(7)
C(26)	2*i*	0.7968(8)	0.6287(7)	0.4526(7)	0.049(5)	0.035(4)	0.093(7)	0.008(4)	0.014(5)	0.009(4)
C(27)	2*i*	1.130(1)	0.7063(9)	0.5930(8)	0.079(5)	0.073(5)	0.103(4)	0.028(4)	0.014(4)	0.016(4)
C(28)	2*i*	1.053(1)	0.685(1)	0.6654(9)	0.106(5)	0.111(6)	0.121(5)	0.035(4)	0.031(4)	0.024(4)
C(29)	2*i*	1.132(1)	0.671(1)	0.7643(9)	0.133(6)	0.127(6)	0.125(5)	0.032(5)	0.029(4)	0.040(5)
C(30)	2*i*	1.074(2)	0.668(2)	0.844(1)	0.174(7)	0.179(7)	0.153(5)	0.034(5)	0.057(5)	0.046(5)
C(31)	2*i*	0.975(1)	0.665(1)	0.3000(9)	0.100(5)	0.096(5)	0.112(4)	0.021(4)	0.040(4)	0.000(4)
C(32)	2*i*	0.984(2)	0.733(1)	0.227(1)	0.136(6)	0.131(6)	0.140(5)	0.027(5)	0.047(5)	0.027(4)
C(33)	2*i*	0.925(2)	0.671(1)	0.115(1)	0.153(7)	0.154(6)	0.137(5)	0.022(5)	0.045(5)	0.029(4)
C(34)	2*i*	0.916(2)	0.743(2)	0.042(1)	0.203(8)	0.191(8)	0.170(6)	0.033(5)	0.050(5)	0.057(5)

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