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Crystal structure of bis[μ_2-benzofuran-2-carboxylato-*O*,*O'*]-bis[μ_2-benzofuran-2-carboxylato-*O*]-octakis(*n*-butyl)-bis(μ_3-oxo)tetratin(IV), $Sn_4O_2(C_4H_9)_8(C_9H_5O_3)_4$

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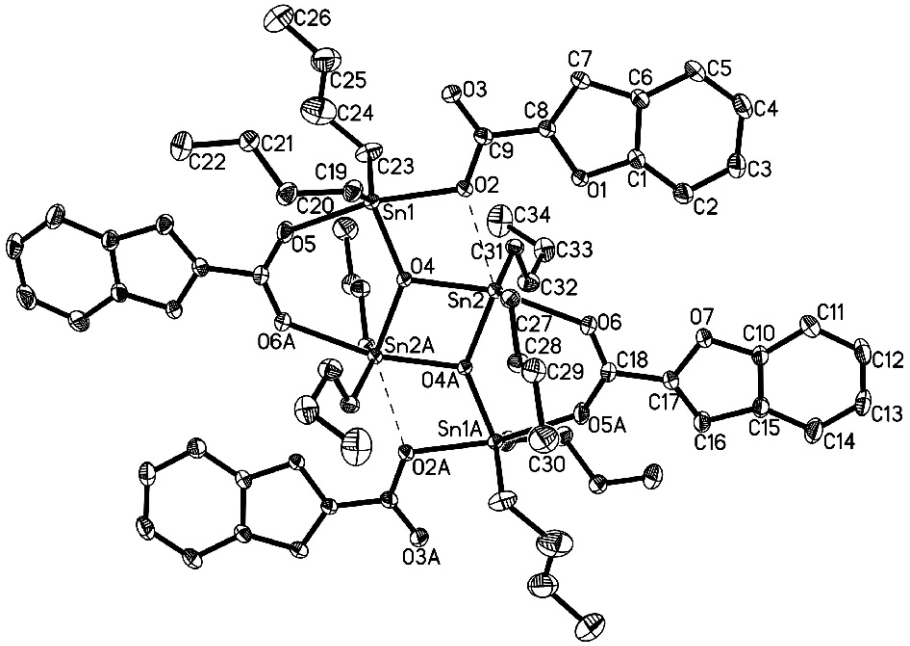

Abstract

$C_{68}H_{92}O_{14}Sn_4$, triclinic, $P\bar{1}$ (no. 2), a = 10.5518(4) Å, b = 12.2122(5) Å, c = 13.6872(5) Å, α = 100.414(1)°, β = 94.248(1)°, γ = 90.322(1)°, V = 1729.6 Å^3, Z = 1, $R_{gt}(F)$ = 0.023, $wR_{ref}(F^2)$ = 0.064, T = 200 K.

Source of material

The chemicals and solvents were commercially available and used without further purification. Di-*n*-butyltin oxide (0.249 g, 1.0 mmol) and benzofuran- 2-carboxylic acid (0.162 g, 1.0 mmol) in toluene (30 mL) were refluxed for 8 h under azeotropic removal of water by a Dean-Stark apparatus. After cooling down to room temperature, the solution was filtered. The filtrate was gradually removed by evaporation under vacuum until solid product was obtained. X-ray quality colorless crystals were obtained by recrystallization from ethanol.

Discussion

The title crystal structure shows a typical organotin ladder framework. The central motif is a four-membered planar Sn_2O_2 ring, which is similar to the corresponding complex [1,2]. The two oxygen atoms O4 and O4A are triply bridging, each linking one exo-cyclic and two endo-cyclic $SnBu_2$ groups. The coordinating fashions of the carboxylate ligands are of two different types. One kind is bidentate bridging, the carboxylate ligand connects the exo- and endo-cyclic tin atoms with both oxygen atoms. The other one is monodentate, but one oxygen atom bridges one exo- and one endo-cyclic tin atom. As a consequence, the exo- and endo-cyclic tin atoms are five-coordinated and six-coordinated, respectively.

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Table 1. Data collection and handling.

Crystal:	colorless block, size 0.28 × 0.34 × 0.36 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	14.88 cm^{-1}
Diffractometer, scan mode:	Bruker Apex CCD, φ/ω
$2\theta_{max}$:	52.2°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	11429, 6768
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 6073
$N(param)_{refined}$:	387
Programs:	SHELXS-97, SHELXL-97, SHELXTL [3]

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

Atom	Site	x	y	z	U_{iso}
H(2)	2*i*	0.2540	0.6073	0.7031	0.050
H(3)	2*i*	0.1559	0.4462	0.6073	0.060
H(4)	2*i*	–0.0096	0.3573	0.6645	0.063
H(5)	2*i*	–0.0846	0.4222	0.8195	0.056

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(7)	2*i*	–0.0365	0.6094	0.9959	0.042
H(11)	2*i*	0.2187	0.5396	0.4593	0.067
H(12)	2*i*	0.2657	0.4512	0.2994	0.073
H(13)	2*i*	0.4562	0.4854	0.2367	0.070
H(14)	2*i*	0.6050	0.6108	0.3267	0.066
H(16)	2*i*	0.6454	0.7656	0.5259	0.046
H(19A)	2*i*	0.1346	1.1044	1.0865	0.045
H(19B)	2*i*	0.0600	1.0361	1.1535	0.045
H(20A)	2*i*	0.1976	1.2373	1.2332	0.043
H(20B)	2*i*	0.0485	1.2329	1.2018	0.060
H(21A)	2*i*	0.1628	1.1306	1.3573	0.051
H(21B)	2*i*	0.0134	1.1278	1.3263	0.051
H(22A)	2*i*	0.0629	1.2669	1.4691	0.087
H(22B)	2*i*	0.0068	1.3235	1.3788	0.087
H(22C)	2*i*	0.1566	1.3276	1.4081	0.087
H(23A)	2*i*	0.3304	0.7955	1.2293	0.060
H(23B)	2*i*	0.4576	0.8681	1.2639	0.060
H(24A)	2*i*	0.3742	0.8512	1.4127	0.109
H(24B)	2*i*	0.3391	0.9754	1.3996	0.109
H(25A)	2*i*	0.1695	0.7908	1.3439	0.097
H(25B)	2*i*	0.1353	0.9171	1.3377	0.097

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(26A)	2*i*	0.0602	0.8679	1.4809	0.157
H(26B)	2*i*	0.1652	0.9656	1.5114	0.157
H(26C)	2*i*	0.2017	0.8400	1.5182	0.157
H(27A)	2*i*	0.1995	0.9125	0.8241	0.039
H(27B)	2*i*	0.2103	1.0169	0.9136	0.039
H(28A)	2*i*	0.3483	1.0020	0.7401	0.044
H(28B)	2*i*	0.3513	1.1077	0.8283	0.044
H(29A)	2*i*	0.1351	1.1404	0.7863	0.054
H(29B)	2*i*	0.1299	1.0330	0.6994	0.054
H(30A)	2*i*	0.1560	1.1956	0.6320	0.097
H(30B)	2*i*	0.2781	1.1195	0.6147	0.097
H(30C)	2*i*	0.2843	1.2267	0.7017	0.097
H(31A)	2*i*	0.4811	0.7573	1.0475	0.038
H(31B)	2*i*	0.4240	0.6866	0.9439	0.038
H(32A)	2*i*	0.6765	0.7768	0.9813	0.052
H(32B)	2*i*	0.6200	0.7134	0.8741	0.052
H(33A)	2*i*	0.5901	0.5494	0.9466	0.062
H(33B)	2*i*	0.7359	0.5835	0.9424	0.062
H(34A)	2*i*	0.7028	0.5377	1.0969	0.121
H(34B)	2*i*	0.5992	0.6329	1.1155	0.121
H(34C)	2*i*	0.7454	0.6659	1.1112	0.121

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*	0.29979(2)	0.98754(1)	1.17253(1)	0.02508(9)	0.0284(1)	0.02384(9)	–0.00307(7)	0.00575(7)	0.00183(7)
Sn(2)	2*i*	0.41222(2)	0.89683(1)	0.93092(1)	0.02530(9)	0.02309(9)	0.02434(9)	–0.00376(6)	0.00314(7)	0.00097(7)
O(1)	2*i*	0.1826(2)	0.7007(2)	0.8837(1)	0.0322(9)	0.0295(9)	0.032(1)	–0.0078(7)	0.0090(8)	–0.0017(8)
O(2)	2*i*	0.2358(2)	0.8611(2)	1.0427(1)	0.034(1)	0.0314(9)	0.032(1)	–0.0115(8)	0.0099(8)	–0.0035(8)
O(3)	2*i*	0.0786(2)	0.8055(2)	1.1232(2)	0.056(1)	0.054(1)	0.043(1)	–0.022(1)	0.025(1)	–0.009(1)
O(4)	2*i*	0.4287(2)	1.0057(1)	1.0744(1)	0.0263(8)	0.0279(9)	0.0232(8)	–0.0053(7)	0.0069(7)	–0.0002(7)
O(5)	2*i*	0.3938(2)	1.1213(2)	1.2838(1)	0.038(1)	0.060(1)	0.032(1)	–0.017(1)	0.0058(8)	–0.0131(9)
O(6)	2*i*	0.4352(2)	0.8041(2)	0.7677(1)	0.047(1)	0.037(1)	0.029(1)	–0.0080(9)	0.0105(8)	–0.0035(8)
O(7)	2*i*	0.3802(2)	0.6834(2)	0.5818(1)	0.044(1)	0.039(1)	0.031(1)	–0.0152(9)	0.0052(8)	–0.0047(8)
C(1)	2*i*	0.1381(2)	0.6086(2)	0.8169(2)	0.031(1)	0.025(1)	0.035(1)	–0.002(1)	–0.001(1)	0.001(1)
C(2)	2*i*	0.1856(3)	0.5705(2)	0.7261(2)	0.040(2)	0.039(2)	0.044(2)	–0.004(1)	0.009(1)	–0.000(1)
C(3)	2*i*	0.1273(3)	0.4757(3)	0.6708(2)	0.060(2)	0.041(2)	0.042(2)	–0.002(2)	0.006(2)	–0.009(1)
C(4)	2*i*	0.0282(3)	0.4222(3)	0.7053(3)	0.060(2)	0.036(2)	0.055(2)	–0.013(1)	–0.001(2)	–0.008(1)
C(5)	2*i*	–0.0172(3)	0.4602(2)	0.7965(2)	0.047(2)	0.037(2)	0.053(2)	–0.017(1)	0.001(1)	0.001(1)
C(6)	2*i*	0.0395(3)	0.5570(2)	0.8546(2)	0.033(1)	0.029(1)	0.039(2)	–0.005(1)	0.001(1)	0.003(1)
C(7)	2*i*	0.0236(3)	0.6225(2)	0.9508(2)	0.033(1)	0.034(1)	0.038(2)	–0.010(1)	0.005(1)	0.004(1)
C(8)	2*i*	0.1103(2)	0.7061(2)	0.9647(2)	0.031(1)	0.028(1)	0.032(1)	–0.004(1)	0.007(1)	0.002(1)
C(9)	2*i*	0.1405(2)	0.7964(2)	1.0501(2)	0.032(1)	0.032(1)	0.036(2)	–0.006(1)	0.008(1)	0.003(1)
C(10)	2*i*	0.3886(3)	0.6253(2)	0.4867(2)	0.051(2)	0.031(1)	0.028(1)	–0.006(1)	0.000(1)	0.001(1)
C(11)	2*i*	0.2970(3)	0.5533(3)	0.4331(3)	0.061(2)	0.056(2)	0.047(2)	–0.028(2)	–0.000(2)	–0.001(2)
C(12)	2*i*	0.3256(4)	0.5021(3)	0.3391(3)	0.084(3)	0.048(2)	0.044(2)	–0.024(2)	–0.012(2)	–0.006(2)
C(13)	2*i*	0.4393(4)	0.5231(3)	0.3013(2)	0.083(3)	0.055(2)	0.028(2)	–0.007(2)	0.002(2)	–0.011(1)
C(14)	2*i*	0.5278(3)	0.5963(3)	0.3541(2)	0.059(2)	0.065(2)	0.034(2)	–0.003(2)	0.010(2)	–0.010(2)
C(15)	2*i*	0.5022(3)	0.6500(2)	0.4501(2)	0.041(2)	0.038(2)	0.029(1)	–0.001(1)	0.001(1)	–0.001(1)
C(16)	2*i*	0.5655(3)	0.7302(3)	0.5278(2)	0.031(1)	0.049(2)	0.031(1)	–0.006(1)	0.003(1)	–0.005(1)
C(17)	2*i*	0.4908(2)	0.7459(2)	0.6038(2)	0.032(1)	0.030(1)	0.029(1)	–0.004(1)	–0.001(1)	0.001(1)
C(18)	2*i*	0.5110(2)	0.8145(2)	0.7043(2)	0.031(1)	0.029(1)	0.026(1)	0.003(1)	0.001(1)	0.000(1)
C(19)	2*i*	0.1356(3)	1.0845(2)	1.1535(2)	0.032(1)	0.045(2)	0.034(2)	0.005(1)	0.005(1)	0.001(1)
C(20)	2*i*	0.1193(3)	1.1908(2)	1.2279(2)	0.047(2)	0.038(2)	0.041(2)	0.010(1)	0.011(1)	0.006(1)
C(21)	2*i*	0.0925(3)	1.1729(3)	1.3308(2)	0.043(2)	0.044(2)	0.040(2)	–0.005(1)	0.011(1)	0.000(1)
C(22)	2*i*	0.0784(4)	1.2826(3)	1.4032(3)	0.069(2)	0.057(2)	0.046(2)	0.011(2)	0.009(2)	–0.002(2)
C(23)	2*i*	0.3637(3)	0.8703(3)	1.2616(2)	0.045(2)	0.061(2)	0.053(2)	0.013(2)	0.017(1)	0.030(2)
C(24)	2*i*	0.3225(5)	0.8956(4)	1.3714(4)	0.089(3)	0.098(4)	0.097(4)	–0.003(3)	–0.012(3)	0.056(3)
C(25)	2*i*	0.1873(5)	0.8694(4)	1.3756(3)	0.088(3)	0.076(3)	0.085(3)	–0.004(2)	0.026(3)	0.023(2)
C(26)	2*i*	0.1504(2)	0.8873(2)	1.4806(2)	0.127(5)	0.120(4)	0.069(3)	–0.023(4)	0.040(3)	0.009(3)
C(27)	2*i*	0.2578(2)	0.9719(2)	0.8614(2)	0.027(1)	0.038(1)	0.031(1)	–0.004(1)	0.001(1)	0.005(1)
C(28)	2*i*	0.2968(3)	1.0459(2)	0.7903(2)	0.033(1)	0.039(2)	0.038(2)	–0.001(1)	0.003(1)	0.010(1)
C(29)	2*i*	0.1856(3)	1.0943(3)	0.7367(2)	0.042(2)	0.049(2)	0.044(2)	0.003(1)	–0.003(1)	0.012(1)
C(30)	2*i*	0.2299(4)	1.1653(3)	0.6649(3)	0.078(3)	0.061(2)	0.058(2)	0.001(2)	–0.009(2)	0.028(2)
C(31)	2*i*	0.4827(2)	0.7490(2)	0.9743(2)	0.033(1)	0.028(1)	0.036(1)	–0.001(1)	0.008(1)	0.006(1)
C(32)	2*i*	0.6159(3)	0.7176(3)	0.9467(2)	0.034(2)	0.044(2)	0.052(2)	0.003(1)	0.009(1)	0.011(1)
C(33)	2*i*	0.6565(3)	0.6066(3)	0.9740(3)	0.043(2)	0.051(2)	0.057(2)	0.010(1)	–0.000(2)	0.002(2)
C(34)	2*i*	0.6778(5)	0.6111(4)	1.0839(3)	0.091(3)	0.077(3)	0.071(3)	0.029(2)	–0.018(2)	0.014(2)

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