

Crystal structure of bis(tetrakis(trimethylsilylmethyl)(μ -hydroxy)(*p*-nitrobenzoato)(μ^3 -oxo)ditin(IV)), [Sn₂O(OH)(NO₂C₆H₄COO){(CH₃)₃SiCH₂}₄]₂

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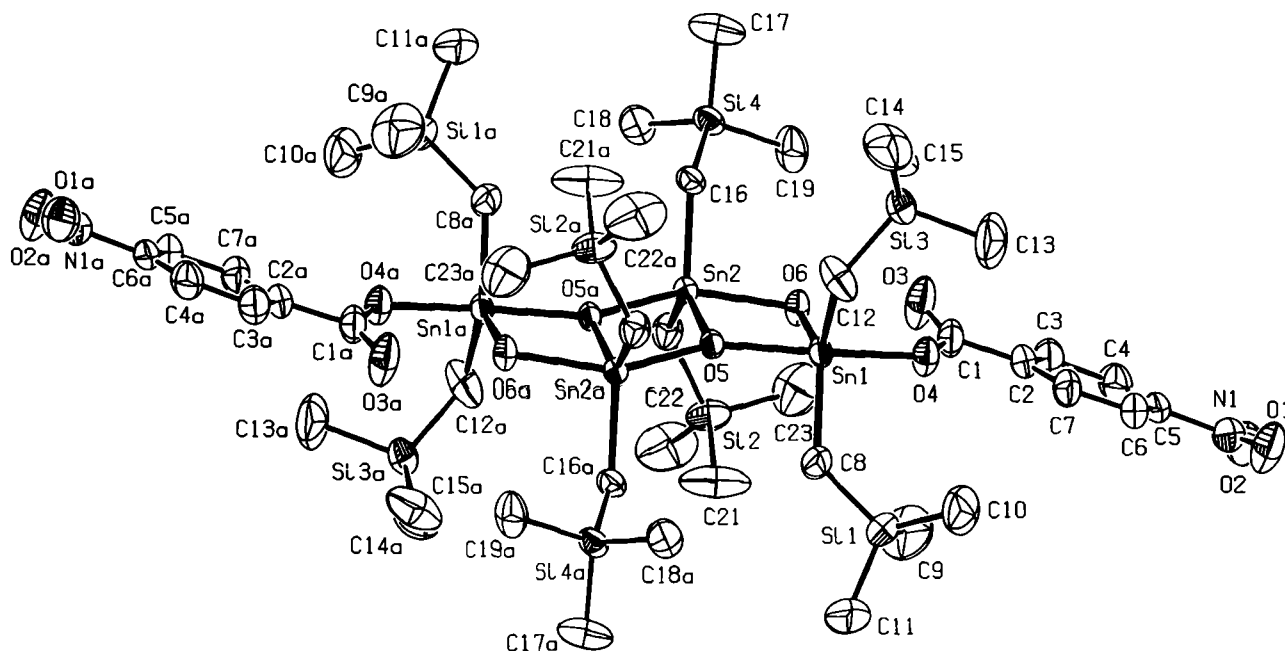
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Abstract

C₄₆H₉₈N₂O₁₂Si₈Sn₄, triclinic, $P\bar{1}$ (no. 2), $a = 12.2616(1)$ Å, $b = 12.4817(1)$ Å, $c = 14.1222(2)$ Å, $\alpha = 110.191(1)^\circ$, $\beta = 100.408(1)^\circ$, $\gamma = 106.679(1)^\circ$, $V = 1846.8$ Å³, $Z = 1$, $R_{\text{gt}}(F) = 0.036$, $wR_{\text{ref}}(F^2) = 0.104$, $T = 273$ K.

Source of material

The crystals were isolated by recrystallization from a toluene solution of the bulk material obtained from the reaction of bis(trimethylsilylmethyl)tin(IV) dichloride and 4-nitrobenzoic acid in an 1:2 molar ratio under reflux. The main product was bis(4-nitrobenzoato)bis(trimethylsilylmethyl)tin(IV) evidenced by supporting methods. In addition, a small number of crystals was isolated and characterized as the title compound (m.p. 531 K).

Experimental details

The H atom on O6 was located from the difference map and refined. The other H atoms were added geometrically.

Discussion

Diorganotin carboxylates have been the focus while owing to their anti-tumor activity [1]. Trimethylsilylmethyl compounds also show a wide range of biological activities [2]. To link the bio-

logical activities of organotin and organosilicon compounds, we prepared bis(*p*-nitrobenzoato)bis(trimethylsilylmethyl)tin(IV), whereas the title compound was also obtained.

The structure features a centrosymmetric Sn₂O₂ core. The carboxylate is monodentate and coordinates each *exo*-cyclic to the tin atoms. Each pair of *endo*- and *exo*-cyclic tin centers is also bridged by a hydroxyl group. Either *endo*- and *exo*-cyclic tin atoms are penta-coordinated with an environment of a distorted trigonal bipyramid. The coordination and the tin polyhedra are comparable to those in the structure of bis[tetraethyl- μ -hydroxyperfluorophenylacetato- μ -3-oxoditin] [3].

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.24 × 0.32 × 0.36 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	15.13 cm ⁻¹
Diffractometer, scan mode:	Bruker APEX II CCD, ϕ/ω
$2\theta_{\text{max}}$:	50°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	23197, 6488
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 5113
$N(\text{param})_{\text{refined}}$:	341
Programs:	SHELXS-97 [4], SHELXL-97 [5]

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Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U _{iso}
H(3)	2i	0.7339	0.8872	0.1737	0.097
H(4)	2i	0.8030	0.9211	0.0393	0.099
H(6)	2i	0.9990	0.7164	0.0517	0.087
H(7)	2i	0.9245	0.6753	0.1800	0.089
H(8A)	2i	0.6724	0.3356	0.2774	0.097
H(8B)	2i	0.5527	0.3511	0.2426	0.097
H(9A)	2i	0.5638	0.4914	0.1085	0.252
H(9B)	2i	0.5058	0.3742	0.0000	0.252
H(9C)	2i	0.6339	0.4707	0.0257	0.252
H(10A)	2i	0.8517	0.4587	0.1763	0.214
H(10B)	2i	0.8073	0.3939	0.0519	0.214
H(10C)	2i	0.8271	0.3182	0.1173	0.214
H(11A)	2i	0.5434	0.1606	-0.0378	0.302
H(11B)	2i	0.4857	0.1558	0.0518	0.302
H(11C)	2i	0.6048	0.1325	0.0532	0.302
H(12A)	2i	0.8429	0.6070	0.5598	0.119
H(12B)	2i	0.8987	0.5506	0.4742	0.119
H(13A)	2i	1.0203	0.7668	0.3946	0.256
H(13B)	2i	1.0981	0.7014	0.4344	0.256
H(13C)	2i	1.1421	0.8456	0.4866	0.256
H(14A)	2i	1.1747	0.8465	0.6981	0.214
H(14B)	2i	1.1220	0.7026	0.6567	0.214
H(14C)	2i	1.0669	0.7825	0.7304	0.214
H(15A)	2i	0.8825	0.8801	0.5326	0.225
H(15B)	2i	1.0109	0.9671	0.6121	0.225

Table 2. Continued.

Atom	Site	x	y	z	U _{iso}
H(15C)	2i	0.9142	0.8953	0.6504	0.225
H(16A)	2i	0.5994	0.7555	0.7045	0.065
H(16B)	2i	0.7011	0.7873	0.6548	0.065
H(17A)	2i	0.7310	1.1272	0.8257	0.187
H(17B)	2i	0.8068	1.0444	0.8061	0.187
H(17C)	2i	0.7114	1.0219	0.8648	0.187
H(18A)	2i	0.4550	0.9196	0.7523	0.137
H(18B)	2i	0.4050	0.8685	0.6285	0.137
H(18C)	2i	0.4610	1.0111	0.6985	0.137
H(19A)	2i	0.6510	1.0676	0.5927	0.145
H(19B)	2i	0.5825	0.9306	0.5083	0.145
H(19C)	2i	0.7205	0.9792	0.5651	0.145
H(20A)	2i	0.2866	0.6031	0.4330	0.083
H(20B)	2i	0.3651	0.7289	0.4386	0.083
H(21A)	2i	0.2697	0.3712	0.2180	0.247
H(21B)	2i	0.4050	0.4450	0.2377	0.247
H(21C)	2i	0.3086	0.4109	0.1316	0.247
H(22A)	2i	0.1323	0.5450	0.1406	0.251
H(22B)	2i	0.1472	0.6474	0.2501	0.251
H(22C)	2i	0.0968	0.5081	0.2295	0.251
H(23A)	2i	0.4404	0.6569	0.1865	0.230
H(23B)	2i	0.4558	0.7625	0.2943	0.230
H(23C)	2i	0.3491	0.7231	0.1934	0.230
H(1)	2i	0.638(6)	0.755(7)	0.395(5)	0.12(2)

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
C(1)	2i	0.7708(5)	0.7479(5)	0.2721(4)	0.084(4)	0.074(3)	0.087(4)	0.024(3)	0.052(3)	0.043(3)
C(2)	2i	0.8195(4)	0.7754(4)	0.1893(4)	0.063(3)	0.060(3)	0.070(3)	0.013(2)	0.029(2)	0.035(2)
C(3)	2i	0.7852(5)	0.8509(5)	0.1482(5)	0.084(4)	0.078(4)	0.095(4)	0.030(3)	0.045(3)	0.044(3)
C(4)	2i	0.8277(6)	0.8724(5)	0.0686(5)	0.093(4)	0.075(4)	0.087(4)	0.023(3)	0.032(3)	0.050(3)
C(5)	2i	0.9049(5)	0.8219(5)	0.0343(4)	0.070(3)	0.061(3)	0.055(3)	-0.001(2)	0.022(2)	0.024(2)
C(6)	2i	0.9444(5)	0.7487(5)	0.0753(4)	0.069(3)	0.076(3)	0.074(3)	0.017(3)	0.035(3)	0.036(3)
C(7)	2i	0.9002(5)	0.7253(5)	0.1522(4)	0.080(4)	0.076(3)	0.078(3)	0.023(3)	0.038(3)	0.044(3)
C(8)	2i	0.6376(6)	0.3845(5)	0.2511(4)	0.103(4)	0.060(3)	0.078(3)	0.024(3)	0.050(3)	0.022(3)
C(9)	2i	0.5795(9)	0.431(1)	0.0552(7)	0.18(1)	0.23(1)	0.119(7)	0.082(9)	0.026(7)	0.102(8)
C(10)	2i	0.8015(8)	0.3835(8)	0.1153(8)	0.140(7)	0.128(7)	0.176(8)	0.054(6)	0.110(7)	0.050(6)
C(11)	2i	0.560(1)	0.1785(8)	0.0363(7)	0.26(1)	0.105(6)	0.127(7)	-0.019(8)	0.094(8)	-0.017(6)
C(12)	2i	0.8667(5)	0.6140(6)	0.4996(6)	0.061(3)	0.091(4)	0.142(6)	0.011(3)	0.008(3)	0.072(4)
C(13)	2i	1.0731(7)	0.770(1)	0.4556(7)	0.091(6)	0.26(1)	0.144(7)	0.021(7)	0.067(6)	0.093(8)
C(14)	2i	1.1029(7)	0.7751(9)	0.6746(7)	0.087(5)	0.169(8)	0.130(6)	0.017(5)	-0.012(5)	0.062(6)
C(15)	2i	0.9445(7)	0.8915(6)	0.5917(8)	0.095(5)	0.082(5)	0.21(1)	0.026(4)	0.014(6)	0.012(6)
C(16)	2i	0.6190(4)	0.7797(4)	0.6494(3)	0.055(2)	0.041(2)	0.057(2)	0.010(2)	0.014(2)	0.020(2)
C(17)	2i	0.7298(7)	1.0455(6)	0.8099(6)	0.129(6)	0.060(4)	0.117(5)	0.028(4)	-0.021(5)	-0.004(4)
C(18)	2i	0.4661(5)	0.9335(5)	0.6913(5)	0.092(4)	0.070(4)	0.120(5)	0.035(3)	0.054(4)	0.034(3)
C(19)	2i	0.6460(6)	0.9843(6)	0.5737(6)	0.117(5)	0.080(4)	0.142(6)	0.049(4)	0.070(4)	0.076(4)
C(20)	2i	0.3534(4)	0.6424(5)	0.4129(4)	0.058(3)	0.095(4)	0.069(3)	0.033(3)	0.024(2)	0.044(3)
C(21)	2i	0.3241(8)	0.4344(7)	0.2064(7)	0.180(9)	0.102(6)	0.121(6)	0.079(6)	-0.052(6)	-0.027(5)
C(22)	2i	0.1521(8)	0.569(1)	0.2158(7)	0.119(7)	0.23(1)	0.123(7)	0.100(7)	-0.015(5)	0.044(7)
C(23)	2i	0.3988(9)	0.6942(9)	0.2310(6)	0.19(1)	0.184(9)	0.109(6)	0.070(8)	0.036(6)	0.096(6)
N(1)	2i	0.9486(5)	0.8426(5)	-0.0511(4)	0.105(4)	0.071(3)	0.073(3)	0.010(3)	0.037(3)	0.029(2)
O(1)	2i	0.8988(5)	0.8889(5)	-0.0992(4)	0.142(4)	0.114(4)	0.098(3)	0.033(3)	0.042(3)	0.071(3)
O(2)	2i	1.0305(6)	0.8110(6)	-0.0692(4)	0.160(5)	0.157(5)	0.122(4)	0.074(4)	0.096(4)	0.086(4)
O(3)	2i	0.7199(5)	0.8143(4)	0.3160(4)	0.181(5)	0.107(3)	0.164(4)	0.087(4)	0.134(4)	0.096(3)
O(4)	2i	0.7844(3)	0.6587(3)	0.2897(3)	0.082(2)	0.075(2)	0.088(2)	0.035(2)	0.051(2)	0.050(2)
O(5)	2i	0.5807(2)	0.5037(2)	0.4545(2)	0.051(2)	0.042(1)	0.059(2)	0.014(1)	0.028(1)	0.020(1)
O(6)	2i	0.6233(3)	0.6832(3)	0.4089(3)	0.062(2)	0.065(2)	0.074(2)	0.029(2)	0.039(2)	0.043(2)
Si(1)	2i	0.6472(2)	0.3447(2)	0.1160(1)	0.096(1)	0.084(1)	0.0637(8)	0.0253(9)	0.0389(8)	0.0205(8)
Si(2)	2i	0.3036(2)	0.5785(2)	0.2670(1)	0.091(1)	0.079(1)	0.0614(8)	0.0460(9)	-0.0026(7)	0.0147(7)
Si(3)	2i	0.9942(1)	0.7622(2)	0.5536(1)	0.0527(8)	0.080(1)	0.0854(9)	0.0169(7)	0.0251(7)	0.0351(8)
Si(4)	2i	0.6146(1)	0.9362(1)	0.6813(1)	0.0604(8)	0.0384(6)	0.0708(8)	0.0117(5)	0.0182(6)	0.0176(6)
Sn(1)	2i	0.70601(3)	0.56130(3)	0.37867(2)	0.0498(2)	0.0494(2)	0.0630(2)	0.0139(1)	0.0295(2)	0.0244(2)
Sn(2)	2i	0.50812(2)	0.63307(2)	0.50123(2)	0.0423(2)	0.0402(2)	0.0456(2)	0.0110(1)	0.0169(1)	0.0175(1)

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