Crystal structure of bis(tetrakis(trimethylsilylmethyl)(μ-hydroxy)(*p*-nitrobenzoato)(μ^3-oxo)ditin(IV)), $[Sn_2O(OH)(NO_2C_6H_4COO)\{(CH_3)_3SiCH_2\}_4]_2$

T. Lei[I], G.-Y. Wang*[,II], Z.-H. Li[I], S. Lin[III] and X.-N. Fang[IV]

[I] Taiyuan University of Science and Technology, Huake Institute, Taiyuan, Shanxi 030024, China
[II] China Research Institute of Daily Chemical Industry, Taiyuan, Shanxi 030001, China
[III] Nanchang University, Department of Chemistry, Nanchang, Jiangxi 330047, China
[IV] JingGangShan College, Department of Chemistry, Ji'an, Jiangxi 343009, China

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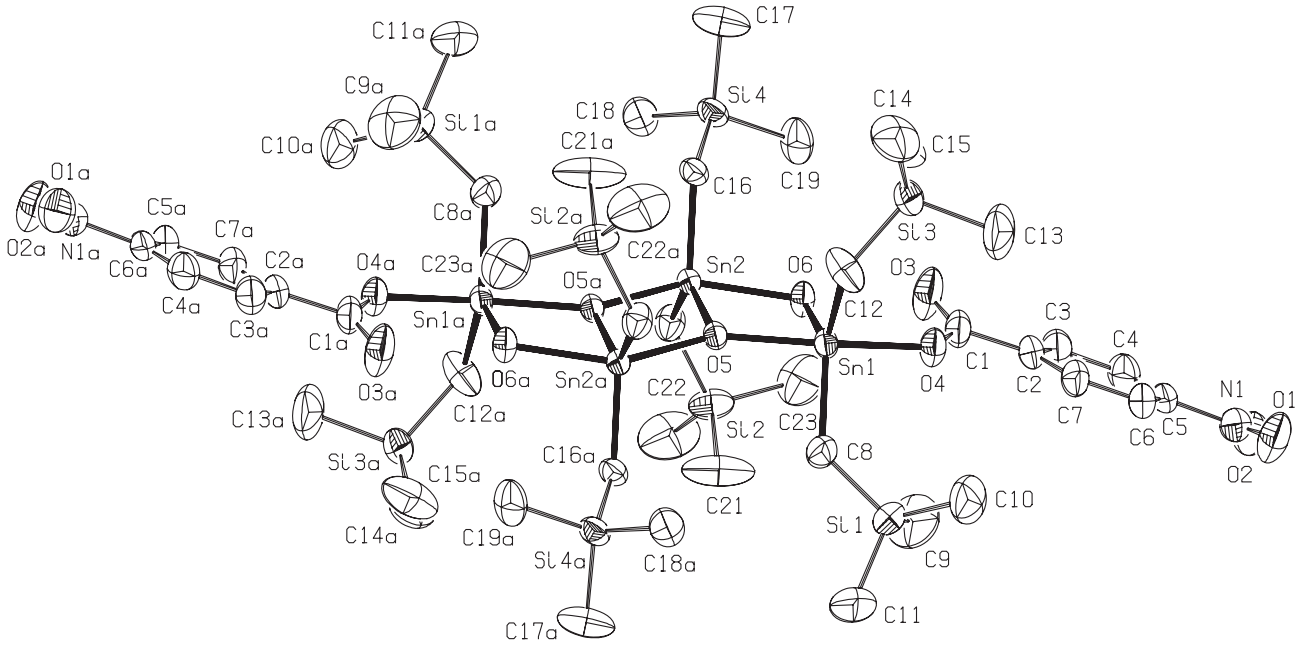

Abstract

$C_{46}H_{98}N_2O_{12}Si_8Sn_4$, triclinic, $P\bar{1}$ (no. 2), a = 12.2616(1) Å, b = 12.4817(1) Å, c = 14.1222(2) Å, α = 110.191(1)°, β = 100.408(1)°, γ = 106.679(1)°, V = 1846.8 Å^3, Z = 1, $R_{gt}(F)$ = 0.036, $wR_{ref}(F^2)$ = 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 biological 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 Sn_2O_2 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
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	23197, 6488
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 5113
$N(param)_{refined}$:	341
Programs:	SHELXS-97 [4], SHELXL-97 [5]

* Correspondence author (e-mail: wang_guoyong@yahoo.com)

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

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

Table 2. Continued.

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

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}
C(1)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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)	2*i*	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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References

1. Sartaj, T.; Claudio, P.: Chemical and biotechnological developments in organotin cancer chemotherapy. J. Organomet. Chem. **691** (2006) 1761-1766.
2. Xie, Q.-L.; Liu, H.: Studies on Biologically Active Organotin Compounds Containing Silicon. Chem. J. Chinese Univ. **19** (1998) 917-920.
3. Gielen, M.; Tiekink, E. R. T.: Crystal structure of bis[tetraethyl-μ-hydroxyperfluorophenylacetato-μ-3-oxoditin], $[(F_5C_6CH_2CO_2)Et_2Sn(OH)OSnEt_2]_2$. Z. Kristallogr. **215** (2000) 255-256.
4. Sheldrick, G. M.: SHELXS-97. Program for the Solution of Crystal Structures. University of Göttingen, Germany 1997.
5. Sheldrick, G. M.: SHELXL-97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1997.