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Crystal structure of bis(μ_3 -oxido)-bis(μ_2 -2,3,4,5-tetrafluorobenzoato- κ^2 O:O')-bis(2,3,4,5-tetrafluorobenzoato- κ O)-oktakis(3-chlorobenzyl- κ C) tetratin(IV), $C_{84}H_{52}Cl_8F_{16}O_{10}Sn_4$

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Abstract

$C_{84}H_{52}Cl_8F_{16}O_{10}Sn_4$, triclinic, $P\bar{1}$ (no. 2), $a = 11.2999(9)$ Å, $b = 14.1471(11)$ Å, $c = 15.2378(12)$ Å, $\alpha = 67.656(1)^\circ$, $\beta = 72.115(1)^\circ$, $\gamma = 69.730(1)^\circ$, $V = 2069.9(3)$ Å³, $Z = 1$, $R_{gt}(F) = 0.0284$, $wR_{ref}(F^2) = 0.0732$, $T = 293(2)$ K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

In a typical experiment, 2,3,4,5-tetrafluorobenzoic acid (0.194 g; 1 mmol) and 3-chlorobenzyltin oxide were dissolved in 18 mL benzene. This mixture was heated and refluxed to

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.19 × 0.15 × 0.11 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	1.55 mm ⁻¹
Diffractometer, scan mode:	Bruker SMART APEX-II,
θ_{max} , completeness:	25.0°, 99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	14986, 7212, 0.022
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 6227
$N(param)_{refined}$:	550
Programs:	Bruker [1], SHELX [2, 3]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	U_{iso}^*/U_{eq}
Sn1	0.88194(2)	1.04167(2)	0.58518(2)	0.02752(7)
Sn2	0.97499(2)	0.75343(2)	0.60816(2)	0.02951(7)
Cl1	0.27012(12)	1.28299(14)	0.66624(14)	0.1067(5)
Cl2	1.28313(14)	0.73452(11)	0.84871(13)	0.0989(5)
Cl3	0.79498(14)	0.71756(13)	1.05807(10)	0.0931(4)
Cl4	0.91403(14)	0.31245(10)	0.74449(12)	0.0914(4)
F1	1.3015(2)	0.78975(16)	0.31669(16)	0.0558(6)
F2	1.5166(2)	0.7292(2)	0.19830(18)	0.0741(8)
F3	1.6545(2)	0.5237(2)	0.23782(19)	0.0810(8)
F4	1.5710(2)	0.37789(18)	0.39652(18)	0.0743(8)
F5	0.5588(2)	1.04105(18)	0.82905(19)	0.0712(7)
F6	0.3646(3)	1.0030(2)	0.9755(2)	0.0894(9)
F7	0.3306(2)	0.8056(2)	1.06083(18)	0.0802(8)
F8	0.4951(3)	0.6449(2)	0.99634(19)	0.0778(8)
O1	0.7793(2)	0.95897(18)	0.72419(16)	0.0397(6)
O2	0.7888(2)	0.80882(18)	0.70410(16)	0.0390(6)
O3	1.1468(2)	0.73686(17)	0.49430(16)	0.0396(6)
O4	1.1508(3)	0.5676(2)	0.5556(2)	0.0584(8)
O5	0.98889(19)	0.90481(15)	0.55332(15)	0.0304(5)
C1	1.1960(3)	0.6391(3)	0.4965(3)	0.0385(8)
C2	1.3175(3)	0.6133(3)	0.4237(2)	0.0362(8)
C3	1.3634(3)	0.6864(3)	0.3411(3)	0.0392(8)
C4	1.4759(3)	0.6566(3)	0.2784(3)	0.0462(9)
C5	1.5455(4)	0.5530(3)	0.2980(3)	0.0511(10)
C6	1.5005(4)	0.4800(3)	0.3792(3)	0.0487(10)
C7	1.3879(3)	0.5081(3)	0.4410(3)	0.0418(8)
H7	1.358249	0.456598	0.495054	0.050*
C8	0.7416(3)	0.8779(3)	0.7472(2)	0.0323(7)
C9	0.6312(3)	0.8616(3)	0.8318(2)	0.0342(7)
C10	0.5487(4)	0.9419(3)	0.8675(3)	0.0449(9)
C11	0.4464(4)	0.9231(3)	0.9436(3)	0.0537(10)
C12	0.4301(4)	0.8233(4)	0.9864(3)	0.0535(10)
C13	0.5128(4)	0.7425(3)	0.9524(3)	0.0492(10)
C14	0.6125(3)	0.7604(3)	0.8749(3)	0.0412(8)

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Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
H14	0.667259	0.705221	0.851241	0.049*
C15	0.7236(3)	1.0904(3)	0.5144(2)	0.0394(8)
H15A	0.717304	1.163531	0.474594	0.047*
H15B	0.747221	1.049085	0.470487	0.047*
C16	0.5913(3)	1.0842(3)	0.5712(2)	0.0369(8)
C17	0.5018(3)	1.1735(3)	0.5918(3)	0.0482(9)
H17	0.524492	1.237029	0.569917	0.058*
C18	0.3798(4)	1.1682(4)	0.6446(3)	0.0630(12)
C19	0.3428(4)	1.0762(4)	0.6776(3)	0.0715(14)
H19	0.260324	1.073605	0.713632	0.086*
C20	0.4290(5)	0.9878(4)	0.6568(4)	0.0702(14)
H20	0.404922	0.924864	0.678898	0.084*
C21	0.5525(4)	0.9918(3)	0.6029(3)	0.0509(10)
H21	0.609564	0.931795	0.588058	0.061*
C22	0.9855(3)	1.0750(3)	0.6620(2)	0.0370(8)
H22A	1.067124	1.084497	0.619577	0.044*
H22B	0.937243	1.142291	0.673449	0.044*
C23	1.0129(3)	0.9968(3)	0.7562(2)	0.0351(7)
C24	1.1201(3)	0.9109(3)	0.7596(3)	0.0411(8)
H24	1.172842	0.899244	0.702395	0.049*
C25	1.1492(4)	0.8426(3)	0.8475(3)	0.0569(11)
C26	1.0715(5)	0.8562(4)	0.9334(3)	0.0716(14)
H26	1.091929	0.809863	0.992387	0.086*
C27	0.9636(5)	0.9395(4)	0.9305(3)	0.0739(14)
H27	0.909552	0.949782	0.987813	0.089*
C28	0.9351(4)	1.0076(4)	0.8431(3)	0.0564(11)
H28	0.860697	1.063110	0.842463	0.068*
C29	1.0688(3)	0.6730(3)	0.7282(3)	0.0442(9)
H29A	1.139058	0.614522	0.713622	0.053*
H29B	1.105941	0.721327	0.735453	0.053*
C30	0.9846(3)	0.6309(3)	0.8225(2)	0.0414(8)
C31	0.9329(3)	0.6862(3)	0.8889(3)	0.0453(9)
H31	0.949667	0.750949	0.874588	0.054*
C32	0.8566(4)	0.6463(4)	0.9765(3)	0.0559(10)
C33	0.8289(4)	0.5520(4)	0.9990(3)	0.0668(13)
H33	0.777258	0.525612	1.058173	0.080*
C34	0.8780(5)	0.4974(4)	0.9333(3)	0.0681(13)
H34	0.858673	0.433727	0.947682	0.082*
C35	0.9559(4)	0.5350(3)	0.8459(3)	0.0576(11)
H35	0.989492	0.496097	0.802371	0.069*
C36	0.8575(4)	0.7103(3)	0.5501(3)	0.0441(9)
H36A	0.789231	0.771793	0.527486	0.053*
H36B	0.909412	0.685649	0.495383	0.053*
C37	0.8000(3)	0.6249(3)	0.6260(3)	0.0428(9)
C38	0.8704(4)	0.5204(3)	0.6445(3)	0.0484(9)
H38	0.950757	0.502073	0.605664	0.058*
C39	0.8214(4)	0.4428(3)	0.7209(3)	0.0535(10)
C40	0.7035(5)	0.4661(4)	0.7788(3)	0.0630(12)
H40	0.673070	0.413113	0.830579	0.076*
C41	0.6308(4)	0.5686(4)	0.7592(4)	0.0681(13)
H41	0.549015	0.585513	0.796854	0.082*
C42	0.6784(4)	0.6475(3)	0.6835(3)	0.0593(11)
H42	0.627832	0.717033	0.670995	0.071*

80 °C, resulting in a colorless solution. The mixture was filtered and recrystallized. Crystals of the title compound were obtained by slow evaporation within one week.

Experimental details

All hydrogen atoms were geometrically positioned. Their U_{iso} values were assigned with common isotropic displacement factors $U_{iso}(H) = 1.2$ times U_{eq} .

Comment

In recent years, organotin(IV) complexes have been focused on acquiring well defined solid-state structures to learn the nature of their versatile coordination chemistry [4]. Moreover, they have received much attention because the tin(IV) is able to form stable bonds to get oligonuclear complexes [5–7].

The symmetric unit of the title structure contains two Sn centers with two carboxylate ligands, four chlorobenzyl moieties and one bridging O atom. Each Sn shows a five-coordinated trigonal bipyramidal geometry. Sn1, O5, C22 and C15 occupy the equatorial planar positions and O1, O5 are in apical positions. The bond angle of O1–Sn1–O5 is 169.07(8)°, showing the distortion. Sn2, O5, C29 and C36 occupy the equatorial planar positions and O2, O3 are in apical positions. The bond angle of O2–Sn1–O3 is 167.00(8)°. The bond lengths of Sn1–O1 is 2.179(2) Å and Sn2–O2 is 2.240(2) Å, which is close to the literature reported [8].

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