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The crystal structure of the cocrystal di- μ_2 -chlorido-octamethyl-di- μ_3 -oxido-bis(2,3,4,5-tetrafluorobenzoato- $\kappa^2 O, O'$)tetratin(IV) – octamethyl-di- μ_3 -oxido-bis(μ_2 -2,3,4,5-tetrafluorobenzoato- $\kappa^2 O:O'$)-bis(μ_2 -2,3,4,5-tetrafluorobenzoato- $\kappa^2 O:O;O'$)tetratin(IV) $C_{58}H_{54}Cl_2F_{24}O_{16}Sn_8$

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.38 × 0.15 × 0.10 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	2.70 mm ⁻¹
Diffractometer, scan mode:	D8, φ and ω
$\theta_{\max}$, completeness:	25.0°, 99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	10,293, 6786, 0.033
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 4166
$N(\text{param})_{\text{refined}}$:	487
Programs:	Bruker [1], SHELX [2]

$\gamma = 79.841(7)^\circ$, $V = 1955.2(15) \text{ \AA}^3$, $Z = 1$, $R_{\text{gt}}(F) = 0.0578$, $wR_{\text{ref}}(F^2) = 0.1660$, $T = 298 \text{ K}$.

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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

The educt, 2,3,4,5-benzoic acid (194 mg, 1 mmol) and sodium ethoxide (74.85 mg) was added to a 50 ml three-necked flask in 25 ml benzene and stirring for half an hour, then dimethyltin dichloride (220 mg, 1 mmol) was added to the mixture. The heating was processed for 4 h and the solution was filtered. The suitable crystal for X-ray diffraction was precipitated by hexane/dichloromethane.

Yield: 65% (base on Sn element), and **elemental analysis**: calc. for $C_{58}H_{54}Cl_2F_{24}O_{16}Sn_8$: C 33.88, H 2.13; found: C 33.52, H 2.28.

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Abstract

$C_{58}H_{54}Cl_2F_{24}O_{16}Sn_8$, triclinic, $P\bar{1}$ (no. 2), $a = 7.609(4) \text{ \AA}$, $b = 13.938(6) \text{ \AA}$, $c = 19.190(9) \text{ \AA}$, $\alpha = 77.599(7)^\circ$, $\beta = 89.846(7)^\circ$,

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
Sn1	0.00283 (11)	0.33048 (6)	0.65011 (4)	0.0420 (2)
Sn2	0.06046 (12)	0.40842 (6)	0.45947 (4)	0.0444 (2)
Sn3	0.93964 (12)	−0.00066 (6)	0.17040 (4)	0.0475 (3)
Sn4	1.03405 (11)	−0.12244 (6)	0.02391 (5)	0.0455 (2)
Cl1	1.0226 (5)	−0.2046 (3)	0.16178 (19)	0.0688 (10)
F1	0.1184 (14)	0.1824 (6)	0.4455 (4)	0.098 (3)
F2	0.2568 (16)	0.0409 (8)	0.3793 (5)	0.117 (4)
F3	0.3893 (15)	−0.1447 (7)	0.4505 (5)	0.115 (4)
F4	0.3830 (15)	−0.1882 (6)	0.5958 (6)	0.114 (4)
F5	−0.2250 (14)	0.7390 (6)	0.7217 (5)	0.096 (3)
F6	−0.3754 (13)	0.7888 (6)	0.8366 (5)	0.099 (3)
F7	−0.4329 (12)	0.6499 (7)	0.9495 (4)	0.091 (3)
F8	−0.3383 (13)	0.4564 (7)	0.9495 (4)	0.093 (3)
F9	0.9387 (12)	0.3416 (6)	0.0618 (4)	0.079 (2)
F10	0.8595 (13)	0.5351 (6)	0.0526 (5)	0.091 (3)
F11	0.6790 (14)	0.6094 (7)	0.1557 (5)	0.099 (3)
F12	0.5972 (12)	0.4856 (7)	0.2734 (4)	0.091 (3)
O1	0.0681 (12)	0.2374 (6)	0.5707 (4)	0.056 (2)
O2	0.0828 (13)	0.1191 (6)	0.6654 (5)	0.065 (3)
O3	−0.0484 (15)	0.4449 (7)	0.7181 (5)	0.085 (3)
O4	−0.1364 (16)	0.5915 (8)	0.6539 (5)	0.093 (3)
O5	−0.0062 (12)	0.4393 (6)	0.5616 (4)	0.055 (2)
O6	0.8995 (12)	0.1594 (6)	0.1313 (4)	0.057 (2)
O7	0.8410 (14)	0.1425 (7)	0.2432 (5)	0.073 (3)
O8	0.9816 (11)	0.0011 (6)	0.0662 (4)	0.051 (2)
C1	0.1018 (16)	0.1470 (8)	0.6015 (7)	0.047 (3)
C2	0.1734 (17)	0.0720 (9)	0.5588 (7)	0.050 (3)
C3	0.1815 (19)	0.0905 (9)	0.4862 (8)	0.062 (4)
C4	0.258 (2)	0.0188 (11)	0.4502 (7)	0.062 (4)
C5	0.3247 (19)	−0.0765 (10)	0.4859 (8)	0.065 (4)
C6	0.317 (2)	−0.0955 (10)	0.5598 (8)	0.069 (4)
C7	0.2426 (17)	−0.0253 (9)	0.5948 (7)	0.058 (3)
H7	0.2371	−0.0422	0.6443	0.070*
C8	−0.1185 (16)	0.5307 (9)	0.7103 (6)	0.047 (3)
C9	−0.1994 (16)	0.5654 (9)	0.7741 (6)	0.044 (3)
C10	−0.2487 (17)	0.6634 (9)	0.7766 (6)	0.049 (3)
C11	−0.3258 (18)	0.6923 (10)	0.8345 (7)	0.057 (3)
C12	−0.3528 (19)	0.6233 (12)	0.8934 (7)	0.062 (4)
C13	−0.3058 (19)	0.5240 (11)	0.8915 (6)	0.060 (4)
C14	−0.2284 (16)	0.4943 (9)	0.8341 (6)	0.048 (3)
H14	−0.1947	0.4266	0.8347	0.058*
C15	0.2689 (14)	0.2972 (9)	0.6923 (7)	0.054 (3)
H15A	0.2786	0.2454	0.7348	0.081*
H15B	0.3482	0.2752	0.6576	0.081*
H15C	0.3005	0.3560	0.7036	0.081*
C16	−0.2658 (18)	0.3110 (10)	0.6624 (7)	0.066 (4)
H16A	−0.2736	0.2596	0.7040	0.098*
H16B	−0.3386	0.3724	0.6677	0.098*
H16C	−0.3072	0.2919	0.6210	0.098*
C17	0.3425 (17)	0.3691 (12)	0.4797 (9)	0.084 (5)
H17A	0.3773	0.2978	0.4901	0.126*
H17B	0.4025	0.3976	0.4384	0.126*
H17C	0.3746	0.3944	0.5197	0.126*
C18	−0.180 (2)	0.3671 (12)	0.4384 (8)	0.103 (7)
H18A	−0.1648	0.2956	0.4472	0.154*

Table 2: (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
H18B	−0.2695	0.3916	0.4689	0.154*
H18C	−0.2174	0.3950	0.3894	0.154*
C19	0.8534 (17)	0.1981 (9)	0.1844 (8)	0.055 (3)
C20	0.8122 (16)	0.3052 (9)	0.1752 (6)	0.047 (3)
C21	0.8550 (16)	0.3706 (10)	0.1171 (7)	0.052 (3)
C22	0.812 (2)	0.4740 (10)	0.1096 (7)	0.062 (4)
C23	0.720 (2)	0.5112 (10)	0.1631 (8)	0.065 (4)
C24	0.6837 (18)	0.4478 (10)	0.2219 (7)	0.057 (3)
C25	0.7244 (17)	0.3469 (10)	0.2302 (7)	0.057 (3)
H25	0.6950	0.3054	0.2717	0.068*
C26	1.1909 (16)	−0.0197 (10)	0.2202 (7)	0.061 (4)
H26A	1.1773	−0.0210	0.2701	0.092*
H26B	1.2617	−0.0816	0.2145	0.092*
H26C	1.2491	0.0346	0.1989	0.092*
C27	0.6676 (15)	−0.0167 (10)	0.1818 (7)	0.056 (3)
H27A	0.6356	−0.0183	0.2304	0.084*
H27B	0.5930	0.0387	0.1509	0.084*
H27C	0.6515	−0.0778	0.1694	0.084*
C28	1.3115 (16)	−0.1758 (11)	0.0259 (7)	0.068 (4)
H28A	1.3338	−0.2340	0.0058	0.102*
H28B	1.3694	−0.1250	−0.0015	0.102*
H28C	1.3578	−0.1928	0.0743	0.102*
C29	0.7975 (19)	−0.1684 (11)	0.0028 (8)	0.076 (4)
H29A	0.8244	−0.2267	−0.0171	0.114*
H29B	0.7358	−0.1840	0.0464	0.114*
H29C	0.7232	−0.1158	−0.0306	0.114*

Experimental details

The hydrogen atoms were placed in calculated positions (C–H = 0.96 and 0.93 Å). Their *U*_{iso} values were assigned with common isotropic displacement factors *U*_{iso} (H) = 1.2 times *U*_{eq} (C, benzene ring) and 1.5 times *U*_{eq} (C, methyl).

Comment

Organotin carboxylates have been the subjects of interest for some time because of their biochemical and commercial applications [3, 4]. The diversity of the structures of these compounds is due to the ability of carboxylate ligands to coordinate Sn in monodentate, bidentate and bridging manner. A typical feature for dicarboxylato-organotin distannoxanes in the solid state is their dimerization that results in the so-called ladder or staircase arrangement that contains a central planar Sn₂O₂ ring. They are monostructural and the Sn atom may show a skew-trapezoidal planar geometry [5–7]. With these knowledge in mind we are now interested in obtaining organotin complexes with carboxylic ligands in order to obtain a novel organotin complex.

Interestingly, there are two different molecular configurations in my title structure. Both are tetranuclear centrosymmetric dimeric complexes containing a central four-membered Sn_2O_2 ring [8–10]. The difference between the two complexes in the title (see the figure) is that one is bridged by two carboxylate ligands and another is bridged by two Cl atoms. For Sn1, two methyl group and μ_3 -O atom occupy the equatorial plane and two carboxylate-O atom in axial position. The angle axial (O1–Sn1–O3) is $170.8(3)^\circ$, showing the distortion from 180° . The bond lengths of Sn–O span a range from to 2.012(8)–2.253(9) Å. As each Sn(IV) metal center shows an individual coordination details of the bond lengths and angles in these two interesting complexes of the title cocrystal are deposited in the attached cif file (Suppl. Material). But all parameters are in the expected ranges for related structures [9, 10].

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