Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

Octabenzyldi-μ_3-oxo-bis(μ-2,3,4,5-tetrafluorobenzoato-κ^2O:O')bis(2,3,4,5-tetrafluorobenzoato-κO)tetratin(IV)

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.009 Å
R factor = 0.040
wR factor = 0.096
Data-to-parameter ratio = 13.9

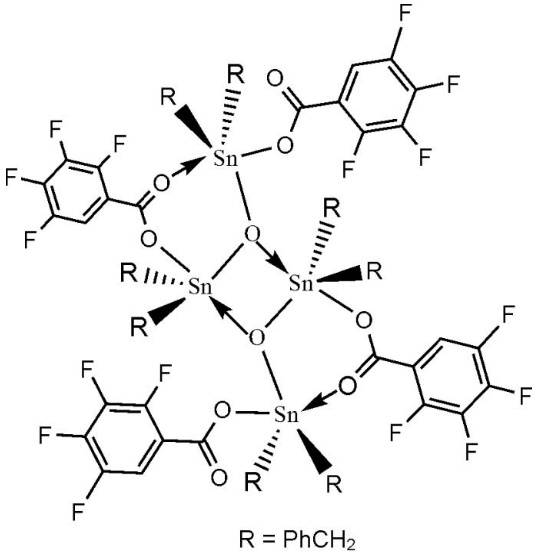
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The structure of the title compound, [$Sn_4(C_7H_7)_8$-$(C_7HF_4O_2)_4O_2$], features a central Sn_2O_2 unit with two additional Sn atoms linked by the μ_3-oxo atoms. Two pairs of Sn atoms are bridged by two bidentate 2,3,4,5-tetrafluorobenzoate ligands. Two of the Sn atoms are also each coordinated by a monodentate 2,3,4,5-tetrafluorobenzoate ligand. The geometry around each five-coordinated Sn center is distorted trigonal bipyramidal.

Received 28 September 2006
Accepted 28 November 2006

Comment

Organotin carboxylate complexes have recently attracted much attention due to their wide industrial applications, such as in catalysis, organic synthesis, PVC stabilization, pesticides, bactericides, antifouling paints, antiseptic materials, as well as their biological activities (Duboy & Roy, 2003). In order to explore the impact of the structure on properties of the complexes, as well as to analyse the structure–activity relationships, a large number of organotin carboxylate complexes have been prepared and studied (Ma *et al.*, 2004). However, only a few fluorinated ligands have been used in organotin complexes (Gielen, 1995). 2,3,4,5-Tetrafluorobenzoic acid (Htfb) is one of the most popular ligands of this type. We report here the structure of the title complex, (I).



(I)

Compound (I) is a tetranuclear organotin complex (Fig. 1). Two oxo groups in the complex are tridentate, linking three Sn centres, two endocyclic and one exocyclic. Further links between the endo- and exocyclic Sn atoms are provided by

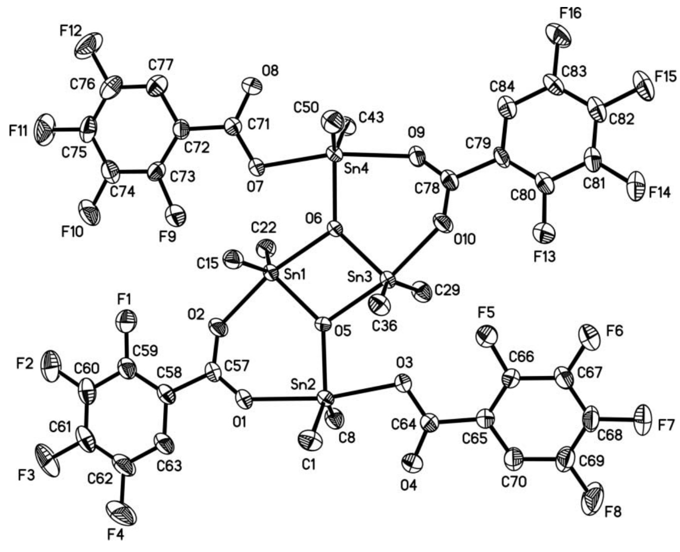


Figure 1

The molecular structure of (I), Displacement ellipsoids are drawn at the 30% probability level. H atoms and benzene rings of the benzyl groups have been omitted for clarity.

bidentate tfb ligands. Each exocyclic Sn atom is also coordinated by a monodentate tfb ligand. The coordination geometry about each of the Sn atoms is best described as distorted trigonal bipyramidal with the axial positions occupied by O atoms. The distortion from an ideal geometry may be attributed in part to the presence of close intramolecular Sn···O interactions. Atoms O4 and O8 form contacts of 2.934 (4) Å and 2.922 (3) Å with Sn2 and Sn4, respectively; this has the effect of opening up the C1—Sn2—C8 [133.2 (2)°] and C43—Sn4—C50 [135.1 (2)°] angles. Similarly, the strong electronegative F atoms (F9 and F5) also affect the C15—Sn1—C22 [137.8 (2)°] and C29—Sn3—C36 [146.5 (2)°] angles. The structural motif is similar to that found in complexes with the general formula $[(R'CO_2)R_2SnOSnR_2(O_2CR')]_2$ (Tiekink, 1991).

Experimental

All reagents and solvents were used as obtained without further purification. The reaction was carried out under a nitrogen atmosphere. Htfb (0.239 g, 1 mmol) and dibenzyltin(IV) oxide (0.220 g, 1 mmol) were added to a solution of dry benzene (30 ml) in a Schlenk flask and refluxed at 353 K for 12 h with stirring. After cooling to room temperature, the solution was filtered. The solvent was removed from the filtrate under vacuum. The solid residue was recrystallized from diethyl ether and colorless crystals of (I) suitable for X-ray diffraction studies were obtained (yield 85%; m.p. 152–154 K). Analysis, calculated for $C_{84}H_{60}F_{16}O_{10}Sn_4$: C 50.24, H 3.01%; found: C 50.52, H 2.86%.

Crystal data

$[Sn_4(C_7H_7)_8(C_7HF_4O_2)_4O_2]$
$M_r = 2008.08$
Triclinic, $P\bar{1}$
$a = 11.9014$ (9) Å
$b = 12.122$ (1) Å
$c = 29.070$ (2) Å
$\alpha = 99.171$ (2)°
$\beta = 91.623$ (2)°
$\gamma = 109.922$ (2)°

$V = 3877.2$ (5) Å^3
$Z = 2$
$D_x = 1.720$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 1.37$ mm^{-1}
$T = 295$ (2) K
Block, colorless
0.27 × 0.13 × 0.08 mm

Data collection

Bruker SMART 1000 CCD area detector diffractometer
φ and ω scans
Absorption correction: multi-scan *SADABS* (Sheldrick, 1996)
$T_{min} = 0.708$, $T_{max} = 0.898$

29322 measured reflections
14288 independent reflections
9736 reflections with $I > 2\sigma(I)$
$R_{int} = 0.031$
$\theta_{max} = 25.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.096$
$S = 1.00$
14288 reflections
1027 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.041P)^2 + 0.1235P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.72$ e Å^{-3}
$\Delta\rho_{min} = -0.64$ e Å^{-3}

H atoms were positioned geometrically and refined as riding, with C—H = 0.93 Å (CH) and 0.97 Å (CH$_2$) and $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

The authors thank the National Natural Science Foundation of China (20271025) for financial support.

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Acta Cryst. (2007). E**63**, m62–m63 Sun *et al.* • $[Sn_4(C_7H_7)_8(C_7HF_4O_2)_4O_2]$ **m63**