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Bis(aquamethyldiphenyltrifluoroacetatotin)–18-crown-6 (1/1)

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The *trans*–C_3SnO_2 trigonal bipyramidal organotin moiety in the centrosymmetric title compound, $[Sn(C_2F_3O_2)(CH_3)(C_6H_5)_2(H_2O)]_2 \cdot C_{12}H_{24}O_6$, is hydrogen bonded to the polyether moiety [O···O = 2.901 (8) and 2.983 (7) Å] through its coordinated water molecule [Sn—O = 2.483 (5) Å].

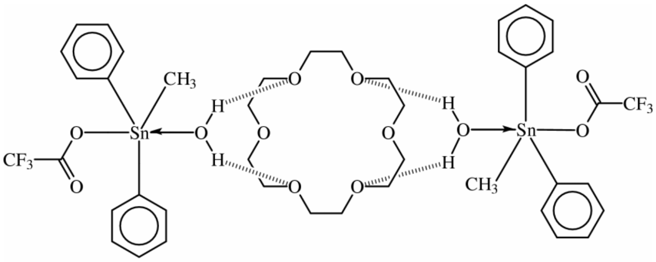
Key indicators

Single-crystal X-ray study
T = 168 K
Mean σ(C–C) = 0.013 Å
R factor = 0.060
wR factor = 0.155
Data-to-parameter ratio = 14.7

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

Comment

Triorganotin(IV) carboxylates commonly exist as either four-coordinate tetrahedral molecules or five-coordinate carboxylate-bridge polymers (Ng *et al.*, 1988; Tiekink, 1991, 1994). Within the triphenyltin carboxylate class, a small number of compounds have sufficiently enhanced Lewis acidicity that they can form complexes with oxygen-donor ligands (Ng, 1998, 1999; Ng & Kumar Das, 1997). For the strongly electron-withdrawing trifluoroacetate anion in particular, the resulting triphenyltin trifluoroacetate is able to accept water into its coordination sphere, and it has been isolated as an 'outer-sphere coordination' complex with 1,10-phenanthroline (Ng *et al.*, 1996). The studies on outer-sphere coordination complexes of hydrated triorganotin carboxylates are now extended to the title mixed alkyldiaryltin analog, methyldiphenyltin trifluoracetate, but with 18-crown-6 as the entity that interacts with the Sn atom through the coordinated water molecule.



(I)

The Sn atom in the title compound, (I), is five-coordinate in a *trans*-C_3SnO_2 trigonal-bipyramidal environment. The two organotin entities are located on opposite sides of the crown ether, across a center of symmetry that lies in the middle of the crown ether. The Sn atom lies out of the equatorial plane by 0.168 (1) Å in the direction of the carboxyl O atom [Sn—O = 2.483 (5) Å]; the C1–C6 phenyl ring is tilted by 77.3 (3)° with respect to the equatorial plane, whereas the other phenyl ring, C7–C12, is tilted by 18.4 (3)°. The O—H···O hydrogen-bonding interactions involve only four of the six O atoms in the crown ether [$O_{water} \cdots O_{crown\ ether}$ = 2.901 (7) and 2.983 (7) Å].

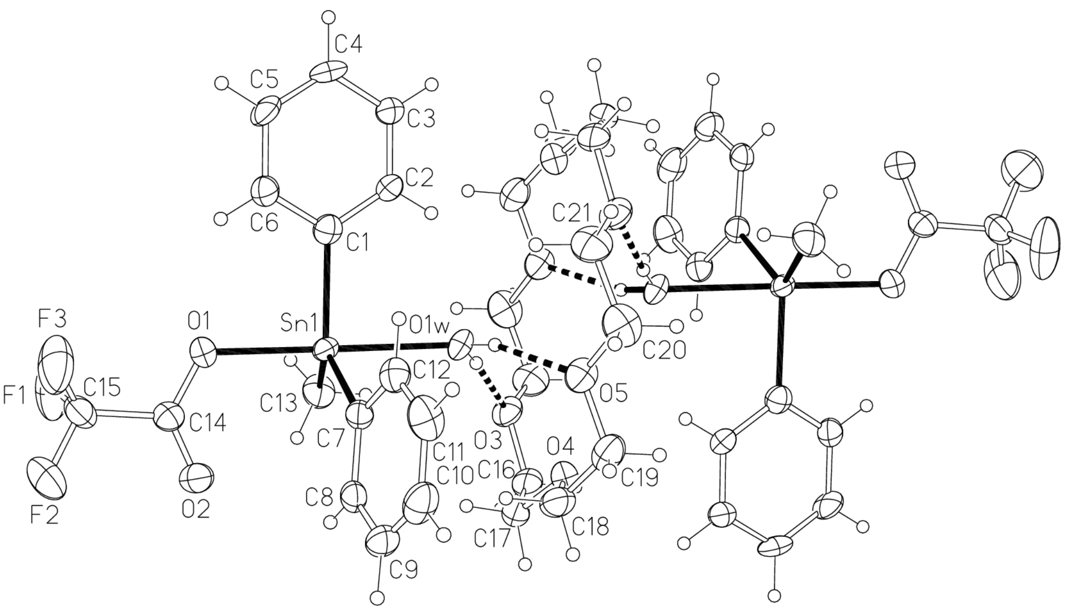


Figure 1
*ORTEP*II (Johnson, 1976) plot of the title compound, with displacement ellipsoids at the 50% probability level.

Experimental

Diphenylmethyltin iodide was synthesized from the cleavage of methyltriphenyltin by elemental iodine in low yield. The halide anion was exchanged for the trifluoroacetate ion by treatment with silver trifluoracetate; the synthesis was adapted from that of dimethylphenyltin acetate, which used dimethyldiphenyltin as the starting reagent (Amini *et al.*, 1989). The disproportionation was carried out in methanol under a nitrogen atmosphere. The silver iodide that resulted from the exchange reaction was removed by filtration, and the filtrate was treated with an equimolar (100% excess) quantity of 18-crown-6. The removal of the solvent gave the desired product, which was then purified by recrystallization from ethanol. The compound melts in the 370–376 K range. The formulation was established by satisfactory ^{1}H NMR integral analysis. The water signal appeared as a broad peak at δ = 4.1 p.p.m.; $^2J(\mathrm{Sn{-}H})$ = 70 Hz in $CDCl_3$; ^{19}F NMR = 75.6 p.p.m. The IR spectrum (KBr disk) showed peaks at 3400 (H_2O), 1703, 1638 (CO) 542, 520 (Sn—C) cm^{-1}.

Crystal data

$[Sn(C_2F_3O_2)(CH_3)(C_6H_5)_2(H_2O)]_2\cdot C_{12}H_{24}O_6$
M_r = 1102.23
Triclinic, $P\overline{1}$
a = 9.3175 (9) Å
b = 12.0263 (11) Å
c = 12.1632 (12) Å
α = 106.156 (1)°
β = 101.177 (1)°
γ = 107.211 (1)°
V = 1192.5 (2) Å^3
Z = 1
D_x = 1.535 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4178 reflections
θ = 1.8–26.5°
μ = 1.13 mm^{-1}
T = 168 (2) K
Plate, colorless
0.30 × 0.15 × 0.05 mm

Data collection

Siemens CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min}$ = 0.729, $T_{\max}$ = 0.946
13839 measured reflections
4135 independent reflections
3066 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.098
$\theta_{\max}$ = 25.0°
$h = -11 \rightarrow 11$
$k = -12 \rightarrow 14$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.155
S = 1.02
4135 reflections
281 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0872P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max}$ = 1.55 e Å^{-3}
$\Delta\rho_{\min}$ = −1.32 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Sn1—C1	2.158 (8)	Sn1—O1	2.193 (5)
Sn1—C7	2.149 (7)	Sn1—O1*w*	2.483 (5)
Sn1—C13	2.135 (8)		
C1—Sn1—C7	115.0 (3)	C7—Sn1—O1	97.4 (2)
C1—Sn1—C13	118.8 (3)	C7—Sn1—O1*w*	82.9 (2)
C1—Sn1—O1	91.2 (3)	C13—Sn1—O1	94.6 (3)
C1—Sn1—O1*w*	88.4 (2)	C13—Sn1—O1*w*	85.5 (3)
C7—Sn1—C13	124.4 (3)	O1—Sn1—O1*w*	179.6 (2)

Although the diffraction intensities were measured beyond θ = 25°, those above this limit were too weak to be of use in the refinement. The water H atoms were placed in calculated positions and refined with a riding model and with $U_{\rm iso}$ = 0.05 Å^2.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

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