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Aqua(trifluoroacetato)triphenyltin–15-crown-5 (2/1)

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In the centrosymmetric title compound, $[Sn(C_2F_3O_2)(C_6H_5)_3\text{-}(H_2O)]_2 \cdot C_{10}H_{20}O_5$, the 1,3–O atoms (positions 1 and 7) of the 1,4,7,10,13-pentaoxacyclopentadecane moiety are hydrogen bonded to the water molecule of the *trans*-C_3SnO_2 trigonal-bipyramidal organotin moiety that lies on one side of the cyclic polyether [O(water)$\cdots$O(crown ether) = 2.800 (4) and 2.837 (4) Å]; the 2,4-O atoms (positions 4 and 10) are hydrogen bonded to the inversion-related organotin moiety that lies on the other side [O(water)$\cdots$O(crown ether) = 2.768 (3) and 2.881 (4) Å].

Comment

The reaction of triphenyltin trifluoroacetate with 18-crown-6 affords a dinuclear compound in which an aquatrifluoro-triphenyltin entity interacts with the polyether through its coordinated water. The compound, $[(C_6H_5)_3SnO_2CCF_3)\cdot H_2O]_2 \cdot C_{12}H_{24}O_6$ [Sn$\leftarrow$O(water) = 2.361 (9) Å], exists as a 1/3 co-crystal with $[(C_6H_5)_3SnO_2CCF_3)\cdot H_2O]_2 \cdot C_{12}H_{24}O_6 \cdot 2H_2O$ (Amini *et al.*, 2003). Disorder in $[(C_6H_5)_3SnO_2CCF_3)\cdot H_2O]_2 \cdot C_{12}H_{24}O_6$, which lies on a threefold axis, precluded the elucidation of the nature of the hydrogen-bonding inter-action. The aquatrifluoroacetatodiphenylmethyltin homolog, $[[(C_6H_5)_2(CH_3)SnO_2CCF_3]\cdot H_2O]_2 \cdot C_{12}H_{24}O_6$ [Sn$\leftarrow$O(water) = 2.483 (5) Å], is not disordered; the cyclic polyether uses its 1,3-substituents to interact with the aquatrifluoroacetato-triorganotin entity that is on one side of the ring, and its 4,6-substituents with the entity on the other side [O(water)$\cdots$O(crown ether) = 2.901 (8), 2.983 (7) Å] (Amini, Yousefi & Ng, 2002).

(I)
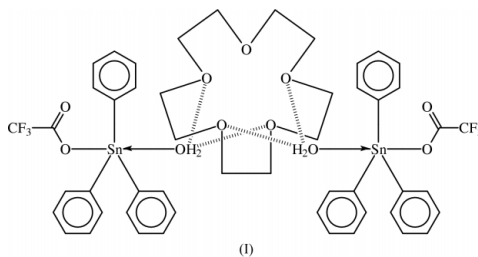
The title compound, (I) (see Scheme), adopts a similar centrosymmetric structure (Fig. 1); as with the 18-crown-6 complex, for which four of the six O atoms are involved in hydrogen-bonding interactions, in this 15-crown-5 complex, four of the five O atoms are linked to the coordinated water molecule by hydrogen bonds. The 15-membered cyclic polyether is disordered over a center of inversion, and the O atoms all point towards the middle of the ring (Fig. 2). On the other hand, in the only other example of 15-crown-5 affording a complex with an organotin compound, the five O atoms point
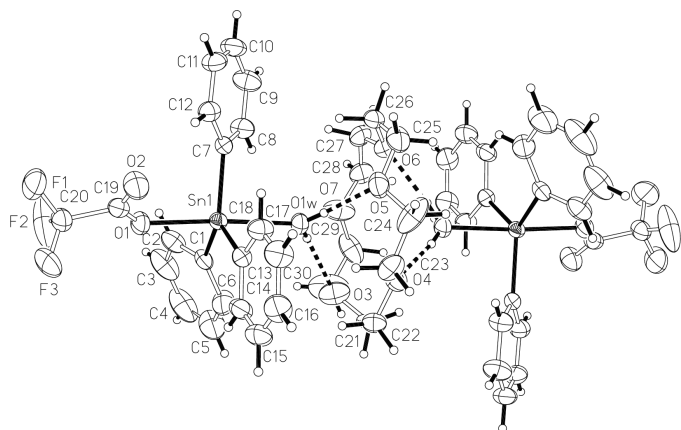
Figure 1
*ORTEP*II (Johnson, 1976) plot of (I), with ellipsoids drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radii. The unlabeled part of the dimer is related to the labeled part by the symmetry operation $(1 - x, 1 - y, 1 - z)$. The second component of the disorder is not shown.
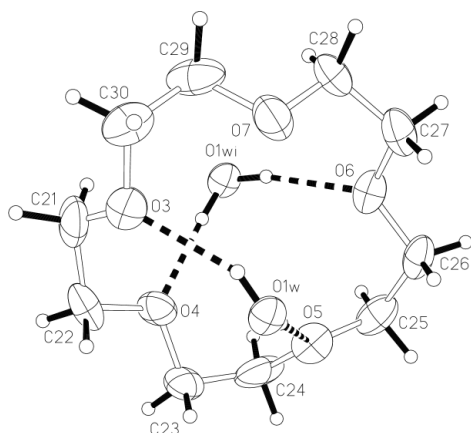

Figure 2
*ORTEP*II (Johnson, 1976) plot of the 15-crown-5·2H$_2$O portion of the title dinuclear complex, without disorder [symmetry code: (i) $1 - x$, $1 - y, 1 - z$]. The ether O atom that is not involved in hydrogen bonding (O7) has a marginally larger equivalent isotropic displacement parameter than the other O atoms.

away from the middle, and there is no interaction with the diaquadichlorodimethyltin entity (Amini *et al.*, 1994*a*; Yap *et al.*, 1996). The tin–water bond distance [Sn←O(water) = 2.407 (1) Å] in the title compound is somewhat shorter than the distances found in the other aquacarboxylatotriorganotin complexes, but much longer than those found in the diaquadichlorodiorganotin adducts of 18-crown-6, *i.e.* [(CH$_3$)$_2$SnCl$_2$·2H$_2$O]$_2$·C$_{12}$H$_{24}$O$_6$ (Amini *et al.*, 1984), [(CH$_3$)-(C$_6$H$_5$)SnCl$_2$·2H$_2$O]$_2$·C$_{12}$H$_{24}$O$_6$ (Amini *et al.*, 1994*b*) and [(C$_6$H$_5$)$_2$SnCl$_2$·2H$_2$O]$_2$·C$_{12}$H$_{24}$O$_6$ (Amini, Amirreza *et al.*, 2002), owing to the weaker Lewis acidity of the triorganotin acceptor.

Experimental

Triphenyltin trifluoroacetate (2.31 g, 5.0 mmol) and 15-crown-5 (0.55 g, 2.5 mmol) were heated in a small volume of acetone. The solvent was removed and the product recrystallized from ethanol to afford the title complex (m.p. 412–414 K). The formulation was established by ^{1}H NMR spectral analysis [^{1}H in CDCl$_3$ (δ in p.p.m.): 4.07 (H$_2$O), 3.15 ($-$CH$_2-$), 7.35–7.79 (C$_6$H$_5$)].

Crystal data

[Sn(C$_2$F$_3$O$_2$)(C$_6$H$_5$)$_3$(H$_2$O)]$_2$·· C$_{10}$H$_{20}$O$_5$	$Z = 1$
$M_r = 1182.31$	$D_x = 1.512$ Mg m^{-3}
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 10.3917$ (6) Å	Cell parameters from 6960 reflections
$b = 11.1154$ (7) Å	$\theta = 1.8$–26.5°
$c = 12.3300$ (8) Å	$\mu = 1.04$ mm^{-1}
$\alpha = 110.611$ (1)°	$T = 168$ (2) K
$\beta = 97.621$ (1)°	Plate, colorless
$\gamma = 97.198$ (1)°	$0.50 \times 0.13 \times 0.12$ mm
$V = 1298.4$ (1) Å^3	

Data collection

Bruker AXS SMART area-detector diffractometer	5179 independent reflections
φ and ω scans	4618 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$R_{\text{int}} = 0.022$
$T_{\min} = 0.571$, $T_{\max} = 0.831$	$\theta_{\max} = 26.5°$
16310 measured reflections	$h = -12 \rightarrow 12$
	$k = -13 \rightarrow 13$
	$l = -12 \rightarrow 15$

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.022$	
$wR(F^2) = 0.056$	$w = 1/[\sigma^2(F_o^2) + (0.0369P)^2]$
$S = 0.98$	where $P = (F_o^2 + 2F_c^2)/3$
5179 reflections	$(\Delta/\sigma)_{\max} = 0.001$
400 parameters	$\Delta\rho_{\max} = 0.69$ e Å^{-3}
	$\Delta\rho_{\min} = -0.57$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Sn1—C1	2.131 (2)	Sn1—O1	2.186 (2)
Sn1—C7	2.137 (2)	Sn1—O1w	2.407 (1)
Sn1—C13	2.124 (2)		
C1—Sn1—C7	117.4 (1)	C7—Sn1—O1	96.7 (1)
C1—Sn1—C13	120.9 (1)	C7—Sn1—O1w	86.4 (1)
C1—Sn1—O1	87.6 (1)	C13—Sn1—O1	96.5 (1)
C1—Sn1—O1w	86.2 (1)	C13—Sn1—O1w	86.5 (1)
C7—Sn1—C13	120.5 (1)	O1—Sn1—O1w	173.8 (1)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O1w—H1w1$\cdots$O3	0.84 (1)	2.07 (3)	2.837 (4)	152 (5)
O1w—H1w2$\cdots$O5	0.85 (1)	1.96 (1)	2.800 (4)	176 (4)
O1w—H1w3$\cdots$O4^i	0.85 (1)	1.93 (1)	2.768 (3)	172 (4)
O1w—H1w4$\cdots$O6^i	0.85 (1)	2.05 (2)	2.881 (4)	168 (4)

Symmetry code: (i) $1 - x, 1 - y, 1 - z$.

The 15-crown-5 ring is disordered over a center of inversion ($\frac{1}{2},\frac{1}{2},\frac{1}{2}$), and it was refined subject to the following restraints: C—C = 1.53 (1), C—O = 1.41 (1) and C—O—C = C—C—O = 2.37 (1) Å. The atoms are of half-site occupancy. The trifluoromethyl group is disordered over two positions; as their occupancy refined to nearly 0.5, the occupancy was fixed as 0.5. The C—F distances were restrained to be approximately equal; the F$\cdots$F lengths were similarly restrained. The displacement parameters of the primed and umprimed F atoms were set equal.

Two pairs of disordered water H atoms were located and refined; the occupancy factor of each of the four H atoms is 0.5. One pair

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(H1w1 and H1w2) interacts with two O atoms (O3 and O5) of the crown ether, and the other pair (H1w3 and H1w4) with two symmetry-related O atoms [O4^i and O6^i; symmetry code: (i) $1 - x$, $1 - y$, $1 - z$]. The O—H distance was restrained to 0.85 (1) Å and the H···H distance to 1.39 (1) Å. The displacement parameters of the H atoms were set to $1.2U_{eq}(O)$.

The carbon-bound H atoms were positioned geometrically, with C—H = 0.95 Å for the aromatic H atoms and C—H = 0.99 Å for the aliphatic H atoms. Their displacement parameters were set to $1.2U_{eq}(C)$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); 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.

References

Amini, M. M., Amirreza, A., Yousefi, M., Foladi, S. & Ng, S. W. (2002). *Acta Cryst.* E**58**, m58–m60.

Amini, M. M., Foladi, S., Aghabozorg, H., Rae, A. D. & Ng, S. W. (2003). *Chin. J. Struct. Chem.* **22**, 77–83.

Amini, M. M., Rheingold, A. L., Taylor, R. & Zuckerman, J. J. (1984). *J. Am. Chem. Soc.* **106**, 7289–7291.

Amini, M. M., Yousefi, M. & Ng, S. W. (2002). *Acta Cryst.* E**58**, m118–m120.

Amini, M. M., Zuckerman, J. J., Rheingold, A. L. & Ng, S. W. (1994a). *Z. Kristallogr.* **209**, 613–614.

Amini, M. M., Zuckerman, J. J., Rheingold, A. L. & Ng, S. W. (1994b). *Z. Kristallogr.* **209**, 682–684.

Bruker (1997). *SAINT* and *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.

Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.

Yap, G. P. A., Counterman, A. E., Amini, M. M., Ng, S. W. & Rheingold, A. L. (1996). *Main Group Chem.* **1**, 359–363.