

Mostafa M. Amini,<sup>a</sup> Amirreza Azadmehr,<sup>a</sup> Shabnam Hossein Abadi<sup>a</sup> and Seik Weng Ng<sup>b\*</sup>

<sup>a</sup>Department of Chemistry, Shahid Beheshti University, Tehran, Iran, and <sup>b</sup>Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia

Correspondence e-mail: seikweng@um.edu.my

#### Key indicators

Single-crystal X-ray study  
 $T = 168$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.010$  Å  
Disorder in main residue  
 $R$  factor = 0.060  
 $wR$  factor = 0.107  
Data-to-parameter ratio = 19.2

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

## Aquachlorodimethylphenyltin–18-crown-6 (2/1)

In the crystal structure of the title compound,  $[\text{SnCl}(\text{CH}_3)_2(\text{C}_6\text{H}_5)(\text{H}_2\text{O})]_2 \cdot \text{C}_{12}\text{H}_{24}\text{O}_8$ , the crown ether, which lies on a center of inversion, interacts with the five-coordinate *trans*- $\text{C}_3\text{SnClO}$  trigonal-bipyramidal aquachlorodimethylphenyltin moieties; the coordinated water molecule, at a distance of 2.445 (4) Å from Sn, forms two hydrogen bonds with the O atoms of the crown ether [ $\text{O}_{\text{water}} \cdots \text{O}_{\text{crown}} = 2.851$  (6) and 2.884 (5) Å].

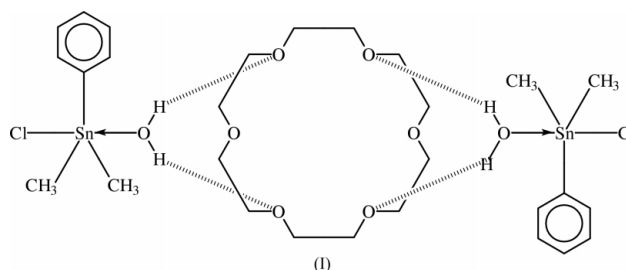
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#### Comment

The coordination status of the Sn atom in mixed triorganotin compounds featuring one methyl group can be investigated from the magnitude of the tin–methyl coupling constants in the NMR. Unfortunately, the synthesis of the mixed triorganotin entity presents difficulties, and only a few such compounds have been synthesized. Earlier, we reported the 2:1 methyl-diphenyltin trifluoroacetate complex of 18-crown-6; this exists as a dihydrate in which the coordinated water molecules interact with the centrosymmetric cyclic polyether through hydrogen bonds [ $\text{Sn}-\text{O}_{\text{water}} = 2.483$  (5) Å;  $\text{O}_{\text{water}} \cdots \text{O}_{18\text{-crown-6}} = 2.901$  (8) and 2.983 (7) Å; Amini *et al.*, 2002]. In this compound, the trifluoroacetate anion serves to increase the Lewis acidity of the Sn atom through its electron-withdrawing ability.



In the title compound, (I), the dimethylphenyltin chloride moiety is also coordinated by a water molecule [ $\text{Sn}-\text{O} = 2.445$  (4) Å], and the water molecule similarly interacts with the cyclic polyether [ $\text{O}_{\text{water}} \cdots \text{O}_{18\text{-crown-6}} = 2.851$  (6) and 2.884 (5) Å]. The two-bond coupling constant of the adduct in solution (83 Hz) is significantly larger than that of the parent Lewis acid (59 Hz), so that the water molecule probably remains attached to the Sn atom even in solution.

#### Experimental

The title adduct was synthesized from the reaction of equimolar quantities of dimethylphenyltin chloride (0.20 g, 0.75 mmol) and 18-crown-6 (0.20 g, 0.75 mmol) in chloroform. The reagents were dissolved in the solvent and crystals of the 2:1 complex (m.p. 344–349 K) separated when the solvent was allowed to evaporate. IR:

3440 (H<sub>2</sub>O), 2880 (C–H), 1099 (C–O–C), 550 (Sn–C, asymmetric), 520 (Sn–C, symmetric) cm<sup>-1</sup>. <sup>1</sup>H NMR in CDCl<sub>3</sub>: 0.90 (CH<sub>3</sub>, 12H), 2.45 (H<sub>2</sub>O, 4H), 3.65 (CH<sub>2</sub>, 24H), 7.46–7.60 (C<sub>6</sub>H<sub>5</sub>, 10H) p.p.m. <sup>2</sup>J[<sup>119</sup>Sn–<sup>1</sup>H] = 83 Hz. Dimethyldiphenyltin chloride was synthesized by the cleavage of dimethyldiphenyltin with hydrogen chloride in a methanol–carbon tetrachloride mixture at 283 K, and was purified by distillation at 363 K/1 Torr (Simmones, 1977).

#### Crystal data

[SnCl(CH <sub>3</sub> ) <sub>2</sub> (C <sub>6</sub> H <sub>5</sub> )(H <sub>2</sub> O)] <sub>2</sub> · C <sub>12</sub> H <sub>24</sub> O <sub>8</sub>	$D_x = 1.541 \text{ Mg m}^{-3}$
$M_r = 822.96$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 4938 reflections
$a = 9.6395 (6) \text{ \AA}$	$\theta = 2.4\text{--}26.5^\circ$
$b = 19.472 (1) \text{ \AA}$	$\mu = 1.60 \text{ mm}^{-1}$
$c = 10.5110 (7) \text{ \AA}$	$T = 168 (2) \text{ K}$
$\beta = 116.015 (1)^\circ$	Block, colorless
$V = 1773.0 (2) \text{ \AA}^3$	$0.50 \times 0.48 \times 0.37 \text{ mm}$
$Z = 2$	

#### Data collection

Siemens CCD area-detector diffractometer	3392 independent reflections
$\omega$ scans	2495 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$R_{\text{int}} = 0.104$
$T_{\text{min}} = 0.502$ , $T_{\text{max}} = 0.589$	$\theta_{\text{max}} = 26.5^\circ$
9926 measured reflections	$h = -10 \rightarrow 11$
	$k = -24 \rightarrow 24$
	$l = -10 \rightarrow 13$

#### Refinement

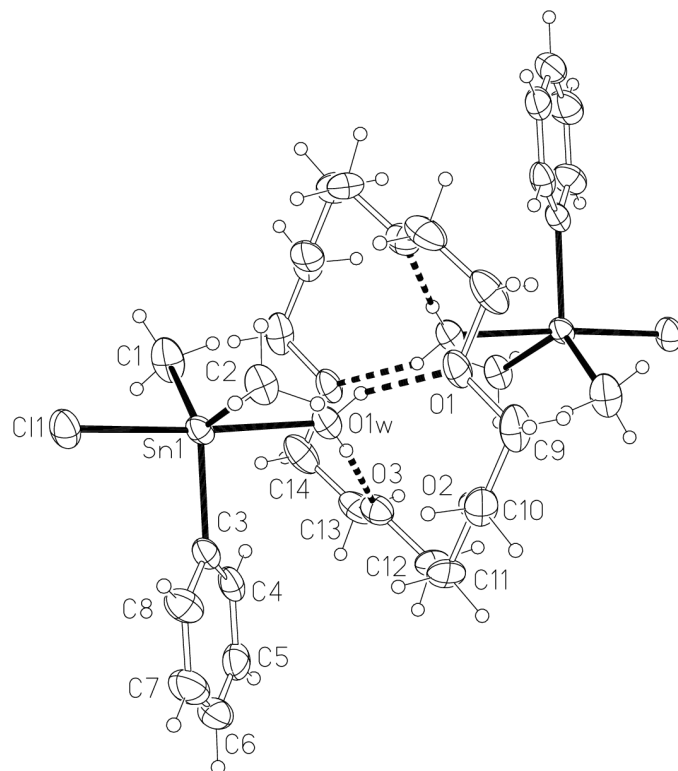
Refinement on $F^2$	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.060$	$wR_{\text{int}} = 0.107$
$wR(F^2) = 0.107$	$w = 1/[\sigma^2(F_o^2) + (0.0348P)^2]$
$S = 0.95$	where $P = (F_o^2 + 2F_c^2)/3$
3392 reflections	$(\Delta/\sigma)_{\text{max}} = 0.001$
177 parameters	$\Delta\rho_{\text{max}} = 2.62 \text{ e \AA}^{-3}$
	$\Delta\rho_{\text{min}} = -1.76 \text{ e \AA}^{-3}$

**Table 1**

Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ).

Sn1–C1	2.111 (6)	Sn1–O1w	2.445 (4)
Sn1–C2	2.119 (5)	Sn1–Cl1	2.484 (2)
Sn1–C3	2.163 (3)		
C1–Sn1–C2	121.9 (3)	C2–Sn1–O1w	82.3 (2)
C1–Sn1–C3	121.2 (2)	C2–Sn1–Cl1	95.2 (2)
C1–Sn1–O1w	84.9 (2)	C3–Sn1–O1w	88.0 (2)
C1–Sn1–Cl1	94.0 (2)	C3–Sn1–Cl1	95.8 (1)
C2–Sn1–C3	114.6 (2)	O1w–Sn1–Cl1	176.1 (1)

The minor disorder in the 18-crown-6 ring was treated by constraining the C–O distances to be approximately equal by a SADI 0.01 instruction in *SHELXL97*; the C–C distances were also constrained by another SADI 0.01 instruction. The phenyl ring was refined as a rigid hexagon. The carbon-bound H atoms were positioned geometrically, and were allowed to ride on the parent C atoms, with  $U(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ . The water H atoms were located and their positions refined, subject to  $\text{O–H} = 0.85 \pm 0.01$ ,  $\text{H} \cdots \text{H} = 1.39 \pm 0.01 \text{ \AA}$ , and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{O})$ . The final difference map had a large peak/hole at about  $1 \text{ \AA}$  from atom Sn1.



**Figure 1**

ORTEP II (Johnson, 1976) plot of the title dinuclear complex, with displacement ellipsoids drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radii.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP II* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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