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Aquatrichloroacetatotriphenyltin-1,10-phenanthroline (2/2)

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Key indicators

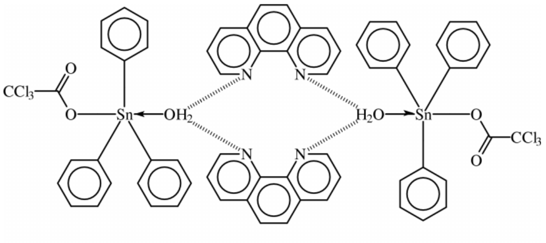
Single-crystal X-ray study
T = 168 K
Mean σ(C–C) = 0.004 Å
R factor = 0.023
wR factor = 0.062
Data-to-parameter ratio = 15.4

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

The 1,10-phenanthroline heterocycle in the centrosymmetric four-molecule aggregate $[Sn(C_2O_2Cl_3)(C_6H_5)_3(H_2O)]_2\cdot 2C_{12}H_8N_2$ interacts with the water molecule of the *trans*-C_3SnO_2 trigonal-bipyramidal aquacarboxylatotriorganotin molecule through the coordinated water molecule through hydrogen bonds [$O_{water}\cdots N$ = 2.755 (2) and 2.758 (2) Å].

Comment

In the centrosymmetric dimeric, five-coordinate complex of aqua(trifluoroacetato)triphenyltin and 1,10-phenanthroline, the coordinated water molecule [$Sn\leftarrow O_{water}$ = 2.335 (4) Å] forms hydrogen bonds to two 1,10-phenanthroline units [$O_{water}\cdots N$ = 2.799 (6) and 2.846 (6) Å] across a center of inversion (Ng *et al.*, 1996). Replacing the trifluoromethyl group by the less electron-withdrawing dichlorofluoromethyl group leads to the formation of a similar compound (Ng, 1997), as does replacement by the trichloromethyl group in this study, (I). The Sn atom shows *trans*-C_3O_2 trigonal bipyramidal coordination (Fig. 1); the apical positions are occupied by the water O atom and the O atom of the trichloroacetato group [Sn—O = 2.208 (1) Å and $Sn\leftarrow O$ = 2.308 (1) Å]. The 1,10-phenanthroline heterocycle interacts indirectly with the organotin entity through the coordinated water molecule *via* somewhat stronger hydrogen bonds [$O_{water}\cdots N$ = 2.755 (2) and 2.758 (2) Å].



(I)

Experimental

Triphenyltin hydroxide (1.84 g, 5 mmol), trichloroacetic acid (0.82 g, 5 mol) and 1,10-phenanthroline (0.99 g, 5 mmol) were heated in a small volume of acetone until the reagents dissolved completely. Colorless crystals separated from the solution upon cooling; m.p. 414–416 K. The use of either methanol or ethanol did not lead to the formation of the adduct. The condensation of the hydroxide with the acid, if carried out in methanol, would yield triphenyltin trichloroacetate methanol solvate (Alcock & Roe, 1989).

Crystal data

$[Sn(C_2Cl_3O_2)(C_6H_5)_3(H_2O)]_2\cdot 2C_{12}H_8N_2$
$M_r = 1421.16$
Triclinic, $P\overline{1}$
$a = 9.1542 (6)$ Å
$b = 12.2739 (7)$ Å
$c = 14.4308 (9)$ Å
$\alpha = 67.559 (1)°$
$\beta = 86.347 (1)°$
$\gamma = 89.490 (1)°$
$V = 1495.4 (2)$ Å^3
$Z = 1$
$D_x = 1.578$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 7435 reflections
$\theta = 1.9–26.4°$
$\mu = 1.16$ mm^{-1}
$T = 168 (2)$ K
Irregular fragment, colorless
$0.76 \times 0.62 \times 0.31$ mm

Data collection

Siemens $P4$/CCD SMART area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.561$, $T_{max} = 0.698$
18009 measured reflections
5840 independent reflections
5252 reflections with $I > 2\sigma(I)$
$R_{int} = 0.021$
$\theta_{max} = 26.4°$
$h = -11 \rightarrow 11$
$k = -15 \rightarrow 15$
$l = -11 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.023$
$wR(F^2) = 0.062$
$S = 1.05$
5840 reflections
378 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0412P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.12$ e Å^{-3}
$\Delta\rho_{min} = -0.75$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Sn1—C1	2.125 (2)	Sn1—O1	2.208 (1)
Sn1—C7	2.126 (2)	Sn1—O1*w*	2.308 (1)
Sn1—C13	2.130 (2)		
C1—Sn1—C7	122.2 (1)	C7—Sn1—O1	97.4 (1)
C1—Sn1—C13	115.5 (1)	C7—Sn1—O1*w*	85.7 (1)
C1—Sn1—O1	87.3 (1)	C13—Sn1—O1	91.7 (1)
C1—Sn1—O1*w*	87.2 (1)	C13—Sn1—O1*w*	90.4 (1)
C7—Sn1—C13	121.8 (1)	O1—Sn1—O1*w*	174.5 (1)

A somewhat large specimen was used for the diffraction measurements, the longest side being almost 0.8 mm, the diameter of the X-ray beam. The absorption correction program probably corrected for those reflections for which the crystal was not entirely bathed by the beam. The refined structure did not show any unacceptable features.

The water H atoms were located and refined; the two O—H distances were restrained to be approximately equal. The aromatic H atoms were placed geometrically, and were included in the refinement in the riding-model approximation with their displacement parameters 1.2 times U_{eq} of the C atoms. Peaks of about 1 e Å^{-3} were found near the Cl atoms.

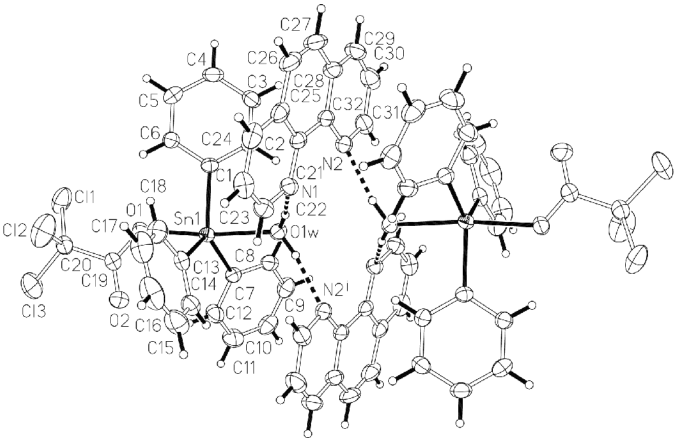


Figure 1
*ORTEP*II (Johnson, 1976) plot of (I), with displacement ellipsoids drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radii. [Symmetry code: (i) $1 - x$, $1 - y$, $1 - z$.]

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