metal-organic papers

μ-Succinato-bis(aquatriphenyltin) bis(o-phenanthroline)

Chin Fei Chee, Kong Mun Lo and Seik Weng Ng*

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 σ(C–C) = 0.002 Å
R factor = 0.017
wR factor = 0.048
Data-to-parameter ratio = 15.1

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

In the crystal structure of the title compound, $[Sn_2(C_6H_5)_6$-$(C_4H_4O_4)(H_2O)_2]\cdot 2C_{12}H_8N_2$, each Sn atom in the centrosymmetric μ-succinato-bis(aquatriphenyltin) molecule interacts with an o-phenanthroline indirectly through the coordinated water molecule [Sn←O$_{water}$ = 2.612 (1) Å], which forms a short hydrogen bond with one of the o-phenanthroline N atoms [O$_{water}$···N = 2.899 (2) Å]; the five-coordinate Sn atom shows trans–C_3SnO_2 trigonal bipyramidal coordination. The weak hydrogen bond involving the second H atom of the water molecule and the carbonyl O atom of an adjacent molecule of μ-succinato-bis(aquatriphenyltin) [O···O^i = 3.123 (2) Å; symmetry code: (i) x, $\frac{1}{2} - y$, $\frac{1}{2} + z$] links the molecules into infinite layers parallel to the bc plane of the crystal.

Comment

The presence of electron-withdrawing substituents in the carboxylate group in triphenyltin carboxylates is usually required in order to increase the Lewis acidity of the triphenyltin acceptor for such triphenyltin carboxylates to afford complexes with oxygen-donor ligands (Ng & Kumar Das, 1997). Bis(triphenyltin) succinate represents an odd exception, as it is capable of forming such adducts although the carboxylate group itself does not appear to have any enhanced electron-withdrawing capacity (Ng, 1998a). When treated with tris(2-pyridyl)-1,3,5-triazine, the compound yields a water-coordinated complex that uses its water H atoms to interact with the N atoms of two pyridyl rings [Sn←O$_{water}$ = 2.487 (3) Å] in the monoclinic modification and 2.441 (3) Å in the triclinic modification] (Ng, 1998b).

(I)
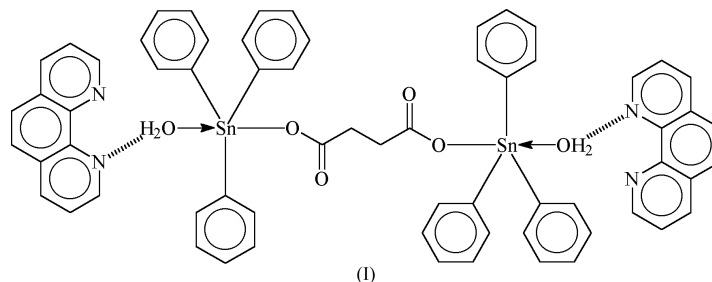
In the structure of the title compound, (I), the coordinated water molecule is only weakly bonded to the Sn atom of the centrosymmetric μ-succinato-bis(aquatriphenyltin) moiety [Sn1←O1W = 2.612 (1) Å], and it uses one of its H atoms to interact with only one N atom of the o-phenanthroline entity [O1W···N1 2.889 (2) Å] (Fig. 1). This hydrogen-bonding distance is similar to that found in o-phenanthroline hydrate (Ng, 1997). The weak hydrogen bond involving the second H
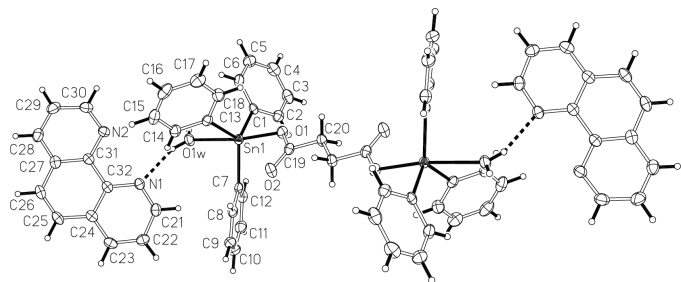
Figure 1
*ORTEP*II (Johnson, 1976) plot of the title compound, with ellipsoids at the 50% probability level. H atoms are drawn as spheres of arbitrary radii.

atom of the water molecule and the carbonyl O atom of an adjacent molecule of μ-succinato-bis(aquatriphenyltin) [O1$W \cdots$O2^i = 3.123 (2) Å; symmetry code: (i) $x, \frac{1}{2} - y, \frac{1}{2} + z$] consolidates the structure by linking the molecules of the complex into infinite layers parallel to the *bc* plane of the crystal.

The Sn atom has a distorted trigonal bipyramidal coordination with both O atoms in apical positions [O1—Sn—O1W = 170.3 (1)°].

Experimental

Bis(triphenyltin) succinate was synthesized by the condensation of triphenyltin hydroxide (0.73 g, 20 mmol) and succinic acid (0.12 g, 10 mmol) in ethyl acetate (Ng, 1998*a*). The compound (0.82 g, 10 mmol) and *o*-phenanthroline monohydrate (0.40 g, 20 mmol) were dissolved in a small volume of hot acetone. Crystals (m.p. 414–416 K) were deposited from the solution when the solvent was allowed to evaporate slowly. The use of ethanol as solvent did not lead to the formation of the complex (Ng, 1998*a*); an ethanol adduct having the empirical formulation [(C₆H₅)₃Sn]₂(O₂CCH₂CH₂CO₂)·2C₂H₅OH was probably formed instead (Ng *et al.*, 1994).

Crystal data

[Sn₂(C₆H₅)₆(C₄H₄O₄)-
  (H₂O)₂]·2C₁₂H₈N₂
M_r = 1212.49
Monoclinic, $P2_1/c$
a = 13.5958 (8) Å
b = 13.6077 (8) Å
c = 14.4444 (9) Å
β = 101.014 (1)°
V = 2623.1 (3) Å³
Z = 2

D_x = 1.535 Mg m⁻³
Mo $K\alpha$ radiation
Cell parameters from 7254
  reflections
θ = 2.1–26.4°
μ = 1.01 mm⁻¹
T = 168 (2) K
Block, colorless
0.50 × 0.45 × 0.20 mm

Data collection

Bruker SMART CCD area-detector
  diffractometer
ω scans
Absorption correction: multi-scan
  (*SADABS*; Sheldrick, 1996)
  T_{min} = 0.632, T_{max} = 0.823
33 422 measured reflections

5303 independent reflections
4570 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 26.4°
$h = -16 \rightarrow 16$
$k = -16 \rightarrow 16$
$l = -15 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.017
$wR(F^2)$ = 0.048
S = 1.02
5303 reflections
351 parameters

H atoms treated by a mixture of
  independent and constrained
  refinement
$w = 1/[\sigma^2(F_o^2) + (0.0316P)^2]$
  where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.27 e Å⁻³
$\Delta\rho_{min}$ = −0.44 e Å⁻³

Table 1
Selected geometric parameters (Å, °).

Sn1—O1	2.113 (1)	Sn1—C7	2.121 (2)
Sn1—O1W	2.612 (1)	Sn1—C13	2.125 (2)
Sn1—C1	2.124 (2)		
C1—Sn1—C7	116.7 (1)	C7—Sn1—O1	104.3 (1)
C1—Sn1—C13	117.3 (1)	C7—Sn1—O1W	82.2 (1)
C1—Sn1—O1	90.2 (1)	C13—Sn1—O1	97.5 (1)
C1—Sn1—O1W	80.4 (1)	C13—Sn1—O1W	84.9 (1)
C7—Sn1—C13	121.0 (1)	O1—Sn1—O1W	170.3 (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$\cdots$N1	0.83 (1)	2.10 (1)	2.899 (2)	161 (2)
O1W—H1W2$\cdots$O2^i	0.84 (1)	2.29 (1)	3.123 (2)	173 (2)

Symmetry code: (i) $x, \frac{1}{2} - y, \frac{1}{2} + z$.

The carbon-bound H atoms were positioned geometrically and were allowed to ride on their parent C atoms, with U_{iso}(H) = $1.2U_{eq}$(C). The water H atoms were located and refined, subject to an O—H = 0.85±0.01 Å restraint.

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

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.
Ng, S. W. (1997). *Z. Kristallogr. New Cryst. Struct.* **212**, 283–284.
Ng, S. W. (1998*a*). *Acta Cryst.* C**54**, 745–750.
Ng, S. W. (1998*b*). *Acta Cryst.* C**54**, 1386–1389.
Ng, S. W. & Kumar Das, V. G. (1997). *Trends Organomet. Chem.* **2**, 107–115.
Ng, S. W., Kumar Das, V. G., Wang, R. J. & Mak, T. C. W. (1994). *Malays. J. Sci.* **15B**, 7–11.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.