Table 1. *Selected geometric parameters (Å, °)*

$Cd1—O1^i$	2.272 (2)	Cd1—N1	2.442 (2)
$Cd1—O3^{ii}$	2.307 (2)	S1—O1	1.521 (2)
Cd1—O1*W*	2.335 (2)	S1—O2	1.525 (2)
Cd1—N2	2.338 (2)	S1—O3	1.543 (2)
Cd1—O3	2.413 (2)		
$O1^i—Cd1—O3^{ii}$	110.50 (8)	N2—Cd1—O3	92.47 (8)
$O1^i—Cd1—O1W$	82.18 (9)	$O1^i—Cd1—N1$	88.73 (9)
$O3^{ii}—Cd1—O1W$	90.70 (8)	$O3^{ii}—Cd1—N1$	160.52 (8)
$O1^i—Cd1—N2$	104.24 (9)	O1*W*—Cd1—N1	88.85 (8)
$O3^{ii}—Cd1—N2$	107.52 (8)	N2—Cd1—N1	68.99 (8)
O1*W*—Cd1—N2	156.50 (8)	O3—Cd1—N1	87.32 (8)
$O1^i—Cd1—O3$	160.14 (8)	O1—S1—O2	105.08 (14)
$O3^{ii}—Cd1—O3$	73.54 (8)	O1—S1—O3	101.13 (13)
O1*W*—Cd1—O3	78.29 (8)	O2—S1—O3	105.87 (12)

Symmetry codes: (i) $x, y, z-1$; (ii) $1-x, -y, 1-z$.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H1*WB*···O2	0.83 (3)	1.93 (3)	2.729 (3)	162 (3)
O1*W*—H1*WA*···O2*W*	0.85 (4)	1.93 (4)	2.761 (4)	166 (3)
$O2W—H2WB\cdots O2^i$	0.87 (4)	1.94 (4)	2.811 (3)	172 (4)
$O2W—H2WA\cdots O2^{ii}$	0.88 (4)	2.12 (4)	2.995 (4)	177 (3)

Symmetry codes: (i) $2-x, -y, 1-z$; (ii) $x, y, z-1$.

Non-H atoms were refined anisotropically on F^2. Those H atoms which were unequivocally defined by the stereochemistry were placed at their calculated positions, riding on their host atoms (C—H 0.96 Å). Those corresponding to water molecules were found in successive ΔF syntheses and refined with restrained O—H and H—H values.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *MSC/AFC Diffractometer Control Software*. Program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997*a*). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997*b*). Molecular graphics: *XP* in *SHELXTL/PC* (Sheldrick, 1994). Software used to prepare material for publication: *PARST* (Nardelli, 1983) and CSD (Allen & Kennard, 1993).

We thank the Spanish Research Council (CSIC) for providing us with a free-of-charge license to the CSD system.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BK1460). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1999). C**55**, 1280–1282

Aqua[4-(4-chlorophenyl)-2-phenylthiazole-5-acetato-*O*]trimethyltin(IV)

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(*Received 22 March 1999; accepted 9 April 1999*)

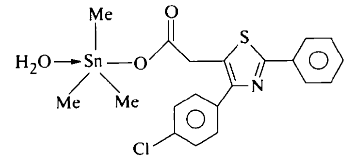
Abstract

The crystal structure of the title compound, $[Sn(CH_3)_3(C_{17}H_{11}ClNO_2S)(H_2O)]$, contains hydrogen-bonded infinite planes, wherein each trimethyltin moiety is coordinated to a water molecule and an O atom of the carboxylate ligand. The Sn atom has a distorted trigonal-bipyramidal geometry with three methyl groups in the equatorial plane [mean Sn—C 2.114 (7) Å]. The O atoms bonded to the Sn atom in the axial positions have significantly different Sn—O bond lengths [2.167 (4) and 2.490 (4) Å].

Comment

The structural chemistry of organotin carboxylic acid esters has been extensively explored in recent years because of the rich diversity of structural motifs in the series (Tiekink, 1994). Only a handful of structures have been cited in the literature (Cambridge Structural Database; Allen *et al.*, 1983) in which Sn atoms are bonded to three organic groups defining the trigonal plane and the axial positions are occupied by O atoms of a carboxylate ligand and a coordinated water molecule. In a continuation of our work on the structural chemistry of organotin carboxylates (Danish, Ali, Mazhar, Badshah, Masood & Tiekink, 1995; Danish, Ali, Mazhar, Badshah & Tiekink, 1995; Danish *et al.*, 1996; Parvez *et al.*, 1997; Tahir, Ülkü, Danish *et al.*, 1997; Tahir, Ülkü, Ali *et al.*, 1997; Bhatti *et al.*, 1999), we now report the crystal structure of aqua[4-(4-chlorophenyl)-2-phenylthiazole-5-acetato-*O*]trimethyltin(IV), (I).

Acta Crystallographica Section C
ISSN 0108-2701 © 1999



(I)

The structure of (I) is presented in Fig. 1. The Sn atom has three methyl groups bonded in the equatorial plane. The Sn—C distances, which are almost identical, lie in the range 2.110 (6)–2.120 (8) Å and are in agreement with the corresponding values reported for related structures (Cambridge Structural Database; Allen *et al.*, 1983). The Sn—O bonds in the axial positions, involving one carboxyl O atom [Sn1—O1 2.167 (4) Å] and a water O atom in the *trans* position [Sn1—O3 2.490 (4) Å], are significantly different from each other. The O—Sn—O angle is 178.30 (14)°. The Sn atom has a distorted trigonal-bipyramidal geometry, with the Sn atom 0.167 (4) Å out of the equatorial plane formed by the three methyl C atoms towards the more strongly bound O1 atom. The structure is stabilized by hydrogen bonds between water H atoms, the N atom of the thiazole ring and the O atom of the CO group not coordinated to Sn, thus forming infinite planes. The molecular dimensions in the 4-(4-chlorophenyl)-2-phenyl-5-thiazoleacetate ligand are normal.

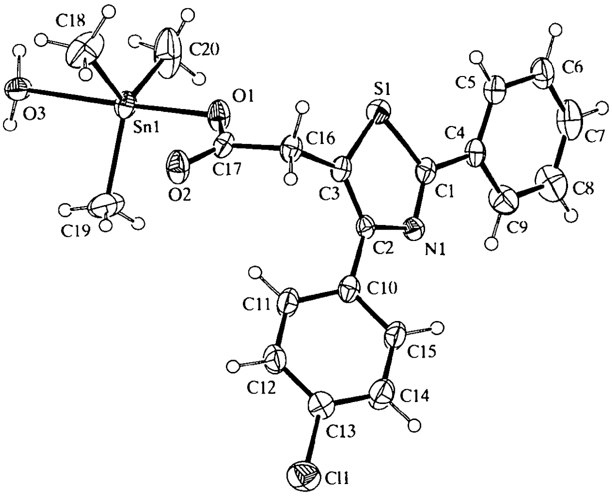


Fig. 1. *ORTEP*II (Johnson, 1976) drawing of (I) with the atomic numbering scheme. Displacement ellipsoids have been plotted at the 30% probability level and H atoms are shown as spheres of arbitrary radii.

Some of the crystal structures of Sn complexes similar to (I) which have been reported include aquatriphenyl(8-quinolyloxyacetato-*O*)tin(IV) (Kumar Das *et al.*, 1987), aquatris(*n*-butyl)(*N*-phthaloylglycinato)tin (Ng *et al.*, 1994), aqua{2-[*N*′-(3-formyl-4-hydroxyphenyl)diazo]benzoato}triphenyltin (Basu Baul *et al.*, 1996), aqua(*p*-chlorophenyl)diphenyl(*N*-phthaloylglycinato)tin(IV) (Lo *et al.*, 1997), bis[aqua(chlorodifluoroacetato-*O*)triphenyltin–1,10-phenanthroline] (Ng, 1997), aqua(3-methyl-4-benzoylpyrazol-5-onato)tri-*n*-butyltin(IV) (Mahon *et al.*, 1996), aquatrimethyl[4-(*p*-methoxybenzoyl)-1-phenyl-3-methylpyrazolon-5-ato]tin(IV) (Marchetti *et al.*, 1996), aqua[bis(*N*,*N*′-dimethyldithiocarbamyl)acetato]triphenyltin monohydrate (Ng & Kumar Das, 1995) and *N*-phthaloylglycinatotrimethylaquatin (Ovsetsina *et al.*, 1993).

Experimental

The title compound was prepared by treating equimolar amounts of the sodium salt of 4-(4-chlorophenyl)-2-phenyl-5-thiazoleacetate with trimethyltin chloride in ethanol under reflux conditions for 5–6 h. The sodium chloride formed was removed by filtration and the solid obtained after removal of the solvent was crystallized from dichloromethane solution to provide crystals of (I).

Crystal data

$[Sn(CH_3)_3(C_{17}H_{11}ClNO_2S)(H_2O)]$
$M_r = 510.59$
Monoclinic
$P2_1/c$
$a = 12.6008\ (11)$ Å
$b = 11.349\ (3)$ Å
$c = 15.634\ (2)$ Å
$\beta = 94.888\ (9)°$
$V = 2227.6\ (7)$ Å^3
$Z = 4$
$D_x = 1.522$ Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
$\lambda = 1.54178$ Å
Cell parameters from 25 reflections
$\theta = 10–15°$
$\mu = 11.26$ mm^{-1}
$T = 293\ (1)$ K
Prismatic
0.30 × 0.22 × 0.18 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical *via* ψ scan (3 reflections) (North *et al.*, 1968)
$T_{min} = 0.074$, $T_{max} = 0.132$
3899 measured reflections
3811 independent reflections

3160 reflections with $I > 2\sigma(I)$
$R_{int} = 0.055$
$\theta_{max} = 68°$
$h = 0 \rightarrow 15$
$k = 0 \rightarrow 12$
$l = -18 \rightarrow 18$
3 standard reflections every 200 reflections intensity decay: 5.03%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.114$
$S = 1.025$
3811 reflections
247 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.056P)^2 + 5.85P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.107$ e Å^{-3}
$\Delta\rho_{min} = -1.306$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Sn1—C18	2.110 (6)	S1—C3	1.713 (6)
Sn1—C19	2.113 (7)	S1—C1	1.730 (5)
Sn1—C20	2.120 (8)	O1—C17	1.272 (6)
Sn1—O1	2.167 (4)	O2—C17	1.220 (6)
Sn1—O3	2.490 (4)	N1—C1	1.290 (7)
Cl1—C13	1.730 (6)	N1—C2	1.383 (6)
C18—Sn1—C19	123.2 (4)	C19—Sn1—O3	84.9 (2)
C18—Sn1—C20	117.3 (4)	C20—Sn1—O3	87.9 (2)
C19—Sn1—C20	117.6 (4)	O1—Sn1—O3	178.30 (14)
C18—Sn1—O1	96.0 (2)	C3—S1—C1	90.3 (3)
C19—Sn1—O1	96.6 (2)	C17—O1—Sn1	119.4 (3)
C20—Sn1—O1	90.8 (2)	C1—N1—C2	111.4 (4)
C18—Sn1—O3	83.7 (2)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H31···O2^{i}	0.82	1.91	2.713 (6)	168
O3—H32···N1^{ii}	0.82	2.13	2.928 (6)	163

Symmetry codes: (i) $2 - x, \frac{1}{2} + y, \frac{3}{2} - z$; (ii) $2 - x, -y, 2 - z$.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1994). Program(s) used to solve structure: *SAPI*91 (Fan, 1991). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *TEXSAN*. Software used to prepare material for publication: *SHELXL*97.

The authors thank the Natural Sciences and Engineering Research Council, Canada, for providing the diffractometer through an equipment grant to the University of Calgary. MHB and SA are grateful to the Pakistan Science Foundation (PSF) for financial support.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1550). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1999). C**55**, 1282–1284

A trinuclear Pd_2Ni complex, $[(C_6H_5)_4P]_2[(PdCl_3)_2\{Ni(C_{12}H_{18}N_2O_2)\}]$

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(*Received 23 February 1999; accepted 20 April 1999*)

Abstract

In the title complex, bis(tetraphenylphosphonium) di-μ-chloro-1:3κ^2*Cl*,2:3κ^2*Cl*-tetrachloro-1κ^2*Cl*,2κ^2*Cl*-μ_3-[4,4′-(1,2-ethanediyldinitrilo)bis(pentan-2-onato)]-1κC^3:2κ^4*O*,*N*,*N*′,*O*′:3$\kappa C^{3'}$-dipalladium(II)nickel(II), $(C_{24}H_{20}P)_2[Pd_2NiCl_6(C_{12}H_{18}N_2O_2)]$, the trinuclear complex anion, $[(PdCl_3)_2\{Ni(acen)\}]^{2-}$, has twofold crystallographic symmetry and consists of a central {Ni(acen)} unit and two terminal $\{PdCl_3\}$ moieties. These are connected through Pd—C [2.120 (2) Å] and Cl—Ni bonds [2.4919 (5) Å], giving an elongated octahedral coordination geometry around the central Ni^{II} ion. The $\{PdCl_3\}$ moiety acts as a pendant arm attached to the {Ni(acen)} unit, and the geometry around the Pd atom is of the square-planar type.

Comment

Schiff base complexes derived from the metal-template reaction of acetylacetone and polyamines have a charge-