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A 1:2 adduct of 3-(piperidin-1-yl)propionic acid and triphenyltin chloride

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Key indicators

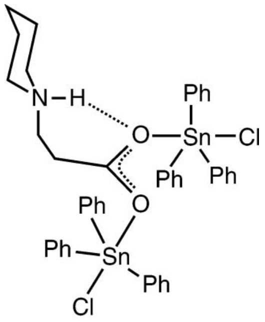
Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.004 Å
R factor = 0.022
wR factor = 0.025
Data-to-parameter ratio = 16.1

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

3-(Piperidin-1-yl)propionic acid, $C_8H_{15}NO_2$, forms a 1:2 adduct, bis-chlorotriphenyl[3-(piperidinium-1-yl)propionato]-tin(IV $(C_8H_{15}NO_2)[Sn(C_6H_5)_3Cl]_2$, with triphenyltin chloride, $[Sn(C_6H_5)_3Cl]$. The acidic H atom is transferred to the imine N atom and an N—H···O intramolecular hydrogen bond is formed. The carboxylate group bridges the two Sn atoms.

Comment

When triphenyltin chloride reacts with Schiff bases, the products are usually 1:1 adducts, in which the Sn is coordinated by the phenolic O atom and the phenolic H atom is transferred to the imine N atom (Hazell *et al.*, 1996). These compounds show promising fungicidal activity (Khoo *et al.*, 1995). We have now prepared and studied the products of the reaction of triphenyltin chloride with 3-(piperidin-1-yl)propionic acid.



(I)

The title compound, (I), a 1:2 adduct of formula $(C_8H_{15}NO_2)[SnPh_3Cl]_2$, can be synthesized directly or obtained by refluxing the chloroform solvate of the 1:1 adduct, $(C_8H_{15}NO_2)[SnPh_3Cl]\cdot CHCl_3$ (Yan & Khoo, 2005), in methanol.

The Sn atoms in (I) are five-coordinated by three phenyl groups, a Cl atom and one O atom of the carboxylate group (Fig. 1). The Sn coordination is trigonal–bipyramidal (tbp) (Table 1), with the three phenyl groups in the equatorial positions. There is significant distortion from ideal tbp geometry: the C—Sn—C angles deviate from 120° and the axial bonds are not perpendicular to the equatorial plane. The Sn atoms are displaced from their equatorial mean planes in the direction of the Cl atom by 0.069 (2) and 0.154 (2) Å for Sn1 and Sn2, respectively.

The piperidinylpropionic acid is zwitterionic, with a protonated N atom and a deprotonated carboxylate group. One of the carboxylate O atoms, O1, acts as the intramolecular hydrogen-bond acceptor for the N—H group (Fig. 1,

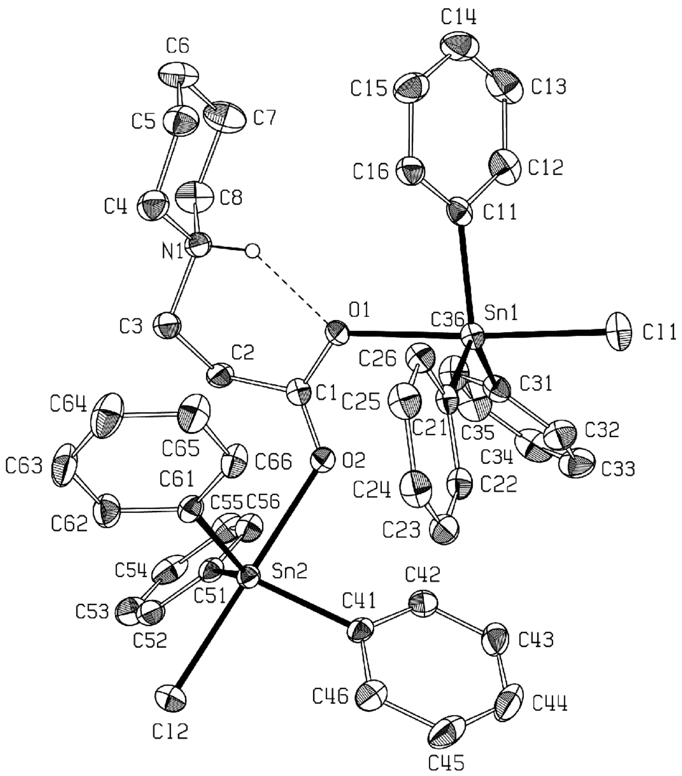


Figure 1
A view of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and all H atoms have been omitted, except for that attached to N1. The dashed line indicates the intramolecular hydrogen bond.

Table 2). The C1—O1 bond is notably longer than the C1—O2 bond.

Experimental

3-(Piperidin-1-yl) acid (0.40 g, 2.5 mmol) in methanol was added to a solution of triphenyltin chloride (1.95 g, 5 mmol) in methanol (20 ml). The mixture was heated to boiling for 5 min and allowed to cool overnight. Crystals of (I) were recrystallized from methanol (m.p. 406–408 K). Analysis, found: C 57.69, H 4.51, N 1.60%; $C_{44}H_{45}Cl_2NO_2Sn_2$ requires: C 56.93, H 4.90, N 1.51%. Spectroscopic analysis: IR (KBr discs, ν, cm^{-1}): 3350 (*w*, *br*, OH), 1600 (*s*, CO), 700, 750 (SnPh); ^{13}C NMR ($CDCl_3$, δ, p.p.m.): 175.09 (C_{CO}), 29.85 (C_α), 52.69 (C_β), 53.55 (C2), 23.41 (C3), 21.09 (C4), 128.92–138.39 (Ph_3Sn); ^{1}H NMR ($CDCl_3$, δ, p.p.m.): 2.64, 2.22 (H_α, H_β, *t*, 2H), 1.38 (H4, *br*, 2H), 1.44 (H3, H5, *m*, 4H), 2.53 (H2, H6, *br*, 4H), 7.3–7.8 (Ph_3Sn, 30H).

Crystal data

$(C_8H_{15}NO_2)[Sn(C_6H_5)_3Cl]_2$
$M_r = 928.13$
Monoclinic, $P2_1/c$
$a = 13.223$ (3) Å
$b = 15.779$ (3) Å
$c = 19.574$ (4) Å
$\beta = 101.065$ (4)°
$V = 4008$ (1) Å^3
$Z = 4$

$D_x = 1.538$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6353 reflections
$\theta = 2.0$–29.8°
$\mu = 1.42$ mm^{-1}
$T = 120$ K
Block, colourless
0.36 × 0.22 × 0.17 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: integration (*XPREP*; Siemens, 1995) $T_{min} = 0.683$, $T_{max} = 0.802$
27 396 measured reflections

10 724 independent reflections
7497 reflections with $I > 3\sigma(I)$
$R_{int} = 0.041$
$\theta_{max} = 29.5°$
$h = -18 \rightarrow 17$
$k = -18 \rightarrow 22$
$l = -27 \rightarrow 23$

Refinement

Refinement on F
$R[F^2 > 3\sigma(F^2)] = 0.022$
$wR(F^2) = 0.025$
$S = 0.81$
7497 reflections
465 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/\{[\sigma_{cs}(F^2) + 1.03F^2]^{1/2} - |F|\}^2$
$(\Delta/\sigma)_{max} = 0.003$
$\Delta\rho_{max} = 0.81$ e Å^{-3}
$\Delta\rho_{min} = -0.65$ e Å^{-3}
Extinction correction: B–C type 1 Lorentzian isotropic (Becker & Coppens, 1974)
Extinction coefficient: 24 (5)

Table 1
Selected geometric parameters (Å, °).

Sn1—Cl1	2.525 (1)	Sn2—O2	2.406 (2)
Sn1—O1	2.382 (2)	Sn2—C41	2.131 (2)
Sn1—C11	2.131 (2)	Sn2—C51	2.140 (2)
Sn1—C21	2.126 (2)	Sn2—C61	2.133 (2)
Sn1—C31	2.140 (2)	C1—O1	1.274 (3)
Sn2—Cl2	2.494 (1)	C1—O2	1.249 (3)
Cl1—Sn1—O1	176.43 (6)	Cl2—Sn2—C41	95.28 (6)
Cl1—Sn1—C11	93.79 (6)	Cl2—Sn2—C51	95.05 (6)
Cl1—Sn1—C21	89.72 (6)	Cl2—Sn2—C61	92.24 (6)
Cl1—Sn1—C31	92.03 (7)	O2—Sn2—C41	84.03 (7)
O1—Sn1—C11	83.10 (7)	O2—Sn2—C51	90.06 (7)
O1—Sn1—C21	90.14 (7)	O2—Sn2—C61	83.68 (7)
O1—Sn1—C31	90.98 (7)	C41—Sn2—C51	114.97 (8)
C11—Sn1—C21	116.76 (9)	C41—Sn2—C61	122.53 (9)
C11—Sn1—C31	118.06 (9)	C51—Sn2—C61	120.95 (8)
C21—Sn1—C31	124.89 (9)	Sn1—O1—C1	124.1 (1)
Cl2—Sn2—O2	174.63 (5)	Sn2—O2—C1	127.5 (1)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N—H1*N*···O1	0.96 (3)	2.05 (3)	2.835 (3)	138 (2)

The N-bound H atom was located in a difference map and freely refined. The other H atoms were located in idealized positions (C—H = 0.95 Å) and refined as riding on their carrier atoms, with $U_{iso}(H) = 1.2U_{eq}$(carrier).

Data collection: *SMART* (Siemens, 1995); cell refinement: *SAINT* (Siemens, 1995); data reduction: *SAINT*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1997) and *KRYSTAL* (Hazell, 1995); program(s) used to refine structure: modified *ORFLS* (Busing *et al.*, 1962) and *KRYSTAL*; molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996) and *KRYSTAL*; software used to prepare material for publication: *KRYSTAL*.

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