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

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Key indicators

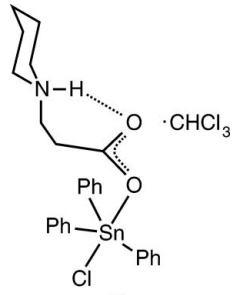
Single-crystal X-ray study
T = 297 K
Mean σ(C–C) = 0.013 Å
H-atom completeness 97%
Disorder in solvent or counterion
R factor = 0.045
wR factor = 0.126
Data-to-parameter ratio = 13.8

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 forms a 1:1 adduct with chlorotriphenyltin, *viz*. chlorotriphenyl[3-(piperidinium-1-yl)propionato]tin(IV) chloroform solvate, $[Sn(C_6H_5)_3(C_8H_{15}O_2)Cl]\cdot CHCl_3$. The acidic H atom is transferred to the piperidine N atom and an N—H···O intramolecular hydrogen bond is formed.

Comment

Triphenyltin chloride forms adducts, usually of 1:1 stoichiometry, with phenolic Schiff bases. These compounds show promising fungicidal activity (Khoo *et al.*, 1995) and, as part of our ongoing studies of these materials, we have prepared and studied the products of the reaction of triphenyltin chloride with 3-(piperidin-1-yl)propionic acid. The title compound, (I), can be synthesized as a 1:1 adduct–methanol solvate. However, refluxing in methanol yields the 1:2 adduct (Khoo & Hazell, 2005), while recrystallization from chloroform yields the title 1:1 adduct, chlorotriphenyl[3-(piperidinium-1-yl)-propionato]tin(IV) chloroform solvate, (I).



(I)

The Sn atom in (I) is pentacoordinated by three phenyl groups, a Cl^- ion and one carboxylate O atom (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 atom is displaced from the equatorial plane in the direction of the Cl atom by 0.094 (4) Å.

As in the 1:2 adduct (Khoo & Hazell, 2005), the piperidinepropionic acid molecule is zwitterionic, with a protonated N atom and a deprotonated carboxylate group. One of the carboxylate O atoms, O1, acts as a hydrogen-bond acceptor for the piperidine NH group. The carboxylate group in (I) is asymmetric, with the uncoordinated CO group (which accepts the hydrogen bond) showing more double-bond character. In the 1:2 adduct (Khoo & Hazell, 2005), the

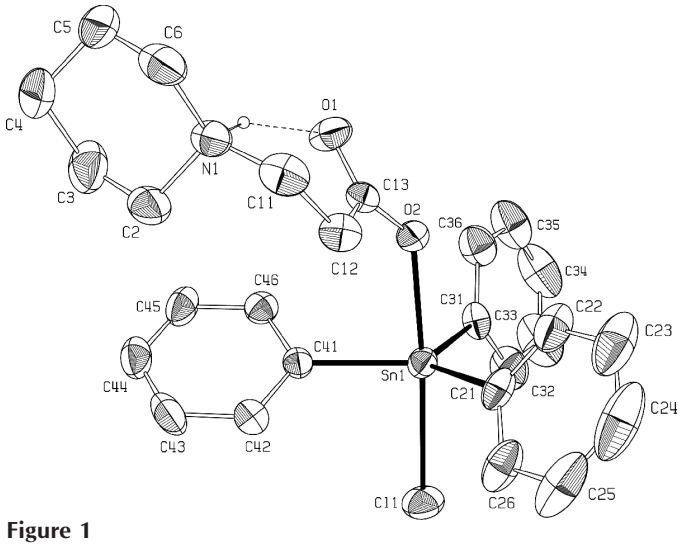


Figure 1
A view of (I), showing 40% displacement ellipsoids. H atoms have been omitted, except for H1*N* attached to N1. The dashed line indicates the intramolecular hydrogen bond. The disordered chloroform solvent molecule is not shown.

hydrogen bond is to the longer of the two carboxylate CO bonds and the H···O separation [2.05 (3) Å] is significantly longer than that in (I) (Table 2).

Experimental

A solution of 3-(piperidin-1-yl)propionic acid (0.80 g, 5.0 mmol) in methanol (20 ml) 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. The solid product was recrystallized from methanol (m.p. 356–359 K). Analysis found: C 57.39, H 5.33, N 2.58%; $C_{27}H_{34}ClNO_2Sn$ requires: C 58.04, H 6.13, N 2.51%. The presence of methanol was confirmed by NMR. IR (cm^{-1}): ν_{NH^+} 2560, 2300; ν_{OH} 3350 (br); ν_{CO} 1600 (*s*); ν_{SnPh} 700, 750. ^{13}C NMR (100.58 MHz, $CDCl_3$, δ, p.p.m.): 175.46 (C_{CO}), 30.21 (C_α), 53.18 (C_β), 54.00 (C2, C6), 23.91 (C3, C5), 22.75 (C4), 51.13 (CH_3OH), 128.76–136.91 (Ph_3Sn); ^1H NMR (400.00 MHz, $CDCl_3$, δ, p.p.m.): 2.92 (H_α, *t*, 2H), 2.53 (H_β, *t*, 2H), 1.65 (H4, *br*, 2H), 1.72 (H3, H5, *m*, 4H), 2.76 (H2, H6, *br*, 4H), 3.6–3.5 (OH *br*, 2H), 7.44–7.77 (Ph_3Sn, 15H), 3.48(CH_3O–, 3H). Recrystallization of the methanol-solvated 1:1 adduct from chloroform in a freezer yielded (I) (m.p. 358–361 K). IR (cm^{-1}): ν_{NH^+} 2600, 2320; ν_{OH} 3350 (br); ν_{CO} 1600; ν_{SnPh} 700, 750. ^{13}C NMR (100.58 MHz, $CDCl_3$, δ, p.p.m.): 175.03 (C_{CO}), 30.15 (C_α), 53.31 (C_β), 54.02 (C2, C6), 24.38 (C3, C5), 22.13 (C4), 129.04–138.88 (Ph_3Sn); ^1H NMR (400.00 MHz, $CDCl_3$, δ, p.p.m.): 2.92 (H_α, *t*, 2H), 2.54 (H_β, *t*, 2H), 1.55 (H4, *br*, 2H), 1.72 (H3, H5, *m*, 4H), 2.70 (H2, H6, *br*, 4H), 7.41–7.76 (Ph_3Sn, 15H).

Crystal data

$[Sn(C_6H_5)_3(C_8H_{15}O_2)Cl]\cdot CHCl_3$
M_r = 662.02
Monoclinic, $P2_1/c$
a = 12.7022 (8) Å
b = 11.705 (1) Å
c = 20.493 (1) Å
β = 105.049 (3)°
V = 2942.5 (4) Å^3
Z = 4
D_x = 1.494 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 44 reflections
θ = 3.0–15.1°
μ = 1.26 mm^{-1}
T = 297 (2) K
Block, colourless
0.4 × 0.4 × 0.3 mm

Data collection

Siemens P4 diffractometer
$2\theta/\omega$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.630, T_{max} = 0.686
5861 measured reflections
4597 independent reflections
3388 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 24.0°
$h = -1 \rightarrow 14$
$k = -1 \rightarrow 13$
$l = -23 \rightarrow 23$
3 standard reflections
every 97 reflections
intensity decay: 8.0%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.126
S = 0.96
4597 reflections
332 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.06P)^2 + 6P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.67 e Å^{-3}
$\Delta\rho_{min}$ = −0.80 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Sn1—C21	2.135 (6)	Sn1—Cl1	2.5517 (16)
Sn1—C41	2.138 (5)	O1—C13	1.236 (7)
Sn1—C31	2.139 (6)	O2—C13	1.264 (7)
Sn1—O2	2.248 (4)		
C21—Sn1—C41	125.6 (2)	C31—Sn1—O2	87.5 (2)
C21—Sn1—C31	116.7 (2)	C21—Sn1—Cl1	91.93 (19)
C41—Sn1—C31	117.2 (2)	C41—Sn1—Cl1	90.22 (15)
C21—Sn1—O2	89.0 (2)	C31—Sn1—Cl1	95.73 (18)
C41—Sn1—O2	85.83 (17)	O2—Sn1—Cl1	175.73 (11)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O1	0.94 (5)	1.83 (6)	2.663 (8)	145 (5)

The NH H atom was located in a difference map and refined with the restraint N—H = 0.950 (1) Å and the constraint U_{iso}(H) = 1.2U_{eq}(N). The CH H atoms were placed in idealized positions and refined as riding on their carrier atoms, with C—H distances in the range 0.93–0.97 Å. The constraint U_{iso}(H) = 1.2U_{eq}(carrier) was applied in all cases. The crystal structure contains a disordered chloroform solvent molecule, which was modelled isotropically in terms of three overlapping positions with occupancies 0.60:0.20:0.20. The C—Cl bond lengths were restrained to be equal within 0.005 Å [final value 1.722 (5) Å]. The H atom of the disordered $CHCl_3$ solvent molecule was not included in the calculations.

Data collection: *XSCANS* (Siemens, 1995); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Siemens, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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