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Acta Cryst. (1997). C**53**, 545–546

Aqua(*p*-chlorophenyl)diphenyl(*N*-phthaloylglycinato)tin(IV)

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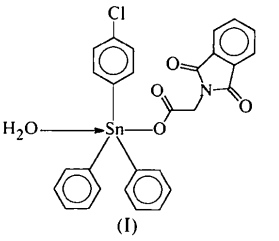
(*Received 24 June 1996; accepted 28 November 1996*)

Abstract

The coordinated water molecule in the title compound, aqua(4-chlorophenyl)(1,3-dioxoisoindoline-2-acetato-*O*)diphenyltin(IV), $[Sn(C_6H_5)_2(C_6H_4Cl)(C_{10}H_6NO_4)(H_2O)]$, is hydrogen bonded to the carboxyl [O···O 2.695 (4) Å] and amido [O···O 2.900 (5) Å] O atoms of adjacent molecules, resulting in a layer structure. The Sn atom shows *trans*-C_3SnO_2 trigonal bipyramidal coordination.

Comment

The condensation of triphenyltin hydroxide and *N*-phthaloylglycine yields triphenyltin *N*-phthaloylglycinate, which crystallizes in a cyclohexameric configuration (Ng, Kumar Das, Pelizzi & Vitali, 1990), an arrangement not known among triorganotin carboxylates (Tiekink, 1991, 1994). This protected amino acid when condensed with (*p*-chlorophenyl)diphenyltin hydroxide yields the corresponding aqua carboxylate, (I); the coordinated water molecule links adjacent molecules (through hydrogen bonding *via* the carboxyl and amido O atoms) into sheets parallel to the *ac* plane.



(I)

The tin–water distance [2.413 (3) Å] is similar to those found in helical aqua(8-quinolyloxylacetato)-triphenyltin [2.388 (7) and 2.391 (6) Å; Kumar Das, Chen, Ng & Mak, 1977], but exceeds that found in dinuclear aquabis[(3-oxapentamethylenethiocarbamoyl-*S*-acetato)triphenyltin] [2.298 (4) Å; Ng, 1995]. Bond dimensions involving the anionic *N*-phthaloylglycinato ligand are similar to those found in the parent carboxylic acid itself (Feeder & Jones, 1994).

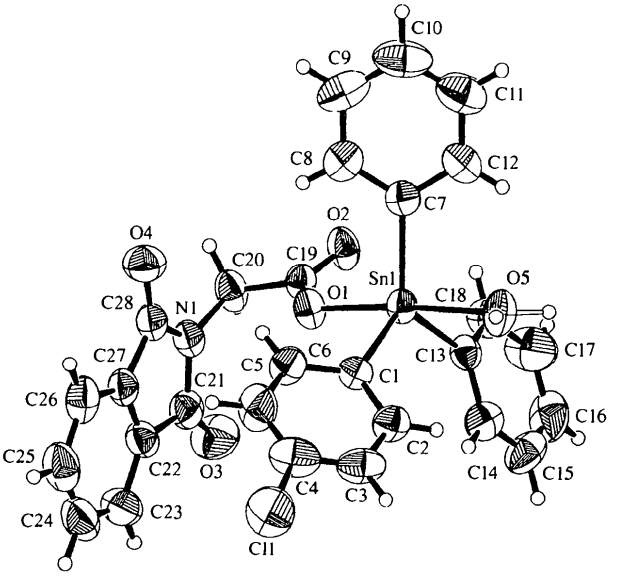


Fig. 1. *ZORTEP* (Zsolnai & Pritzkow, 1996) plot of aqua(*p*-chlorophenyl)diphenyl(*N*-phthaloylglycinato)tin(IV). Displacement ellipsoids are shown at the 50% probability level and H atoms are drawn as small circles of arbitrary radii.

Experimental

The title complex was synthesized by condensing diphenyl(*p*-chlorophenyl)tin hydroxide and *N*-phthaloylglycine. Equimolar amounts of the reactants were dissolved in a small

volume of hot ethanol and slow cooling of the filtered solution yielded large crystals of (I).

Crystal data

$[Sn(C_6H_5)_2(C_6H_4Cl)(C_{10}H_6NO_4)(H_2O)]$
M_r = 606.61
Monoclinic
$P2_1/n$
a = 10.085 (2) Å
b = 19.660 (2) Å
c = 13.096 (2) Å
β = 96.683 (8)°
V = 2578.9 (7) Å^3
Z = 4
D_x = 1.562 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 13–15°
μ = 1.133 mm^{-1}
T = 298 K
Block
0.28 × 0.26 × 0.22 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: ψ scans (North, Phillips & Mathews, 1968)
T_{min} = 0.741, T_{max} = 0.779
4793 measured reflections
4523 independent reflections

3539 reflections with $I > 2\sigma(I)$
R_{int} = 0.0126
θ_{max} = 24.98°
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 23$
$l = -15 \rightarrow 15$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R(F)$ = 0.0342
$wR(F^2)$ = 0.0878
S = 1.031
4523 reflections
351 parameters
H atoms riding with $U(H) = 1.5U_{eq}(C)$; water H atoms were located and refined

$w = 1/[\sigma^2(F_o^2) + (0.0453P)^2 + 1.0185P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.656 e Å^{-3}
$\Delta\rho_{min}$ = −0.451 e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

Sn1—C1	2.134 (4)	Sn1—O5	2.413 (3)
Sn1—C7	2.126 (4)	O5—O2^i	2.695 (4)
Sn1—C13	2.123 (4)	O5—O4	2.900 (5)
Sn1—O1	2.142 (3)		
C1—Sn1—C7	118.2 (1)	C7—Sn1—O1	96.8 (1)
C1—Sn1—C13	116.9 (2)	C7—Sn1—O5	86.7 (1)
C1—Sn1—O1	87.4 (1)	C13—Sn1—O1	99.2 (1)
C1—Sn1—O5	85.4 (1)	C13—Sn1—O5	84.1 (1)
C7—Sn1—C13	123.0 (2)	O1—Sn1—O5	172.8 (1)

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

The Cl atom was disordered over the three phenyl rings. Refinement of its occupancy gave factors of 0.550, 0.325 and 0.125 for the C1–C6, C7–12 and C13–C18 rings. The disorder resulted in somewhat less satisfactory angles at the *para*-C atoms of the C7–C12 and C13–C18 rings.

Data collection: *CAD-4 VAX/PC* (Enraf–Nonius, 1988). Cell refinement: *CAD-4 VAX/PC*. Data reduction: *NRCVAX* (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ZORTEP* (Zsolnai & Pritzkow, 1996). Software used to prepare material for publication: *SHELXL93*.

The authors thank the University of Malaya (F102/66 and F667/96) for supporting this work.

Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: KH1124). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1997). C**53**, 546–548

Tricyclohexyl(*N*-phthaloylglycinato)tin(IV)

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(*Received 24 June 1996; accepted 28 November 1996*)

Abstract

An amido O atom in the title compound, tricyclohexyl(1,3-dioxoisoindoline-2-acetato-*O*)tin(IV), $[Sn(C_6H_{11})_3(C_{10}H_6NO_4)]$, bridges adjacent molecules to form a