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

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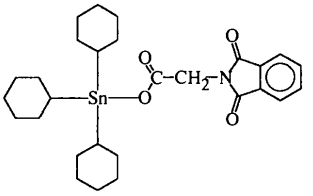
Abstract

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

helical chain parallel to the *b* axis. The Sn atom has a tetrahedral geometry which is distorted towards a trigonal bipyramid.

Comment

In triphenyltin *N*-phthaloylglycinate, six molecules are bridged *via* carboxylate ligands to form a cyclic hexamer (Ng, Kumar Das, Pelizzi & Vitali, 1990). The present tricyclohexyltin analog, (I), does not exhibit this type of bridging, but the molecules are instead linked into a helical chain through an amido O atom. The bridging interaction [Sn←O 3.013 (4) Å] is weak, however, and the distance exceeds the carboxylate-bridging distance [2.810 (7) Å] found in tricyclohexyltin butyrate (Wang, Wang, Yao, Xie, Wang & Li, 1989).



(I)

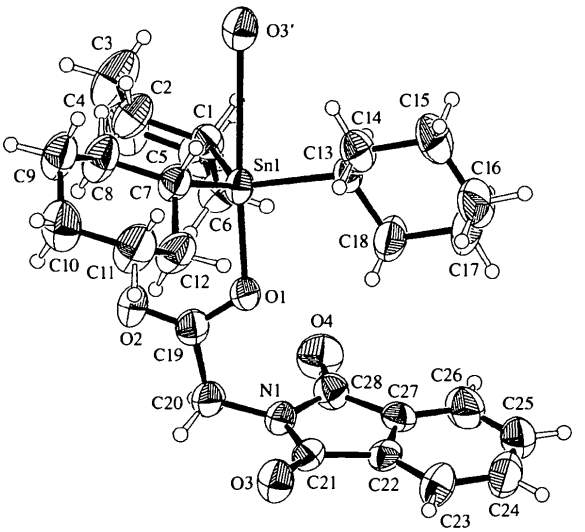


Fig. 1. *ZORTEP* (Zsolnai & Pritzkow, 1996) plot of the title compound at the 50% probability level. H atoms are drawn as spheres of arbitrary radii and the symmetry-related O3 atom is shown.

Experimental

The title compound was prepared by treating dicyclohexylammonium *N*-phthaloylglycinate (Ng, 1995) with tricyclohexyltin chloride in ethanol. The reactants were heated briefly in the solvent and the mixture was then filtered. The resulting solution, once cool, afforded large crystals of (I).

Crystal data

$[Sn(C_6H_{11})_3(C_{10}H_6NO_4)]$
M_r = 572.29
Monoclinic
$P2_1/n$
a = 11.003 (1) Å
b = 13.3701 (9) Å
c = 18.408 (2) Å
β = 96.734 (5)°
V = 2689.4 (5) Å^3
Z = 4
D_x = 1.413 Mg m^{-3}
D_m not measured
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 9.0–11.0°
μ = 0.983 mm^{-1}
T = 298 (2) K
Block
0.28 × 0.25 × 0.22 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: refined from ΔF (*DIFABS*; Walker & Stuart, 1983)
T_{min} = 0.673, T_{max} = 0.806
4714 measured reflections
4714 independent reflections
3336 reflections with $I > 2\sigma(I)$
θ_{max} = 24.97°
$h = -13 \rightarrow 12$
$k = 0 \rightarrow 15$
$l = 0 \rightarrow 21$
3 standard reflections
frequency: 60 min
intensity decay: 2.4%

Refinement

Refinement on F^2
$R(F)$ = 0.0446
$wR(F^2)$ = 0.0942
S = 0.980
4714 reflections
307 parameters
H atoms riding with U(H) = $1.5U_{eq}$(C)
$w = 1/[\sigma^2(F_o^2) + (0.039P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.303 e Å^{-3}
$\Delta\rho_{min}$ = −0.448 e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

Sn1—C1	2.154 (5)	Sn1—O1	2.095 (3)
Sn1—C7	2.145 (5)	Sn1—O3^i	3.013 (4)
Sn1—C13	2.166 (4)		
C1—Sn1—C7	122.4 (2)	C7—Sn1—O1	104.4 (2)
C1—Sn1—C13	115.8 (2)	C7—Sn1—O3^i	81.0 (2)
C1—Sn1—O1	101.7 (2)	C13—Sn1—O1	95.1 (2)
C1—Sn1—O3^i	75.4 (2)	C13—Sn1—O3^i	82.2 (2)
C7—Sn1—C13	111.8 (2)	O1—Sn1—O3^i	174.5 (1)

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

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 (F102/96 and F677/96) for supporting this work.

Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: KH1125). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Tricyclohexyl[(*N*,*N*-diethylthiocarbamoylthio)acetato-*O*]tin(IV)

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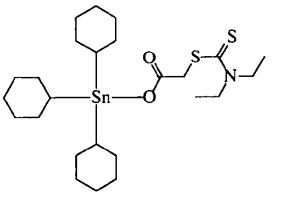
(*Received 24 June 1996; accepted 2 December 1996*)

Abstract

The Sn atom in the title compound, $[Sn(C_6H_{11})_3(C_7H_{12}NO_2S_2)]$, is four-coordinate in a distorted tetrahedral arrangement.

Comment

The tricyclohexyltin derivatives of *N*,*N*-dithiocarbamoylacetic acids have been assigned a tetrahedral geometry on the basis of spectroscopic measurements (Ng & Kumar Das, 1991) and this has been confirmed by the present study on the *N*,*N*-diethyldithiocarbamoylacetate derivative, (I). Intermolecular distances exceed 3.5 Å and bond dimensions involving the Sn atom are similar to those found in four-coordinate triorganotin carboxylates (Tiekink, 1991, 1994).



(I)

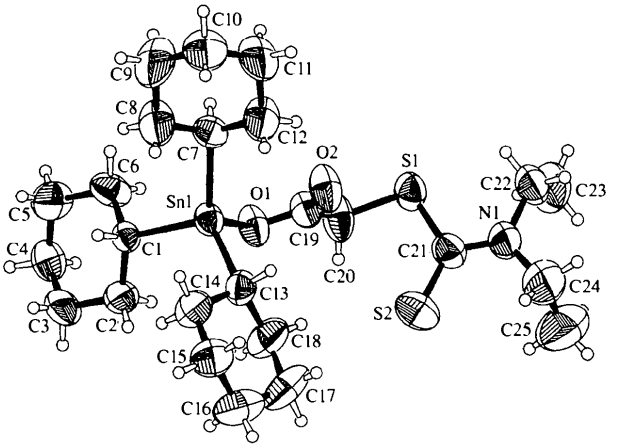


Fig. 1. *ZORTEP* (Zsolnai & Pritzkow, 1996) plot of compound (I). Displacement ellipsoids are shown at the 50% probability level and H atoms are drawn as small circles of arbitrary radii.

Experimental

The title compound was synthesized from tricyclohexyltin hydroxide and *N*,*N*-diethyldithiocarbamoylacetic acid in ethanol (Ng & Kumar Das, 1991).

Crystal data

$[Sn(C_6H_{11})_3(C_7H_{12}NO_2S_2)]$
M_r = 574.43
Triclinic
$P\bar{1}$
a = 9.3375 (8) Å
b = 10.0667 (9) Å
c = 17.456 (2) Å
α = 79.866 (9)°
β = 81.874 (9)°
γ = 62.776 (7)°
V = 1432.8 (3) Å^3
Z = 2
D_x = 1.331 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 14.5–15.0°
μ = 1.057 mm^{-1}
T = 300 K
Block
0.51 × 0.43 × 0.36 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer

4451 reflections with $I > 2\sigma(I)$