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Tetra(*p*-chlorophenyl)tin

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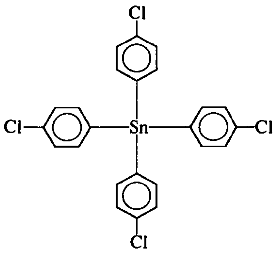
Abstract

The crystal structure of tetra(*p*-chlorophenyl)tin, [Sn(C_6H_4Cl)$_4$], consists of discrete molecules having 1 molecular symmetry.

Comment

Except for tetra(*p*-methylsulfonlylphenyl)tin, which has 2 molecular symmetry (Wharf, Simard & Lamparski, 1990), other R_4Sn tetraaryltin compounds, *i.e.* tetraphenyltin (Akhmed & Aleksandrov, 1970; Belsky, Simonenko, Reikhsfeld & Saratov, 1983; Chieh & Trotter, 1970; Engelhardt, Leung, Ratson & White, 1982), tetra(*o*-tolyl)tin (Belsky *et al.*, 1983), tetra(*m*-tolyl)tin (Karipides & Oertel, 1977), tetra(*p*-tolyl)tin (Karipides & Wolfe, 1975) and tetra(*p*-anisyl)tin (Ismailzade, 1958), as well as tetrakis(pentafluorophenyl)tin (Karipides, Forman, Thomas & Reed, 1974) and tetra(2-thienyl)tin (Karipides, Reed, Haller & Hayes, 1977), have $\bar{4}$ molecular symmetry in the crystalline state. Tetra(*p*-chlorophenyl)tin, (I), is the first example of a tetraaryltin compound having only 1 molecular symmetry.



(I)

The tetrahedral molecules of (I) are separated by normal van der Waals contacts (Cl$\cdots$Cl 3.36–3.96 Å). The C—Sn—C angles [107.9 (2)–113.9 (2)°] do not differ significantly from the idealized tetrahedral angle of 109.5°. The Sn—C bond distances [2.125 (4)–2.149 (4) Å] are similar to that [2.136 (4) Å] found in tetra(*p*-anisyl)tin (Wharf & Simard, 1987), which possesses an electron-donating substituent, and that found in tetraphenyltin [2.143 (5) Å; Engelhardt *et al.*, 1982]. These distances are somewhat longer than that found in tetramethyltin [2.102 (8) Å; Krebs, Henkel & Dartmann, 1989].

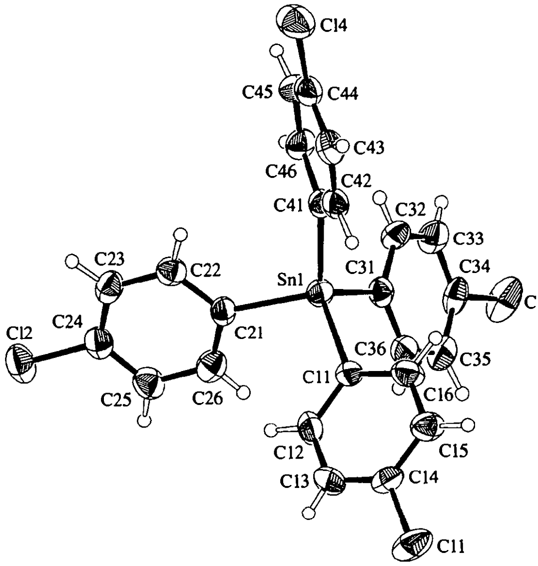


Fig. 1. A *ZORTEP* (Zsolnai & Pritzkow, 1996) plot of [Sn(*p*-ClC_6H_4)$_4$] at the 50% probability level. H atoms are drawn as small circles of arbitrary radii.

Experimental

Tetra(*p*-chlorophenyl)tin was synthesized from stannic chloride and (*p*-chlorophenyl)magnesium chloride by a Grignard synthesis and was recrystallized from ethanol.

Crystal data

[Sn(C_6H_4Cl)$_4$]
M_r = 564.86
Triclinic
$P\bar{1}$
a = 10.035 (4) Å
b = 11.090 (8) Å
c = 12.325 (4) Å
α = 65.28 (9)°
β = 70.50 (3)°
γ = 70.37 (5)°
V = 1143 (1) Å^3
Z = 2
D_x = 1.641 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 19 reflections
θ = 5.0–12.5°
μ = 1.593 mm^{-1}
T = 163 (2) K
Parallelepiped
0.60 × 0.30 × 0.25 mm
Colourless

Data collection

Siemens *P*4 diffractometer
ω-scans
Absorption correction: ψ scans (North, Phillips & Mathews, 1968)
$T_{\min}$ = 0.643, $T_{\max}$ = 0.671
3534 measured reflections
3459 independent reflections
2998 reflections with $I > 2\sigma(I)$

$R_{\rm int}$ = 0.1116
$\theta_{\max}$ = 24.97°
$h = 0 \rightarrow 10$
$k = -11 \rightarrow 12$
$l = -13 \rightarrow 14$
3 standard reflections every 97 reflections intensity decay: 4.6%

Refinement

Refinement on F^2
$R(F) = 0.0288$
$wR(F^2) = 0.0787$
$S = 1.068$
3458 reflections
326 parameters
H atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0465P)^2 + 0.0113P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.787$ e Å^{-3}
$\Delta\rho_{\min} = -0.628$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

Sn1—C11	2.144 (4)	Sn1—C31	2.141 (4)
Sn1—C21	2.149 (4)	Sn1—C41	2.125 (4)
C11—Sn1—C21	107.9 (2)	C21—Sn1—C31	108.1 (1)
C11—Sn1—C31	107.4 (2)	C21—Sn1—C41	108.3 (2)
C11—Sn1—C41	111.2 (1)	C31—Sn1—C41	113.9 (2)

Data collection: *XSCANS* (Siemens, 1991). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ZORTEP* (Zsolnai & Pritzkow, 1996). Software used to prepare material for publication: *SHELXL*93.

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Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: KH1137). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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[Bis(*N*,*N*-dimethylthiocarbamoylthio)-acetato-*O*]triphenyl(quinoline *N*-oxide-*O*)tin

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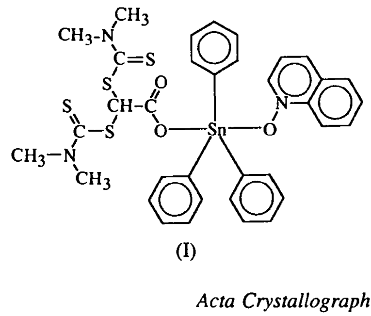
Abstract

The Sn atom in the title compound, $[Sn(C_6H_5)_3(C_8H_{13}N_2O_2S_4)(C_9H_7NO)]$, shows *trans*-$C_3SnO_2$ trigonal bipyramidal coordination [ΣC—Sn—C 357.5 (6), O—Sn←O 171.4 (2)° and Sn←O 2.446 (4) Å].

Comment

Triphenyltin carboxylates generally do not yield complexes with oxygen-donor ligands unless the anion possesses electron-withdrawing groups that can raise the Lewis acidity of tin. Bis(*N*,*N*-dimethyldithiocarbamyl)-acetate displays enhanced acceptor properties as it is able to form 1:1 complexes with hexamethylphosphoramide and triphenylphosphine oxide (Ng, 1995). The Sn atom in the title complex, (I), is also five-coordinate in a trigonal bipyramidal geometry, but the Sn←O bond [2.446 (4) Å] is much longer than those in the phosphorus donors, as N-atom donors are weaker Lewis bases than P-atom donors. The bond distance is also longer than that [2.319 (6) Å] found in the quinoline *N*-oxide complex of *N*-triphenylstannyl-1,2-benzisothiazol-3(2*H*)-one 1,1-dioxide (Ng, 1994), but is similar to that [2.459 (5) Å] found in the quinoline *N*-oxide complex of tri(*p*-tolyl)tin bromide (Kumar Das, Yap, Ng, Chen & Mak, 1986).



(I)