

Tin–halide compounds. IV¹.
tert-Butyltrichlorotin(IV)Hans Reuter,^{a*} Henning Eickmeier,^a Heinrich Puff,^b
Norbert Beckermann^b and Dieter Hänssgen^b^aInstitut für Chemie, Universität Osnabrück, Barbarastraße 7, D-49069 Osnabrück, Germany, and ^bInstitut für Anorganische Chemie, Universität Bonn, Gerhard-Domagk-Straße 1, D-53121 Bonn, Germany
Correspondence e-mail: hreuter@rz.uni-osnabrueck.de

Received 12 December 2001

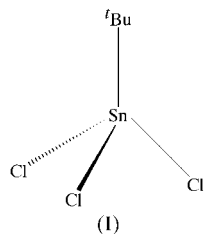
Accepted 28 January 2002

Online 12 March 2002

The molecules of the title compound, [Sn(C₄H₉)Cl₃], adopt an eclipsed conformation with respect to the C and Cl atoms, and possess crystallographic C_s symmetry. The molecular structure and geometric parameters are comparable with those of related organotin trihalides.

Comment

Among the organotin halides R_nSnX_{4-n}, with simple hydrocarbon groups R = C_mH_{2m+1}, the organotin trihalides RSnX₃ (n = 1) are the least structurally characterized, and only for the methyl compounds (R = CH₃) have the structures of the trichloride (Frank *et al.*, 1994), tribromide (Zhang *et al.*, 1991) and triiodide (Tse *et al.*, 1986) been published. In these compounds, the Sn atom exhibits tetrahedral coordination, and interactions between the molecules are restricted to van der Waals attraction, which accounts for their physical properties. The title compound, ^tBuSnCl₃, (I), originally prepared by Beckermann (Hänssgen *et al.*, 1985), now represents the second R group in the R = C_mH_{2m+1} series to be structurally characterized as the tin trichloride.



In the unit cell of (I), there is half a molecule in the asymmetric unit: the Sn atom, one Cl atom, the α-C atom and

¹ Part III: Reuter & Pawlak (2001).

one β-C atom are on a mirror plane, whereas one further C and a Cl atom are in general positions. The molecule shows local C_{3v} symmetry, but only crystallographic C_s symmetry is realised by the space-group symmetry. In the solid state, (I) adopts an eclipsed conformation with respect to the methyl-C and Cl atoms (Fig. 1).

The Sn atom within the isolated molecule of (I) is tetrahedrally coordinated by three Cl atoms and one *tert*-butyl group. This geometry is distorted by a smaller bond angle between the Cl atoms [mean 102.0 (1)^o] and a greater bond angle between the organic ligand and the Cl atoms [mean 116.2 (2)^o]. Similar values were observed in the methyl compound, MeSnCl₃ (102.3 and 115.8^o, respectively; Frank *et al.*, 1994).

The Sn–C bond (Table 1) is considerably longer and the mean value of the Sn–Cl distance [2.320 (5) Å] is marginally longer than the corresponding distances found in the methyl compound [2.074 (8) and 2.307 (16) Å, respectively], probably because of the electronic and steric effects of the *tert*-butyl ligand. For SnCl₄ itself, a mean Sn–Cl bond length of 2.279 (3) Å is observed (Reuter & Pawlak, 2000).

The same molecular structure as (I) is observed in RSnX₃ compounds with more complex organic groups, such as Ph₅Cp or Me₅Cp, which have been synthesized as the chloride (Janiak *et al.*, 1997) and iodide (Bartlett *et al.*, 1992), respectively. Even with heteroatom-substituted organic groups, this situation does not change, as long as the heteroatom does not belong to a polar group. However, if the heteroatom is part of a highly polar group, intramolecular interactions between this group and the Sn atom are observed in most cases, leading to the formation of five- or six-membered rings and a trigonal-bipyramidal coordination around the Sn atom. In some cases, intermolecular interactions occur between the polar group and the Sn atoms, leading to polymeric structures, as in the case of HO(CH₂)₅SnCl₃ (Biesemans *et al.*, 1998).

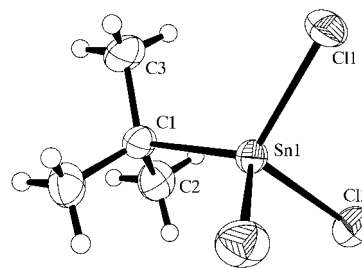


Figure 1

A view of the molecular structure of (I) showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

Experimental

Single crystals of (I) were grown by cooling the reaction liquor (dichloromethane) to 193 K. The compound decomposes at room temperature (Hänssgen *et al.*, 1985).

Crystal data

[Sn(C₄H₉)Cl₃]
M_r = 282.15
 Monoclinic, *P*2₁/*m*
a = 7.632 (4) Å
b = 10.341 (5) Å
c = 6.289 (5) Å
 β = 112.77 (4)°
V = 457.7 (5) Å³
Z = 2

D_x = 2.047 Mg m⁻³
 Mo *K*α radiation
 Cell parameters from 25 reflections
 θ = 9.1–20.3°
 μ = 3.58 mm⁻¹
T = 203 (2) K
 Irregular prism, colourless
 0.20 × 0.14 × 0.14 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
 $\omega/2\theta$ scans
 1715 measured reflections
 836 independent reflections
 835 reflections with *I* > 2σ(*I*)
*R*_{int} = 0.026
 θ_{\max} = 25°

h = -9 → 8
k = 0 → 12
l = 0 → 7
 3 standard reflections every 100 reflections
 frequency: 60 min
 intensity decay: 13.5%

Refinement

Refinement on *F*²
R[*F*² > 2σ(*F*²)] = 0.036
wR(*F*²) = 0.101
S = 1.15
 836 reflections
 45 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0633P)^2 + 1.6121P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 1.82 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -1.06 \text{ e } \text{Å}^{-3}$
 Extinction correction: *SHELXL97* (Sheldrick, 1997)
 Extinction coefficient: 0.011 (3)

Table 1

Selected geometric parameters (Å, °).

Sn1—C1	2.170 (8)	C1—C2	1.519 (8)
Sn1—Cl1	2.313 (3)	C1—C3	1.523 (11)
Sn1—Cl2	2.324 (2)		
C1—Sn1—C11	117.8 (2)	C2—C1—C2 ⁱ	110.4 (7)
C1—Sn1—Cl2	115.40 (11)	C2—C1—C3	112.0 (4)
Cl1—Sn1—Cl2	102.01 (6)	C2—C1—Sn1	107.3 (4)
Cl2 ⁱ —Sn1—Cl2	101.97 (10)	C3—C1—Sn1	107.6 (5)
Cl1—Sn1—C1—C3	0.00 (4)		

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

All H atoms were fixed in geometrically idealized positions and were allowed to ride on their parent atoms (C—H = 0.96–0.97 Å), while a common isotropic displacement parameter was refined. The maximum peak of residual electron density is located 0.92 Å from Sn1.

Data collection: *CAD-4 Operations Manual* (Enraf–Nonius, 1977); cell refinement: *CAD-4 Operations Manual*; data reduction: *CADSHEL* (Kopf & Ruebcke, 1987); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1997); software used to prepare material for publication: *SHELXL97*.

This work was supported by the Deutsche Forschungsgemeinschaft and the Fonds der Chemischen Industrie.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: LN1131). Services for accessing these data are described at the back of the journal.

References

- Bartlett, R. A., Cowley, A., Jutzi, P., Olmstead, M. M. & Stammer, H.-G. (1992). *Organometallics*, **11**, 2837–2840.
- Biesemans, M., Willem, R., Damoun, S., Geerlings, P., Tiekink, E. R. T., Jaumier, P., Lahcini, M. & Jousseume, B. (1998). *Organometallics*, **17**, 90–97.
- Enraf–Nonius (1977). *CAD-4 Operations Manual*. Enraf–Nonius, Delft, The Netherlands.
- Frank, W., Reiss, G. J. & Kuhn, D. (1994). *Acta Cryst.* **C50**, 1904–1906.
- Hänssgen, D., Puff, H. & Beckermann, N. (1985). *J. Organomet. Chem.* **293**, 191–195.
- Janiak, C., Weimann, R. & Görlitz, F. (1997). *Organometallics*, **16**, 4933–4936.
- Kopf, J. & Ruebcke, H.-C. (1987). *CADSHEL*. Version 3.10. University of Hamburg, Germany.
- Reuter, H. & Pawlak, R. (2000). *Z. Anorg. Allg. Chem.* **626**, 925–929.
- Reuter, H. & Pawlak, R. (2001). *Z. Kristallogr.* **216**, 56–59.
- Sheldrick, G. M. (1997). *SHELXS97*, *SHELXL97* and *SHELXTL* (Version 5.10). Bruker AXS Inc., Madison, Wisconsin, USA.
- Tse, J. S., Collins, M. J., Lee, F. L. & Gabe, E. J. (1986). *J. Organomet. Chem.* **310**, 169–178.
- Zhang, D., Shi-Qi, D. & Weiss, A. (1991). *Z. Naturforsch. Teil A*, **46**, 337–343.