1993). Anisotropic displacement parameters were refined for all non-H atoms. All H atoms were located from a difference Fourier map and refined with free isotropic displacement parameters. Geometrical analysis was performed using *PARST* (Nardelli, 1983) and *SHELXL*93. There is a pseudo-twofold axis passing through the Zn atoms approximately parallel to the *c* axis. It brings one ligand (sald) into the other, however, with significant atomic displacements varying from 0.25 to 0.31 Å. In addition, the water molecules are unaffected by this local pseudosymmetry, which excludes any canonical transformation of the monoclinic unit cell into a higher crystal system.

Data collection: Stadi-4 software. Cell refinement: Stadi-4 software. Data reduction: *X-RED*. Program(s) used to solve structure: *SHELXS*86. Program(s) used to refine structure: *SHELXL*93. Molecular graphics: *ORTEP* (Johnson, 1965). Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1207). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Tribenzyltin Chloride

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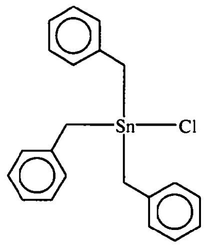
Abstract

The crystal structure of tribenzyltin chloride consists of [SnCl(C_7H_7)$_3$] molecules linked axially by a tin–chlorine bridge [Sn—Cl 2.387 (2), Sn···Cl 3.531 (2) Å and Cl—Sn···Cl 180°] into a linear chain along the *c* axis.

Comment

Among the triorganotin halides, the fluorides universally exist as bridged polymers, whereas the heavier bromides and iodides generally crystallize as monomeric solids (Smith, 1981; Harrison, 1994). The coordination number of tin in the chlorides lies between those in the fluorides and bromides.

The crystal structure of tribenzyltin chloride, (I), consists of ($C_6H_5CH_2$)$_3$SnCl molecules (Fig. 1) linked axially by a tin–chlorine bridge [Sn—Cl 2.387 (2), Sn···Cl 3.531 (2) Å and Cl—Sn···Cl 180°] into a linear chain along the *c* axis.



(I)

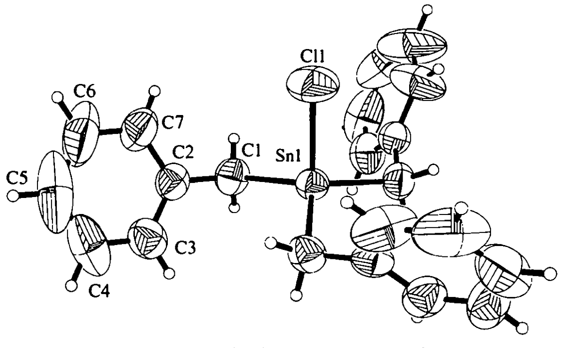


Fig. 1. *ORTEP* [(Johnson, 1965) in *SHELXTL/PC* (Sheldrick, 1990*b*)] plot of tribenzyltin chloride with ellipsoids at the 50% probability level. H atoms are drawn as spheres of arbitrary radii.

Trimethyltin chloride (Lefferts, Molloy, Hossain, van der Helm & Zuckerman, 1982) shows an intermolecular Sn···Cl distance of 3.269 (2) Å [Sn—Cl 2.430 (2) Å and Cl—Sn···Cl 176.9 (1)°]; the structure has been interpreted in terms of a chlorine-bridged chain. Tricyclohexyltin chloride (Calogero, Ganis, Peruzzo & Tagliavini, 1979) has a similar chain structure [Sn—Cl 2.407 (5), Sn···Cl 3.306 (5) Å and Cl—Sn···Cl 180°], the chain propagating by translations along the *c* axis [c = 5.713 (3) Å]. On the other hand, both the monoclinic (Bokii, Zakharova & Struchkov, 1971; Tse, Lee & Gabe, 1986) and rhombohedral (Ng, 1996) modifications of triphenyltin chloride, a much weaker Lewis acid compared with the trialkyltin chlorides, consist of discrete tetrahedral molecules.

Experimental

Tribenzyltin chloride was prepared from metallic tin and benzyl chloride in water (Sisido, Takeda & Kinugawa, 1961), and was recrystallized from ethanol.

Crystal data

$[\mathrm{SnCl(C_7H_7)_3}]$
M_r = 427.52
Trigonal
$R3$
a = 16.942 (1) Å
c = 5.9187 (4) Å
V = 1471.3 (2) Å^3
Z = 3
D_x = 1.448 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 12.25–12.75°
μ = 1.436 mm^{-1}
T = 298 (2) K
Block
0.50 × 0.45 × 0.35 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
Absorption correction: ψ scan (North, Phillips & Mathews, 1968)
$T_{\min}$ = 0.56, $T_{\max}$ = 0.60
2305 measured reflections
1490 independent reflections

1490 observed reflections [$I > 2\sigma(I)$]
$R_{\rm int}$ = 0.0291
$\theta_{\max}$ = 27.48°
$h = 0 \rightarrow 22$
$k = -22 \rightarrow 18$
$l = -7 \rightarrow 7$
3 standard reflections
frequency: 30 min
intensity decay: 5.1%

Refinement

Refinement on F^2
$R(F)$ = 0.0229
$wR(F^2)$ = 0.0563
S = 1.065
1490 reflections
70 parameters
H atoms: U(H) = 1.5$U_{\rm eq}$(C)
$w = 1/[\sigma^2(F_o^2) + (0.0363P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max}$ = 0.790 e Å^{-3}
$\Delta\rho_{\min}$ = −0.174 e Å^{-3}

Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration: Flack & Schwarzenbach (1988)
Flack parameter = −0.09 (4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Sn1	0	0	0.0029	0.0493 (1)
Cl1	0	0	−0.4004 (3)	0.0971 (6)
C1	0.0187 (2)	−0.1127 (2)	0.0809 (7)	0.0631 (7)
C2	−0.0642 (3)	−0.2016 (2)	0.0313 (6)	0.0593 (7)
C3	−0.1357 (3)	−0.2405 (3)	0.1814 (9)	0.081 (1)
C4	−0.2132 (4)	−0.3221 (4)	0.134 (2)	0.119 (2)
C5	−0.2197 (7)	−0.3648 (4)	−0.067 (2)	0.156 (5)
C6	−0.1500 (8)	−0.3278 (4)	−0.217 (1)	0.160 (4)
C7	−0.0718 (6)	−0.2468 (3)	−0.1690 (9)	0.111 (2)

Table 2. *Geometric parameters (Å, °)*

Sn1—C1	2.136 (3)	C2—C3	1.376 (6)
Sn1—C1$^{\rm i}$	2.136 (3)	C2—C7	1.382 (6)
Sn1—C1$^{\rm ii}$	2.136 (3)	C3—C4	1.378 (8)
Sn1—Cl1	2.387 (2)	C4—C5	1.37 (2)
Sn1—Cl1$^{\rm iii}$	3.531 (2)	C5—C6	1.36 (2)
C1—C2	1.487 (5)	C6—C7	1.38 (1)
C1—Sn1—C1$^{\rm i}$	115.46 (8)	C2—C1—Sn1	112.1 (2)
C1—Sn1—C1$^{\rm ii}$	115.46 (8)	C3—C2—C7	118.2 (4)
C1—Sn1—Cl1	102.5 (1)	C1—C2—C3	121.1 (4)
C1—Sn1—Cl1$^{\rm iii}$	77.5 (1)	C1—C2—C7	120.7 (4)
C1$^{\rm i}$—Sn1—C1$^{\rm ii}$	115.46 (8)	C2—C3—C4	121.1 (6)
C1$^{\rm i}$—Sn1—Cl1	102.5 (1)	C3—C4—C5	119.6 (7)
C1$^{\rm i}$—Sn1—Cl1$^{\rm iii}$	77.5 (1)	C4—C5—C6	120.2 (6)
C1$^{\rm ii}$—Sn1—Cl1	102.5 (1)	C5—C6—C7	120.6 (7)
C1$^{\rm ii}$—Sn1—Cl1$^{\rm iii}$	77.5 (1)	C2—C7—C6	120.3 (7)
Cl1—Sn1—Cl1$^{\rm iii}$	180		

Symmetry codes: (i) $-y, x - y, z$; (ii) $-x + y, -x, z$; (iii) $x, y, 1 + z$.

H atoms were placed at calculated positions and refined in riding mode.

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: *SHELXS*86 (Sheldrick, 1990*a*). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC* (Sheldrick, 1990*b*). Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: SK1046). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Bis{μ-chloro-[2(3*H*)-benzothiazolethione](triphenylphosphine)copper(I)} Dihydrate

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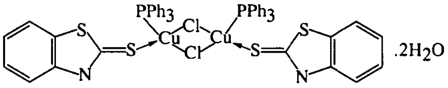
Abstract

The chloro-bridged dimeric structure of the title compound, bis[benzothiazole-2(3*H*)-thione]-1κS;2κS-di-μ-chloro-1:2$\kappa^4 Cl$-bis(triphenylphosphine)-1κP;2κP-dicopper(I) dihydrate, [Cu$_2$Cl$_2$(C$_7$H$_5$NS$_2$)$_2$(Ph$_3$P)$_2$].2H$_2$O, is reported. The molecule is centrosymmetric with a Cu···Cu distance of 3.400 (2) Å and two Cu—μ-Cl distances of 2.396 (2) and 2.516 (2) Å. The heterocyclic ligand benzothiazole-2(3*H*)-thione acts as a monodentate ligand coordinating to copper through the S atom, with a Cu—S distance of 2.304 (3) Å. A hydrogen bond between the μ-Cl and heterocyclic N atom is also found, with a Cl···N distance of 3.134 (6) Å and a Cl···H—N angle of 162.4°.

Comment

The coordination chemistry of heterocyclic thiones, especially with copper, continues to draw attention (Raper, 1985). The known structures of the complexes are almost all monomers or S-bridged dimers (Raper, 1994). Some examples with halogen-bridged dimers are [Cu$_2$(merhod)$_4$Cl$_2$] (Moers, Beurskens, Noordik & Smits, 1986), [Cu$_2$(pu6tH)$_2$Cl$_4$] (Dubler & Gyr, 1988) and [Cu$_2$(mepu6tH)$_2$Cl$_4$] (Sletten & Apeland, 1975). As for benzothiazole-2(3*H*)-thione (bztztH), there are some reports of Cu$^{\rm I}$ complexes, such as [Cu(bztztH){(bztz)$_2$S$_3$}]ClO$_4$.2CHCl$_3$ (Jeannin, Jeannin & Lavigne, 1979) and [{Cu(bztzt)(dppe)}$_2$(μ-dppe)] (dppe = diphenylphosphinoethane) (Ainscough, Baker, Bingham, Brodie & Smith, 1989). The title compound, (I), is the first example of a halogen-bridged binuclear complex of this ligand.



(I)

The Cu atom adopts a tetrahedral coordination and the CuCl$_2$Cu nucleus is almost planar and nearly forms a square. The Cu···Cu distance is 3.400 (2) Å, which is shorter than the distance of 3.501 (1) Å found in [Cu$_2$(pu6tH)$_2$Cl$_4$] (Dubler & Gyr, 1988) and longer than the distance of 2.906 (1) found in [Cu$_2$(merhod)$_4$Cl$_2$] (Moers *et al.*, 1986). The Cl1—Cu1 and Cl1$^{\rm i}$—Cu1 distances are 2.516 (2) and 2.396 (2) Å, respectively [symmetry code: (i) $1-x$, $-y$, $-z$]. The difference here is larger than is the case in [Cu$_2$(pu6tH)$_2$Cl$_4$], in which the two Cu—μ-Cl distances are 2.465 (1) and 2.493 (1) Å.

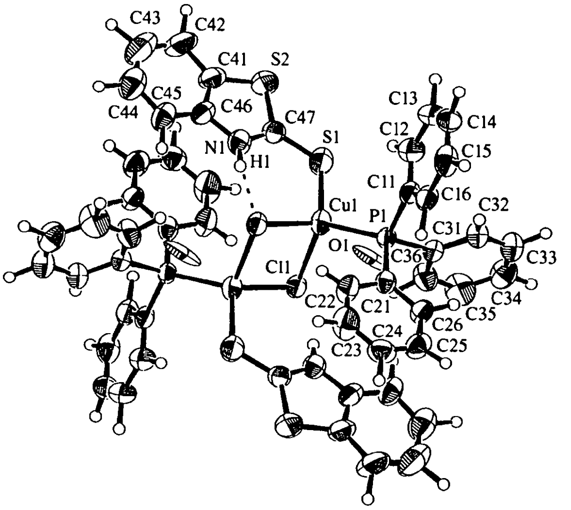


Fig. 1. A view of the title complex (*ORTEP*II; Johnson, 1976) with displacement ellipsoids shown at the 40% probability level. The intramolecular hydrogen bond is shown as a dashed line.