$wR = 0.0570$
$S = 4.119$
2424 reflections
217 parameters
H-atom parameters not refined
$w = 4.178/\sigma^2(F)$
$\Delta\rho_{max} = 0.80$ e Å^{-3}
$\Delta\rho_{min} = -0.72$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

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

	x	y	z	U_{eq}
Zn	0.24633 (14)	0.13936 (3)	0.08967 (5)	0.0302 (2)
S1	0.6422 (3)	0.1490 (1)	0.1216 (1)	0.0339 (6)
C11	0.6999 (11)	0.1829 (3)	0.2325 (4)	0.0319 (13)
N11	0.5533 (10)	0.2179 (3)	0.2578 (4)	0.0482 (13)
N12	0.8970 (10)	0.1730 (3)	0.2902 (4)	0.0411 (13)
S2	0.0371 (3)	0.0960 (1)	−0.0451 (1)	0.0367 (6)
C21	0.2472 (11)	0.0558 (3)	−0.0826 (4)	0.0293 (13)
N21	0.2280 (10)	−0.0006 (2)	−0.0884 (4)	0.0421 (14)
N22	0.4172 (10)	0.0819 (3)	−0.1086 (5)	0.0434 (15)
O31	0.1963 (8)	0.0996 (2)	0.2077 (3)	0.0338 (13)
O32	0.3552 (8)	0.0219 (2)	0.1590 (3)	0.0413 (12)
C31	0.2811 (10)	0.0492 (3)	0.2168 (5)	0.0287 (15)
C32	0.2869 (12)	0.0217 (3)	0.3154 (5)	0.0404 (14)
Cl31	0.0137 (4)	0.0227 (1)	0.3414 (2)	0.0701 (8)
Cl32	0.3908 (8)	−0.0496 (1)	0.3222 (2)	0.0898 (10)
Cl33	0.4756 (4)	0.0636 (1)	0.4017 (2)	0.0749 (9)
O41	0.1347 (8)	0.2189 (2)	0.1109 (3)	0.0363 (12)
O42	0.0260 (13)	0.2525 (3)	−0.0361 (4)	0.0916 (14)
C41	0.0533 (12)	0.2560 (3)	0.0498 (5)	0.0421 (14)
C42	−0.0216 (11)	0.3141 (3)	0.0897 (5)	0.0376 (14)
Cl41	0.0587 (6)	0.3191 (1)	0.2131 (2)	0.0961 (9)
Cl42	−0.3253 (4)	0.3175 (1)	0.0568 (2)	0.0795 (10)
Cl43	0.0859 (5)	0.3722 (1)	0.0372 (2)	0.0782 (9)
O1	0.4433 (11)	0.2087 (2)	−0.0920 (4)	0.0673 (14)

Table 2. *Selected geometric parameters* (Å, °)

Zn—S1	2.316 (2)	Zn—O31	2.012 (5)
Zn—S2	2.291 (2)	Zn—O41	2.000 (5)
O31—Zn—O41	100.0 (2)	S1—Zn—O41	104.0 (2)
S2—Zn—O41	113.2 (2)	S1—Zn—O31	102.3 (1)
S2—Zn—O31	111.1 (2)	S1—Zn—S2	123.3 (1)

Table 3. *Hydrogen-bonding geometry* (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N11—H112···O41	1.01 (1)	1.975 (4)	2.892 (7)	150 (4)
N22—H221···O1	0.90 (1)	2.058 (5)	2.940 (8)	166 (5)
O1—H11···S1	1.08 (1)	2.384 (2)	3.338 (6)	146 (3)
O1—H12···O42	0.97 (1)	2.125 (8)	2.963 (10)	143 (4)
N11—H111···O1^{i}	0.77 (1)	2.197 (6)	2.936 (9)	162 (5)
N12—H121···O42^{ii}	1.03 (1)	2.049 (6)	2.992 (9)	151 (4)
N12—H122···O31^{iii}	0.96 (1)	1.973 (5)	2.904 (8)	162 (4)
N21—H211···O32^{iv}	1.06 (1)	1.970 (5)	2.933 (8)	149 (3)
N22—H222···O32^{iv}	0.96 (1)	2.020 (5)	2.927 (8)	156 (4)

Symmetry codes: (i) $x, \frac{1}{2}-y, \frac{1}{2}+z$; (ii) $1+x, \frac{1}{2}-y, \frac{1}{2}+z$; (iii) $1+x, y, z$; (iv) $1-x, -y, -z$.

Programs used to solve and refine the structure were *SDS* (Petříček & Malý, 1990) and *SHELX*76 (Sheldrick, 1976). Anisotropic displacement parameters were refined for all non-H atoms. All H atoms were located from the final difference Fourier map and were not refined. Geometrical analysis was performed using *PARST* (Nardelli, 1983). *ORTEP* (Johnson, 1965) was employed for drawing the molecular structure.

This research was carried out under grant 1/990917/93 from the Slovak Ministry of Education and Sciences.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry, including distances involving H atoms, have been deposited with the IUCr (Reference: KA1068). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Trichloromethyltin(IV)

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Abstract

The crystal structure of trichloromethyltin(IV), $[SnCl_3(CH_3)]$, has been solved from single-crystal X-ray diffraction data. The compound crystallizes isostructurally with tribromomethyltin(IV), $[SnBr_3(CH_3)]$. The structure consists of compressed tetrahedral molecules that are arranged in infinite chains along the *b* axis. The molecules exhibit point-group symmetry $C_s(m)$ with only a small deviation from the higher $C_{3v}(3m)$ point-group symmetry. The molecules are linked through extremely weak chlorine–tin interactions.

Comment

The structure determination of the title compound was undertaken in order to verify a prediction, derived from the results of ^{35}Cl nuclear quadrupole resonance (NQR) measurements in the solid state (Petrosyan, Yashina, Reutov, Bryuchova & Semin, 1973), that the Sn atoms are almost octahedrally coordinated.

The compound crystallizes in the centrosymmetric orthorhombic space group *Pnma* (No. 62) with $Z = 4$. Fig. 1 shows the geometry of the molecules that make up the structure. They exhibit $C_s(m)$ symmetry, with all atoms except Cl2 and H2 lying on the mirror plane. The intramolecular Sn—Cl distances differ only slightly from each other (Table 2). The angle between the Sn—C bond and the normal of the plane composed of atoms Cl1, Cl2, Cl2*a* [symmetry code (*a*): $x, \frac{1}{2}-y, z$; Fig. 2] is only 3.5° and the Cl$\cdots$Cl distances are all very similar. Thus, the coordination polyhedron of the Sn atom is a compressed tetrahedron with approximate $C_{3v}(3m)$ point symmetry. [$SnCl_3(CH_3)$] suffers only slight distortion in the solid state, relative to the geometric data determined by gas-phase electron diffraction (Beagley, McAloon & Freeman, 1974). Fig. 2 shows a part of the infinite-chain structure with intermolecular Sn$\cdots$Cl distances of 3.714 (2) Å. The intermolecular Sn$\cdots$Cl distance is slightly shorter than the sum of the van der Waals radii (Bondi, 1964). It can be assumed, therefore, that these interactions are responsible for the arrangement of the molecules in the infinite chains. The title structure is essentially isostructural with tribromomethyltin(IV) (Weiss, Zhang & Dou, 1991).

Based on a concept introduced by Pauling (1960) and refined by Brown (Brown & Altermatt, 1985), bond valences for the Sn—*X* (*X* = Cl, C) interactions have been calculated, the *B* parameter being set at 0.37 (Table 2). Sn—C and Sn—Cl distances from tetramethylstannane and tetrachlorostannane, determined by gas-phase electron diffraction (Fuji & Kimura, 1970, 1971), were taken as standard bond lengths (r_0). The valence sum at the Sn atom has been calculated as 4.02, which is close to the expected value.

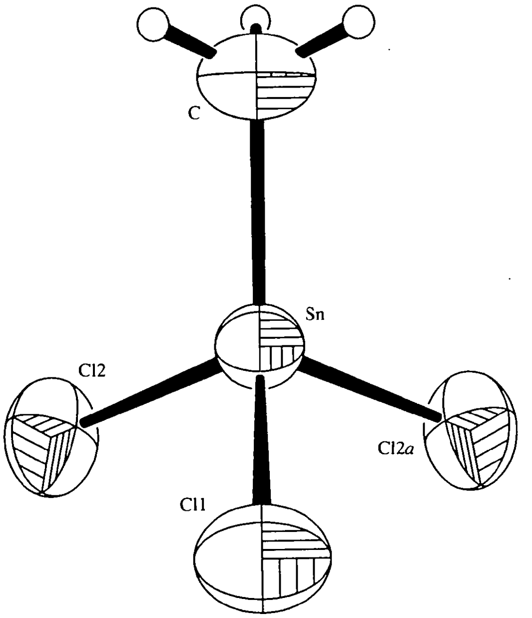

Fig. 1. Diagram of [$SnCl_3(CH_3)$] showing the atom-numbering scheme (displacement ellipsoids at the 50% probability level).

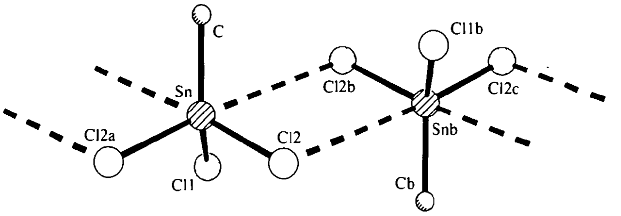

Fig. 2. Chain structure along the *b* axis [symmetry codes: (*a*) $x, \frac{1}{2}-y, z$; (*b*) $-x, 1-y, 1-z$; (*c*) $-x, \frac{1}{2}+y, 1-z$].

Taking into account the calculated bond valence of 0.02 for the interactions between the molecules in the solid state, it is not surprising that these are far too weak to produce a significant effect on the coordination environment of the monomeric species. This was not to be expected *a priori*, since the structure of dichlorodimethyltin(IV) was found, by X-ray structure determination (Davies, Milledge, Puxley & Smith, 1970), to have a distorted octahedral Sn environment and a similar octahedral coordination geometry had been postulated for the title compound, derived from ^{35}Cl NQR measurements (Petrosyan *et al.*, 1973).

A refinement of the structure in the alternative non-centrosymmetric space group *Pna*2$_1$ (No. 33) has been attempted. The resulting *R* values, bond lengths and angles show no significant differences to those derived from the refinement in the higher symmetric space group *Pnma*. However, together with the reported absence of tunnel splitting in inelastic neutron scattering (Weiss, Zhang & Dou, 1991) and the two observed ^{35}Cl NQR resonances (Petrosyan *et al.*, 1973), the centrosymmetric choice seems to be much more plausible.

Experimental

The air-sensitive [$SnCl_3(CH_3)$] compound was synthesized according to procedures given in the literature (Pfeiffer & Lehnhardt, 1903). Colourless needles were grown from hydrocarbon solution at low temperature.

Crystal data

[$SnCl_3(CH_3)$]
$M_r = 240.07$
Orthorhombic
Pnma
$a = 6.411$ (1) Å
$b = 9.564$ (2) Å
$c = 10.637$ (2) Å
$V = 652.2$ (2) Å^3
$Z = 4$
$D_x = 2.445$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 12.97$–$17.10°$
$\mu = 5.003$ mm^{-1}
$T = 293$ (2) K
Transparent needle
0.7 × 0.4 × 0.3 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical
$T_{\min} = 0.598$, $T_{\max} = 0.999$
1985 measured reflections
983 independent reflections
905 observed reflections $[I > 2\sigma(I)]$

$R_{\rm int} = 0.0394$
$\theta_{\max} = 29.85°$
$h = -9 \rightarrow 1$
$k = -13 \rightarrow 11$
$l = -14 \rightarrow 1$
3 standard reflections
frequency: 60 min
intensity variation: none

Refinement

Refinement on F^2
$R(F) = 0.0477$
$wR(F^2) = 0.0860$
$S = 4.139$
983 reflections
35 parameters
H atoms refined with common $U_{\rm iso}$
$w = 1/\sigma^2(F_o^2)$
$(\Delta/\sigma)_{\max} = -0.319$

$\Delta\rho_{\max} = 2.27$ e Å^{-3}
$\Delta\rho_{\min} = -1.52$ e Å^{-3}
Extinction correction: *SHELXL93*
Extinction coefficient: 0.0038 (10)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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	$U_{\rm eq}$
Sn	0.02310 (8)	1/4	0.52960 (4)	0.0488 (2)
Cl1	0.2001 (3)	1/4	0.3434 (2)	0.0746 (6)
Cl2	0.1692 (3)	0.4354 (2)	0.63900 (14)	0.0781 (5)
C	−0.3004 (13)	1/4	0.5314 (9)	0.073 (2)

Table 2. *Selected geometric parameters* (Å, °)

Values in square brackets are calculated bond orders.

Sn—C	2.074 (8) [1.18]	Sn—Cl2	2.318 (1) [0.90]
Sn—Cl1	2.283 (2) [1.00]	Sn—Cl2$^{\rm i}$	3.714 (2) [0.02]
C—Sn—Cl1	120.3 (3)	Cl1—Sn—Cl2	103.58 (6)
C—Sn—Cl2	113.5 (1)		

Symmetry code: (i) $-x, 1-y, 1-z$.

Dispersion corrections and absorption coefficients were taken from *International Tables for Crystallography* (1992, Vol. C) Tables 4.2.6.8 and 4.2.4.2, respectively.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1994). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991). Software used to prepare material for publication: *SHELXL93*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, including H-atom geometry, have been deposited with the IUCr (Reference: HU1116). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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mer-Tris[2-(5-bromo-2-hydroxyphenyl)-2-oxazolinato]aluminium(III) Methanol Solvate

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Abstract

The title complex, (*OC*-6-21)-tris[4-bromo-2-(4,5-dihydro-1,3-oxazol-2-yl)phenolato-*N*,*O*]aluminium(III) methanol solvate, $[\mathrm{Al(C_9H_7BrNO_2)_3].CH_4O}$, has a slightly distorted octahedral coordination geometry with a meridional arrangement of the ligands. Pertinent Al—O and Al—N bond distances are 1.839 (2)–1.876 (2) and 2.015 (3)–2.024 (3) Å, respectively. The methanol solvent is hydrogen bonded to one of the coordinated O atoms.

Comment

The synthesis of the title compound, (I), has been published elsewhere (Hoveyda, Karunaratne, Rettig & Orvig, 1992). The meridional hexacoordinate geometry