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Poly[μ_2-chlorido-nonamethyl-μ_3-nitrato-tritin(IV)]

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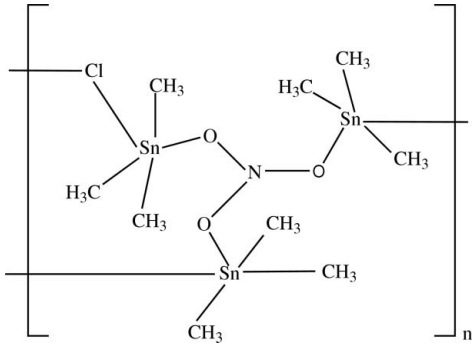
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(O–N) = 0.008 Å; R factor = 0.049; wR factor = 0.131; data-to-parameter ratio = 22.9.

The crystal structure of the title compound, $[Sn_3(CH_3)_9Cl(NO_3)]$, contains trigonal–bipyramidal Sn atoms with three methyl groups bonded in the equatorial plane and an O atom of the NO_3 group and a Cl atom in the axial sites. The Cl atom, which lies on a threefold axis, is bridged between three Sn atoms, with Sn—Cl distances of 2.9298 (13) Å. The N atom of the NO_3 group also lies on a threefold axis, with its O atoms bonded to three Sn atoms. Thus, polymeric sheets are formed parallel to the *ab* plane.

Related literature

For related literature, see: Anderson *et al.* (1984); Gielen (1994); Shahzadi *et al.* (2006); de Vos *et al.* (1998).



Experimental

Crystal data

$[Sn_3(CH_3)_9Cl(NO_3)]$
M_r = 588.84
Trigonal, $R\overline{3}$
a = 9.843 (4) Å
c = 33.073 (5) Å
V = 2775.0 (12) Å^3
Z = 6
Mo $K\alpha$ radiation
μ = 4.16 mm^{-1}
T = 100 (2) K
0.35 × 0.30 × 0.10 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.360, T_{max} = 1.000
(expected range = 0.237–0.660)
5290 measured reflections
1282 independent reflections
1240 reflections with $I > 2\sigma(I)$
R_{int} = 0.040

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.131
S = 1.17
1282 reflections
56 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 4.50 e Å^{-3}
$\Delta\rho_{min}$ = −1.38 e Å^{-3}

Table 1
Selected bond lengths (Å).

Sn1—C2	2.127 (9)	Sn1—O1	2.142 (7)
Sn1—C3	2.134 (10)	Sn1—Cl1	2.9298 (13)
Sn1—C1	2.138 (11)		

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: EZ2088).

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supplementary materials

Acta Cryst. (2007). E**63**, m2329 [**doi:10.1107/S1600536807038792**]

Poly[μ_2-chlorido-nonamethyl-μ_3-nitrato-tritin(IV)]

Sadiq-ur-Rehman, S. Sherzaman, S. Ali, S. Shahzadi and M. Helliwell

Comment

The synthesis and structural chemistry of organotin compounds are fertile areas of research because of their extensive biological and pharmaceutical applications (Anderson *et al.*, 1984). The biological applications of organotin compounds as antitumour and anticancer agents (Gielen, 1994; de Vos *et al.*, 1998) have been well documented. We report here the crystal structure of the title compound (I), in a continuation of our efforts in the synthesis and structural characterization of organotin complexes (Shahzadi *et al.*, 2006). The polymeric structure of (I) which contains trigonal bipyramidal Sn atoms is shown in Fig. 1. The Sn atom is bonded to three methyl groups in equatorial positions with Sn—C distances in the range of 2.127 (9)–2.138 (11) Å. In the axial direction, the Sn atom is bonded to a nitrate O atom and a Cl atom. The nitrate N and the Cl atoms each lie on 3-fold axes. The structure forms polymeric sheets parallel to the *ab* plane, Fig. 1 and 2.

Experimental

4-Hydroxy piperidine (1 mmol) and trimethyltin chloride (1 mmol) were suspended in dry methanol (150 ml) in a round bottom two necked flask. The mixture was stirred at room temperature. Solid product obtained was filtered off and recrystallized from chloroform to obtain colourless crystals suitable for X-ray analysis (yield 80°; m.p. 59 °C).

Refinement

Methyl H atoms were included in calculated positions using the riding method, with C—H distances of 0.96 Å, U_{iso}(H) = 1.5U_{eq}(C) and torsion angles optimized to give the best fit to the electron density.

Figures

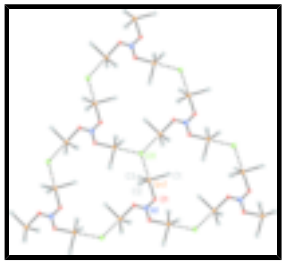


Fig. 1. Structure of (I) showing part of a polymeric sheet with atoms of the asymmetric unit labelled. Dashed lines indicate long Sn—Cl bonds. H atoms are omitted for clarity.

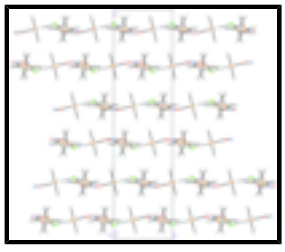


Fig. 2. Packing of (I) viewed down the *a* axis showing the polymeric sheets parallel to the *ab* plane. Dashed lines indicate long Sn—Cl bonds. H atoms are omitted for clarity.

Poly[μ_2-chlorido-nonamethyl-μ_3-nitrato-tritin(IV)]

Crystal data

$[Sn_3(CH_3)_9Cl(NO_3)]$	$Z = 6$
$M_r = 588.84$	$F_{000} = 1674$
Trigonal, $R\overline{3}$	$D_x = 2.114$ Mg m^{-3}
Hall symbol: -R 3	Mo $K\alpha$ radiation $\lambda = 0.71069$ Å
$a = 9.843$ (4) Å	Cell parameters from 2484 reflections
$b = 9.843$ Å	$\theta = 2.7–26.4°$
$c = 33.073$ (5) Å	$\mu = 4.16$ mm^{-1}
$\alpha = 90°$	$T = 100$ (2) K
$\beta = 90°$	Plate, colourless
$\gamma = 120°$	0.35 × 0.30 × 0.10 mm
$V = 2775.0$ (12) Å^3	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	1282 independent reflections
Radiation source: fine-focus sealed tube	1240 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.040$
$T = 100(2)$ K	$\theta_{max} = 26.4°$
φ and ω scans	$\theta_{min} = 1.9°$
Absorption correction: multi-scan (SADABS; Bruker, 2001)	$h = -12\rightarrow12$
$T_{min} = 0.360$, $T_{max} = 1.000$	$k = -10\rightarrow12$
5290 measured reflections	$l = -40\rightarrow36$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.050$	H-atom parameters constrained
$wR(F^2) = 0.131$	$w = 1/[\sigma^2(F_o^2) + (0.0707P)^2 + 62.3389P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.17$	$(\Delta/\sigma)_{max} < 0.001$
1282 reflections	$\Delta\rho_{max} = 4.50$ e Å^{-3}
56 parameters	$\Delta\rho_{min} = -1.38$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of *F*^2^ against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on *F*^2^, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative *F*^2^. The threshold expression of *F*^2^ > σ(*F*^2^) is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on *F*^2^ are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Sn1	0.10596 (7)	0.32791 (9)	0.08605 (2)	0.0215 (2)
Cl1	0.0000	0.0000	0.06588 (14)	0.0316 (10)
O1	0.1849 (8)	0.5680 (8)	0.1007 (2)	0.0246 (14)
N1	0.3333	0.6667	0.1019 (4)	0.025 (3)
C1	−0.1303 (13)	0.2811 (11)	0.0814 (3)	0.031 (2)
H1A	−0.1317	0.3658	0.0674	0.046*
H1B	−0.1734	0.2712	0.1080	0.046*
H1C	−0.1920	0.1852	0.0667	0.046*
C2	0.1779 (12)	0.2725 (12)	0.1413 (3)	0.023 (2)
H2A	0.2700	0.2646	0.1367	0.035*
H2B	0.0951	0.1743	0.1514	0.035*
H2C	0.2010	0.3537	0.1608	0.035*
C3	0.2335 (13)	0.3779 (13)	0.0307 (3)	0.027 (2)
H3A	0.3362	0.3931	0.0359	0.041*
H3B	0.2430	0.4714	0.0189	0.041*
H3C	0.1788	0.2916	0.0123	0.041*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sn1	0.0180 (4)	0.0224 (4)	0.0251 (3)	0.0109 (3)	−0.0007 (2)	−0.0006 (2)
Cl1	0.0258 (14)	0.0258 (14)	0.043 (2)	0.0129 (7)	0.000	0.000
O1	0.015 (3)	0.015 (3)	0.043 (4)	0.007 (3)	0.001 (3)	−0.002 (3)
N1	0.029 (5)	0.029 (5)	0.015 (6)	0.015 (3)	0.000	0.000
C1	0.029 (6)	0.018 (5)	0.044 (6)	0.011 (4)	−0.005 (5)	0.000 (4)
C2	0.020 (5)	0.020 (5)	0.031 (5)	0.010 (4)	−0.003 (4)	−0.003 (4)
C3	0.031 (6)	0.028 (5)	0.026 (5)	0.017 (5)	−0.003 (4)	−0.004 (4)

Geometric parameters (Å, °)

Sn1—C2	2.127 (9)	C1—H1B	0.9600
Sn1—C3	2.134 (10)	C1—H1C	0.9600
Sn1—C1	2.138 (11)	C2—H2A	0.9600

Sn1—O1	2.142 (7)	C2—H2B	0.9600
Sn1—Cl1	2.9298 (13)	C2—H2C	0.9600
O1—N1	1.288 (7)	C3—H3A	0.9600
N1—O1^{i}	1.288 (7)	C3—H3B	0.9600
N1—O1^{ii}	1.288 (7)	C3—H3C	0.9600
C1—H1A	0.9600		
C2—Sn1—C3	124.3 (4)	H1A—C1—H1B	109.5
C2—Sn1—C1	118.2 (4)	Sn1—C1—H1C	109.5
C3—Sn1—C1	116.0 (4)	H1A—C1—H1C	109.5
C2—Sn1—O1	96.0 (3)	H1B—C1—H1C	109.5
C3—Sn1—O1	95.3 (3)	Sn1—C2—H2A	109.5
C1—Sn1—O1	90.6 (3)	Sn1—C2—H2B	109.5
C2—Sn1—Cl1	83.9 (3)	H2A—C2—H2B	109.5
C3—Sn1—Cl1	84.4 (3)	Sn1—C2—H2C	109.5
C1—Sn1—Cl1	89.8 (3)	H2A—C2—H2C	109.5
O1—Sn1—Cl1	179.64 (19)	H2B—C2—H2C	109.5
N1—O1—Sn1	119.2 (4)	Sn1—C3—H3A	109.5
O1^{i}—N1—O1^{ii}	119.91 (8)	Sn1—C3—H3B	109.5
O1^{i}—N1—O1	119.91 (8)	H3A—C3—H3B	109.5
O1^{ii}—N1—O1	119.91 (8)	Sn1—C3—H3C	109.5
Sn1—C1—H1A	109.5	H3A—C3—H3C	109.5
Sn1—C1—H1B	109.5	H3B—C3—H3C	109.5
C2—Sn1—O1—N1	71.1 (9)	Sn1—O1—N1—O1^{i}	−11.0 (17)
C3—Sn1—O1—N1	−54.3 (9)	Sn1—O1—N1—O1^{ii}	163.0 (7)
C1—Sn1—O1—N1	−170.5 (9)		

Symmetry codes: (i) −*y*+1, *x*−*y*+1, *z*; (ii) −*x*+*y*, −*x*+1, *z*.

Fig. 1

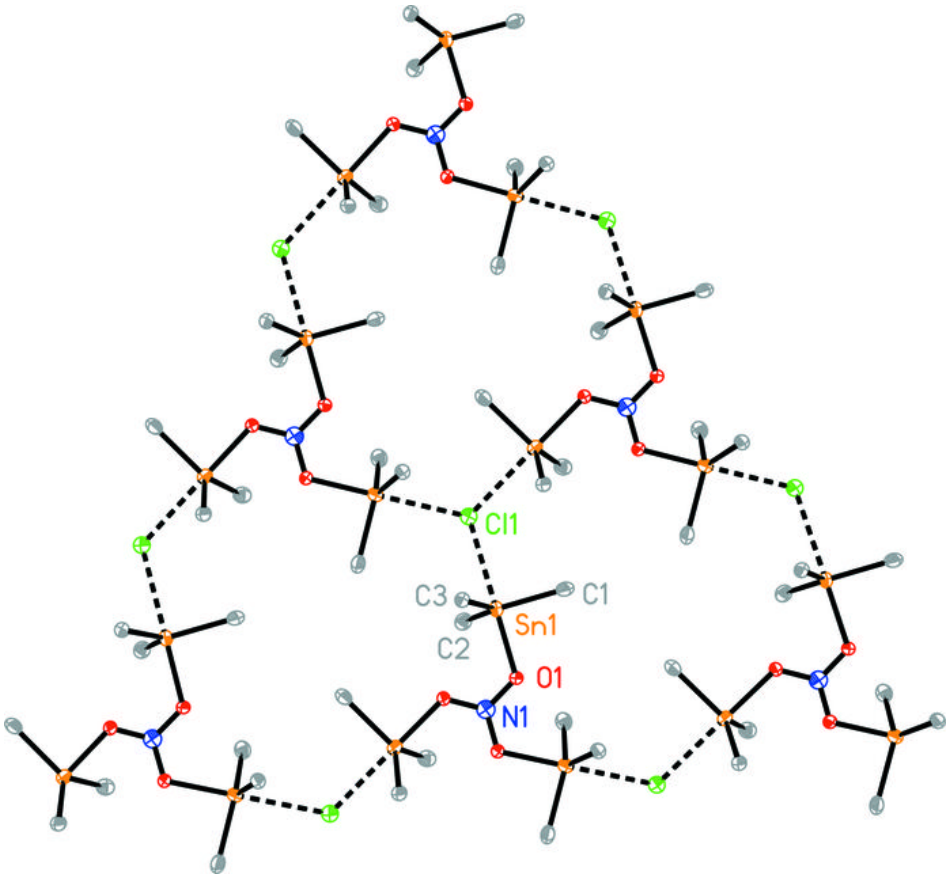
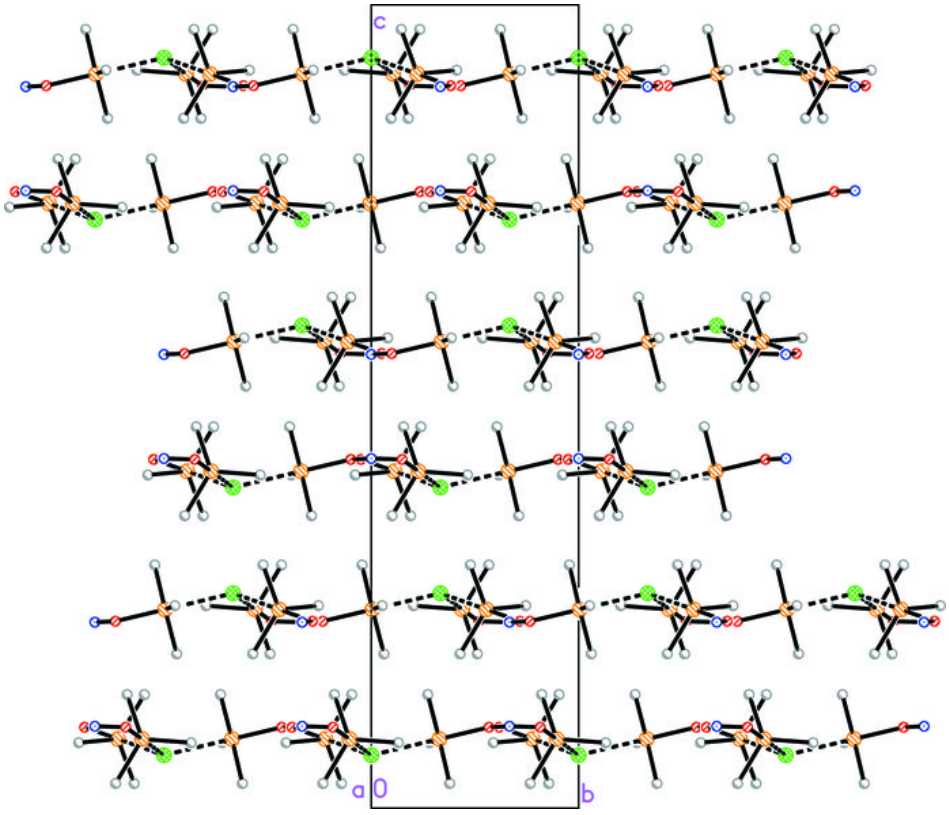


Fig. 2



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Poly[μ_2-chlorido-nonamethyl-μ_3-nitrato-tritin(IV)]. Corrigendum

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An error in the original formulation of the title compound in the paper by Sadiq-ur-Rehman, Sherzaman, Ali, Shahzadi & Helliwell [*Acta Cryst.* (2007), E**63**, m2329] is corrected.

The title compound in the paper by Sadiq-ur-Rehman, Sherzaman, Ali, Shahzadi & Helliwell [*Acta Cryst.* (2007), E**63**, m2329] was an unexpected product which seemed to have nitrate coordinated to three Sn atoms. However, it was noticed that the charges do not balance and that it is most likely that the nitrate is in fact a carbonate. Regrettably, there is no material to carry out microanalysis, but a plausible mechanism has been suggested to explain the unexpected formation of the product. Trimethyltin chloride will react with methanol in the presence of a base (4-hydroxypiperidine) to give trimethyltin methoxide, which will rapidly hydrolyze in air to give the hydroxide. Both the methoxide and the hydroxide will react with atmospheric CO_2 to give the carbonate (Bloodworth *et al.*, 1967; Blunden *et al.*, 1984; Sato, 1967).

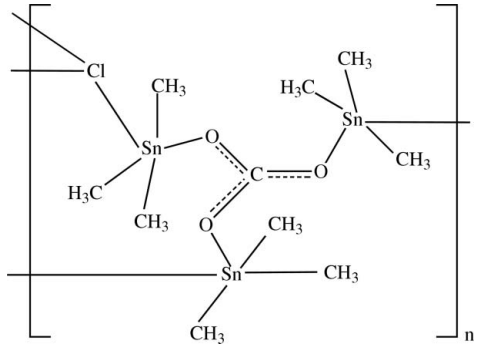
$Me_3SnCl + MeOH + base \rightarrow Me_3SnOMe + base{\cdot}HCl$

$Me_3SnOMe + H_2O \rightarrow Me_3SnOH + MeOH$

$Me_3SnOH + CO_2 \rightarrow Me_3SnOCO_2H$

$Me_3SnOCO_2H + Me_3SnOMe \rightarrow Me_3SnOCO_2SnMe_3 + MeOH.$

The carbonate then forms a coordination copolymer with trimethyltin chloride. The name of the title compound is corrected to poly[μ_3-carbonato-μ_3-chlorido-nonamethyltritin(IV)], $[Sn_3(CH_3)_9(CO_3)Cl]$ (M_r = 586.84).



We thank Professor Alwyn G. Davies (Department of Chemistry, University College London, UK) for providing the mechanism to explain the unexpected formation of the product.

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