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

Sadiq-ur-Rehman,[a] Saira Sherzaman,[a] Saqib Ali,[b]* Saira Shahzadi[c] and Madeleine Helliwell[d]

[a]Department of Chemistry, University of Azad Jammu and Kashmir, Muzaffarabad, Pakistan, [b]Department of Chemistry, Quaid-i-Azam University, Islamabad 45320, Pakistan, [c]Department of Chemistry, G.C. University, Faisalabad, Pakistan, and [d]School of Chemistry, The University of Manchester, Manchester M13 9PL, England
Correspondence e-mail: drsa54@yahoo.com

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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).

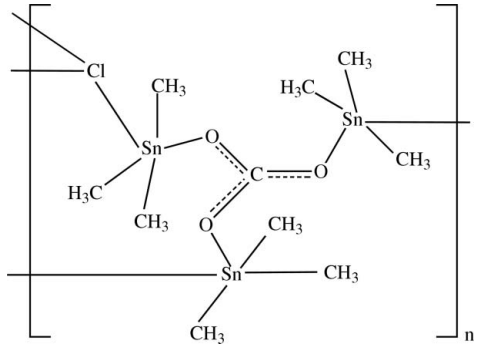
$Me_3SnCl + MeOH + base \rightarrow Me_3SnOMe + base{\cdot}HCl$

$Me_3SnOMe + H_2O \rightarrow Me_3SnOH + MeOH$

$Me_3SnOH + CO_2 \rightarrow Me_3SnOCO_2H$

$Me_3SnOCO_2H + Me_3SnOMe \rightarrow Me_3SnOCO_2SnMe_3 + MeOH.$

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.

References

Bloodworth, A. J., Davies, A. G. & Vasishtha, S. V. (1967). *J. Chem. Soc. C*, pp. 1309–1313.
Blunden, S. J., Hill, R. & Ruddick, J. N. R. (1984). *J. Organomet. Chem.* **267**, C5.
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supporting information

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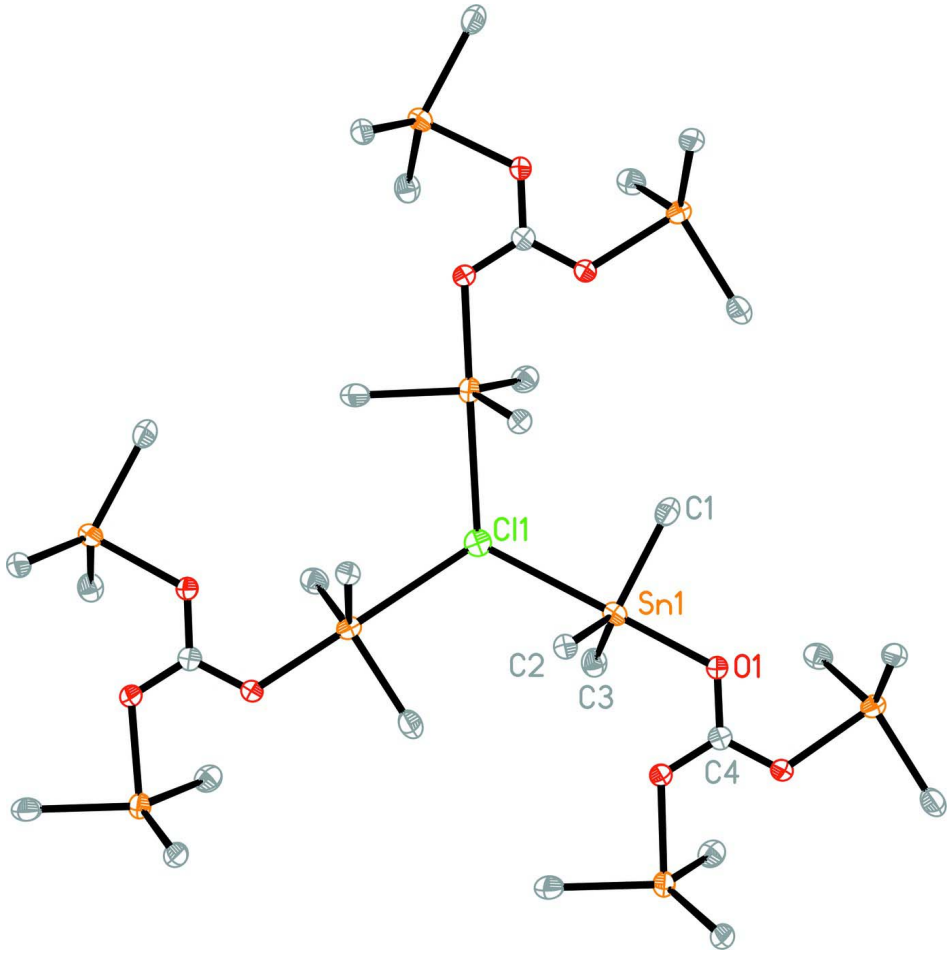

Figure 1

The structure of (I), showing part of a polymeric sheet with atoms of the asymmetric unit labelled. H atoms have been omitted for clarity.

Poly[μ_3-carbonato-μ_3-chlorido-nonamethyltritin(IV)]

Crystal data

$[Sn_3(CH_3)_9(CO_3)Cl]$
M_r = 586.84
Rhombohedral, $R\bar{3}$
Hall symbol: -R 3

a = 9.843 (4) Å
c = 33.073 (5) Å
α = 90°
γ = 120°

V = 2775.0 (12) Å^3
Z = 6
F(000) = 1668
D_x = 2.107 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71069 Å
Cell parameters from 2484 reflections
θ = 2.7–26.4°
μ = 4.16 mm^{-1}
T = 100 K
Plate, colourless
0.35 × 0.30 × 0.10 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.360, T_{max} = 1.000
5290 measured reflections
1282 independent reflections
1240 reflections with $I > 2\sigma(I)$
R_{int} = 0.040
θ_{max} = 26.4°, θ_{min} = 1.9°
h = −12→12
k = −10→12
l = −40→36

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.130
S = 1.17
1282 reflections
56 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0701P)^2 + 60.0465P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 4.50 e Å^{-3}
$\Delta\rho_{min}$ = −1.40 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 > 2sigma(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	0.77263 (7)	0.66125 (9)	0.41938 (2)	0.0215 (2)
Cl1	0.6667	0.3333	0.39922 (14)	0.0318 (10)
O1	0.8520 (8)	0.9015 (8)	0.4340 (2)	0.0245 (14)
C4	1.0000	1.0000	0.4352 (4)	0.015 (3)
C1	0.5364 (13)	0.6144 (12)	0.4148 (3)	0.031 (2)
H1A	0.5349	0.6982	0.4003	0.046*
H1B	0.4940	0.6065	0.4414	0.046*
H1C	0.4743	0.5176	0.4005	0.046*
C2	0.8446 (12)	0.6058 (12)	0.4746 (3)	0.023 (2)
H2A	0.9396	0.6031	0.4703	0.035*
H2B	0.7640	0.5052	0.4840	0.035*

H2C	0.8627	0.6843	0.4945	0.035*
C3	0.9003 (13)	0.7112 (13)	0.3640 (3)	0.027 (2)
H3A	1.0026	0.7256	0.3692	0.041*
H3B	0.9105	0.8051	0.3523	0.041*
H3C	0.8451	0.6253	0.3456	0.041*

Atomic displacement parameters (Å²)

	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	0.0260 (14)	0.0260 (14)	0.043 (2)	0.0130 (7)	0.000	0.000
O1	0.017 (3)	0.016 (3)	0.041 (4)	0.009 (3)	0.001 (3)	−0.002 (3)
C4	0.019 (5)	0.019 (5)	0.005 (6)	0.010 (2)	0.000	0.000
C1	0.029 (6)	0.018 (5)	0.044 (6)	0.011 (4)	−0.005 (5)	−0.001 (4)
C2	0.020 (5)	0.019 (5)	0.032 (5)	0.011 (4)	−0.003 (4)	−0.003 (4)
C3	0.031 (6)	0.028 (5)	0.025 (5)	0.017 (5)	−0.003 (4)	−0.004 (4)

Geometric parameters (Å, º)

Sn1—C2	2.126 (9)	C1—H1A	0.9600
Sn1—C3	2.134 (10)	C1—H1B	0.9600
Sn1—C1	2.138 (11)	C1—H1C	0.9600
Sn1—O1	2.142 (7)	C2—H2A	0.9600
Sn1—Cl1	2.9298 (13)	C2—H2B	0.9600
Cl1—Sn1^{i}	2.9298 (15)	C2—H2C	0.9600
Cl1—Sn1^{ii}	2.9298 (16)	C3—H3A	0.9600
O1—C4	1.285 (7)	C3—H3B	0.9600
C4—O1^{iii}	1.285 (7)	C3—H3C	0.9600
C4—O1^{iv}	1.285 (7)		
C2—Sn1—C3	124.2 (4)	Sn1—C1—H1B	109.5
C2—Sn1—C1	118.2 (4)	H1A—C1—H1B	109.5
C3—Sn1—C1	116.1 (4)	Sn1—C1—H1C	109.5
C2—Sn1—O1	96.0 (3)	H1A—C1—H1C	109.5
C3—Sn1—O1	95.3 (3)	H1B—C1—H1C	109.5
C1—Sn1—O1	90.6 (3)	Sn1—C2—H2A	109.5
C2—Sn1—Cl1	83.9 (3)	Sn1—C2—H2B	109.5
C3—Sn1—Cl1	84.4 (3)	H2A—C2—H2B	109.5
C1—Sn1—Cl1	89.8 (3)	Sn1—C2—H2C	109.5
O1—Sn1—Cl1	179.56 (19)	H2A—C2—H2C	109.5
Sn1^{i}—Cl1—Sn1^{ii}	114.98 (7)	H2B—C2—H2C	109.5
Sn1^{i}—Cl1—Sn1	114.98 (7)	Sn1—C3—H3A	109.5
Sn1^{ii}—Cl1—Sn1	114.98 (7)	Sn1—C3—H3B	109.5
C4—O1—Sn1	119.3 (4)	H3A—C3—H3B	109.5
O1^{iii}—C4—O1^{iv}	119.91 (8)	Sn1—C3—H3C	109.5
O1^{iii}—C4—O1	119.91 (8)	H3A—C3—H3C	109.5
O1^{iv}—C4—O1	119.91 (8)	H3B—C3—H3C	109.5
Sn1—C1—H1A	109.5		

C2—Sn1—Cl1—Sn1^{i}	−44.3 (3)	O1—Sn1—Cl1—Sn1^{ii}	168 (100)
C3—Sn1—Cl1—Sn1^{i}	81.1 (3)	C2—Sn1—O1—C4	71.1 (9)
C1—Sn1—Cl1—Sn1^{i}	−162.6 (3)	C3—Sn1—O1—C4	−54.2 (9)
O1—Sn1—Cl1—Sn1^{i}	31 (25)	C1—Sn1—O1—C4	−170.5 (9)
C2—Sn1—Cl1—Sn1^{ii}	92.7 (3)	Cl1—Sn1—O1—C4	−4 (26)
C3—Sn1—Cl1—Sn1^{ii}	−141.9 (3)	Sn1—O1—C4—O1^{iii}	−11.1 (17)
C1—Sn1—Cl1—Sn1^{ii}	−25.7 (3)	Sn1—O1—C4—O1^{iv}	163.0 (7)

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