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N,N,N',N'-Tetramethylguanidinium tetrachloroantimonate(III) at 295 and 92 K

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Abstract

The crystal structure of *N,N,N',N'*-tetramethylguanidinium tetrachloroantimonate(III), (C₅H₁₄N₃)[SbCl₄], has been determined at 295 and 92 K. Each Sb atom is surrounded by six Cl atoms forming an irregular [SbCl₆]³⁻ octahedron. The octahedra are connected with each other in infinite zigzag chains. There is one crystallographically independent *N,N,N',N'*-tetramethylguanidinium cation in the crystal structure. It is linked to the [SbCl₆]³⁻ octahedra through N—H...Cl hydrogen bonds. The deformation of the octahedral coordination of the Sb^{III} atom is related to the presence of N—H...Cl hydrogen bonds.

Comment

Many halogenoantimonates(III), organic–inorganic salts defined by the general formula *R_aSb_bX_c* (*R* is an organic cation; *X* = chlorine, bromine or iodine; *a*, *b* and *c* are stoichiometric coefficients, where *c* = 3*b* + *a*), show a number of phase transitions, some of

them to ferroelectric, ferrielectric and ferroelastic phases (Jakubas & Sobczyk, 1990). These phase transitions are related mostly to the molecular dynamics of the cations embedded in the anionic sublattices. One of the subgroups of halogenoantimonates(III) constitutes salts of *RSbX₄* stoichiometry. In this group, several different anionic sublattices were found composed either of polyanions or of isolated units. The polyanionic chains may be built in three different ways: (i) as [SbX₆]³⁻ octahedra connected by edges, possessing four bridging and two terminal halogen atoms (Porter & Jacobson, 1970), (ii) as [SbX₆]³⁻ octahedra linked in such a way that two out of three independent Sb atoms have three bridging and three terminal Sb—X bonds, while the third independent Sb atom has five bridging and one terminal Sb—X bond (Lipka, 1980), and (iii) as [SbX₅]²⁻ square pyramids connected by corners (Ensinger *et al.*, 1983).

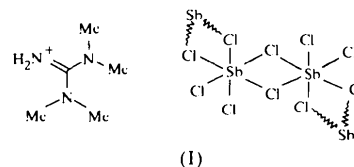
Larger cations break the polyanionic chains, leading to the formation of three different isolated units composed of (i) four octahedra forming [Sb₄X₁₆]⁴⁻ units (Ensinger *et al.*, 1982; Kozawa & Uchida, 1990), (ii) three octahedra connected by faces, forming [Sb₃X₁₂]³⁻ anions (Borgsen *et al.*, 1991), and (iii) two square pyramids linked by an edge (Belz *et al.*, 1992).

Until now, guanidinium chloroantimonates(III) have been analysed by IR, NMR, differential scanning calorimetry (DSC), X-ray, dielectric, pyroelectric and dilatometric methods (Pająk & Zaleski, 1994; Zaleski *et al.*, 1994, 1995). The compounds were characterized by a number of solid–solid phase transitions attributed to changes in the dynamics of the cationic sublattices.

We have synthesized *N,N,N',N'*-tetramethylguanidinium chloroantimonates(III) to study the structure and properties of the resulting salts. Our interest was directed towards the effect of the presence of phase transitions and the deformation of the octahedral coordination of the Sb^{III} atom on the N—H...Cl hydrogen-bonding scheme.

In our earlier work, we noticed that significant changes in Sb—Cl bond lengths take place at phase transitions and are caused by changes in the hydrogen-bonding scheme (Zaleski & Pietraszko, 1996). It should be noted however that changes in Sb—Cl bond lengths also take place with decreasing temperature (Bujak & Zaleski, 1998, 1999). They are much smaller, but are also correlated with changes in strength of the N—H...Cl hydrogen bonds.

The title compound, (I), has the most common anionic sublattice structure for salts of *RSbX₄* stoichiometry, namely polymeric one-dimensional chains.



Each [SbCl₆]³⁻ octahedron shares two *cis* edges with two other neighbours, forming an infinite chain parallel to the [100] direction. The *N,N,N',N'*-tetramethylguanidinium cations are between the inorganic chains, with their NH₂⁺ groups facing the polyanions (Fig. 1). The CN₃ (guanidine) part of each cation is planar, with a mean deviation of 0.005 (3) Å from this plane at both temperatures. Three N—C bonds have partial double-bond character. The *N,N*- and *N',N'*-dimethylammonium groups are twisted with respect to the CN₃ plane by 27(3)°. All the C—N distances and angles in the *N,N,N',N'*-tetramethylguanidinium cation are comparable with those found in [*N,N,N',N'*-tetramethylguanidinium(1+) pentachlorophenolate(1-) pentachlorophenol]₂ (Kanters *et al.*, 1992). The *N,N,N',N'*-tetramethylguanidinium cation is linked to the [SbCl₆]³⁻ octahedra through weak bifurcated N—H...Cl hydrogen bonds. At 295 K, two of the terminal methyl C atoms (C3 and C5) are characterized by large thermal motions, which decrease about three times at 92 K.

A 'non-distorted' [SbCl₆]³⁻ octahedron as part of the infinite chain of *RSbCl₄* stoichiometry was found in pyridinium tetrachloroantimonate(III) (Porter & Jacob-

son, 1970). There is one independent pyridinium cation in the crystal structure. It is dynamically disordered and rotates around a pseudo-sixfold axis, with the position of the N atom distributed over six sites. There are no N—H...Cl hydrogen bonds present. In this way, we may treat the anionic lattice as 'non-distorted' from the point of view of hydrogen bonds. The differences which are observed in the Sb—Cl bond lengths result only from the formation of the polyanionic [SbCl₄]_{*n*} chains. In each [SbCl₆]³⁻ octahedron, there are three pairs of opposite Sb—Cl bonds which may be divided into two groups, *i.e.* one consisting of two pairs involving one bridging and one terminal bond, with the characteristic Sb—Cl bond lengths equal to 2.365 (3) Å for terminal and 3.117 (4) Å for bridging contacts, and the second involving two opposite bridging chlorines, with an Sb—Cl bond length of 2.640 (9) Å, intermediate between the other two lengths.

In (I), the polyanionic chains are more distorted, since here the cations are bound to the anionic sublattice by N—H...Cl hydrogen bonds, which additionally deforms the [SbCl₆]³⁻ octahedra. At 295 K, the two short Sb—Cl terminal distances are 2.362 (2) and 2.442 (2) Å

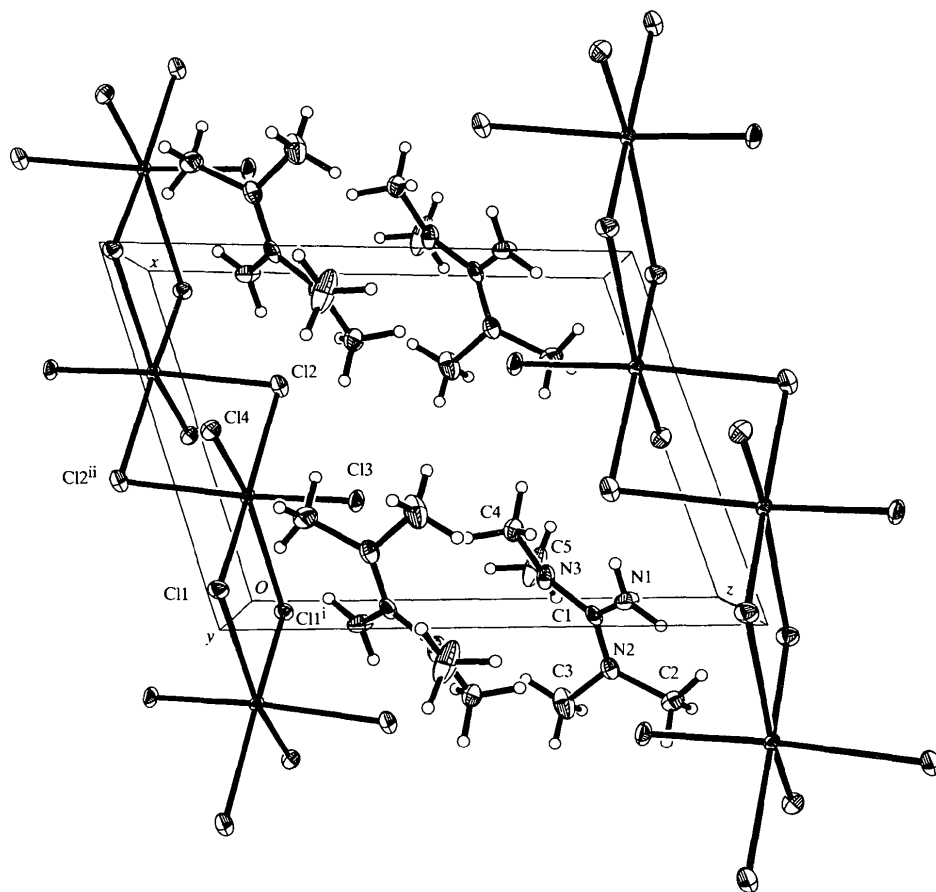


Fig. 1. The crystal structure of *N,N,N',N'*-tetramethylguanidinium tetrachloroantimonate(III) as seen along the *y* axis at 92 K. Displacement ellipsoids are at 50% probability. [Symmetry codes: (i) 2 - *x*, 2 - *y*, 2 - *z*; (ii) 1 - *x*, 2 - *y*, 2 - *z*.]

(Table 1); the first is the same as in $(C_5H_5NH)[SbCl_4]$, while the other is 0.077 (5) Å longer. The two *trans* Sb—Cl bridging distances are 3.255 (2) and 2.978 (2) Å, respectively. They deviate above and below the 'non-distorted' value of 3.117 (4) Å. The third pair, which involves two bridging Sb—Cl bonds in *trans* positions, has lengths of 2.509 (2) and 2.790 (2) Å; here again the first is shorter and the second longer. In this pair, we observe the largest changes in Sb—Cl bond lengths [shortening by 0.13 (1) Å for Sb1—Cl1 and elongation by 0.15 (1) Å for Sb1—Cl2]. This is understandable since bridging chlorines form much longer and therefore weaker bonds in comparison with terminal chlorines. As a result, they may generally be more easily displaced in the direction of the positive charge. Similar to the situation in other halogenoantimonates(III), the longest Sb—X bonds are directly opposite the shortest ones (Zaleski & Pietraszko, 1996). It should be noted that the sums of the bond lengths between the Sb and the opposite Cl atoms, involving two bridging Cl atoms [5.299 (4) Å], are shorter than the two other pairs involving terminal and bridging Cl atoms [5.617 (4) and 5.420 (4) Å]. At room temperature, the Cl—Sb—Cl angles (for Cl atoms *cis* to one another) are in the range 76.63 (6)–112.24 (6)° (Table 1).

The $[SbCl_6]^{3-}$ octahedron and the hydrogen-bonding scheme are shown in Fig. 2. Arrows denote relative shortening/elongation of Sb—Cl bonds in relation to the 'non-distorted' octahedron. The $N1^{iii}—H1B^{iii} \cdots Cl1$ [symmetry code: (iii) $2-x, 1-y, 2-z$] hydrogen bond leads to a shortening of the Sb1—Cl1 bond, while the two others lead to an elongation of the Sb1—Cl1ⁱ [symmetry code: (i) $2-x, 2-y, 2-z$] and Sb1—Cl2 bonds. The Sb1—Cl4 bond length remains unchanged

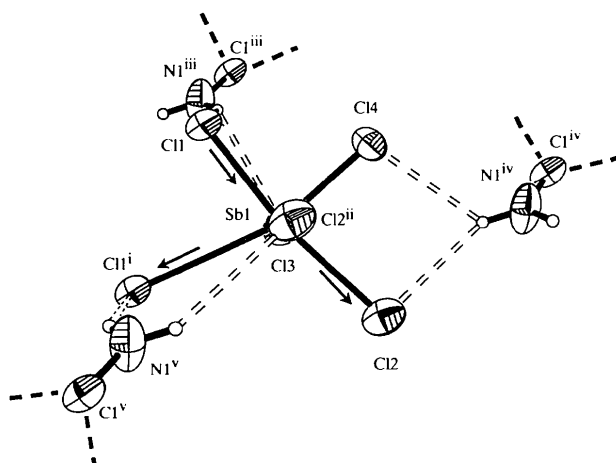


Fig. 2. Hydrogen-bonding scheme showing the $[SbCl_6]^{3-}$ octahedron at 295 K. Arrows denote relative shortening/elongation of Sb—Cl bonds in relation to the 'non-distorted' octahedron (Porter & Jacobson, 1970). Displacement ellipsoids are at 50% probability. [Symmetry codes: (i) $2-x, 2-y, 2-z$; (ii) $1-x, 2-y, 2-z$; (iii) $2-x, 1-y, 2-z$; (iv) $1-x, 1-y, 2-z$; (v) $x, 1+y, z$.]

probably because the force required to change the position of Cl4 is higher since the Sb1—Cl4 distance is much shorter than the others.

At 92 K, the Sb—Cl bond lengths to terminal (Cl3 and Cl4) and two other bridging (Cl1 and Cl2) chlorines do not change significantly [average change 0.007 (3) Å; Table 1]. In contrast, Sb—Cl distances to two other bridging Cl atoms significantly decrease, by 0.025 (3) Å for Sb1—Cl1ⁱ and 0.024 (3) Å for Sb1—Cl2ⁱⁱ [symmetry code: (ii) $1-x, 2-y, 2-z$]. The largest changes in Cl—Sb—Cl bond angles also involve the Cl1ⁱ and Cl2ⁱⁱ atoms (Table 1). The $N \cdots Cl$ contacts are in the range 3.447 (7)–3.618 (7) Å at room temperature, decreasing to 3.395 (3)–3.544 (3) Å at 92 K (Table 2). The largest changes involve the $N1—H1A \cdots Cl2^{ii}$ bond, in which the $N \cdots Cl$ contact decreases by 0.074 (10) Å. This is correlated with the changes in the Sb1—Cl2ⁱⁱ bond length.

Experimental

N,N,N',N'-Tetramethylguanidinium tetrachloroantimonate(III) (m.p. 384 K) was obtained by crystallization from an aqueous solution of hydrochloric acid containing antimony trichloride and *N,N,N',N'*-tetramethylguanidine (molar ratio from 1:1 to 1:10). *N,N,N',N'*-Tetramethylguanidinium tetrachloroantimonate(III) was studied by the DSC method between 300 and 470 K with no phase transitions detected.

Compound (I) at 295 K

Crystal data

$(C_5H_{14}N_3)[SbCl_4]$

$M_r = 379.74$

Triclinic

$P\bar{1}$

$a = 7.873 (2) \text{ \AA}$

$b = 8.985 (2) \text{ \AA}$

$c = 10.412 (2) \text{ \AA}$

$\alpha = 101.55 (3)^\circ$

$\beta = 104.51 (3)^\circ$

$\gamma = 103.52 (3)^\circ$

$V = 666.6 (3) \text{ \AA}^3$

$Z = 2$

$D_x = 1.892 \text{ Mg m}^{-3}$

$D_m = 1.88 (1) \text{ Mg m}^{-3}$

D_m measured by flotation

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 35

reflections

$\theta = 7\text{--}17^\circ$

$\mu = 2.838 \text{ mm}^{-1}$

$T = 295 (2) \text{ K}$

Irregular

$0.40 \times 0.40 \times 0.30 \text{ mm}$

Colourless

Data collection

Kuma KM-4 diffractometer

ω – θ scans

Absorption correction:

empirical *via* ψ scans

(Sheldrick, 1990)

$T_{\min} = 0.356, T_{\max} = 0.427$

5177 measured reflections

2612 independent reflections

2307 reflections with

$I > 2\sigma(I)$

$R_{\text{int}} = 0.020$

$\theta_{\max} = 26.05^\circ$

$h = -9 \rightarrow 9$

$k = -10 \rightarrow 10$

$l = -12 \rightarrow 12$

2 standard reflections

every 50 reflections

intensity decay: 0.8%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.035$
 $wR(F^2) = 0.096$
 $S = 1.244$
 2612 reflections
 121 parameters
 H-atom parameters
 constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0326P)^2 + 2.2466P]$
 where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.879 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.663 \text{ e } \text{\AA}^{-3}$
 Extinction correction:
SHELX97
 Extinction coefficient:
 0.258 (8)
 Scattering factors from
*International Tables for
 Crystallography* (Vol. C)

C11—Sb1—C11 ¹	76.63 (6)	77.02 (3)
C11—Sb1—C12	170.38 (6)	169.48 (2)
C11—Sb1—C12 ¹¹	94.85 (6)	93.37 (3)
C11—Sb1—C13	89.16 (7)	89.30 (3)
C11—Sb1—C14	86.56 (7)	86.27 (4)
C12—Sb1—C11 ¹	112.24 (6)	112.69 (3)
C12—Sb1—C12 ¹¹	87.98 (6)	89.42 (3)
C12 ¹¹ —Sb1—C11 ¹	96.34 (7)	97.42 (4)
C13—Sb1—C11 ¹	87.24 (7)	86.54 (4)
C13—Sb1—C12	87.63 (7)	87.34 (4)
C13—Sb1—C12 ¹¹	175.15 (6)	175.63 (2)
C14—Sb1—C11 ¹	163.18 (5)	163.28 (2)
C14—Sb1—C12	84.57 (7)	83.95 (4)
C14—Sb1—C12 ¹¹	84.36 (7)	83.81 (4)
C14—Sb1—C13	93.16 (8)	92.92 (4)

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

Compound (I) at 92 K

Crystal data

(C₅H₁₄N₃)[SbCl₄]
 $M_r = 379.74$
 Triclinic
 $P\bar{1}$
 $a = 7.818 (2) \text{ \AA}$
 $b = 8.849 (2) \text{ \AA}$
 $c = 10.333 (2) \text{ \AA}$
 $\alpha = 101.18 (3)^\circ$
 $\beta = 104.81 (3)^\circ$
 $\gamma = 103.72 (3)^\circ$
 $V = 646.3 (3) \text{ \AA}^3$
 $Z = 2$
 $D_x = 1.951 \text{ Mg m}^{-3}$
 D_m not measured

Mo $K\alpha$ radiation
 $\lambda = 0.71073 \text{ \AA}$
 Cell parameters from 34
 reflections
 $\theta = 7-21^\circ$
 $\mu = 2.927 \text{ mm}^{-1}$
 $T = 92 (2) \text{ K}$
 Irregular
 $0.28 \times 0.25 \times 0.20 \text{ mm}$
 Colourless

Data collection

Kuma KM-4 diffractometer
 ω - θ scans
 Absorption correction:
 empirical *via* ψ scans
 (Sheldrick, 1990)
 $T_{\min} = 0.462, T_{\max} = 0.557$
 2985 measured reflections
 2789 independent reflections
 2587 reflections with
 $I > 2\sigma(I)$

$R_{\text{int}} = 0.010$
 $\theta_{\max} = 27.06^\circ$
 $h = -9 \rightarrow 0$
 $k = -10 \rightarrow 11$
 $l = -11 \rightarrow 12$
 2 standard reflections
 every 50 reflections
 intensity decay: 1.46%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.020$
 $wR(F^2) = 0.052$
 $S = 1.110$
 2789 reflections
 120 parameters
 H-atom parameters
 constrained

$w = 1/[\sigma^2(F_o^2) + (0.0266P)^2 + 0.8892P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.545 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.466 \text{ e } \text{\AA}^{-3}$
 Extinction correction: none
 Scattering factors from
*International Tables for
 Crystallography* (Vol. C)

Table 1. Selected geometric parameters ($\text{\AA}, ^\circ$)

	295 K	92 K
Sb1—C11	2.509 (2)	2.503 (1)
Sb1—C11 ¹	3.255 (2)	3.230 (1)
Sb1—C12	2.790 (2)	2.797 (1)
Sb1—C12 ¹¹	2.978 (2)	2.954 (1)
Sb1—C13	2.442 (2)	2.451 (1)
Sb1—C14	2.362 (2)	2.368 (1)

Table 2. Hydrogen-bonding geometry ($\text{\AA}, ^\circ$)

In the table below, for ease of comparison, the first line of data for each hydrogen bond is for the structure at 92 K, the second for the structure at 295 K.

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
N1—H1B \cdots C11 ¹	0.90	2.67	3.466 (3)	149
	0.90	2.68	3.479 (6)	149
N1—H1B \cdots C13 ¹	0.90	2.91	3.482 (3)	124
	0.90	2.96	3.542 (7)	124
N1—H1A \cdots C12 ¹¹	0.90	2.76	3.544 (3)	148
	0.90	2.82	3.618 (7)	148
N1—H1A \cdots C14 ¹¹	0.90	2.82	3.395 (3)	124
	0.90	2.86	3.447 (7)	125

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

For both compounds, data collection: *KM-4 Software* (Kuma, 1996); cell refinement: *KM-4 Software*; data reduction: *KM-4 Software*; program(s) used to solve structures: *SHELX97* (Sheldrick, 1997); program(s) used to refine structures: *SHELX97*; molecular graphics: *SHELXTL* (Sheldrick, 1990); software used to prepare material for publication: *SHELX97*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: GD1040). Services for accessing these data are described at the back of the journal.

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Bis(dicyclohexylammonium) tributyl-(nitrilotriacetato)stannate†

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Abstract

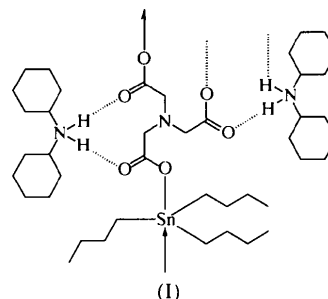
In the title compound, $(C_{12}H_{24}N)_2[Sn(C_4H_9)_3(C_6H_5NO_6)]$, the tributyltin groups are linked by the nitrilotriacetate groups [$Sn-O = 2.281(4)$ and $2.258(3)$ Å] into a linear helical chain. Adjacent chains are linked by one of the two dicyclohexylammonium counter-ions [$N \cdots O = 2.727(5)$ and $2.858(5)$ Å] into layers.

Comment

Unlike other dicyclohexylammonium hydrogen dicarboxylates (oxalate, malonate and succinate) that condense with bis(tributyltin) oxide to afford ammonium stannate compounds having complex formulations (Ng *et al.*, 1990, 1992; Ng & Holeček, 1998), the mono-(dicyclohexylammonium) derivative of the aminopolycarboxylic acid 2,6-pyridinedicarboxylic acid furnishes a simple ammonium stannate. In this compound, the tributyltin groups are linked axially by the 2,6-pyridinedicarboxylate groups into linear chains. The dicyclohexylammonium cations surround the chains and the cation forms hydrogen bonds with the two carbonyl O atoms of the same carboxylate unit (Ng *et al.*, 1991). The bis(dicyclohexylammonium) salt of tribasic nitrilotriacetic acid also yields a simple ammonium stannate when treated with the organotin oxide. The nitrilotriacetate trianion has been used to chelate to a plethora of metal complexes (Oliver *et al.*, 1984); however, few organometallic derivatives have been prepared and only one, 3-dimethylaminopropyl(nitrilotriacetato)-

tin *N*-oxide, has been crystallographically documented (Dakternieks *et al.*, 1993). In this compound, the nitrilotriacetate group displays tetradentate ($N+3O$) ligating behavior.

In the chiral title compound, bis(dicyclohexylammonium) tributyl(nitrilotriacetato)stannate, (I), the tributyltin and nitrilotriacetate groups are covalently



bonded into a linear helical chain (Fig. 1). The Sn atom shows $trans-C_3SnO_2$ trigonal-bipyramidal coordination [$\Sigma_{C-Sn-C} = 359.8(9)^\circ$ and $O-Sn-O = 176.8(1)^\circ$]. The two Sn—O bonds are longer than the covalent Sn—O bonds but shorter than the carboxylate bridges found in five-coordinate triorganotin carboxylates (Tiekink, 1991, 1994), in agreement with a 1.5 bond order for the Sn—O bond in this stannate.

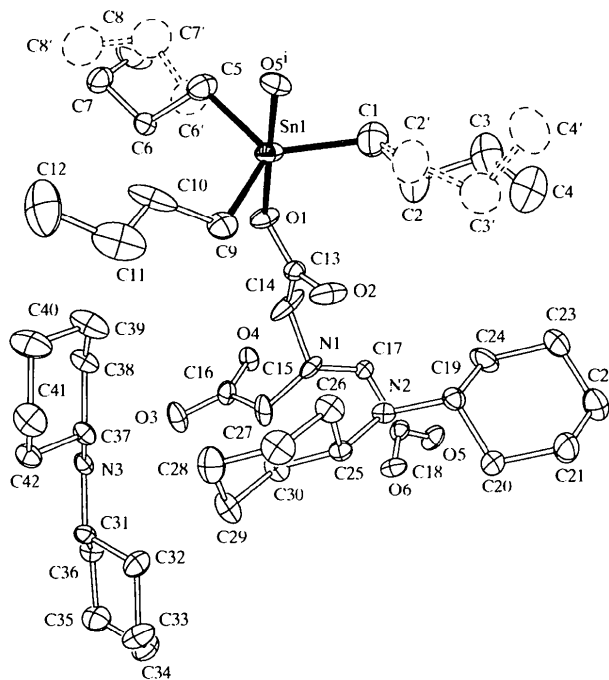


Fig. 1. ORTEP (Johnson, 1976) plot of bis(dicyclohexylammonium) tributyl(nitrilotriacetato)stannate at the 50% probability level. H atoms are not shown. The two orientations of the disordered *n*-butyl groups are shown. [Symmetry code: (i) $2 - x, \frac{1}{2} + y, \frac{1}{2} - z$].

† Alternative name: *catena*-poly[bis(dicyclohexylammonium) [[tributyltin(IV)]- μ -[nitrilotriacetato(3-)-*O*:*O'*]]].