

## Isostructural crystal packing and hydrogen bonding in alkylammonium tin(IV) chloride compounds

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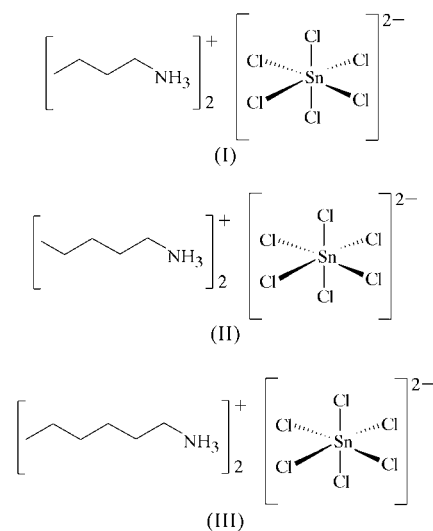
The three isostructural compounds butylammonium hexachloridotin(IV), pentylammonium hexachloridotin(IV) and hexylammonium hexachloridotin(IV),  $(C_nH_{2n+1}NH_3)_2[SnCl_6]$ , with  $n = 4, 5$  and  $6$ , respectively, crystallize as inorganic–organic hybrids. As such, the structures consist of layers of  $[SnCl_6]^{2-}$  octahedra, separated by hydrocarbon layers of interdigitated butylammonium, pentylammonium or hexylammonium cations. Corrugated layers of cations alternate with tin(IV) chloride layers. The asymmetric unit in each compound consists of an anionic component comprising one Sn and two Cl atoms on a mirror plane, and two Cl atoms in general positions; the two cations lie on another mirror plane. Application of the mirror symmetry generates octahedral coordination around the Sn atom. All compounds exhibit bifurcated and simple hydrogen-bonding interactions between the ammonium groups and the Cl atoms, with little variation in the hydrogen-bonding geometries.

### Comment

The bis-ammonium tetrahalometallate inorganic–organic hybrid materials of general formula  $(R-NH_3)_2[MX_4]$  [ $R = C_nH_{2n+1}$ – and  $Ar-(CH_2)_m$ –, with  $n = 1$ – $18$ ,  $m = 1$ – $3$ ,  $M = Pb, Sn, Cu, Mn$  and  $Cd$ , and  $X = Cl, Br$  and  $I$ ] are characterized by the presence of two-dimensional layers of corner-sharing  $MX_6$  octahedra, each sandwiched between two hydrocarbon layers (Mitzi, 1999). The overall structure exhibits an alternation of inorganic and organic layers. The interface between the inorganic and hydrocarbon layers consists of  $Cl^-$  and  $-NH_3^+$  ions, in which strong charge-assisted hydrogen bonds connect the separate layers. If the metal is tetravalent, with general formula  $(R-NH_3)_2[MX_6]$ , the dimensionality is reduced and discrete  $MX_6$  octahedra exist. This phenomenon, where the motif of the inorganic part depends on the valency of the metal, has been observed for Sn, and extensive investigations of divalent Sn have been reported (Koutselas *et al.*, 1996; Mitzi *et al.*, 1998; Kagan *et al.*, 1999; Yin & Yo, 1998). However,

tetravalent Sn has been less well studied and generally few compounds of the type  $(C_nH_{2n+1}NH_3)_2[SnCl_6]$  (Lee *et al.*, 1998, 2002; Elleuch *et al.*, 1999; Aruta *et al.*, 2005, and references therein) and  $[C_6H_5-(CH_2)_n-NH_3]_2[SnCl_6]$  [ $n = 0$  (Rademeyer, 2004a) and  $n = 1$  (Rademeyer, 2004b)] have been reported in the literature. It has been found that the separation between the Sn atoms is sufficiently long, at 7.3–7.5 Å, that the hydrocarbon chains can interdigitate (Lee *et al.*, 2002).

The aim of the present report is to describe the single-crystal structures of the three isostructural compounds containing tetravalent tin(IV) chloride and butylammonium, (I), pentylammonium, (II), or hexylammonium, (III), counterions. The atomic numbering scheme of the asymmetric units in all three compounds is given in Fig. 1. Other compounds with two or fewer H atoms on the N atom have been reported (Knop *et al.*, 1983) but are not considered in this study.



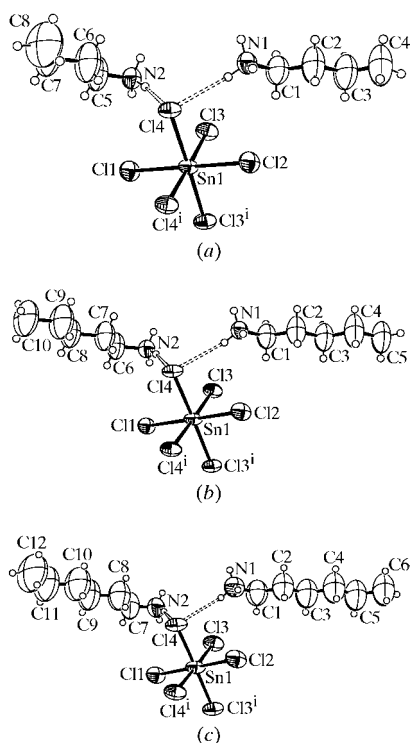
The structure of  $(C_4H_9NH_3)_2[SnCl_6]$ , (I), has been reported previously by Elleuch *et al.* (1996) but was only briefly discussed. The packing arrangement is related to that of  $(C_2H_5NH_3)_2[SnCl_6]$ , which crystallizes in the space group  $P\bar{3}m1$  (Knop *et al.*, 1983). Compounds (I), (II) and (III) contain alternating hydrocarbon layers of butylammonium molecules and inorganic layers of isolated  $SnCl_6$  octahedra. The layers stack along the  $c$  axis (Fig. 2). In the directions of the  $a$  and  $b$  axes, cohesion between the inorganic and hydrocarbon layers is achieved by  $N-H\cdots Cl$  hydrogen bonds, related to the  $NH_3$  polar groups.

The inorganic component of the asymmetric unit of (I) contains an  $Sn^{IV}$  centre, two Cl atoms (Cl1 and Cl2) on a site of  $m$  symmetry at  $y = \frac{1}{4}$ , and two Cl atoms (Cl3 and Cl4) on general positions (Fig. 1). Mirror symmetry generates another two Cl atoms [Cl3<sup>v</sup> and Cl4<sup>v</sup>; symmetry code: (v)  $x, \frac{1}{2} - y, z$ ] to complete the octahedral coordination around the Sn atom. A consequence of the mirror symmetry within the octahedra are four unique Sn–Cl bond lengths, in a narrow range from 2.4075 (15) to 2.4273 (10) Å, and *cis* Cl–Sn–Cl bond angles varying from 88.76 (4) to 92.05 (5)°. The  $SnCl_6$  octahedra are tilted with respect to the layer they occupy; the vector through

Cl1 and Cl2 makes an angle of  $49.08(2)^\circ$  with the normal to the layers. Successive inorganic layers tilt in opposite directions and are separated by an interlayer spacing of  $10.7418(4) \text{ \AA}$ , corresponding to half of the unit-cell length extending perpendicular to the inorganic layer.

The asymmetric unit in (I) is completed by two symmetry-independent butylammonium cations lying across the same mirror plane at  $\frac{3}{4}$ , identified as cat1 (containing N1) and cat2 (containing N2). The mirror symmetry implies an all-*trans* geometry for the hydrocarbon chains. The cations lie parallel to the (010) plane, with the  $C4 \cdots N1$  vector making an angle of  $25.7(1)^\circ$  with the *c* axis.

The two ammonium groups in the asymmetric unit of (I) display the same pattern of hydrogen-bonding interactions with  $Cl^-$  ions. Atom H1A on atom N1 forms a single hydrogen bond to  $Cl4^i$ , which exhibits the shortest  $H \cdots A$  distance of  $2.52 \text{ \AA}$  (symmetry codes as in Table 1). Atoms H1B and H1C both form bifurcated hydrogen bonds to the acceptor atoms  $Cl2^{iii}$ , Cl3, Cl4 and  $Cl4^{ii}$ , which are significantly longer than the simple hydrogen bond (see Table 1 and Fig. 3). Atom H2C on atom N2 forms a single hydrogen bond to Cl4, while atoms H2A and H2B both form bifurcated hydrogen bonds, to the acceptor atoms  $Cl1^{iii}$ ,  $Cl1^{iv}$ ,  $Cl3^{iv}$  and  $Cl3^{ii}$ . Cat1 forms no hydrogen bonds to Cl1 and, similarly, cat2 forms no hydrogen bonds to Cl2. The hydrogen-bonding geometry is repeated for the pentylammonium cation in (II) (see Table 2). This pattern is interrupted in (III), where atom N1 forms two single hydrogen bonds *via* atoms H1A and H1B, to Cl4 and  $Cl4^i$ , and

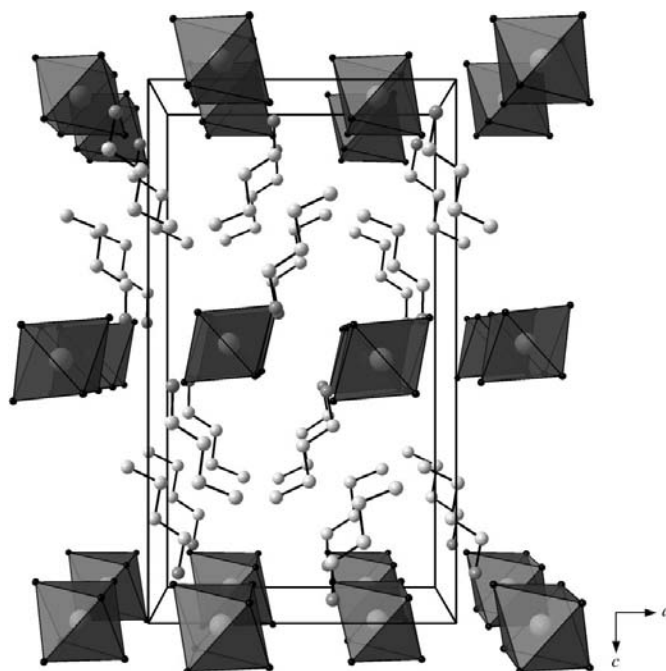


**Figure 1**  
The asymmetric units of (a) (I), (b) (II) and (c) (III), showing the atomic numbering schemes. Displacement ellipsoids are shown at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. [Symmetry code: (v)  $x, -y + \frac{1}{2}, z$ .]

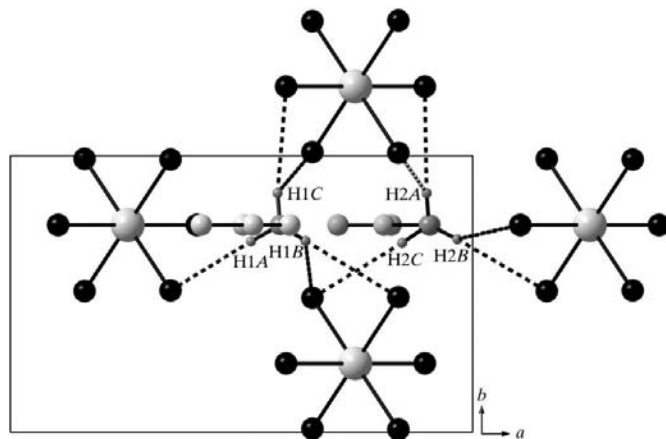
a bifurcated hydrogen bond to  $Cl4^{ii}$  and  $Cl2^{iii}$ , such that cat1 forms no hydrogen bonds to either Cl1 or Cl3. Atom N2 repeats the same pattern as in (I) and (II) (Table 3 and Fig. 4).

The differences in the geometries of the inorganic layers and the  $SnCl_6$  octahedra among the three compounds are summarized in Table 4. The shortest Sn—Cl bond length [ $2.400(3) \text{ \AA}$ ] is found for (III) and the longest [ $2.4311(11) \text{ \AA}$ ] for (II), with the average Sn—Cl bond lengths increasing from (I) to (III). The widest bond angle involving mutually *cis* chloride ions varies from  $92.05(5)^\circ$  in (I) to  $92.21(6)^\circ$  in (II).

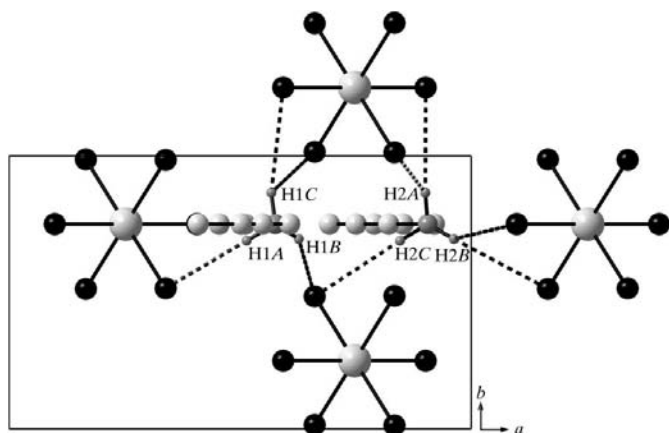
The interlayer spacing increases as a function of increasing chain length and the tilt of the  $SnCl_6$  octahedra decreases



**Figure 2**  
The packing of (I), viewed along the *b* axis.



**Figure 3**  
A magnified view of the hydrogen bonding of the two butylammonium cations in (I). Each cation has two bifurcated and one simple  $H \cdots Cl$  bond. The same geometry is seen in (II).


**Figure 4**

A magnified view of the hydrogen bonding of the two hexylammonium cations in (III). Cat1 has one bifurcated and two simple H···Cl bonds, while cat2 has two bifurcated and one simple H···Cl bond as in (I) and (II).

simultaneously. There is a greater increase in the interlayer spacing in going from (I) to (II) than from (II) to (III). The tilt of the cations decreases consistently by approximately 4°, whereas the tilt of the SnCl<sub>6</sub> octahedra is greatest in (I) and decreases to approximately 39° for both (II) and (III). The average N···Cl distance within the hydrogen bonds is remarkably consistent.

## Experimental

For the preparation of (I), C<sub>4</sub>H<sub>9</sub>NH<sub>2</sub> (0.098 g, 1.34 mmol) was combined with SnCl<sub>2</sub> (0.119 g, 0.628 mmol) in 33% aqueous HCl (2 ml). The resulting precipitate was dissolved by refluxing for 48 h at 353 K. The solution was then cooled slowly to room temperature at a rate of 2 K h<sup>-1</sup>, and colourless block-shaped crystals were harvested. For the preparation of (II), C<sub>5</sub>H<sub>11</sub>NH<sub>2</sub> (0.123 g, 1.41 mmol) was combined with SnCl<sub>2</sub> (0.119 g, 0.570 mmol) and dissolved in aqueous HCl (33%, 5 ml). The resulting solution was left open to the atmosphere, and colourless plate-like crystals were grown by slow evaporation. For the preparation of (III), C<sub>6</sub>H<sub>13</sub>NH<sub>2</sub> (0.155 g, 1.53 mmol) was combined with SnCl<sub>2</sub> (0.119 g, 0.533 mmol) and dissolved in aqueous HCl (33%, 5 ml). The resulting solution was left open to the atmosphere, and colourless plate-like crystals were grown by slow evaporation.

### Compound (I)

#### Crystal data

(C<sub>4</sub>H<sub>12</sub>N)<sub>2</sub>[SnCl<sub>6</sub>] Z = 4  
*M<sub>r</sub>* = 479.68 *D<sub>x</sub>* = 1.675 Mg m<sup>-3</sup>  
 Orthorhombic, *Pnma* Mo *K*α radiation  
*a* = 12.1624 (4) Å *μ* = 2.17 mm<sup>-1</sup>  
*b* = 7.2782 (2) Å *T* = 243 (2) K  
*c* = 21.4837 (7) Å Block, colourless  
*V* = 1901.75 (10) Å<sup>3</sup> 0.26 × 0.24 × 0.1 mm

#### Data collection

Bruker SMART APEXII CCD 14713 measured reflections  
 area-detector diffractometer 2470 independent reflections  
 $\omega$  scans 2088 reflections with *I* > 2σ(*I*)  
 Absorption correction: integration *R<sub>int</sub>* = 0.062  
 (*XPREP*; Bruker, 2004) *θ<sub>max</sub>* = 28°  
*T<sub>min</sub>* = 0.535, *T<sub>max</sub>* = 0.822

#### Refinement

Refinement on *F*<sup>2</sup> *w* = 1/[σ<sup>2</sup>(*F<sub>o</sub>*<sup>2</sup>) + (0.0367*P*)<sup>2</sup>  
 $R[F^2 > 2\sigma(F^2)] = 0.038$  + 3.3848*P*  
 $wR(F^2) = 0.103$  where *P* = (*F<sub>o</sub>*<sup>2</sup> + 2*F<sub>c</sub>*<sup>2</sup>)/3  
*S* = 1.13 ( $\Delta/\sigma$ )<sub>max</sub> < 0.001  
 2470 reflections  $\Delta\rho_{max}$  = 0.54 e Å<sup>-3</sup>  
 98 parameters  $\Delta\rho_{min}$  = -0.53 e Å<sup>-3</sup>  
 H-atom parameters constrained Extinction correction: *SHELXL97*  
Extinction coefficient: 0.0113 (7)

**Table 1**

Hydrogen-bond geometry (Å, °) for (I).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
N1—H1A···Cl4 <sup>i</sup>	0.90	2.52	3.423 (5)	177
N1—H1B···Cl4	0.90	2.67	3.365 (5)	135
N1—H1B···Cl3	0.90	2.88	3.651 (5)	145
N1—H1C···Cl4 <sup>ii</sup>	0.90	2.73	3.365 (5)	128
N1—H1C···Cl2 <sup>iii</sup>	0.90	2.85	3.6502 (5)	149
N2—H2A···Cl1 <sup>iii</sup>	0.90	2.81	3.6408 (2)	155
N2—H2A···Cl3 <sup>ii</sup>	0.90	2.86	3.519 (5)	131
N2—H2B···Cl3 <sup>iv</sup>	0.90	2.77	3.564 (5)	147
N2—H2B···Cl1 <sup>iv</sup>	0.90	2.83	3.533 (6)	136
N2—H2C···Cl4	0.90	2.76	3.630 (5)	163

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

### Compound (II)

#### Crystal data

(C<sub>5</sub>H<sub>14</sub>N)<sub>2</sub>[SnCl<sub>6</sub>] Z = 4  
*M<sub>r</sub>* = 507.73 *D<sub>x</sub>* = 1.558 Mg m<sup>-3</sup>  
 Orthorhombic, *Pnma* Mo *K*α radiation  
*a* = 12.2351 (5) Å *μ* = 1.91 mm<sup>-1</sup>  
*b* = 7.2021 (3) Å *T* = 243 (2) K  
*c* = 24.5723 (9) Å Plate, colourless  
*V* = 2165.27 (15) Å<sup>3</sup> 0.32 × 0.3 × 0.06 mm

#### Data collection

Bruker SMART APEXII CCD 16581 measured reflections  
 area-detector diffractometer 2820 independent reflections  
 $\omega$  scans 2038 reflections with *I* > 2σ(*I*)  
 Absorption correction: integration *R<sub>int</sub>* = 0.043  
 (*XPREP*; Bruker, 2004) *θ<sub>max</sub>* = 28°  
*T<sub>min</sub>* = 0.571, *T<sub>max</sub>* = 0.893

#### Refinement

Refinement on *F*<sup>2</sup> *w* = 1/[σ<sup>2</sup>(*F<sub>o</sub>*<sup>2</sup>) + (0.0464*P*)<sup>2</sup>  
 $R[F^2 > 2\sigma(F^2)] = 0.043$  + 4.3427*P*  
 $wR(F^2) = 0.118$  where *P* = (*F<sub>o</sub>*<sup>2</sup> + 2*F<sub>c</sub>*<sup>2</sup>)/3  
*S* = 1.07 ( $\Delta/\sigma$ )<sub>max</sub> = 0.001  
 2820 reflections  $\Delta\rho_{max}$  = 0.82 e Å<sup>-3</sup>  
 109 parameters  $\Delta\rho_{min}$  = -0.59 e Å<sup>-3</sup>  
 H-atom parameters constrained

**Table 2**

Hydrogen-bond geometry (Å, °) for (II).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
N1—H1A···Cl4 <sup>i</sup>	0.90	2.54	3.438 (5)	177
N1—H1B···Cl4	0.90	2.68	3.411 (5)	139
N1—H1B···Cl3	0.90	2.89	3.636 (5)	142
N1—H1C···Cl4 <sup>ii</sup>	0.90	2.80	3.411 (5)	126
N1—H1C···Cl2 <sup>iii</sup>	0.90	2.79	3.6062 (4)	151
N2—H2A···Cl1 <sup>iii</sup>	0.90	2.76	3.6024 (2)	156
N2—H2A···Cl3 <sup>ii</sup>	0.90	2.90	3.546 (5)	130
N2—H2B···Cl3 <sup>iv</sup>	0.90	2.92	3.661 (5)	141
N2—H2B···Cl1 <sup>iv</sup>	0.90	2.71	3.437 (6)	139
N2—H2C···Cl4	0.90	2.64	3.519 (5)	167

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

Compound (III)

Crystal data

(C<sub>6</sub>H<sub>16</sub>N)<sub>2</sub>[SnCl<sub>6</sub>] Z = 4  
 M<sub>r</sub> = 535.79 D<sub>x</sub> = 1.478 Mg m<sup>-3</sup>  
 Orthorhombic, Pnma Mo Kα radiation  
 a = 12.3622 (3) Å μ = 1.72 mm<sup>-1</sup>  
 b = 7.3179 (2) Å T = 293 (2) K  
 c = 26.6245 (6) Å Plate, colourless  
 V = 2408.59 (10) Å<sup>3</sup> 0.36 × 0.28 × 0.09 mm

Data collection

Bruker SMART APEXII CCD 18912 measured reflections  
 area-detector diffractometer 3129 independent reflections  
 ω scans 2094 reflections with I > 2σ(I)  
 Absorption correction: integration R<sub>int</sub> = 0.055  
 XPREP (Bruker, 2004) θ<sub>max</sub> = 28°  
 T<sub>min</sub> = 0.603, T<sub>max</sub> = 0.856

Refinement

Refinement on F<sup>2</sup> w = 1/[σ<sup>2</sup>(F<sub>o</sub><sup>2</sup>) + (0.0677P)<sup>2</sup>  
 R[F<sup>2</sup> > 2σ(F<sup>2</sup>)] = 0.075 + 14.1291P]  
 wR(F<sup>2</sup>) = 0.217 where P = (F<sub>o</sub><sup>2</sup> + 2F<sub>c</sub><sup>2</sup>)/3  
 S = 1.11 (Δ/σ)<sub>max</sub> < 0.001  
 3129 reflections Δρ<sub>max</sub> = 0.68 e Å<sup>-3</sup>  
 121 parameters Δρ<sub>min</sub> = -0.91 e Å<sup>-3</sup>  
 H-atom parameters constrained

Table 3

Hydrogen-bond geometry (Å, °) for (III).

D—H...A	D—H	H...A	D...A	D—H...A
N1—H1A...Cl4 <sup>i</sup>	0.89	2.54	3.434 (10)	177
N1—H1B...Cl4	0.89	2.68	3.443 (10)	144
N1—H1C...Cl4 <sup>ii</sup>	0.89	2.86	3.443 (10)	125
N1—H1C...Cl2 <sup>iii</sup>	0.89	2.87	3.6680 (9)	150
N2—H2A...Cl1 <sup>iii</sup>	0.89	2.83	3.6597 (3)	156
N2—H2A...Cl3 <sup>ii</sup>	0.89	2.88	3.537 (10)	132
N2—H2B...Cl3 <sup>iv</sup>	0.89	2.92	3.660 (12)	142
N2—H2B...Cl1 <sup>iv</sup>	0.89	2.76	3.484 (13)	139
N2—H2C...Cl4	0.89	2.73	3.599 (11)	167

Symmetry codes: (i) -x + 1, -y + 1, -z + 1; (ii) x, -y + ½, z; (iii) x, y + 1, z; (iv) -x + 2, -y + 1, -z + 1.

Table 4

Comparative geometric parameters (Å, °) in (I), (II) and (III).

Parameter	(I)	(II)	(III)
Sn1—Cl1	2.4235 (15)	2.4303 (11)	2.423 (3)
Sn1—Cl2	2.4075 (15)	2.4109 (17)	2.400 (3)
Sn1—Cl3	2.4172 (10)	2.4096 (11)	2.409 (2)
Sn1—Cl4	2.4273 (10)	2.4311 (11)	2.430 (2)
Sn1—Cl3 <sup>i</sup>	2.4172 (10)	2.4096 (11)	2.409 (2)
Sn1—Cl4 <sup>i</sup>	2.4273 (10)	2.4311 (11)	2.430 (2)
Average Sn—Cl	2.4200 (11)	2.4204 (11)	2.417 (2)
Cl3—Sn1—Cl3 <sup>i</sup>	92.05 (5)	92.21 (6)	92.18 (12)
Tilt of SnCl <sub>6</sub>	49.08 (2)	38.25 (2)	39.11 (4)
Tilt of cations	25.7 (1)	21.8 (1)	17.8 (2)
Interplanar spacing	10.7415 (7)	12.2862 (9)	13.3123 (6)
Packing efficiency	0.674	0.643	0.632

Symmetry code: (i) x, -y + ½, z.

H atoms were placed geometrically and refined using a riding model, with C—H distances in the range 0.96–0.98 Å, N—H distances of either 0.90 [for (I) and (II)] or 0.89 Å [for (III)], and U<sub>iso</sub>(H) values of 1.5U<sub>eq</sub>(C,N) or 1.2U<sub>eq</sub>(C). The highest residual peak was 0.83 Å

from atom Sn1 in (I), 0.25 Å from C8 in (II) and 2.19 Å from Cl3 in (III). The ammonium end groups on each of the two cations have their three H atoms [labelled H1A/H1B/H1C (cat1) and H2A/H2B/H2C (cat2)] disordered over two sets of positions, each with a site-occupancy factor of 50%, related by the mirror plane on which the two cations are situated. Only the positions of the H atoms as they occur in the asymmetric unit are shown in the figures. The disorder is a result of the long, and by inference weak, N—H...Cl hydrogen bonds, which limits their directionality.

For all three compounds, data collection: APEX2 (Bruker, 2005); cell refinement: SAINT-Plus (Bruker, 2004); data reduction: SAINT-Plus and XPREP (Bruker, 2004); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997) and DIAMOND (Brandenburg, 1999); software used to prepare material for publication: WinGX (Farrugia, 1999) and PLATON (Spek, 2003).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM3022). Services for accessing these data are described at the back of the journal.

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