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Key indicators

Single-crystal X-ray study
 T = 120 K
 Mean $\sigma(\text{C}-\text{C}) = 0.007 \text{ \AA}$
 R factor = 0.038
 wR factor = 0.105
 Data-to-parameter ratio = 22.7

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

(2-Amidoethyl- $\kappa^2\text{C},\text{O}$)trichloro(3-chloropropionamide- κO)stannane

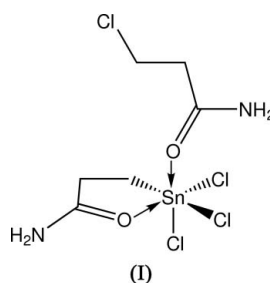
The Sn atom in the title compound, $[\text{Sn}(\text{C}_3\text{H}_6\text{NO})\text{Cl}_3(\text{C}_3\text{H}_6\text{ClNO})]$, exists within a *fac*- CCl_3O_2 donor set that defines an octahedral geometry and features a negatively charged chelating 2-amidoethyl ligand as well as a neutral 3-chloropropionamide ligand that coordinates exclusively *via* the carbonyl-O atom. Extensive $\text{N}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{Cl}$ hydrogen bonding leads to a layer structure.

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Comment

Functionally substituted organotin compounds, $X_3\text{SnCR}_2\text{CH}_2\text{COY}$ (1) and the less well studied $X_2\text{Sn}(\text{CR}_2\text{CH}_2\text{COY})_2$ (2), for $X = \text{halide}$, $R = \text{H}$ or alkyl, and $Y = \text{alkyl}$, aryl, alkoxy or NH_2 , are readily available from reactions of $R_2\text{C}=\text{CHCOY}$, HX and SnX_2 (generally for 1) or Sn (generally for 2) (Hutton & Oakes, 1976; Hutton *et al.*, 1978; Burley *et al.*, 1979). Original interest in these compounds was primarily involved with their industrial potential as precursors of PVC stabilizers, but much attention was also paid to their coordination chemistry (Milne *et al.*, 2005, and references therein). The title compound (I) was an unexpected product isolated from the reaction between Sn, $\text{H}_2\text{C}=\text{CHCONH}_2$ and HCl in diethyl ether solution.



The structure of (I) (Fig. 1 and Table 1) features an Sn atom within a distorted octahedral geometry defined by three Cl atoms, arranged facially, C and O of the chelating 2-amidoethyl ligand and carbonyl-O from 3-chloropropionamide. The 2-amidoethyl ligand in (I) coordinates in a similar fashion to that found in the only other structure of an amidotin compound, *viz.* $\text{Cl}_2\text{Sn}(\text{CH}_2\text{CH}_2\text{CONH}_2)_2$ (Harrison *et al.*, 1979; also see Marsh (1997) for space-group revision).

The crystal structure is stabilized by hydrogen-bonding interactions as summarized in Table 2. Adjacent molecules form inversion-related dimers with an eight-membered $\{\cdots\text{H}-\text{N}-\text{C}=\text{O}\}_2$ ring *via* $\text{N1}-\text{H1a}\cdots\text{O1}$ hydrogen bonds shown as '(a)' in Fig. 2. These pairs associate with adjacent pairs *via* $\text{N}-\text{H}\cdots\text{Cl}^{\text{iii}}$ interactions involving the second $\text{N1}-\text{H}$ amide H atom so as to form a double chain aligned along

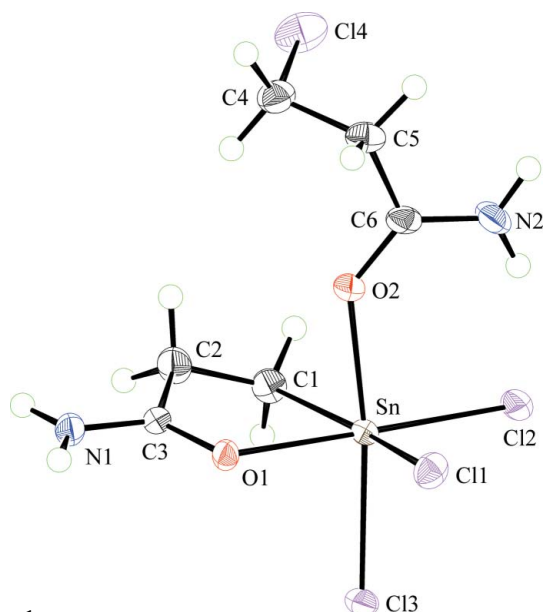


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

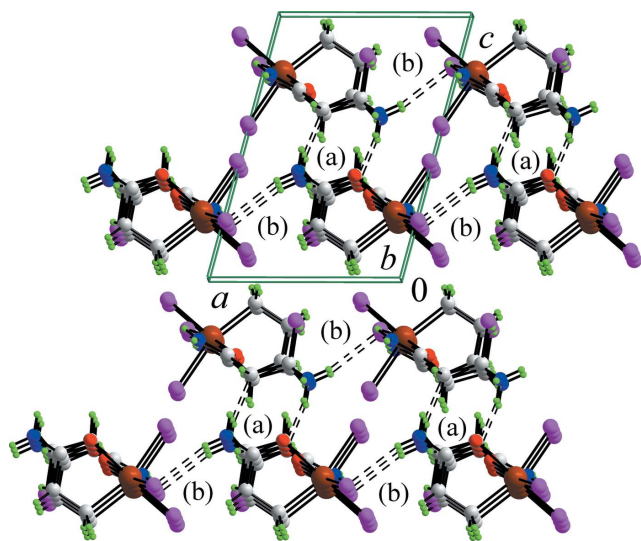


Figure 2
Packing diagram for (I), viewed down the *b* axis. Colour code: Sn (brown), Cl (pink), O (red), N (blue), C (grey) & H (green). Hydrogen bonds are shown as dashed lines.

the *a* axis, '(b)' in Fig. 2. N2—H1_a forms an intramolecular hydrogen bond to Cl2 and N2—H1_b forms an interaction with Cl3ⁱⁱⁱ so that this Cl atom forms two hydrogen bonds. As these latter interactions extend in the *b*-axis direction, a 2-dimensional supramolecular array is formed. Connections between layers are made primarily via C4—H4_a···Cl^{iv} interactions. It is the nature of the Cl···H interactions that readily accounts for the disparity in the Sn—Cl distances that span the range 2.3730 (11) to 2.4735 (10) Å. The Sn—Cl bond distances systematically elongate in accord with the number of such interactions so that Sn—Cl1, with the Cl1 atom forming only a weak C—H···Cl contact, is significantly shorter than the Sn—Cl2 bond, with the Cl2 atom forming a single N—H···Cl

contact, which in turn is significantly shorter than the Sn—Cl3 bond, with the Cl3 atom involved in two N—H···Cl contacts.

Experimental

The title compound (I) was isolated from a reaction between Sn, acrylonitrile and HCl in diethyl ether solution following a general procedure (Hutton & Oakes, 1976). HCl was bubbled through a well stirred suspension of granulated Sn (0.1 mol) and H₂C=CHCONH₂ (0.22 mol) in Et₂O (40 ml), maintained at 273–283 K until all the Sn had reacted. The reaction mixture was stirred for a further 2 h and all volatiles removed under vacuum. The thick oily liquid was extracted into CH₂Cl₂ and hexane (1:) added. On leaving the mixture at 268 K, a small amount of crystalline (I) was initially deposited, m. p. 524–528 K (decomposition).

Crystal data

[Sn(C₃H₅NO)Cl₃(C₃H₆ClNO)]
M_r = 404.67
 Triclinic, *P*1̄
a = 7.3582 (3) Å
b = 9.0387 (5) Å
c = 10.4342 (6) Å
 α = 92.005 (2)°
 β = 104.529 (3)°
 γ = 96.222 (3)°
V = 666.42 (6) Å³
Z = 2

D_x = 2.017 Mg m⁻³
 Mo *K*α radiation
 Cell parameters from 2904 reflections
 θ = 2.9–27.5°
 μ = 2.70 mm⁻¹
T = 120 (2) K
 Needle, colourless
 0.14 × 0.04 × 0.02 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 2003)
T_{min} = 0.704, *T_{max}* = 0.948
 13278 measured reflections

3086 independent reflections
 2721 reflections with *I* > 2σ(*I*)
R_{int} = 0.068
 θ_{max} = 27.8°
h = -9 → 8
k = -11 → 11
l = -13 → 13

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.038
wR (*F*²) = 0.105
S = 1.09
 3086 reflections
 136 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0474P)^2 + 1.4199P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 (Δ/*σ*)_{max} < 0.001
 Δρ_{max} = 1.11 e Å⁻³
 Δρ_{min} = -1.54 e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Sn—Cl1	2.3730 (11)	Cl4—C4	1.786 (5)
Sn—Cl2	2.4038 (11)	O1—C3	1.263 (5)
Sn—Cl3	2.4735 (10)	O2—C6	1.257 (5)
Sn—O1	2.239 (3)	N1—C3	1.313 (5)
Sn—O2	2.240 (3)	N2—C6	1.307 (6)
Sn—Cl	2.138 (4)		
Cl1—Sn—Cl2	93.44 (4)	Cl3—Sn—O1	87.47 (8)
Cl1—Sn—Cl3	90.04 (4)	Cl3—Sn—O2	168.47 (9)
Cl1—Sn—O1	84.52 (8)	Cl3—Sn—C1	96.48 (13)
Cl1—Sn—O2	83.93 (9)	O1—Sn—O2	82.18 (11)
Cl1—Sn—C1	162.38 (13)	O1—Sn—C1	79.47 (14)
Cl2—Sn—Cl3	93.97 (4)	O2—Sn—C1	86.66 (16)
Cl2—Sn—O1	177.51 (8)	Sn—O1—C3	112.5 (3)
Cl2—Sn—O2	96.20 (8)	Sn—O2—C6	135.8 (3)
Cl2—Sn—C1	102.37 (12)		

Table 2
Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1a \cdots O1 ⁱ	0.88	2.06	2.924 (5)	167
N1—H1b \cdots Cl3 ⁱⁱ	0.88	2.47	3.326 (4)	166
N2—H2a \cdots Cl2	0.88	2.40	3.227 (4)	156
N2—H2b \cdots Cl3 ⁱⁱⁱ	0.88	2.40	3.250 (4)	163
C4—H4a \cdots Cl1 ^{iv}	0.99	2.79	3.742 (5)	163
C4—H4b \cdots O2	0.99	2.59	2.923 (6)	100

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

All H atoms were allowed to ride on their parent atoms in the riding-model approximation at distances of 0.99 (C—H) and 0.88 Å (N—H), and with $U_{\text{iso}}(\text{H})$ values of $1.2U_{\text{eq}}(\text{C}, \text{N})$. The maximum residual electron density peak was located 1.26 Å from the Sn atom and the deepest hole was located 0.77 Å also from the Sn atom.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* and *COLLECT* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS97*; program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976) and *DIAMOND* (Crystal Impact, 2006); software used to prepare material for publication: *SHELXL97*.

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