(2-Amidoethyl-κ^2C,O)trichloro(3-chloropropionamide-κO)stannane

Edward R. T. Tiekink,[a]* James L. Wardell[b,c] and Solange M. S. V. Wardell[d]*

[a]Department of Chemistry, The University of Texas at San Antonio, 6900 North Loop 1604 West, San Antonio, Texas 78249-0698, USA, [b]Department of Chemistry, University of Aberdeen, Old Aberdeen, AB24 3UE, Scotland, [c]Instituto de Química, Universidade Federal do Rio de Janeiro, 21945-970 Rio de Janeiro, RJ, Brazil, and [d]Complexo Tecnológico de Medicamentos Farmanguinhos, Av. Comandante Guaranys 447, Jacarepaguá - Rio de Janeiro, RJ, Brazil

Correspondence e-mail:
Edward.Tiekink@utsa.edu,
solangewardell@yahoo.co.uk

Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.007 Å
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.

The Sn atom in the title compound, [Sn(C_3H_6NO)Cl_3-(C_3H_6ClNO)], 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 N—H···O and N—H···Cl hydrogen bonding leads to a layer structure.

Comment

Functionally substituted organotin compounds, X_3SnCR_2-CH_2COY (1) and the less well studied X_2Sn(CR_2CH$_2$COY)$_2$ (2), for X = halide, R = H or alkyl, and Y = alkyl, aryl, alkoxy or NH$_2$, are readily available from reactions of R_2C=CH-COY, 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, H$_2$C=CHCONH$_2$ and HCl in diethyl ether solution.

(I)
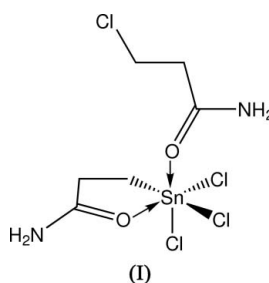
The structure of (I) (Fig. 1 and Table 1) features an Sn atom within a disorted octahedral geometry defined by three Cl atoms, arranged facially, C and O of the chelating 2-amidoethyl ligand and carbonyl-O from 3-chloropropionamide. The 2-amidoethyl ligand in (I) coordinates in a similar fashion to that found in the only other structure of an amidotin compound, *viz.* Cl$_2$Sn(CH$_2$CH$_2$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 {···H—N—C=O}$_2$ ring *via* N1—H1a···O1 hydrogen bonds shown as '(a)' in Fig. 2. These pairs associate with adjacent pairs *via* N—H···Cl3[ii] interactions involving the second N1—H amide H atom so as to form a double chain aligned along
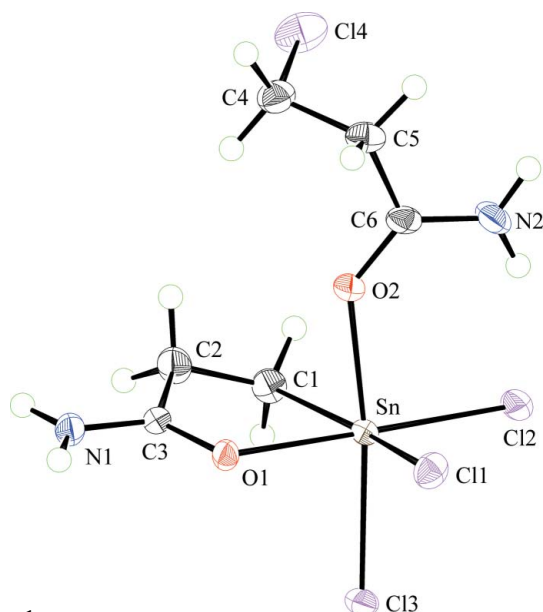
Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.
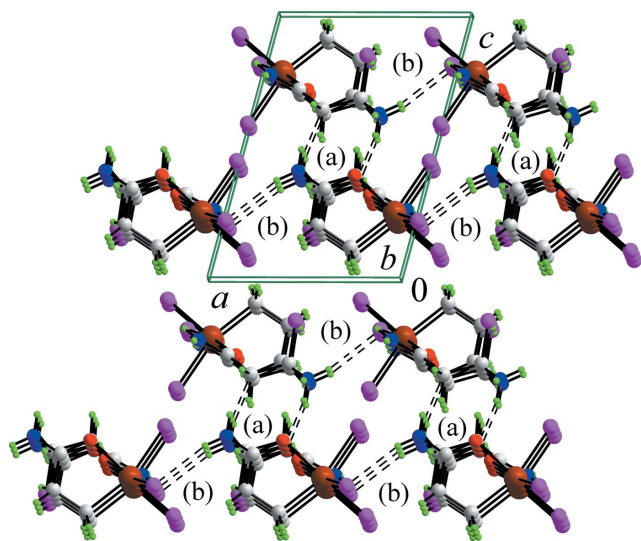

Figure 2
Packing diagram for (I), viewed down the *b* axis. Colour code: Sn (brown), Cl (pink), O (red), N (blue), C (grey) & H (green). Hydrogen bonds are shown as dashed lines.

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 Cl3[iii] 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 $H_2C=CHCONH_2$ (0.22 mol) in Et_2O (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 CH_2Cl_2 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

$[Sn(C_3H_6NO)Cl_3(C_3H_6ClNO)]$	$D_x = 2.017$ Mg m^{-3}
$M_r = 404.67$	Mo $K\alpha$ radiation
Triclinic, $P\bar{1}$	Cell parameters from 2904
$a = 7.3582$ (3) Å	reflections
$b = 9.0387$ (5) Å	$\theta = 2.9$–$27.5°$
$c = 10.4342$ (6) Å	$\mu = 2.70$ mm^{-1}
$\alpha = 92.005$ (2)°	$T = 120$ (2) K
$\beta = 104.529$ (3)°	Needle, colourless
$\gamma = 96.222$ (3)°	$0.14 \times 0.04 \times 0.02$ mm
$V = 666.42$ (6) Å^3	
$Z = 2$	

Data collection

Bruker–Nonius KappaCCD diffractometer	3086 independent reflections
	2721 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.068$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)	$\theta_{max} = 27.8°$
	$h = -9 \rightarrow 8$
$T_{min} = 0.704$, $T_{max} = 0.948$	$k = -11 \rightarrow 11$
13278 measured reflections	$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0474P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.038$	$+ 1.4199P]$
$wR(F^2) = 0.105$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.09$	$(\Delta/\sigma)_{max} < 0.001$
3086 reflections	$\Delta\rho_{max} = 1.11$ e Å^{-3}
136 parameters	$\Delta\rho_{min} = -1.54$ e Å^{-3}
H-atom parameters constrained	

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—C1	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_{iso}(H) values of $1.2U_{eq}$(C,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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