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

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Key indicators

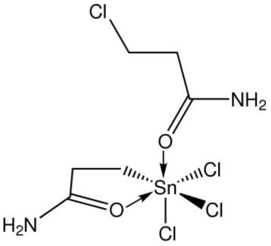
Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.007 Å
R factor = 0.038
wR factor = 0.105
Data-to-parameter ratio = 22.7

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, $X_3SnCR_2CH_2COY$ (1) and the less well studied $X_2Sn(CR_2CH_2COY)_2$ (2), for X = halide, R = H or alkyl, and Y = alkyl, aryl, alkoxy or NH_2, are readily available from reactions of $R_2C{=}CHCOY$, 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, $H_2C{=}CHCONH_2$ 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.* $Cl_2Sn(CH_2CH_2CONH_2)_2$ (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 $\{\cdots H{-}N{-}C{=}O\}_2$ ring *via* N1—H1*a*···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

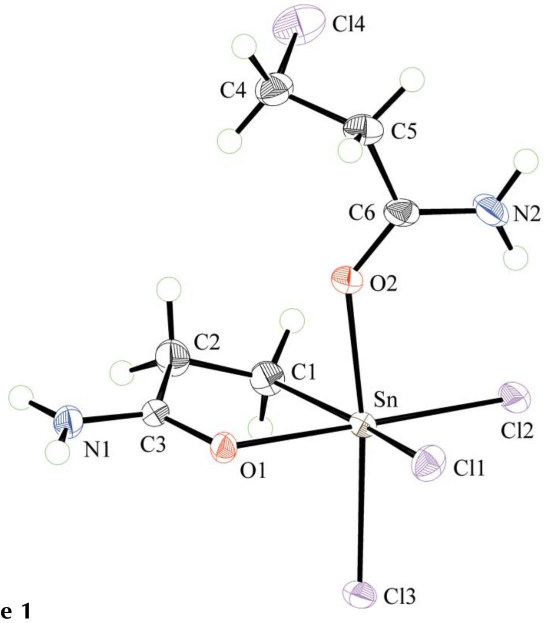


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

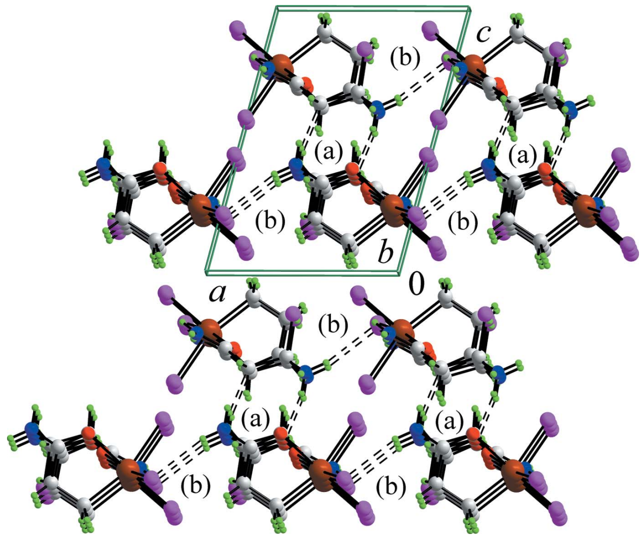


Figure 2
Packing diagram for (I), viewed down the *b* axis. Colour code: Sn (brown), Cl (pink), O (red), N (blue), C (grey) & H (green). Hydrogen bonds are shown as dashed lines.

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

$[Sn(C_3H_6NO)Cl_3(C_3H_6ClNO)]$
$M_r = 404.67$
Triclinic, $P\bar{1}$
$a = 7.3582$ (3) Å
$b = 9.0387$ (5) Å
$c = 10.4342$ (6) Å
$\alpha = 92.005$ (2)°
$\beta = 104.529$ (3)°
$\gamma = 96.222$ (3)°
$V = 666.42$ (6) Å^3
$Z = 2$
$D_x = 2.017$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2904 reflections
$\theta = 2.9$–27.5°
$\mu = 2.70$ 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 $I > 2\sigma(I)$
$R_{int} = 0.068$
$\theta_{max} = 27.8°$
$h = -9 \rightarrow 8$
$k = -11 \rightarrow 11$
$l = -13 \rightarrow 13$

Refinement

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

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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 $U_{\rm iso}$(H) values of 1.2$U_{\rm eq}$(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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supporting information

Acta Cryst. (2006). E**62**, m971–m973 [https://doi.org/10.1107/S1600536806010543]

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(I)

Crystal data

[Sn(C_3H_6ClNO)(C_3H_6NO)Cl_3]
$M_r = 404.67$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.3582$ (3) Å
$b = 9.0387$ (5) Å
$c = 10.4342$ (6) Å
$\alpha = 92.005$ (2)°
$\beta = 104.529$ (3)°
$\gamma = 96.222$ (3)°
$V = 666.42$ (6) Å^3

$Z = 2$
$F(000) = 392$
$D_x = 2.017$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2904 reflections
$\theta = 2.9$–$27.5°$
$\mu = 2.70$ mm^{-1}
$T = 120$ K
Plate, colourless
0.14 × 0.04 × 0.02 mm

Data collection

Bruker-Nonius 95mm CCD camera on a κ-goniostat diffractometer
Radiation source: Bruker-Nonius FR591 rotating anode
10cm confocal mirrors monochromator
Detector resolution: 9.091 pixels mm^{-1}
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 $I > 2\sigma(I)$
$R_{\text{int}} = 0.068$
$\theta_{\max} = 27.8°$, $\theta_{\min} = 2.9°$
$h = -9 \rightarrow 8$
$k = -11 \rightarrow 11$
$l = -13 \rightarrow 13$

Refinement

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

Special details

Experimental. IR (CsI, cm^{-1}): *ν* 3396, 3254, 1652, 1576, 1409, 1295, 1166, 970. 740, 666, 616, 584, 480, 367, 314.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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 $F^2 > \sigma(F^2)$ 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	$U_{\rm iso}$*/$U_{\rm eq}$
Sn	0.10766 (4)	0.75079 (3)	0.23109 (3)	0.01770 (12)
Cl1	−0.00142 (15)	0.72255 (13)	0.42547 (11)	0.0257 (2)
Cl2	−0.16772 (15)	0.60455 (12)	0.09151 (12)	0.0266 (3)
Cl3	−0.02963 (15)	0.98881 (11)	0.19741 (11)	0.0243 (2)
Cl4	0.5294 (2)	0.26714 (17)	0.17569 (13)	0.0413 (3)
O1	0.3632 (4)	0.8805 (3)	0.3682 (3)	0.0197 (6)
O2	0.2650 (4)	0.5578 (3)	0.3009 (3)	0.0251 (7)
N1	0.6626 (5)	0.9740 (4)	0.3833 (4)	0.0227 (8)
H1A	0.6735	1.0141	0.4633	0.027*
H1B	0.7590	0.9852	0.3474	0.027*
N2	0.0551 (6)	0.3494 (4)	0.2481 (4)	0.0297 (9)
H2A	−0.0356	0.3990	0.2056	0.036*
H2B	0.0331	0.2524	0.2532	0.036*
C1	0.2878 (6)	0.7847 (5)	0.1001 (4)	0.0234 (9)
H1C	0.2785	0.6926	0.0434	0.028*
H1D	0.2486	0.8659	0.0418	0.028*
C2	0.4886 (7)	0.8253 (6)	0.1821 (5)	0.0281 (10)
H2C	0.5566	0.8945	0.1336	0.034*
H2D	0.5525	0.7339	0.1931	0.034*
C3	0.5025 (6)	0.8973 (5)	0.3175 (4)	0.0173 (8)
C4	0.5541 (7)	0.3544 (6)	0.3364 (5)	0.0283 (10)
H4A	0.6551	0.3132	0.4018	0.034*
H4B	0.5918	0.4628	0.3353	0.034*
C5	0.3718 (7)	0.3298 (5)	0.3779 (5)	0.0265 (10)
H5A	0.3978	0.3566	0.4741	0.032*
H5B	0.3224	0.2226	0.3627	0.032*
C6	0.2227 (6)	0.4197 (5)	0.3038 (4)	0.0226 (9)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sn	0.01735 (18)	0.01386 (17)	0.02147 (19)	0.00121 (11)	0.00494 (12)	−0.00209 (11)
Cl1	0.0240 (5)	0.0293 (6)	0.0262 (6)	0.0040 (4)	0.0102 (4)	0.0052 (4)
Cl2	0.0222 (5)	0.0192 (5)	0.0339 (6)	0.0013 (4)	0.0003 (4)	−0.0066 (4)
Cl3	0.0262 (5)	0.0149 (5)	0.0345 (6)	0.0035 (4)	0.0125 (4)	0.0021 (4)

Cl4	0.0492 (8)	0.0498 (8)	0.0325 (7)	0.0197 (6)	0.0182 (6)	0.0047 (6)
O1	0.0161 (14)	0.0207 (15)	0.0216 (15)	−0.0011 (11)	0.0061 (12)	−0.0067 (12)
O2	0.0208 (15)	0.0153 (15)	0.0381 (19)	0.0038 (12)	0.0046 (13)	0.0029 (13)
N1	0.0200 (18)	0.0232 (19)	0.025 (2)	−0.0005 (14)	0.0090 (15)	−0.0054 (15)
N2	0.030 (2)	0.0147 (18)	0.039 (2)	0.0007 (15)	0.0000 (18)	0.0020 (16)
C1	0.027 (2)	0.026 (2)	0.020 (2)	0.0012 (18)	0.0121 (18)	−0.0029 (17)
C2	0.028 (2)	0.028 (2)	0.028 (3)	−0.0013 (19)	0.012 (2)	−0.009 (2)
C3	0.0157 (19)	0.0165 (19)	0.020 (2)	0.0027 (15)	0.0052 (16)	0.0006 (15)
C4	0.026 (2)	0.027 (2)	0.031 (3)	0.0083 (18)	0.004 (2)	0.0024 (19)
C5	0.033 (3)	0.021 (2)	0.027 (2)	0.0085 (19)	0.009 (2)	0.0050 (18)
C6	0.029 (2)	0.018 (2)	0.026 (2)	0.0051 (17)	0.0173 (19)	0.0007 (17)

Geometric parameters (Å, º)

Sn—Cl1	2.3730 (11)	N2—H2B	0.8800
Sn—Cl2	2.4038 (11)	C1—C2	1.509 (6)
Sn—Cl3	2.4735 (10)	C1—H1C	0.9900
Sn—O1	2.239 (3)	C1—H1D	0.9900
Sn—O2	2.240 (3)	C2—C3	1.510 (6)
Sn—C1	2.138 (4)	C2—H2C	0.9900
Cl4—C4	1.786 (5)	C2—H2D	0.9900
O1—C3	1.263 (5)	C4—C5	1.506 (7)
O2—C6	1.257 (5)	C4—H4A	0.9900
N1—C3	1.313 (5)	C4—H4B	0.9900
N1—H1A	0.8800	C5—C6	1.508 (6)
N1—H1B	0.8800	C5—H5A	0.9900
N2—C6	1.307 (6)	C5—H5B	0.9900
N2—H2A	0.8800		
Cl1—Sn—Cl2	93.44 (4)	Sn—C1—H1D	110.0
Cl1—Sn—Cl3	90.04 (4)	H1C—C1—H1D	108.4
Cl1—Sn—O1	84.52 (8)	C1—C2—C3	113.5 (4)
Cl1—Sn—O2	83.93 (9)	C1—C2—H2C	108.9
Cl1—Sn—C1	162.38 (13)	C3—C2—H2C	108.9
Cl2—Sn—Cl3	93.97 (4)	C1—C2—H2D	108.9
Cl2—Sn—O1	177.51 (8)	C3—C2—H2D	108.9
Cl2—Sn—O2	96.20 (8)	H2C—C2—H2D	107.7
Cl2—Sn—C1	102.37 (12)	O1—C3—N1	120.5 (4)
Cl3—Sn—O1	87.47 (8)	O1—C3—C2	120.3 (4)
Cl3—Sn—O2	168.47 (9)	N1—C3—C2	119.2 (4)
Cl3—Sn—C1	96.48 (13)	C5—C4—Cl4	111.2 (3)
O1—Sn—O2	82.18 (11)	C5—C4—H4A	109.4
O1—Sn—C1	79.47 (14)	Cl4—C4—H4A	109.4
O2—Sn—C1	86.66 (16)	C5—C4—H4B	109.4
Sn—O1—C3	112.5 (3)	Cl4—C4—H4B	109.4
Sn—O2—C6	135.8 (3)	H4A—C4—H4B	108.0
C3—N1—H1A	120.0	C4—C5—C6	113.0 (4)
C3—N1—H1B	120.0	C4—C5—H5A	109.0

H1A—N1—H1B	120.0	C6—C5—H5A	109.0
C6—N2—H2A	120.0	C4—C5—H5B	109.0
C6—N2—H2B	120.0	C6—C5—H5B	109.0
H2A—N2—H2B	120.0	H5A—C5—H5B	107.8
C2—C1—Sn	108.6 (3)	O2—C6—N2	123.7 (4)
C2—C1—H1C	110.0	O2—C6—C5	118.5 (4)
Sn—C1—H1C	110.0	N2—C6—C5	117.8 (4)
C2—C1—H1D	110.0		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1a···O1^{i}	0.88	2.06	2.924 (5)	167
N1—H1b···Cl3^{ii}	0.88	2.47	3.326 (4)	166
N2—H2a···Cl2	0.88	2.40	3.227 (4)	156
N2—H2b···Cl3^{iii}	0.88	2.40	3.250 (4)	163
C4—H4a···Cl1^{iv}	0.99	2.79	3.742 (5)	163
C4—H4b···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.