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[*N*-(2-Aminocyclohexyl)salicylaldiminato-κ^3O,N,N']-dichlorophenyltin(IV) chloroform solvate

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Key indicators

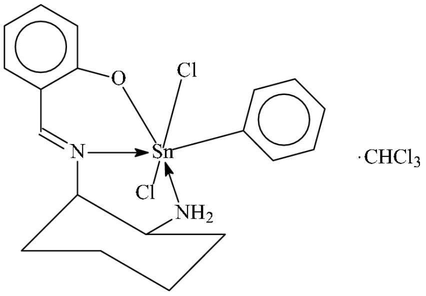
Single-crystal X-ray study
T = 223 K
Mean σ(C–C) = 0.007 Å
R factor = 0.041
wR factor = 0.114
Data-to-parameter ratio = 21.4

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

In the title compound, $[SnCl_2(C_{19}H_{22}N_2O)]\cdot CHCl_3$, the deprotonated *N*-(2-aminocyclohexyl)salicylaldimine Schiff base chelates in an *O*,*N*,*N*′-tridentate manner to the Sn atom. The octahedral tin coordination environment is completed by a phenyl ring and two chloride ions; the bonded C atom of the phenyl ring along with the three chelating atoms comprise an approximate square plane. The molecules of the complex are linked by N—H⋯O and N—H⋯Cl hydrogen bonds to form a helical chain running along the *b* axis; the solvent molecules occupy the space between the chains but are not hydrogen bonded to them.

Comment

Deprotonated Schiff bases that are derived from the condensation of salicylaldehyde (and its analogs) with primary amines furnish an enormous number of complexes with metal ions; among these are a number of organotin salicylaldiminates such as, for example, the dimethyltin (Calligaris *et al.*, 1972; Dey *et al.*, 1999; Evans & Penfold, 1975; Yeap *et al.*, 1992), divinyltin (Pettinari *et al.*, 2001), dibutyltin (Smith *et al.*, 1992), methoxycarbonylethyltin (Tian, Zhao *et al.*, 1998; Tian, Zhou *et al.*, 1998; Tian *et al.*, 1999) and diphenyltin (Diamantis *et al.*, 1999; Preut *et al.*, 1974, 1976; Wang *et al.*, 1993, 1996) derivatives. In the present study, the attempt to synthesize an adduct of the neutral double-Schiff base *N*,*N*′-cyclohexane-1,2-diylsalicylaldimine (Cannadine *et al.*, 1996) with diphenyltin dichloride gave, instead, a Schiff base complex with one fewer phenyl ring. The organic reagent also underwent cleavage of one of the imine bonds; the resulting compound crystallizes as a chloroform solvate, (I) (Fig. 1). Sn—C bond cleavage, though unusual in a reaction carried out under mild conditions, has been previously noted (Ng & Kumar Das, 1993). The metal atom in (I) exists in an approximately octahedral environment, with the two chloride ions in a *trans* configuration.



(I)

Compound (I) features an unusual primary amino–tin linkage [Sn—N = 2.243 (3) Å]. Organotin compounds having a

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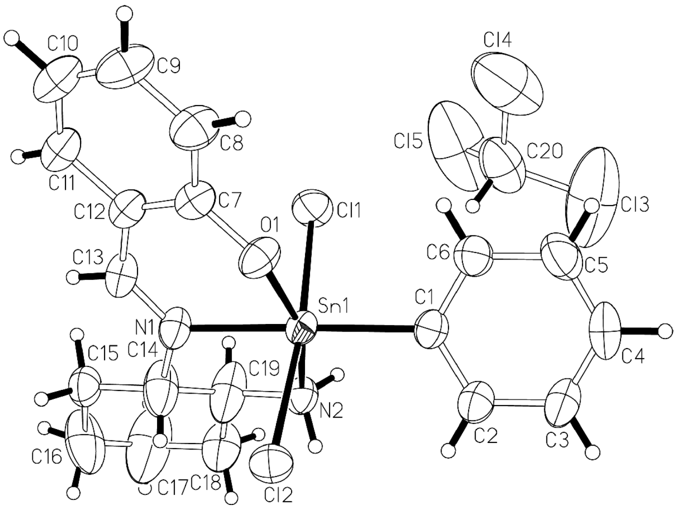

Figure 1
View of (I), with displacement ellipsoids drawn at the 50% probability level and H atoms drawn as spheres of arbitrary radii.

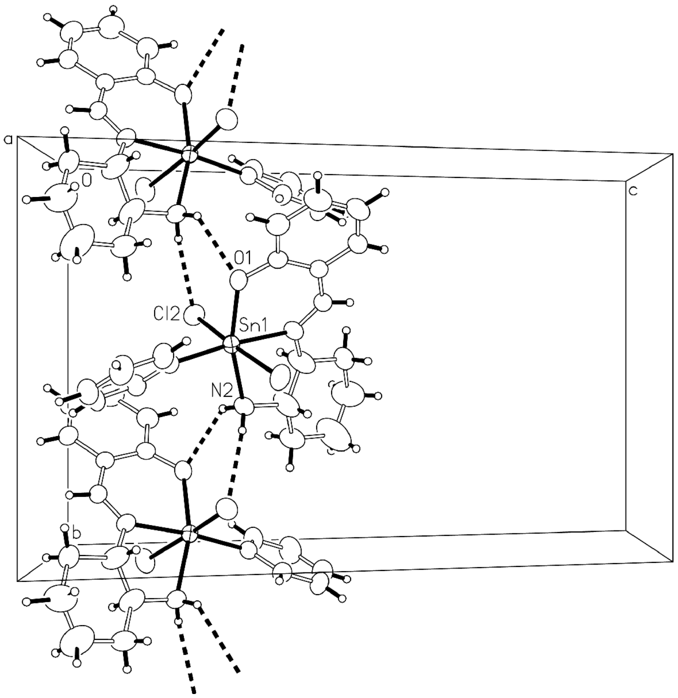

Figure 2
Detail of (I), showing how the hydrogen bonds (dashed lines) result in a chain.

similar Sn—N bond are rare, as amines tend to promote the hydrolysis of the starting organotin halide reagent. Other than the cysteine derivative (Domazetis *et al.*, 1979; Domazetis & Mackay, 1979), the only structurally authenticated examples are a dichlorobutyltin complex of a substituted salicylaldimine (Yearwood *et al.*, 2002), the chlorodimethyltin complex of a hydrazine (Baul *et al.*, 1996), as well as two 8-aminoquinoline adducts of diorganotin dichlorides (Bengston *et al.*, 1996; Hazell *et al.*, 1997). The Sn—N bond length in the title complex is similar to those found in these two adducts.

Experimental

N,*N*′-Cyclohexane-1,2-diylsalicylaldimine (Cannadine *et al.*, 1996) was prepared by condensing salicylaldehyde with 1,2-diaminocyclohexane in a 2:1 molar ratio. The compound (0.21 g, 0.6 mmol) and diphenyltin dichloride (0.21 g, 0.6 mol) were dissolved separately in chloroform and the two solutions were mixed. The solvent was evaporated to give a white compound, which was purified by recrystallization from chloroform to give colorless crystals of (I) that melt at 564–567 K. The presence of the amino substituent in the compound was proven by the IR peak at 3199 cm^{-1}, while the presence of chloroform was shown by the peak at 7.24 p.p.m. in the solution ^{1}H NMR spectrum.

Crystal data

$[SnCl_2(C_{19}H_{22}N_2O)]\cdot CHCl_3$
$M_r = 603.34$
Monoclinic, $P2_1/n$
$a = 11.877$ (1) Å
$b = 11.559$ (1) Å
$c = 18.766$ (2) Å
$\beta = 108.449$ (2)°
$V = 2443.9$ (4) Å^3
$Z = 4$

$D_x = 1.640$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 967 reflections
$\theta = 2.4$–28.3°
$\mu = 1.61$ mm^{-1}
$T = 223$ (2) K
Block, colorless
0.40 × 0.36 × 0.24 mm

Data collection

Bruker SMART CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{min} = 0.545$, $T_{max} = 0.678$
16789 measured reflections

5614 independent reflections
4850 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$
$\theta_{max} = 27.5°$
$h = -15 \rightarrow 15$
$k = -14 \rightarrow 15$
$l = -24 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.114$
$S = 1.01$
5614 reflections
262 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0648P)^2 + 3.2538P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.31$ e Å^{-3}
$\Delta\rho_{min} = -0.72$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Sn1—Cl1	2.474 (1)	Sn1—C1	2.129 (4)
Sn1—Cl2	2.479 (1)	Sn1—N1	2.188 (3)
Sn1—O1	2.048 (2)	Sn1—N2	2.243 (3)
Cl1—Sn1—Cl2	171.21 (3)	Cl2—Sn1—C1	95.1 (1)
Cl1—Sn1—O1	91.9 (1)	O1—Sn1—N1	85.1 (1)
Cl1—Sn1—C1	92.5 (1)	O1—Sn1—N2	161.4 (1)
Cl1—Sn1—N1	85.3 (1)	O1—Sn1—C1	99.7 (1)
Cl1—Sn1—N2	87.0 (1)	N1—Sn1—N2	76.3 (1)
Cl2—Sn1—O1	91.2 (1)	N1—Sn1—C1	174.8 (1)
Cl2—Sn1—N1	86.8 (1)	N2—Sn1—C1	99.0 (1)
Cl2—Sn1—N2	87.4 (1)		

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N2—H2n1$\cdots$Cl2^{i}	0.91	2.50	3.372 (3)	162
N2—H2n2$\cdots$O1^{i}	0.91	2.39	3.065 (4)	131

Symmetry code: (i) $\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$.

H atoms were placed at calculated positions, the C—H distance being 0.94 Å for the aromatic C atoms, 0.98 Å for the methylene C atoms and 0.99 Å for the methine C atoms, and were included in the refinement in the riding-model approximation, with the constraint $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}$(carrier atom) in all cases. The amino H atoms were similarly constrained [N—H = 0.91 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm N})$]. The largest peak in the final difference Fourier map was 0.88 Å from atom C14.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

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