Absorption correction:
  refined from ΔF
  (*DIFABS*; Walker &
  Stuart, 1983)
  $T_{min} = 0.974$, $T_{max} = 1.000$
1723 measured reflections
1596 independent reflections

$h = 0 \rightarrow 9$
$k = -11 \rightarrow 11$
$l = -7 \rightarrow 7$
3 standard reflections
  monitored every 400
  reflections
  intensity decay: 3%

Refinement

Refinement on F
$R = 0.0339$
$wR = 0.0283$
$S = 1.791$
1596 reflections
121 parameters
H-atom parameters not
  refined

Weighting scheme based
  on measured e.s.d.'s;
  $w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{max} = 0.0015$
$\Delta\rho_{max} = 0.41$ e Å^{-3}
$\Delta\rho_{min} = -0.29$ e Å^{-3}
Atomic scattering factors
  from *International Tables
  for X-ray Crystallography*
  (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters ($\mathring{A}^2$)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Co	0	0	0	0.01702 (9)
Na	0	1/2	1/2	0.0261 (3)
O(1)	−0.0076 (2)	0.1272 (1)	−0.2189 (2)	0.0216 (3)
O(2)	0.1741 (2)	0.3488 (2)	−0.2415 (3)	0.0356 (4)
O(3)	−0.0965 (2)	0.2744 (2)	0.1629 (3)	0.0335 (4)
O(4)	−0.2798 (3)	0.0656 (2)	0.1866 (3)	0.0377 (5)
N(1)	0.2578 (2)	0.1464 (2)	0.2184 (3)	0.0188 (4)
N(4)	0.6354 (3)	0.3716 (2)	0.4624 (3)	0.0265 (4)
N(5)	−0.1442 (3)	0.1279 (2)	0.1347 (3)	0.0235 (4)
C(2)	0.3133 (3)	0.2565 (2)	0.1140 (3)	0.0203 (5)
C(3)	0.5022 (3)	0.3670 (2)	0.2374 (4)	0.0251 (5)
C(5)	0.5744 (3)	0.2656 (2)	0.5667 (4)	0.0262 (5)
C(6)	0.3851 (3)	0.1509 (2)	0.4467 (3)	0.0229 (5)
C(7)	0.1504 (3)	0.2469 (2)	−0.1361 (3)	0.0221 (5)

Table 2. *Selected geometric parameters ($\mathring{A}$, °)*

Co—O(1)	1.889 (1)	N(1)—C(2)	1.344 (2)
Co—N(1)	1.899 (2)	N(1)—C(6)	1.335 (2)
Co—N(5)	1.954 (2)	N(4)—C(3)	1.331 (3)
O(1)—C(7)	1.283 (2)	N(4)—C(5)	1.332 (3)
O(2)—C(7)	1.225 (2)	C(2)—C(3)	1.376 (3)
O(3)—N(5)	1.251 (2)	C(2)—C(7)	1.507 (3)
O(4)—N(5)	1.220 (2)	C(5)—C(6)	1.394 (3)
O(1)—Co—N(1)	85.85 (6)	O(3)—N(5)—O(4)	120.9 (2)
O(1)—Co—N(5)	89.77 (6)	N(1)—C(2)—C(3)	120.2 (2)
N(1)—Co—N(5)	89.84 (7)	N(1)—C(2)—C(7)	113.9 (2)
Co—O(1)—C(7)	114.3 (1)	C(3)—C(2)—C(7)	125.8 (2)
Co—N(1)—C(2)	111.5 (1)	N(4)—C(3)—C(2)	122.4 (2)
Co—N(1)—C(6)	129.7 (1)	N(4)—C(5)—C(6)	122.7 (2)
C(2)—N(1)—C(6)	118.8 (2)	N(1)—C(6)—C(5)	119.3 (2)
C(3)—N(4)—C(5)	116.5 (2)	O(1)—C(7)—O(2)	125.6 (2)
Co—N(5)—O(3)	118.6 (1)	O(1)—C(7)—C(2)	114.1 (2)
Co—N(5)—O(4)	120.6 (1)	O(2)—C(7)—C(2)	120.3 (2)

The values of T obtained from *DIFABS* (Walker & Stuart, 1983) have been normalized by multiplying them by $1/T(m)$, where $T(m)$ was the maximum value of T obtained by the program. It is recognized that the range of T values thus obtained may not be as great as might be expected from crystals of this shape.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduc-

tion: *TEXSAN* (Molecular Structure Corporation, 1992). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

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Benzyltrichloro(1,10-phenanthroline-N,N')-tin(IV) Benzene Solvate (1/1)

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Abstract

The octahedral Sn atom in the title complex, $[SnCl_3(C_7H_7)(C_{12}H_8N_2)].C_6H_6$, is coordinated by one C, three Cl and two N atoms such that the two N atoms occupy positions *trans* to two of the Cl atoms, leaving one Cl atom *trans* to the C atom. This has the result that there are two classes of Sn—Cl interaction.

Comment

The Sn atom in [BzSnCl$_3$(phen)], (I), where Bz is benzyl and phen is 1,10-phenanthroline, exists in a distorted octahedral geometry defined by a C atom of the benzyl group, three Cl atoms and the two N atoms of a chelating 1,10-phenanthroline ligand. The greatest

distortion from ideal geometry is found in the N(1)—Sn—N(2) angle of 72.7 (1)°, reflecting the restricted bite angle of the chelate. The Cl atoms *trans* to the N atoms form longer Sn—Cl bonds [Sn—Cl(1) 2.428 (1) and Sn—Cl(2) 2.430 (1) Å] than the Cl atom *trans* to the C atom [Sn—Cl(3) 2.400 (1) Å]. The phenyl portion of the benzyl substituent is orientated so as to lie over the phenanthroline ligand; the dihedral angle between the two planes is 155.4 (2)°.

(I)
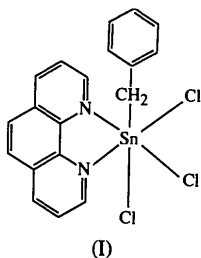
In the lattice, there is a benzene solvent molecule of crystallization which does not interact significantly with the adduct. The closest intermolecular contact in the lattice occurs between the C(13) and C(15^i) atoms at a distance of 3.503 (5) Å [symmetry code: (i) $-x$, $-y$, $1-z$].

The crystal structure of the related compound [^{n}BuSnCl$_3$\{C$_5$H$_4$NC(H)=NC$_6$H$_5$-p-OCH$_3$\}] has been determined (Teoh, Teo, Lee, Chong & Tiekink, 1995). In this structure, however, one N-donor atom is *trans* to the organic substituent, leading to disparate Sn—N bond distances in contrast to the present report.
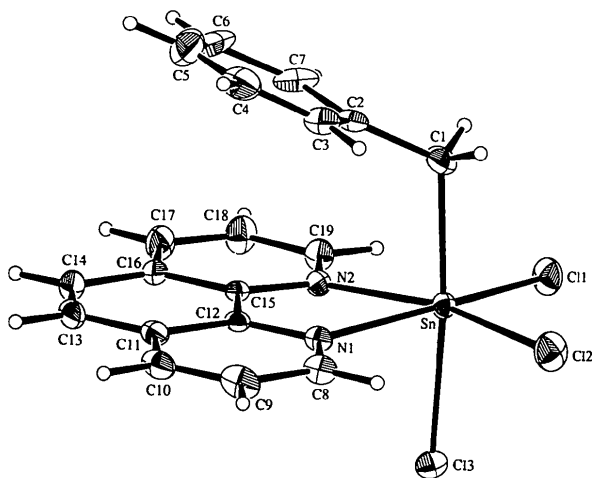

Fig. 1. The molecular structure of [BzSnCl$_3$(phen)] showing 15% probability ellipsoids (*ORTEP*II; Johnson, 1976).

Experimental

Crystals of [BzSnCl$_3$(phen)].C$_6$H$_6$ were isolated unexpectedly from the 1:1 reaction of [Bz$_2$SnCl$_2$] with 1,10-phenanthroline

in an acetonitrile/ethanol (9/1 *v/v*) solution at 333 K (m.p. 498–500 K).

Crystal data

[SnCl$_3$(C$_7$H$_7$)(C$_{12}$H$_8$N$_2$)].- C$_6$H$_6$	Mo $K\alpha$ radiation $\lambda = 0.7107$ Å
$M_r = 574.50$	Cell parameters from 25
Monoclinic	reflections
$P2_1/c$	$\theta = 23.0–24.5°$
$a = 10.941$ (3) Å	$\mu = 1.386$ mm^{-1}
$b = 17.658$ (5) Å	$T = 293$ K
$c = 13.516$ (2) Å	Block
$\beta = 110.40$ (2)°	0.47 × 0.32 × 0.15 mm
$V = 2447$ (1) Å^3	Yellow
$Z = 4$	
$D_x = 1.559$ Mg m^{-3}	

Data collection

Rigaku AFC-6R diffractometer	3817 observed reflections [$I \geq 2.0\sigma(I)$]
$\omega/2\theta$ scans	$R_{int} = 0.066$
Absorption correction:	$\theta_{max} = 27.5°$
refined from ΔF (Walker	$h = 0 \rightarrow 14$
& Stuart, 1983)	$k = 0 \rightarrow 22$
$T_{min} = 0.959$, $T_{max} =$	$l = -17 \rightarrow 16$
1.000	3 standard reflections
6103 measured reflections	monitored every 400
5811 independent reflections	reflections
	intensity decay: 0.17%

Refinement

Refinement on F	Weighting scheme based
$R = 0.040$	on measured e.s.d.'s;
$wR = 0.034$	$w = 1/\sigma^2(F)$
$S = 1.753$	$(\Delta/\sigma)_{max} = 0.0546$
3817 reflections	$\Delta\rho_{max} = 0.36$ e Å^{-3}
280 parameters	$\Delta\rho_{min} = -0.59$ e Å^{-3}
H-atom parameters not	Atomic scattering factors
refined	from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Sn	−0.25868 (3)	0.05760 (2)	0.72527 (2)	0.04320 (8)
Cl(1)	−0.2085 (1)	0.01174 (9)	0.90446 (9)	0.0736 (4)
Cl(2)	−0.4950 (1)	0.06600 (9)	0.6668 (1)	0.0847 (5)
Cl(3)	−0.2797 (1)	−0.07049 (7)	0.6621 (1)	0.0611 (4)
N(1)	−0.2519 (3)	0.0853 (2)	0.5624 (3)	0.045 (1)
N(2)	−0.0465 (3)	0.0472 (2)	0.7357 (3)	0.044 (1)
C(1)	−0.2254 (5)	0.1751 (3)	0.7758 (4)	0.074 (2)
C(2)	−0.1529 (5)	0.2227 (3)	0.7264 (3)	0.053 (1)
C(3)	−0.2159 (5)	0.2644 (3)	0.6373 (4)	0.067 (2)
C(4)	−0.1494 (8)	0.3094 (4)	0.5919 (5)	0.094 (2)
C(5)	−0.0215 (9)	0.3137 (4)	0.6302 (7)	0.116 (3)
C(6)	0.0480 (6)	0.2745 (4)	0.7186 (7)	0.112 (3)
C(7)	−0.0178 (6)	0.2293 (3)	0.7685 (4)	0.082 (2)
C(8)	−0.3548 (4)	0.1011 (3)	0.4769 (4)	0.059 (2)
C(9)	−0.3401 (6)	0.1220 (3)	0.3821 (4)	0.074 (2)
C(10)	−0.2186 (6)	0.1258 (3)	0.3756 (4)	0.069 (2)
C(11)	−0.1087 (5)	0.1089 (2)	0.4640 (3)	0.053 (1)
C(12)	−0.1308 (4)	0.0886 (2)	0.5561 (3)	0.042 (1)

C(13)	0.0215 (6)	0.1101 (3)	0.4635 (4)	0.066 (2)
C(14)	0.1230 (5)	0.0922 (3)	0.5495 (4)	0.066 (2)
C(15)	−0.0217 (4)	0.0689 (2)	0.6482 (3)	0.042 (1)
C(16)	0.1055 (4)	0.0710 (2)	0.6456 (4)	0.051 (1)
C(17)	0.2068 (4)	0.0511 (3)	0.7383 (4)	0.071 (2)
C(18)	0.1814 (4)	0.0301 (3)	0.8251 (4)	0.074 (2)
C(19)	0.0534 (4)	0.0289 (3)	0.8218 (3)	0.059 (1)
C(100)	−0.544 (1)	0.1173 (7)	0.0665 (8)	0.133 (4)
C(101)	−0.4995 (8)	0.1172 (6)	−0.018 (1)	0.134 (4)
C(102)	−0.531 (1)	0.179 (1)	−0.0815 (8)	0.151 (5)
C(103)	−0.605 (2)	0.239 (1)	−0.051 (2)	0.267 (9)
C(104)	−0.646 (3)	0.225 (1)	0.031 (3)	0.45 (2)
C(105)	−0.615 (1)	0.175 (1)	0.077 (1)	0.210 (7)

Table 2. *Selected geometric parameters (Å, °)*

Sn—Cl(1)	2.428 (1)	N(1)—C(8)	1.332 (5)
Sn—Cl(2)	2.430 (1)	N(1)—C(12)	1.358 (5)
Sn—Cl(3)	2.400 (1)	N(2)—C(15)	1.357 (5)
Sn—N(1)	2.281 (3)	N(2)—C(19)	1.329 (5)
Sn—N(2)	2.283 (3)	C(1)—C(2)	1.466 (6)
Sn—C(1)	2.175 (5)		
Cl(1)—Sn—Cl(2)	100.91 (5)	Cl(3)—Sn—C(1)	174.6 (1)
Cl(1)—Sn—Cl(3)	90.04 (5)	N(1)—Sn—N(2)	72.7 (1)
Cl(1)—Sn—N(1)	164.14 (9)	N(1)—Sn—C(1)	92.3 (2)
Cl(1)—Sn—N(2)	92.29 (9)	N(2)—Sn—C(1)	90.1 (2)
Cl(1)—Sn—C(1)	92.9 (1)	Sn—N(1)—C(8)	125.5 (3)
Cl(2)—Sn—Cl(3)	89.00 (5)	Sn—N(1)—C(12)	115.5 (3)
Cl(2)—Sn—N(1)	93.56 (9)	C(8)—N(1)—C(12)	118.9 (4)
Cl(2)—Sn—N(2)	165.57 (9)	Sn—N(2)—C(15)	115.5 (3)
Cl(2)—Sn—C(1)	95.0 (2)	Sn—N(2)—C(19)	125.6 (3)
Cl(3)—Sn—N(1)	83.71 (9)	C(15)—N(2)—C(19)	118.5 (4)
Cl(3)—Sn—N(2)	85.16 (9)	Sn—C(1)—C(2)	117.4 (3)

The values of T obtained from *DIFABS* (Walker & Stuart, 1983) have been normalized by multiplying them by $1/T(m)$, where $T(m)$ was the maximum value of T obtained by the program. It is recognized that the range of T values thus obtained may not be as great as might be expected from crystals of this shape.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1992). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

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Bis(diethylammonium) Tetrachloro-divinylstannate(IV)

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Abstract

The Sn atom in the anion of the title compound, $(C_4H_{12}N)_2[SnCl_4(C_2H_3)_2]$, exists in a distorted *trans*-C_2SnCl_4 octahedral geometry. Differences in the Sn—Cl separations within the anion are related to hydrogen bonding between the Cl(2) atoms of the anion and the diethylammonium cations. The average Sn—Cl distance in this and related compounds is correlated with the Lewis acidity of the diorganotin moiety.

Comment

The Sn atom in the anion of $(H_2NEt_2)_2[(vin)_2SnCl_4]$, (I), where vin is vinyl, is located on a centre of symmetry and the unit cell comprises two anions and four cations. The Sn atom exists in a distorted C_2Cl_4 octahedral geometry. The independent Sn—Cl bond distances of 2.583 (2) and 2.602 (2) Å arise as a result of a hydrogen-bonding contact between atoms H(10b) and Cl(2) at a distance of 2.41 Å; the Cl(2)···N(10) distance is 3.199 (6) Å and the angle at H(10b) is 138°.

(I)
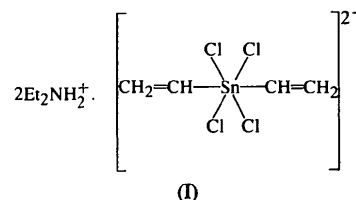
Similar *trans*-C_2SnCl_4 geometries have been observed previously in the structures of the $[Ph_2SnCl_4]^{2-}$ (Teoh, Teo, Yeap & Declercq, 1992) and $[Me_2SnCl_4]^{2-}$ (Valle, González, Ettore & Plazzogna, 1988) anions. The average Sn—Cl bond distances in the three structures follow the expected trends in the Lewis acidities