identified within the asymmetric unit. All non-H atoms were refined anisotropically, and H atoms were included in fixed calculated positions with d(C—H) 0.96 Å and U_{iso}(H) = U_{iso}(C) of the parent C atom.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1993). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN*. Program(s) used to solve structure: *SIR92*. Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

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(8-Aminoquinoline-N,N')dichlorodimethyltin(IV)

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Abstract

The title compound, [SnCl$_2$(CH$_3$)$_2$(C$_9$H$_8$N$_2$)], has been prepared from dimethyltin dichloride and 8-aminoquinoline. The coordination of the Sn atom is octahedral,

with the Cl atoms *trans* to the N atoms of 8-aminoquinoline, with bond distances Sn—Cl 2.547 (3) and 2.619 (3), Sn—N$_{sp^3}$ 2.322 (7), Sn—N$_{sp^2}$ 2.357 (8), and Sn—C 2.115 (10) and 2.122 (10) Å. Both amino protons are intermolecularly hydrogen bonded to Cl atoms of neighbouring molecules.

Comment

Extensive studies have been carried out on the 1:1 adducts of diorganotin dichloride with symmetric (*e.g.* 1,10-phenanthroline) and asymmetric *N,N'*-chelating agents (*e.g.* 2-aminomethylpyridine) owing to a potential link between antitumour activity, the Cl—Sn—Cl angle and the Sn—N bond distance (Crowe, Smith & Atassi, 1984; Saxena & Huber, 1989). However, there have been no structural reports on diorganotin dihalide adducts of 2-aminomethylpyridine even though some of these adducts were reported to possess antitumour activity (Crowe *et al.*, 1984). We have studied the reactions of 8-aminoquinoline, (I), which is an asymmetric *N,N'*-bidentate ligand closely resembling 2-aminomethylpyridine, (II).

(I)   (II)   (III)
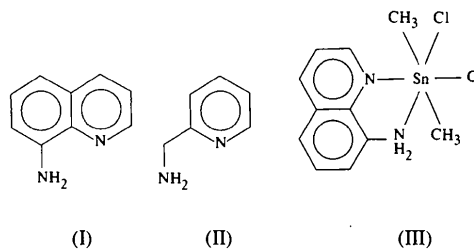
An X-ray structural determination of diphenyldichloro(8-aminoquinoline)tin(IV) revealed that the Cl atoms occupied the two axial positions of the six-coordinated Sn atom (Bengtson *et al.*, 1996). In a continuation of our attempts to prepare compounds displaying antitumour activity, we now report the synthesis and crystal structure of the dimethyldichloro(8-aminoquinoline)tin(IV) adduct, (III). IR measurements show two medium intensity bands (567 and 494 cm^{-1}) arising from the Sn—CH$_3$ stretching vibrations, suggesting that the two methyl groups attached to the Sn atom are *trans* to each other (Petrosyan, Yashina & Reutov, 1976). Furthermore, calculations using equations (1) and (3) of Lockhart & Manders (1986), based on the $^2J_{Sn-CH_3}$ coupling constant value (113.4 Hz) obtained from the proton spectrum of the title adduct give a Me—Sn—Me angle of 166.8°, while the $^1J_{Sn-C}$ coupling constant of 987.0 Hz gives an angle of 163.3°. An X-ray structure analysis confirmed the proposed *trans* configuration but found a C—Sn—C angle closer to 180°. The Sn—Cl distances are longer than those found for diphenyldichloro(8-aminoquinoline)tin(IV), 2.502 (1) and 2.518 (1) Å. This is to be expected (Hall & Tiekink, 1996) because dimethyltin dichloride is a weaker Lewis

acid than diphenyltin dichloride and because the Sn—Cl bonds participate in Sn—Cl···N hydrogen bonds for the dimethyltin dichloride adduct. The Cl—Sn—Cl angle is however larger than the minimum of 103° which Kabanos *et al.* (1992) consider to be an added requirement for antitumour activity in these compounds. Related dimethyltin dichloride adducts in which the ligand is related to 2-aminomethylpyridine but with the amino group replaced by an imino group (Kabanos *et al.*, 1992; Teoh, Teo, Lee & Fun, 1994) show the reverse situation, *i.e.* Sn—N bonds greater than 2.39 Å and Cl—Sn—Cl angles less than 103°.
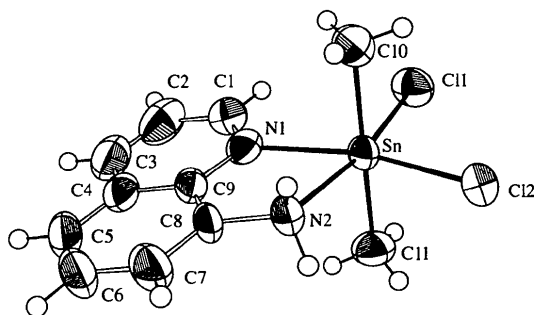

Fig. 1. View showing the labelling of the non-H atoms. Displacement ellipsoids are shown at 50% probability levels and H atoms are drawn as small circles of arbitrary radii.

The molecules are linked in infinite chains, parallel to the *c* axis, by Sn—Cl1···N2(x, y, $z-1$) hydrogen bonds, the chains are themselves linked in pairs by Sn—Cl2···N2($2-x$, $1-y$, $2-z$) hydrogen bonds. The Sn—Cl bonds which form the infinite chains are significantly longer than those in the cross-links. The N···Cl distances are similar to those in bis(3-methyladenine)-dichlorodimethyltin(IV) [3.211 (4) Å; Hazell, Ouyang & Khoo, 1997] and in 8-aminoquinolium chloride [3.030 (7) Å; Bengtson *et al.*, 1996]. As with the di-
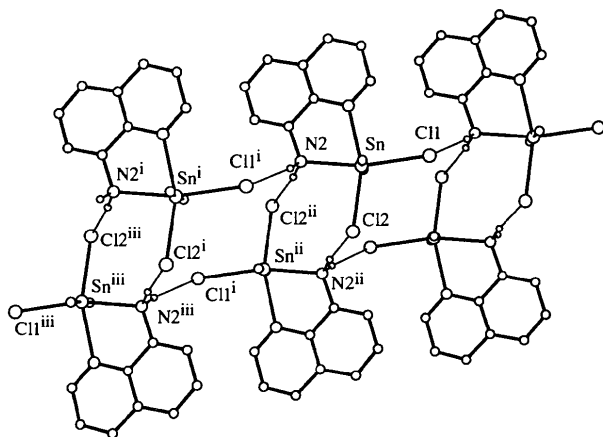
phenyl compound, the Sn—N$_{sp^2}$ distance is longer than Sn—N$_{sp^3}$, but both are shorter than the limiting value of 2.39 Å which Crowe, Smith & Atassi (1984) suggest must be exceeded for this type of compounds to exhibit antitumour activity.

Experimental

The title compound was synthesized by mixing equimolar amounts of dimethyltin dichloride and 8-aminoquinoline in anhydrous benzene. The product upon recrystallization in benzene yielded the title adduct (m.p. 399–402 K) which has the following microanalytical data: C 36.20, H 4.01 and N 7.84%; calculated for $C_{11}H_{14}N_2Cl_2Sn$: C 36.31, H 3.88 and N 7.70%.

Crystal data

[SnCl₂(CH₃)₂(C₉H₈N₂)]	Mo $K\alpha$ radiation
$M_r = 363.84$	$\lambda = 0.71073$ Å
Triclinic	Cell parameters from 50
$P\bar{1}$	reflections
$a = 9.678\,(8)$ Å	$\theta = 10.0{-}14.3°$
$b = 10.701\,(9)$ Å	$\mu = 2.21$ mm^{-1}
$c = 7.280\,(6)$ Å	$T = 294$ K
$\alpha = 90.15\,(5)°$	Block
$\beta = 104.32\,(5)°$	$0.325 \times 0.300 \times 0.200$ mm
$\gamma = 71.59\,(5)°$	Brown
$V = 691\,(1)$ Å³	
$Z = 2$	
$D_x = 1.749$ Mg m^{-3}	
D_m not measured	

Data collection

Huber four-circle diffractom- eter	$R_{int} = 0.025$ $\theta_{max} = 25.0°$
$\theta/2\theta$ scans	$h = 0 \rightarrow 11$
Absorption correction:	$k = -11 \rightarrow 12$
numerical by integration	$l = -8 \rightarrow 8$
$T_{min} = 0.432$, $T_{max} = 0.706$	2 standard reflections
2642 measured reflections	every 50 reflections
2432 independent reflections	intensity decay: 1%
1925 reflections with	
$I > 3\sigma(I)$	

Refinement

Refinement on F	$(\Delta/\sigma)_{max} = 0.005$
$R = 0.055$	$\Delta\rho_{max} = 1.6\,(2)$ e Å^{-3}
$wR = 0.066$	$\Delta\rho_{min} = -1.8\,(2)$ e Å^{-3}
$S = 1.671$	Extinction correction: none
1925 reflections	Scattering factors from *Inter-*
145 parameters	*national Tables for X-ray*
H atoms constrained	*Crystallography* (Vol. IV)
$w = 1/\{[\sigma_{cs}(F^2)$	
$+ 1.03F^2]^{1/2} - |F|\}^2$	

Fig. 2. The hydrogen-bonding scheme. Symmetry codes (i) x, y, $z-1$; (ii) $2-x$, $1-y$, $2-z$; (iii) $2-x$, $1-y$, $1-z$.

Table 1. *Selected geometric parameters (Å, °)*

Sn—C10	2.115 (10)	Sn—N2	2.322 (7)
Sn—C11	2.122 (10)	Sn—Cl1	2.619 (3)
Sn—N1	2.357 (8)	Sn—Cl2	2.547 (3)

Cl1—Sn—Cl2	105.4 (1)	Cl2—Sn—C11	91.2 (3)
Cl1—Sn—N1	94.4 (2)	N1—Sn—N2	73.0 (3)
Cl1—Sn—N2	167.4 (2)	N1—Sn—C10	90.7 (3)
Cl1—Sn—C10	86.9 (3)	N1—Sn—C11	88.6 (3)
Cl1—Sn—C11	90.1 (3)	N2—Sn—C10	93.2 (3)
Cl2—Sn—N1	160.2 (2)	N2—Sn—C11	89.4 (4)
Cl2—Sn—N2	87.2 (2)	C10—Sn—C11	176.9 (4)
Cl2—Sn—C10	90.5 (3)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—HN2A···Cl1^i	0.950	2.369	3.285 (9)	161.7
N2—HN2B···Cl2^{ii}	0.950	2.332	3.274 (8)	171.2

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

H atoms of the aminoquinoline were kept fixed at calculated positions (C—H = 0.95 Å), those of the methyl groups were kept fixed at their observed positions. U_{iso} was set to be 20% larger than U_{eq} for the atom to which they were attached. The distances from Sn to the minimum and maximum values of $\Delta\rho$ are 0.94 and 1.22 Å, respectively.

Data collection: *MAD* (Allibon, 1995). Cell refinement: *MAD*. Data reduction: *KRYSTAL* (Hazell, 1995). Program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994) and *KRYSTAL*. Program(s) used to refine structure: modified *ORFLS* (Busing, Martin & Levy, 1962). Molecular graphics: *ORTEP*III (Burnett & Johnson, 1996) and *KRYSTAL*. Software used to prepare material for publication: *KRYSTAL*.

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Chlorodimethyl(N-pyrrolidinecarbodithioato-S,S')tin(IV)

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Abstract

The Sn atom in $[SnCl(C_5H_8NS_2)(CH_3)_2]$ is in a distorted trigonal bipyramidal environment, with the two methyl C atoms and an S atom in the equatorial plane, and the Cl and other S atom occupying axial positions. The carbodithioate ligand is unsymmetrically coordinated to the Sn atom with Sn—S distances of 2.7555 (10) and 2.4825 (12) Å.

Comment

Although a number of pentadentate diorganotin complexes of the type R_2ClSnS_2C have been reported, examples of structural studies are limited to $Me_2SnCl(S_2CNMe_2)$, $PhBuSnCl(S_2CNEt_2)$, $Ph_2SnCl(S_2CO^iPr)$ (Furue, Kimura, Yasuoka, Kasai & Masao, 1970; Wei, Kumar Das & Sinn, 1985; Dakternieks, Hoskins, Jackson, Tiekink & Winter, 1985). In these complexes, the Sn atom is asymmetrically bonded to the two S atoms with an average difference in the Sn—S bond lengths of 0.3 Å, similar to that observed in the title compound, $Me_2ClSn[S_2CN(CH_2)_4]$, (1). However, the Sn—S distances in the related 1,2-dithio anionic complex 2-chloro-2,2-diphenyl-5-methyl-1,3,2-benzodithiastannole are similar (Sau, Day & Holmes, 1981). As in (1), the chloro group in all these complexes invariably occupies an axial position with the two organo groups in equatorial positions.

(1)
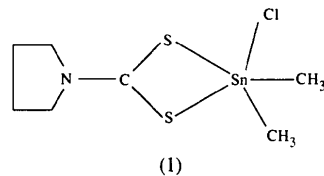
The displacement ellipsoid plot of (1) with the numbering scheme is shown in Fig. 1. The geometry about the Sn atom is a distorted trigonal bipyramid with the Cl and S1 atoms in axial positions and the two methyl C atoms and S2 in the equatorial plane. Despite