$a = 17.491\,(4)$ Å
$b = 14.236\,(3)$ Å
$c = 18.656\,(4)$ Å
$\beta = 100.24\,(2)°$
$V = 4571.4\,(16)$ Å^3
$Z = 4$
$D_x = 1.650$ Mg m^{-3}
D_m not measured

$\mu = 5.438$ mm^{-1}
$T = 293\,(2)$ K
Prism
$0.21 \times 0.16 \times 0.15$ mm
Colourless

Data collection

Siemens *R*3*m* diffractometer
ω–2θ scans
Absorption correction:
  ψ scans (North, Phillips & Mathews, 1968)
  $T_{min} = 0.389$, $T_{max} = 0.442$
5451 measured reflections
5290 independent reflections
3502 reflections with $I > 2\sigma(I)$

$R_{int} = 0.0352$
$\theta_{max} = 27.56°$
$h = -22 \rightarrow 22$
$k = 0 \rightarrow 18$
$l = 0 \rightarrow 24$
3 standard reflections every 400 reflections intensity decay: 4%

Refinement

Refinement on F^2
$R(F) = 0.0424$
$wR(F^2) = 0.0635$
$S = 0.944$
5290 reflections
245 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0192P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.630$ e Å^{-3}
$\Delta\rho_{min} = -0.709$ e Å^{-3}
Extinction correction: *SHELXTL*
Extinction coefficient: 0.00004 (2)
Scattering factors from *International Tables for Crystallography* (Vol. C)

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}
Re1	0.040342 (13)	0.28762 (2)	0.178908 (12)	0.02716 (8)
P1	0	0.1563 (2)	1/4	0.0259 (5)
P2	−0.08685 (9)	0.32913 (12)	0.10207 (8)	0.0330 (4)
C1	0.0737 (3)	0.2156 (6)	0.1031 (3)	0.043 (2)
O1	0.0950 (3)	0.1709 (4)	0.0592 (3)	0.071 (2)
C2	0.0771 (3)	0.4044 (5)	0.1452 (3)	0.039 (2)
O2	0.1051 (3)	0.4719 (4)	0.1269 (3)	0.072 (2)
C3	0.1442 (3)	0.2704 (4)	0.2328 (3)	0.0327 (14)
O3	0.2086 (2)	0.2634 (3)	0.2595 (2)	0.0526 (13)
C11	0.0822 (3)	0.0808 (4)	0.2944 (3)	0.0291 (13)
C12	0.0591 (3)	0.0061 (4)	0.3465 (3)	0.041 (2)
C13	0.1322 (4)	−0.0412 (5)	0.3895 (4)	0.050 (2)
C14	0.1814 (4)	−0.0840 (5)	0.3399 (4)	0.059 (2)
C15	0.2029 (4)	−0.0116 (5)	0.2873 (4)	0.055 (2)
C16	0.1306 (3)	0.0350 (4)	0.2437 (3)	0.040 (2)
C21	−0.1087 (3)	0.4576 (4)	0.0971 (3)	0.038 (2)
C22	−0.1134 (5)	0.4962 (5)	0.1715 (4)	0.067 (2)
C23	−0.1315 (5)	0.6021 (5)	0.1703 (4)	0.073 (2)
C24	−0.2026 (4)	0.6267 (6)	0.1162 (4)	0.067 (2)
C25	−0.1949 (4)	0.5923 (5)	0.0421 (4)	0.070 (2)
C26	−0.1800 (4)	0.4862 (5)	0.0427 (4)	0.052 (2)
C31	−0.1080 (4)	0.2897 (5)	0.0063 (3)	0.0427 (15)
C32	−0.0513 (4)	0.3315 (5)	−0.0391 (3)	0.049 (2)
C33	−0.0767 (5)	0.3016 (6)	−0.1192 (4)	0.075 (3)
C34	−0.0820 (6)	0.1989 (6)	−0.1275 (4)	0.088 (3)
C35	−0.1390 (5)	0.1585 (6)	−0.0825 (4)	0.073 (2)
C36	−0.1164 (5)	0.1861 (5)	−0.0021 (4)	0.063 (2)

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

Re1—P1	2.467 (2)	Re1—Re1^i	3.2185 (8)
Re1—P2	2.494 (2)	Re1—H1	1.84 (4)
P1—Re1—P2	100.22 (4)	C11^i—P1—C11	109.6 (4)
P1—Re1—Re1^i	49.28 (3)	Re1^i—P1—Re1	81.45 (7)
P2—Re1—Re1^i	89.81 (4)	Re1^i—H1—Re1	122 (5)

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

H atoms were refined riding on their carrier atoms, except for H1 and H2, which were located from the difference map and their positions refined.

Program(s) used to solve structure: *SHELXTL* (Sheldrick, 1995). Program(s) used to refine structure: *SHELXTL*.

Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: SK1060). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1997). **C53**, 406–408

Dichlorodimethylbis(3-methyladenine-*N*^{7})-tin(IV)

Alan Hazell,[a] Jiexiang Ouyang[b] and Lian Ee Khoo[b]

[a]*Department of Chemistry, Aarhus University, Langelandsgade 140, DK-8000 Århus C, Denmark, and* [b]*School of Science, Nanyang Technological University, 469 Bukit Timah Road, Singapore 1025, Singapore. E-mail: ach@kemi.aau.dk*

(*Received 26 September 1996; accepted 9 December 1996*)

Abstract

The title compound, [$SnCl_2(CH_3)_2(C_6H_7N_5)_2$], has been prepared from dimethyltin dichloride and 3-methyladenine in methanol. The coordination of the Sn atom

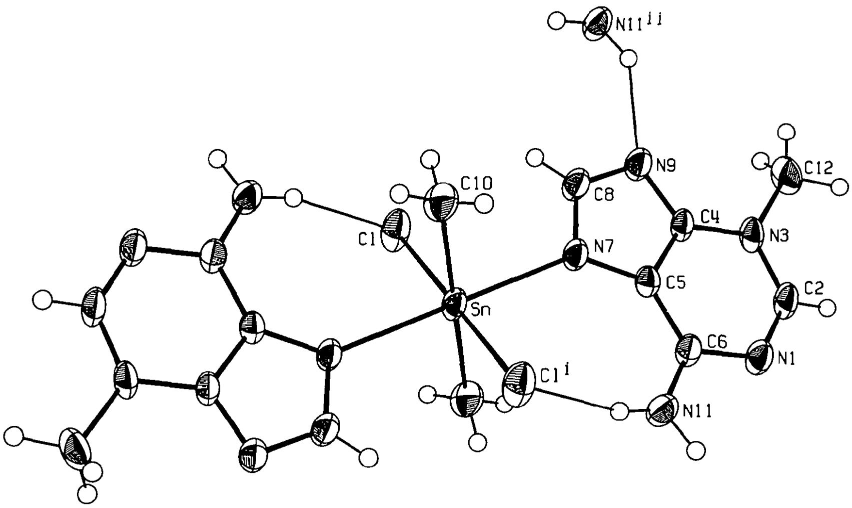
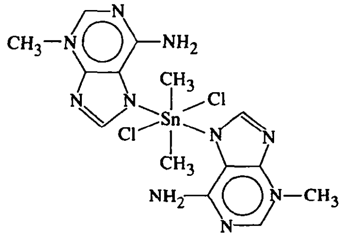


Fig. 1. View of the title molecule showing the hydrogen bonding and the labelling of the non-H atoms. Displacement ellipsoids are shown at the 50% probability level and H atoms are drawn as small circles of arbitrary radii. [Symmetry codes: (i) $2-x$, $-y$, $2-z$; (ii) x, y, $z-1$.]

is all-*trans* octahedral, with bond distances Sn—Cl 2.596 (1), Sn—N 2.383 (3) and Sn—C 2.120 (4) Å. The 3-methyladenine ligands are bonded to the Sn atom through their N7 atoms. The two amino protons are hydrogen bonded, one in an intermolecular bond to the N9 atom of a neighbouring molecule, the other in an intramolecular bond to a Cl atom.

Comment

^{1}H NMR studies (Cardin & Roy, 1985) of the 1:2 adducts of dimethyltin dichloride with adenine, adenosine and 9-methyladenine suggest that coordination takes place at the N7 atom. In the present study of the title compound, (I), we also found that tin is coordinated to the N7 atom and that the two amino protons are involved in hydrogen bonds, one in an intramolecular bond to Cl^- (at $2-x$, $-y$, $2-z$) and the other in an intermolecular hydrogen bond to the N9 atom of a neighbouring molecule (at x, y, $z+1$), thus forming chains of molecules parallel to the c axis.



(I)

The Sn atom lies on a symmetry centre and is bonded to two Cl atoms, two methyl groups and the N7 atoms of the two ligands, giving an all-*trans* octahedral geometry. The coordination angles between *trans* groups are exactly 180°, the other coordination angles are in the range 88.1 (1)–91.9 (1)°. The Sn—C, Sn—Cl and Sn—N distances lie within the ranges for similar compounds (Hazell, Goh & Khoo, 1994; Kamwaya & Khoo, 1985; Pettinari *et al.*, 1995). The geometry of the adenine moiety differs from that of adenine itself (Taylor & Kennard, 1982) in that N1—C2, C8—C9, C4—C9 and C6—N11 are shorter than in adenine, while N3—C4 and C5—N7 are longer. These results are similar to those found for 3-methyladenine hydrochloride, which is protonated at N7 (Yamagata & Tomita, 1987) and for metal complexes, *e.g. cis*-diamminebis(3-methyladenine)platinum(II) nitrate trihydrate (Orbell, Solorzano, Marzilli & Kistenmacher, 1982). The maximum deviation from planarity of the nine-membered purine ring is 0.023 (5) Å. Atoms N11 and C12 lie −0.066 (5) and −0.048 (5) Å, respectively, out of this plane, which roughly bisects the Cl—Sn—C10 angle and is twisted towards the Cl^- (at $2-x$, $-y$, $2-z$) atom, to which it is hydrogen bonded.

Experimental

3-Methyladenine was prepared according to the reported procedure of Barlin (1981). A mixture of 3-methyladenine (0.3 g, 2 mmol) in 50 ml methanol and dimethyltin dichloride (0.44 g, 2 mmol) in 35 ml methanol was refluxed for 2 d. The mixture was concentrated to *ca* 10 ml using a rotatory evaporator. On cooling in a freezer overnight, a white solid (0.34 g, 66% yield; m.p. 569–571 K) was obtained and filtered off. Crystals suitable for X-ray analysis were obtained by recrystallization from methanol. The title complex was obtained regardless of the starting ratio of the reagents. Analysis found: C 32.5, H 4.02, N 27.3%; calculated for $C_{14}H_{20}Cl_2N_{10}Sn$: C 32.46, H 3.89, N 27.04%.

Crystal data

[$SnCl_2(CH_3)_2(C_6H_7N_5)_2$]
$M_r = 518.0$
Monoclinic
$P2_1/a$
$a = 7.435$ (2) Å
$b = 18.709$ (3) Å
$c = 7.5618$ (13) Å
$\beta = 113.078$ (14)°
$V = 967.7$ (4) Å^3
$Z = 2$
$D_x = 1.778$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 60 reflections
$\theta = 12.00$–15.95°
$\mu = 1.619$ mm^{-1}
$T = 294$ K
Tabular
0.70 × 0.70 × 0.18 mm
Colourless

Data collection

Huber four-circle diffractometer
$\theta/2\theta$ scans
Absorption correction: by integration
$T_{\min} = 0.347$, $T_{\max} = 0.766$
1978 measured reflections
1761 independent reflections
1658 reflections with $I > 0$

$R_{\rm int} = 0.079$
$\theta_{\max} = 25°$
$h = -8 \rightarrow 8$
$k = 0 \rightarrow 22$
$l = 0 \rightarrow 8$
2 standard reflections every 50 reflections intensity decay: 3%

Refinement

Refinement on F
$R = 0.043$
$wR = 0.054$
$S = 1.81$
1658 reflections
165 parameters
All H atoms refined
$w = 1/\{[\sigma_{\rm cs}(F^2) + 1.03F^2]^{1/2} - |F|\}^2$
$(\Delta/\sigma)_{\max} = 0.028$

$\Delta\rho_{\max} = 1.2$ (2) e Å^{-3}
$\Delta\rho_{\min} = -1.5$ (2) e Å^{-3}
Extinction correction: type 1 Lorentzian isotropic (Becker & Coppens, 1975)
Extinction coefficient: 1.0 (3) × 10^3
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

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

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

	x	y	z	$U_{\rm eq}$
Sn	1	0	1	0.0286 (3)
Cl	1.22355 (18)	−0.01780 (6)	0.81218 (17)	0.0440 (7)
N1	0.9368 (5)	0.2903 (2)	1.1277 (4)	0.034 (2)
C2	0.9341 (7)	0.3297 (2)	0.9839 (5)	0.035 (2)
N3	0.9294 (5)	0.3055 (1)	0.8145 (4)	0.031 (2)
C4	0.9296 (5)	0.2329 (2)	0.7933 (5)	0.027 (2)
C5	0.9344 (5)	0.1873 (2)	0.9390 (5)	0.026 (2)
C6	0.9342 (5)	0.2172 (2)	1.1105 (5)	0.029 (2)
N7	0.9378 (5)	0.1184 (1)	0.8742 (4)	0.030 (2)
C8	0.9315 (6)	0.1291 (2)	0.6959 (5)	0.035 (3)
N9	0.9261 (5)	0.1970 (2)	0.6380 (4)	0.034 (2)
C10	0.7542 (7)	−0.0353 (2)	0.7583 (6)	0.041 (3)
N11	0.9320 (6)	0.1797 (2)	1.2576 (5)	0.039 (2)
C12	0.9176 (8)	0.3534 (2)	0.6560 (6)	0.044 (3)

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

Sn—Cl	2.596 (1)	C4—N9	1.344 (4)
Sn—N7	2.384 (3)	C4—C5	1.383 (5)
Sn—C10	2.121 (4)	C5—N7	1.383 (4)
N1—C2	1.308 (5)	C5—C6	1.413 (5)
N1—C6	1.372 (4)	C6—N11	1.321 (5)
C2—N3	1.346 (5)	N7—C8	1.345 (4)
N3—C4	1.367 (4)	C8—N9	1.338 (5)
N3—C12	1.472 (5)		
Sn—N7—C8	117.6 (2)	C5—N7—C8	102.6 (3)
Sn—N7—C5	138.8 (2)	N7—C8—N9	117.0 (3)
C5—C4—N9	111.9 (3)	C4—N9—C8	101.6 (3)
C4—C5—N7	106.9 (3)		

Table 3. *Hydrogen-bonding geometry (Å, °)*

D—H···A	D—H	H···A	D···A	D—H···A
N11—HN11A···Cl$^{\rm i}$	0.87 (6)	2.37 (6)	3.211 (4)	163 (5)
N11—HN11B···N9$^{\rm ii}$	0.94 (5)	2.08 (5)	2.912 (4)	147 (4)

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

Cell dimensions were determined from reflections measured at ±2θ. A very large crystal (0.70 × 0.70 × 0.18 mm) was used in order to increase the number of significant reflections.

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) and *KRYSTAL*. Molecular graphics: *ORTEP*III (Burnett & Johnson, 1996) and *KRYSTAL*. Software used to prepare material for publication: *KRYSTAL*.

Financial support from Nanyang Technological University (Grant No. RP9/92KLE) is gratefully acknowledged. AH is indebted to the Carlsberg Foundation and to the Danish Science Research Council for the diffractometer.

Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: AB1424). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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