

Di- μ -methanolato- κ^4 O:O-bis[trichlorido-(dimethylformamide- κ O)tin(IV)]

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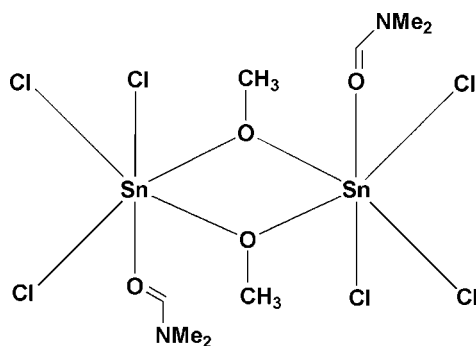
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 Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{N}-\text{C}) = 0.011$ Å; R factor = 0.044; wR factor = 0.117; data-to-parameter ratio = 18.5.

The title compound, $[\text{Sn}_2(\text{CH}_3\text{O})_2\text{Cl}_6(\text{C}_3\text{H}_7\text{NO})_2]$, contains two hexacoordinated Sn^{IV} atoms symmetrically bridged by two deprotonated methanol ligands, with an inversion center in the middle of the planar Sn_2O_2 ring. The other sites of the distorted octahedral coordination geometry of the Sn^{IV} atom are occupied by three Cl atoms and one O atom from a dimethylformamide molecule. The complex molecules are connected by weak $\text{C}-\text{H}\cdots\text{Cl}$ hydrogen bonds into a two-dimensional supramolecular network parallel to $(10\bar{1})$.

Related literature

For related tin(IV) compounds, see: Mao & You (1990); Reuter & Schröder (1992).



Experimental

Crystal data

 $[\text{Sn}_2(\text{CH}_3\text{O})_2\text{Cl}_6(\text{C}_3\text{H}_7\text{NO})_2]$
 $M_r = 658.34$

 Monoclinic, $P2_1/n$
 $a = 8.589$ (8) Å

 $b = 11.4444$ (13) Å

 $c = 11.8453$ (10) Å

 $\beta = 111.155$ (1)°

 $V = 1085.9$ (10) Å³
 $Z = 2$

 Mo $K\alpha$ radiation

 $\mu = 3.05$ mm⁻¹
 $T = 298$ K

 $0.22 \times 0.17 \times 0.16$ mm

Data collection

Bruker APEX CCD diffractometer

Absorption correction: multi-scan

(SADABS; Sheldrick, 1996)

 $T_{\text{min}} = 0.553$, $T_{\text{max}} = 0.641$

5512 measured reflections

1903 independent reflections

 1554 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.063$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.044$
 $wR(F^2) = 0.117$
 $S = 1.14$

1903 reflections

103 parameters

13 restraints

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 1.54$ e Å⁻³
 $\Delta\rho_{\text{min}} = -2.07$ e Å⁻³
Table 1

Selected bond lengths (Å).

Sn1—O1	2.106 (5)	Sn1—Cl1	2.372 (2)
Sn1—O1 ⁱ	2.101 (5)	Sn1—Cl2	2.3743 (18)
Sn1—O2	2.108 (4)	Sn1—Cl3	2.368 (2)

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

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C1—H1C \cdots Cl3	0.96	2.72	3.356 (8)	124
C3—H3A \cdots Cl3 ⁱⁱ	0.96	2.95	3.895 (11)	170
C4—H4B \cdots Cl1 ⁱⁱⁱ	0.96	2.90	3.837 (9)	164

 Symmetry codes: (ii) $x + \frac{1}{2}, -y + \frac{1}{2}, z + \frac{1}{2}$; (iii) $-x + \frac{3}{2}, y + \frac{1}{2}, -z + \frac{1}{2}$.

Data collection: SMART (Bruker, 2007); cell refinement: SAINT (Bruker, 2007); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008) and X-SEED (Barbour, 2001); software used to prepare material for publication: SHELXTL.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HY2391).

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supplementary materials

Acta Cryst. (2011). E67, m146 [doi:10.1107/S1600536810053997]

Di- μ -methanolato- κ^4 O:O-bis[trichlorido(dimethylformamide- κ O)tin(IV)]

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Comment

The title compound (Fig.1) consists of six Cl anions, two methoxo anions, two Sn^{IV} ions and two dimethylformamide molecules. The molecule has an inversion center in the middle of the Sn₂O₂ ring. This ring is planar and can be described as rhombic, with the endocyclic angles at the O atoms larger than those at the Sn atoms [Sn1ⁱ—O1—Sn1 = 106.9 (2), O1ⁱ—Sn1—O1 = 73.1 (2)°. Symmetry code: (i) -x+2, -y, -z+1]. The Sn1—O1 distance [2.106 (5) Å] is very close to the Sn1—O1ⁱ distance [2.101 (5) Å] (Table 1). Each Sn^{IV} atom is hexacoordinated with two methoxo anions, three Cl anions and one dimethylformamide molecule in a distorted octahedral geometry.

As is indicated from Fig. 2 and Table 2, the intramolecular interactions, C1—H1Cⁱⁱⁱ···Cl3, strengthen the dimeric unit and the intermolecular ones, C3—H3Aⁱⁱ···Cl3ⁱⁱ and C4—H4Bⁱⁱⁱ···Cl1ⁱⁱⁱ [symmetry codes: (ii) x+1/2, -y+1/2, z+1/2; (iii) -x+3/2, y+1/2, -z+1/2], give rise to a two-dimensional polymer-like supramolecular network.

Experimental

Stannic chloride hydrate (0.4 mmol, 0.14 g) was dissolved in methanol (20 ml) and dimethylformamide (5 ml) was added with stirring at room temperature. The mixture was allowed to react for 6 h and was then filtered. Colorless crystals suitable for X-ray analysis were obtained by slow evaporation of methanol over a period of two weeks (yield: 60%). Analysis, calculated for C₈H₂₀Cl₆N₂O₄Sn₂: C 14.59, H 3.06, N 4.25%; found: C 14.58, H 3.04, N 4.27%.

Refinement

H atoms were placed in calculated positions and treated as riding on their parent atoms, with C—H = 0.93 (CH) and 0.96 (CH₃) Å and with $U_{\text{iso}}(\text{H}) = 1.2(1.5 \text{ for methyl})U_{\text{eq}}(\text{C})$. The highest residual electron density was found at 0.72 Å from H1A atom and the deepest hole at 1.01 Å from Sn1 atom.

Figures

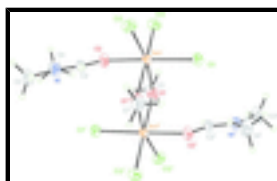


Fig. 1. The molecular structure of the title compound, with 50% probability displacement ellipsoids. [Symmetry code: (i) -x+2, -y, -z+1.]

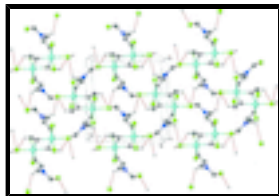


Fig. 2. A view of the two-dimensional polymer-like supramolecular network in the title compound.

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Crystal data

[Sn₂(CH₃O)₂Cl₆(C₃H₇NO)₂]

$M_r = 658.34$

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

$a = 8.589$ (8) Å

$b = 11.4444$ (13) Å

$c = 11.8453$ (10) Å

$\beta = 111.155$ (1)°

$V = 1085.9$ (10) Å³

$Z = 2$

$F(000) = 632$

$D_x = 2.013$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 2694 reflections

$\theta = 2.6$ – 27.0 °

$\mu = 3.05$ mm⁻¹

$T = 298$ K

Block, colourless

$0.22 \times 0.17 \times 0.16$ mm

Data collection

Bruker APEX CCD
diffractometer

Radiation source: fine-focus sealed tube
graphite

φ and ω scans

Absorption correction: multi-scan
(*SADABS*; Sheldrick, 1996)

$T_{\min} = 0.553$, $T_{\max} = 0.641$

5512 measured reflections

1903 independent reflections

1554 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.063$

$\theta_{\max} = 25.0$ °, $\theta_{\min} = 2.6$ °

$h = -6$ → 10

$k = -13$ → 12

$l = -14$ → 14

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.044$

$wR(F^2) = 0.117$

$S = 1.14$

1903 reflections

103 parameters

13 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

$w = 1/[\sigma^2(F_o^2) + (0.065P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.012$

$\Delta\rho_{\max} = 1.54$ e Å⁻³

$\Delta\rho_{\min} = -2.07$ e Å⁻³

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Sn1	0.99552 (5)	0.07346 (3)	0.37511 (4)	0.0383 (2)
Cl1	1.1769 (3)	-0.06088 (15)	0.3320 (2)	0.0619 (5)
Cl2	1.1500 (2)	0.24014 (15)	0.35788 (19)	0.0586 (5)
Cl3	0.7989 (3)	0.07190 (15)	0.17440 (19)	0.0629 (5)
N1	0.7652 (7)	0.3252 (5)	0.5241 (5)	0.0498 (14)
O1	0.8762 (6)	-0.0591 (4)	0.4372 (5)	0.0511 (10)
O2	0.8269 (5)	0.1862 (4)	0.4134 (4)	0.0461 (10)
C1	0.7018 (10)	-0.0904 (6)	0.3757 (7)	0.0556 (11)
H1A	0.6323	-0.0359	0.3974	0.083*
H1B	0.6828	-0.1677	0.3993	0.083*
H1C	0.6750	-0.0881	0.2897	0.083*
C2	0.8701 (8)	0.2657 (5)	0.4921 (6)	0.0438 (15)
H2	0.9832	0.2824	0.5290	0.053*
C3	0.8233 (14)	0.4147 (7)	0.6175 (10)	0.078 (3)
H3A	0.9420	0.4236	0.6411	0.117*
H3B	0.7968	0.3919	0.6864	0.117*
H3C	0.7696	0.4876	0.5864	0.117*
C4	0.5869 (9)	0.3028 (7)	0.4732 (9)	0.072 (2)
H4A	0.5679	0.2218	0.4517	0.109*
H4B	0.5372	0.3501	0.4023	0.109*
H4C	0.5377	0.3217	0.5319	0.109*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sn1	0.0438 (3)	0.0350 (3)	0.0425 (3)	0.00087 (17)	0.0231 (2)	0.00141 (16)
Cl1	0.0809 (14)	0.0551 (10)	0.0707 (13)	0.0188 (9)	0.0527 (11)	0.0088 (8)
Cl2	0.0610 (11)	0.0488 (9)	0.0764 (13)	-0.0084 (8)	0.0371 (10)	0.0094 (8)
Cl3	0.0709 (13)	0.0691 (12)	0.0452 (10)	0.0061 (9)	0.0165 (9)	0.0006 (8)
N1	0.056 (4)	0.044 (3)	0.059 (4)	0.008 (3)	0.032 (3)	0.000 (3)
O1	0.0531 (15)	0.0515 (15)	0.0514 (15)	-0.0033 (12)	0.0221 (12)	0.0004 (12)
O2	0.043 (2)	0.044 (2)	0.056 (3)	-0.001 (2)	0.023 (2)	-0.008 (2)
C1	0.0560 (14)	0.0550 (13)	0.0553 (14)	-0.0025 (9)	0.0195 (9)	0.0009 (9)
C2	0.048 (4)	0.044 (3)	0.044 (3)	0.003 (3)	0.022 (3)	-0.004 (3)
C3	0.097 (7)	0.065 (5)	0.083 (7)	0.001 (4)	0.046 (6)	-0.030 (4)
C4	0.051 (5)	0.071 (5)	0.099 (7)	0.016 (4)	0.031 (4)	0.000 (5)

Geometric parameters (\AA , $^\circ$)

Sn1—O1	2.106 (5)	O2—C2	1.259 (7)
Sn1—O1 ⁱ	2.101 (5)	C1—H1A	0.9600
Sn1—O2	2.108 (4)	C1—H1B	0.9600
Sn1—Cl1	2.372 (2)	C1—H1C	0.9600
Sn1—Cl2	2.3743 (18)	C2—H2	0.9300

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Sn1—Cl3	2.368 (2)	C3—H3A	0.9600
N1—C2	1.291 (8)	C3—H3B	0.9600
N1—C4	1.452 (9)	C3—H3C	0.9600
N1—C3	1.457 (10)	C4—H4A	0.9600
O1—C1	1.455 (9)	C4—H4B	0.9600
O1—Sn1 ⁱ	2.101 (5)	C4—H4C	0.9600
O1 ⁱ —Sn1—O2	87.65 (18)	O1—C1—H1A	109.5
O1 ⁱ —Sn1—O1	73.1 (2)	O1—C1—H1B	109.5
O2—Sn1—O1	84.69 (19)	H1A—C1—H1B	109.5
O1 ⁱ —Sn1—Cl3	166.73 (15)	O1—C1—H1C	109.5
O2—Sn1—Cl3	85.64 (14)	H1A—C1—H1C	109.5
O1—Sn1—Cl3	94.86 (16)	H1B—C1—H1C	109.5
O1 ⁱ —Sn1—Cl1	92.45 (15)	O2—C2—N1	123.2 (6)
O2—Sn1—Cl1	177.31 (12)	O2—C2—H2	118.4
O1—Sn1—Cl1	92.76 (15)	N1—C2—H2	118.4
Cl3—Sn1—Cl1	93.72 (9)	N1—C3—H3A	109.5
O1 ⁱ —Sn1—Cl2	93.32 (14)	N1—C3—H3B	109.5
O2—Sn1—Cl2	88.59 (13)	H3A—C3—H3B	109.5
O1—Sn1—Cl2	165.05 (15)	N1—C3—H3C	109.5
Cl3—Sn1—Cl2	97.94 (8)	H3A—C3—H3C	109.5
Cl1—Sn1—Cl2	94.09 (8)	H3B—C3—H3C	109.5
C2—N1—C4	121.9 (6)	N1—C4—H4A	109.5
C2—N1—C3	120.6 (7)	N1—C4—H4B	109.5
C4—N1—C3	117.4 (6)	H4A—C4—H4B	109.5
C1—O1—Sn1 ⁱ	124.2 (4)	N1—C4—H4C	109.5
C1—O1—Sn1	123.0 (4)	H4A—C4—H4C	109.5
Sn1 ⁱ —O1—Sn1	106.9 (2)	H4B—C4—H4C	109.5
C2—O2—Sn1	124.0 (4)		
O1 ⁱ —Sn1—O1—C1	154.0 (6)	Cl2—Sn1—O1—Sn1 ⁱ	-25.5 (7)
O2—Sn1—O1—C1	64.8 (5)	O1 ⁱ —Sn1—O2—C2	42.9 (5)
Cl3—Sn1—O1—C1	-20.3 (5)	O1—Sn1—O2—C2	116.2 (5)
Cl1—Sn1—O1—C1	-114.3 (5)	Cl3—Sn1—O2—C2	-148.5 (5)
Cl2—Sn1—O1—C1	128.5 (6)	Cl2—Sn1—O2—C2	-50.4 (5)
O1 ⁱ —Sn1—O1—Sn1 ⁱ	0.0	Sn1—O2—C2—N1	-171.9 (5)
O2—Sn1—O1—Sn1 ⁱ	-89.1 (2)	C4—N1—C2—O2	1.6 (10)
Cl3—Sn1—O1—Sn1 ⁱ	-174.30 (17)	C3—N1—C2—O2	178.8 (7)
Cl1—Sn1—O1—Sn1 ⁱ	91.72 (19)		

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

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C1—H1C \cdots Cl3	0.96	2.72	3.356 (8)	124
C3—H3A \cdots Cl3 ⁱⁱ	0.96	2.95	3.895 (11)	170
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Symmetry codes: (ii) $x+1/2, -y+1/2, z+1/2$; (iii) $-x+3/2, y+1/2, -z+1/2$.

Fig. 1

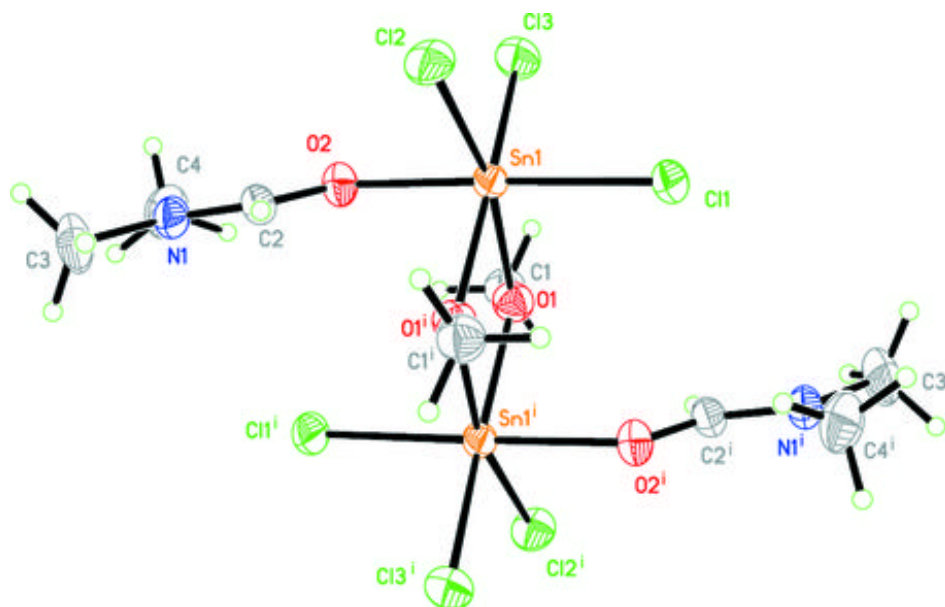


Fig. 2

