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## Structure Reports

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# Di- $\mu$ -methanolato- $\kappa^4$ O:O-bis[trichlorido-(dimethylformamide- $\kappa$ O)tin(IV)]

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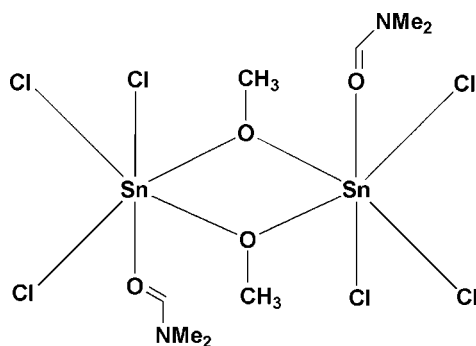
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Key indicators: single-crystal X-ray study;  $T = 298$  K; mean  $\sigma(\text{N}-\text{C}) = 0.011$  Å;  $R$  factor = 0.044;  $wR$  factor = 0.117; data-to-parameter ratio = 18.5.

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  $\text{Sn}^{\text{IV}}$  atoms symmetrically bridged by two deprotonated methanol ligands, with an inversion center in the middle of the planar  $\text{Sn}_2\text{O}_2$  ring. The other sites of the distorted octahedral coordination geometry of the  $\text{Sn}^{\text{IV}}$  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) Å<sup>3</sup> $Z = 2$ Mo  $K\alpha$  radiation $\mu = 3.05$  mm<sup>-1</sup> $T = 298$  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<sup>-3</sup> $\Delta\rho_{\text{min}} = -2.07$  e Å<sup>-3</sup>

Table 1

Selected bond lengths (Å).

Sn1—O1	2.106 (5)	Sn1—Cl1	2.372 (2)
Sn1—O1 <sup>i</sup>	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 <sup>ii</sup>	0.96	2.95	3.895 (11)	170
C4—H4B $\cdots$ Cl1 <sup>iii</sup>	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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## supporting information

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

**Di- $\mu$ -methanolato- $\kappa^4$ O:O-bis[trichlorido(dimethylformamide- $\kappa$ O)tin(IV)]**

Qijun Zhang, Handong Yin and Daqi Wang

**S1. Comment**

The title compound (Fig.1) consists of six Cl anions, two methoxo anions, two Sn<sup>IV</sup> ions and two dimethylformamide molecules. The molecule has an inversion center in the middle of the Sn<sub>2</sub>O<sub>2</sub> 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 [Sn1<sup>i</sup>—O1—Sn1 = 106.9 (2), O1<sup>i</sup>—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—O1<sup>i</sup> distance [2.101 (5) Å] (Table 1). Each Sn<sup>IV</sup> atom is hexacoordinated with two methoxo anions, three Cl anions and one dimethylformamide molecule in a distorted octahedral geometry.

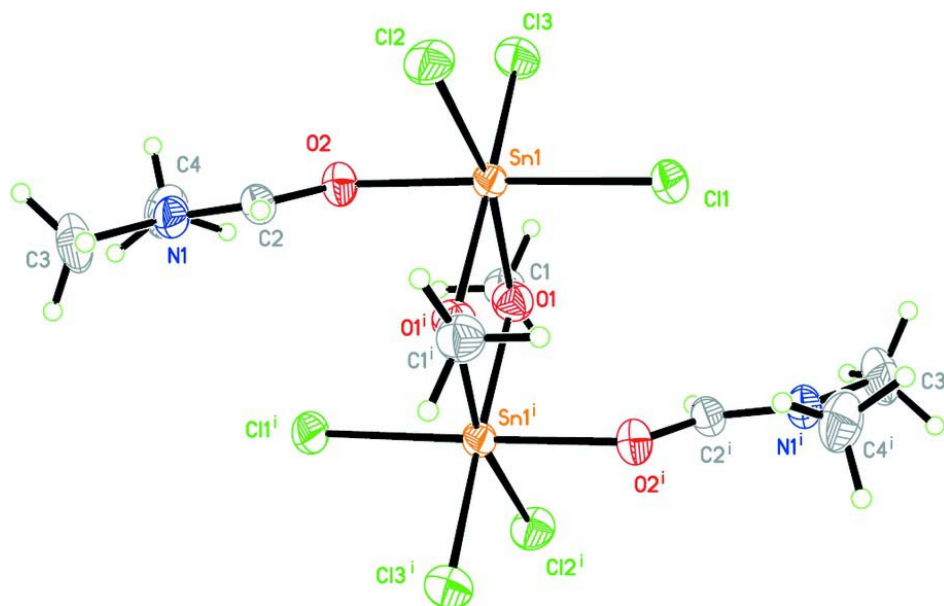
As is indicated from Fig. 2 and Table 2, the intramolecular interactions, C1—H1C···Cl3, strengthen the dimeric unit and the intermolecular ones, C3—H3A···Cl3<sup>ii</sup> and C4—H4B···Cl1<sup>iii</sup> [symmetry codes: (ii) x+1/2, -y+1/2, z+1/2; (iii) -x+3/2, y+1/2, -z+1/2], give rise to a two-dimensional polymer-like supramolecular network.

**S2. Experimental**

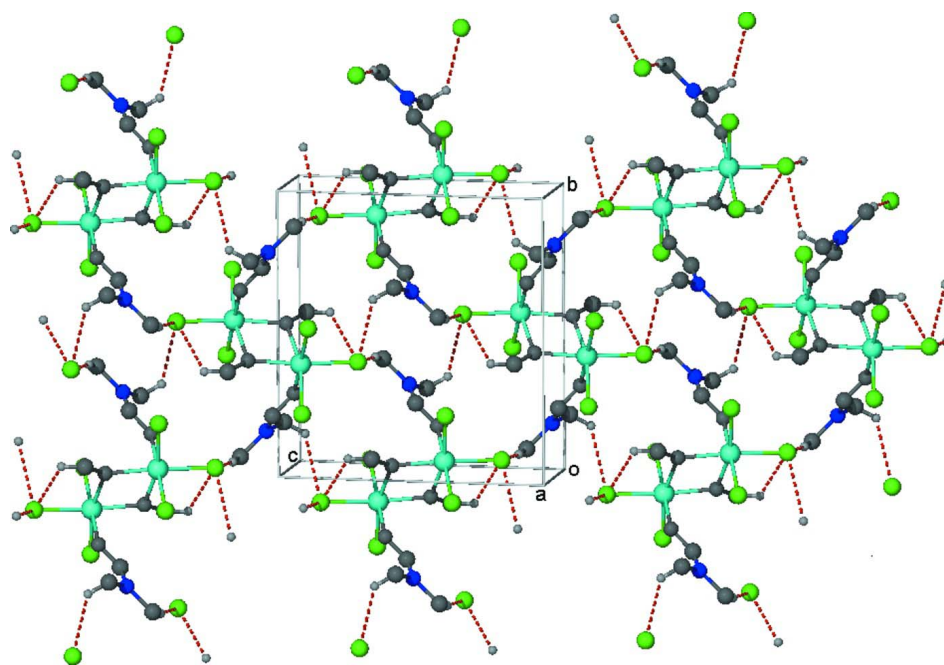
Stannic chloride hydrate (0.4 mmol, 0.14 g) was dissolved in methanol (20 ml) and dimethylformamide (5 ml) was added with stirring at room temperature. The mixture was allowed to react for 6 h and was then filtered. Colorless crystals suitable for X-ray analysis were obtained by slow evaporation of methanol over a period of two weeks (yield: 60%). Analysis, calculated for C<sub>8</sub>H<sub>20</sub>Cl<sub>6</sub>N<sub>2</sub>O<sub>4</sub>Sn<sub>2</sub>: C 14.59, H 3.06, N 4.25%; found: C 14.58, H 3.04, N 4.27%.

**S3. 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 (CH<sub>3</sub>) 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.

**Figure 1**

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

**Figure 2**

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

**Di- $\mu$ -methanolato- $\kappa^4$ O:O-bis[trichlorido(dimethylformamide- $\kappa$ O)tin(IV)]***Crystal data*[Sn<sub>2</sub>(CH<sub>3</sub>O)<sub>2</sub>Cl<sub>6</sub>(C<sub>3</sub>H<sub>7</sub>NO)<sub>2</sub>] $M_r = 658.34$ 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) Å<sup>3</sup> $Z = 2$  $F(000) = 632$  $D_x = 2.013$  Mg m<sup>-3</sup>Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 2694 reflections

 $\theta = 2.6$ – $27.0$ ° $\mu = 3.05$  mm<sup>-1</sup> $T = 298$  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 monochromator

 $\varphi$  and  $\omega$  scansAbsorption correction: multi-scan  
(SADABS; Sheldrick, 1996) $T_{\min} = 0.553$ ,  $T_{\max} = 0.641$ 

5512 measured reflections

1903 independent reflections

1554 reflections with  $I > 2\sigma(I)$  $R_{\text{int}} = 0.063$  $\theta_{\text{max}} = 25.0$ °,  $\theta_{\text{min}} = 2.6$ ° $h = -6 \rightarrow 10$  $k = -13 \rightarrow 12$  $l = -14 \rightarrow 14$ *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 methodsSecondary atom site location: difference Fourier  
mapHydrogen site location: inferred from  
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.065P)^2]$ where  $P = (F_o^2 + 2F_c^2)/3$  $(\Delta/\sigma)_{\text{max}} = 0.012$  $\Delta\rho_{\text{max}} = 1.54$  e Å<sup>-3</sup> $\Delta\rho_{\text{min}} = -2.07$  e Å<sup>-3</sup>*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å<sup>2</sup>)*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
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 (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
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 (Å, °)*

Sn1—O1	2.106 (5)	O2—C2	1.259 (7)
Sn1—O1 <sup>i</sup>	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
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 <sup>i</sup>	2.101 (5)	C4—H4C	0.9600
O1 <sup>i</sup> —Sn1—O2	87.65 (18)	O1—C1—H1A	109.5
O1 <sup>i</sup> —Sn1—O1	73.1 (2)	O1—C1—H1B	109.5
O2—Sn1—O1	84.69 (19)	H1A—C1—H1B	109.5
O1 <sup>i</sup> —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 <sup>i</sup> —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 <sup>i</sup> —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 <sup>i</sup>	124.2 (4)	N1—C4—H4C	109.5
C1—O1—Sn1	123.0 (4)	H4A—C4—H4C	109.5
Sn1 <sup>i</sup> —O1—Sn1	106.9 (2)	H4B—C4—H4C	109.5
C2—O2—Sn1	124.0 (4)		
O1 <sup>i</sup> —Sn1—O1—C1	154.0 (6)	Cl2—Sn1—O1—Sn1 <sup>i</sup>	-25.5 (7)
O2—Sn1—O1—C1	64.8 (5)	O1 <sup>i</sup> —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 <sup>i</sup> —Sn1—O1—Sn1 <sup>i</sup>	0.0	Sn1—O2—C2—N1	-171.9 (5)
O2—Sn1—O1—Sn1 <sup>i</sup>	-89.1 (2)	C4—N1—C2—O2	1.6 (10)
Cl3—Sn1—O1—Sn1 <sup>i</sup>	-174.30 (17)	C3—N1—C2—O2	178.8 (7)
Cl1—Sn1—O1—Sn1 <sup>i</sup>	91.72 (19)		

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

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

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

Symmetry codes: (ii)  $x+1/2, -y+1/2, z+1/2$ ; (iii)  $-x+3/2, y+1/2, -z+1/2$ .