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## Chlorodimethyl(*N*-methylpyrrolidinone)tin(IV)- $\mu$ -chloro-dichlorodimethyltin(IV)

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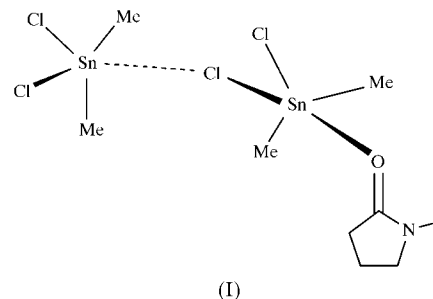
The synthesis and the X-ray structural analysis of the title compound,  $\mu$ -chloro-1:2 $\kappa^2$ Cl-trichloro-1 $\kappa$ Cl,2 $\kappa^2$ Cl-tetramethyl-1 $\kappa^2$ C,2 $\kappa^2$ C-(*N*-methylpyrrolidin-2-one)-1 $\kappa$ O-ditin(IV), [Sn<sub>2</sub>Cl<sub>4</sub>(CH<sub>3</sub>)<sub>4</sub>(C<sub>5</sub>H<sub>9</sub>NO)], are described. The title compound is found to exhibit a distorted trigonal-bipyramidal geometry at both Sn<sup>IV</sup> atoms. The Sn—Cl—Sn angle involving the bridging chlorine ligand is 135.56 (5)°, with the Sn—Cl bond lengths being 2.5704 (13) and 3.1159 (13) Å.

### Comment

One of the most interesting topics in structural organotin chemistry is the exhibition of pentacoordination at the Sn atom. The complexation of organotin compounds with *N*-methylpyrrolidinone (NMP) is well established. Articles describing molecular complexes derived from 1:1 and 1:2 complexation of dimethyltin dihalides by NMP have been published (König *et al.*, 2000*a,b*). A 2:1 complexation of dimethyltin dichloride by NMP should help in the investigation of whether NMP is able to react as a bidentate ligand, which would lead to two different pentacoordinated dimethyltin dihalide adducts. The results show that, as predicted, NMP belongs to the group of monodentate ligands. Nevertheless, there are two pentacoordinated Sn atoms (Sn1 and Sn2) in the crystal of the title compound, (I). Both exhibit a distorted trigonal-bipyramidal geometry.

Atom Sn2 has Cl4 and NMP as apical ligands with an angle of 177.35 (7)° close to the ideal angle of 180°. The deviation from ideal geometry is illustrated more clearly by the widened C3—Sn2—C4 angle of 146.29 (17)° and by the narrowed C3—Sn2—Cl3 and C4—Sn2—Cl3 angles of 106.58 (11) and 106.37 (13)°, respectively, compared with a value of 120° for an ideal angle in the equatorial plane. This plane contains two methyl groups and one chlorine. These ligands are somewhat displaced towards the axial NMP ligand. This becomes obvious on comparing the values of the angles between the

equatorial and axial ligands: (a) C3—Sn2—O1 89.91 (14)°, C4—Sn2—O1 83.59 (15)°, O1—Sn2—Cl3 89.24 (8)°; (b) C3—Sn2—Cl4 91.98 (12)°, C4—Sn2—Cl4 93.81 (14)°, Cl3—Sn2—Cl4 92.00 (5)°. The Sn—O bond length in this compound [2.278 (3) Å] is shorter than in other pentacoordinated tin complexes (Dey *et al.*, 1999; Cunningham *et al.*, 1993). The two Sn2—Cl bond lengths differ; Sn2—Cl3 2.3777 (13) Å and Sn2—Cl4 2.5704 (13) Å. The Sn—Cl bond in the equatorial plane is a little longer than in known pentacoordinated tin complexes (Dey *et al.*, 1999; Cunningham *et al.*, 1993; Einstein & Penfold, 1968). The long axial Sn—Cl bond may be due to its bridging character, similar features having been observed in the structure of the anion of [Me<sub>2</sub>SnCl(terpy)]<sup>+</sup>[Me<sub>2</sub>SnCl<sub>3</sub>]<sup>−</sup> (Einstein & Penfold, 1968). The values of the bond lengths and bond angles in the NMP ligand are comparable with those observed in other NMP coordinated organometallic compounds (Churchill & Rotella, 1979) and in free NMP (Müller *et al.*, 1996)



The other part of the complex also exhibits a distorted trigonal-bipyramidal coordination geometry around tin. Atom Sn1 has Cl1 and Cl4 as apical ligands with an angle of 175.67 (4)° close to the ideal angle of 180°. The deviation from ideal geometry is demonstrated most clearly by the widened C2—Sn1—Cl1 angle of 142.33 (17)° and by the narrowed C1—Sn1—Cl2 and C2—Sn2—Cl2 angles of 103.99 (12) and 105.64 (11)°, respectively, compared with a value of 120° for an ideal angle in the equatorial plane. This plane also consists of two methyl groups and one chlorine. The ligands are displaced towards the axial Cl4 ligand. This becomes evident when the angles between the axial and equatorial ligands are compared: (a) C1—Sn1—Cl4 82.82 (12)°, C2—Sn1—Cl4 76.30 (12)°, Cl2—Sn1—Cl4 86.58 (5)°; (b) C1—Sn1—Cl1 100.18 (12)°, C2—Sn1—Cl1 99.50 (12)°, Cl2—Sn1—Cl1 95.67 (5)°. There are three Sn—Cl bonds in this part of the complex represented by the following values: (a) Sn1—Cl1 2.4197 (12) Å, (b) Sn1—Cl2 2.3702 (13) Å, and (c) Sn1—Cl4 3.1159 (13) Å. As in the part of the complex described above, the bond between tin and the chlorine in the equatorial plane is a little longer than in known pentacoordinated tin complexes (Dey *et al.*, 1999; Cunningham *et al.*, 1993; Einstein & Penfold, 1968). On the other hand, the Sn—Cl1 bond is only a little longer than the Sn—Cl2 bond and therefore short.

Also, the bond between Sn1 and the bridging chlorine Cl4 is short compared with other complexes containing bridging Cl ligands such as Me<sub>3</sub>SnCl (Hossain *et al.*, 1979) and Me<sub>2</sub>SnCl<sub>2</sub> (Davies *et al.*, 1970).

## Experimental

The title compound was prepared by the reaction of *N*-methylpyrrolidinone (0.71 g, 0.69 ml, 7.3 mmol) with freshly sublimed dichlorodimethylstannane (3.20 g, 14.6 mmol) derived from the reaction of dimethyltin oxide with HCl (Pfeiffer, 1902) in 10 ml of dry diethyl ether. The reaction mixture was stirred for 30 min and afterwards stored in a refrigerator at 278 K. Colourless crystals are obtained in quantitative yield after filtration and drying *in vacuo* (m.p. 329 K). A solution of the complex (80 mg) in C<sub>6</sub>D<sub>6</sub> (470 mg) gives the following values for the structure-relevant NMR parameters: <sup>2</sup>J(<sup>119</sup>Sn, <sup>1</sup>H) = 79 Hz, <sup>1</sup>J(<sup>119</sup>Sn-<sup>13</sup>C) = 590 Hz and d(<sup>119</sup>Sn) = 30.1 p.p.m.

### Crystal data

[Sn<sub>2</sub>Cl<sub>4</sub>(CH<sub>3</sub>)<sub>4</sub>(C<sub>5</sub>H<sub>9</sub>NO)]  
*M<sub>r</sub>* = 538.45  
 Triclinic, *P*1  
*a* = 7.3650 (15) Å  
*b* = 10.311 (2) Å  
*c* = 12.311 (3) Å  
 α = 78.45 (3)°  
 β = 84.36 (3)°  
 γ = 84.38 (3)°  
*V* = 908.6 (3) Å<sup>3</sup>

*Z* = 2  
*D<sub>x</sub>* = 1.968 Mg m<sup>-3</sup>  
 Mo *K*α radiation  
 Cell parameters from 5442 reflections  
 θ = 2.38–27.06°  
 μ = 3.324 mm<sup>-1</sup>  
*T* = 173 (2) K  
 Parallelepiped, colourless  
 0.10 × 0.08 × 0.08 mm

### Data collection

Nonius KappaCCD diffractometer  
 Method: 278 frames *via* ω-rotation  
 (Δω = 1°) at different κ values  
 and two times 25 s per frame  
 5442 measured reflections  
 3557 independent reflections  
 2466 reflections with *I* > 2σ(*I*)

*R*<sub>int</sub> = 0.046  
 θ<sub>max</sub> = 27.06°  
*h* = -9 → 9  
*k* = -12 → 13  
*l* = -14 → 15  
 Intensity decay: none

### Refinement

Refinement on *F*<sup>2</sup>  
*R* [*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.030  
*wR* (*F*<sup>2</sup>) = 0.063  
*S* = 0.804  
 3557 reflections  
 159 parameters

H-atom parameters constrained  
*w* = 1/[σ<sup>2</sup>(*F<sub>o</sub>*<sup>2</sup>)]  
 where *P* = (*F<sub>o</sub>*<sup>2</sup> + 2*F<sub>c</sub>*<sup>2</sup>)/3  
 (Δ/σ)<sub>max</sub> = 0.012  
 Δρ<sub>max</sub> = 0.76 e Å<sup>-3</sup>  
 Δρ<sub>min</sub> = -0.85 e Å<sup>-3</sup>

**Table 1**

Selected geometric parameters (Å, °).

Sn2—C3	2.100 (4)	Sn1—C2	2.100 (4)
Sn2—C4	2.103 (4)	Sn1—C1	2.109 (4)
Sn2—O1	2.278 (3)	Sn1—Cl1	2.4197 (12)
Sn2—Cl3	2.3777 (13)	Sn1—Cl4	3.1159 (13)
Sn2—Cl4	2.5704 (13)		
C3—Sn2—C4	146.29 (17)	Cl3—Sn2—Cl4	92.00 (5)
C3—Sn2—O1	89.91 (14)	C2—Sn1—C1	142.33 (17)
C4—Sn2—O1	83.59 (15)	C2—Sn1—Cl2	105.64 (11)
C3—Sn2—Cl3	106.58 (11)	C1—Sn1—Cl2	103.99 (12)
C4—Sn2—Cl3	106.37 (13)	C2—Sn1—Cl1	99.50 (12)
O1—Sn2—Cl3	89.24 (8)	C1—Sn1—Cl1	100.18 (12)
C3—Sn2—Cl4	91.98 (12)	Cl2—Sn1—Cl1	95.67 (5)
C4—Sn2—Cl4	93.81 (14)	Sn1—Cl4—Sn2	135.56 (5)
O1—Sn2—Cl4	177.35 (7)		

The H atoms were placed in calculated positions with *U*<sub>iso</sub> constrained to be 1.2*U*<sub>eq</sub> of the carrier atom.

Data collection: *COLLECT* (Nonius, 1998); cell refinement and data reduction: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve and refine structure: *SHELXS97* (Sheldrick, 1990) and *SHELXL97* (Sheldrick, 1997); software used to prepare material for publication: *SHELXL97* and *PARST95* (Nardelli, 1995).

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