

Two coordination compounds of SnCl₂ with 4-methylpyridine *N*-oxide

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Received 8 December 2020

Accepted 1 January 2021

Edited by M. Weil, Vienna University of
Technology, Austria

Keywords: crystal structure; SnCl₂ coordination compounds; 4-methylpyridine *N*-oxide; bond-valence calculations; coordination geometry.

CCDC references: 2053460; 2053459

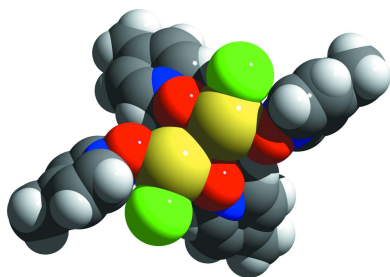
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In the solid-state structures of *catena*-poly[[dichloridotin(II)]- μ_2 -(4-methylpyridine *N*-oxide)- κ^2 O:O], [SnCl₂(C₆H₇NO)]_n, **1**, and dichloridobis(4-methylpyridine *N*-oxide- κ O)tin(II), [SnCl₂(C₆H₇NO)₂], **2**, the bivalent tin atoms reveal a seesaw coordination with both chlorine atoms in equatorial and the Lewis base molecules in axial positions. While the Sn–Cl distances are almost identical, the Sn–O distances vary significantly as a result of the different bonding modes (μ_2 for **1**, μ_1 for **2**) of the 4-methylpyridin-*N*-oxide molecules, giving rise to a one-dimensional coordination polymer for the 1:1 adduct, **1**, and a molecular structure for the 1:2 adduct, **2**. The different coordination modes also influence the bonding parameters within the almost planar ligand molecules, mostly expressed in N–O-bond lengthening and endocyclic bond-angle widening at the nitrogen atoms. Additional supramolecular features are found in the crystal structure of **2** as two adjacent molecules form dimers *via* additional, weak O \cdots Sn interactions.

1. Chemical context

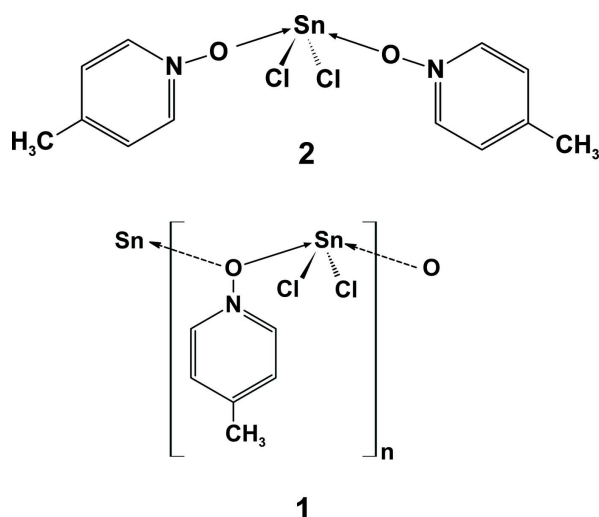
Tin(II) halides, SnHal₂, are nominally electron-deficient compounds and therefore strong Lewis acids. Corresponding Lewis acid/Lewis base adducts, however, have been structurally characterized in only small numbers so far. Examples are known with Lewis base molecules bearing nitrogen [SnCl₂·*t*BuNH₂ (Veith *et al.*, 1988)], phosphorus [SnCl₂·Ph₃P (Lukic *et al.*, 2019); SnHal₂·Et₃P (Arp *et al.*, 2013)], or sulfur [SnCl₂·thiourea (Harrison *et al.*, 1983)] atoms as possible donor atoms, but the most prominent ones are those with oxygen atoms. Triphenylphosphine oxide (TPPO), dimethylsulfoxide (DMSO) and *N,N*-dimethylformide (DMF) are representative examples for such O-bearing Lewis base molecules. Typically, the tin(II) dihalides form 1:1 adducts [*e.g.* SnHal₂·DMF with Hal = Cl, Br, I, and SnI₂·DMSO (Ozaki *et al.*, 2017)] where the tin(II) atoms in these complexes reach an electron octet by taking up the two donor electrons of the Lewis base molecule. In the case of 1:2 adducts [*e.g.* SnF₂·2DMSO (Gurnani *et al.*, 2013); SnCl₂·2TPPO (Selvaraju *et al.*, 1998); SnCl₂·2DMSO (Barbul *et al.*, 2011); SnBr₂·2DMSO, SnBr₂·2THF, SnBr₂·2acetone (Schrenk *et al.*, 2009)] the tin(II) atoms exceed the electron octet as a result of the two additional donor electrons. Both 1:1 and 1:2 compositions of one and the same tin(II) halide with one and the same Lewis base molecule have been previously reported only for SnI₂ with DMSO (Ozaki *et al.*, 2017).

Pyridin-*N*-oxide, PyNO, and its derivatives such as 4-methylpyridin-*N*-oxide, MePyNO, are excellent Lewis bases, which act as electron-pair donors *via* their exocyclic single-



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bonded oxygen atom in numerous inorganic and organometallic compounds of transition metals [*i.e.* CdHal₂·PyNO with Hal = Cl (Beyeh & Puttreddy, 2015), Hal = I (Sawitzki & von Schnering, 1974), CuCl₂·2MePyNO (Johnson & Watson, 1971), Ni(BF₄)₂·6PyNO (Ingen Schenau *et al.*, 1974), Au(CF₃)₃·PyNO (Pérez-Bitrián *et al.*, 2017), MoO(O₂)₂·2MePyNO (Griffith *et al.*, 1994)] as well as of *p*-block metals [*i.e.* TlBr₃·PyNO (Bermejo *et al.*, 1991); TlBr₃·2PyNO (Hiller *et al.*, 1988); TlBrI₂·MePyNO (Hiller *et al.*, 1988); SnI₄·2PyNO (Właźlak *et al.*, 2016), Me₂SnCl₂·2PyNO (Blom *et al.*, 1969), Ph₃SnCl·PyNO (Kumar *et al.*, 2020). With the exception of SbF₃·PyNO (Benjamin *et al.*, 2012) and BiI₃·PyNO (Właźlak *et al.*, 2020), no complexes of low-valent post-transition-metal elements have been crystallographically determined so far.



Here we report the crystal structures of two complexes of MePyNO with tin in oxidation state +II having different compositions, *viz.* SnCl₂·MePyNO, **1**, and SnCl₂·2MePyNO, **2**. Both compounds were obtained simultaneously in the same micro-scale experiment from SnCl₂ and MePyNO in excess using *N,N*-dimethylformamide as solvent. As reactions were performed on reaction plates we were able to inspect the progress of the reaction by microscopy, which allowed us to observe the intermediate compound formation as well as to study the crystal growth. No scaling-up experiments were performed but **1** has previously been mentioned in the literature with respect to its elemental analysis, X-ray-powder diffraction and IR data (Kauffman *et al.*, 1977), giving hints of a low symmetric crystal system and coordination number of three for tin. Mössbauer investigations have been performed by Ichiba *et al.* (1978).

2. Structural commentary

Compound **1** crystallizes in the monoclinic space group *P*2₁/*c*, and **2** in the orthorhombic space group *Pbcn*, each with one formula unit in the asymmetric unit and all atoms in general positions. In both compounds, the bivalent tin atoms adopt a seesaw coordination, which results from a μ_2 -coordination mode of the MePyNO-molecule in **1**, giving rise to a one-

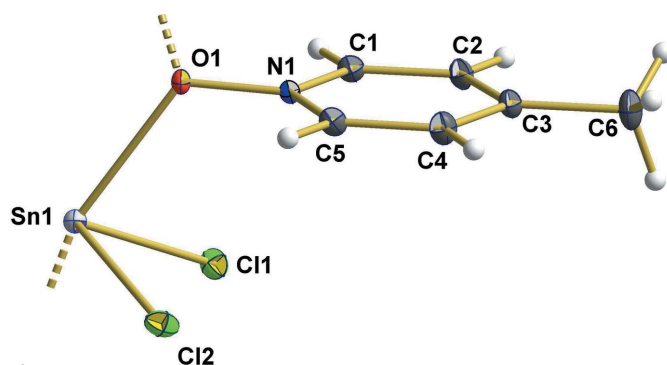


Figure 1
The asymmetric unit of SnCl₂·MePyNO, **1**, with the atom-numbering scheme; with the exception of the hydrogen atoms (which are shown as spheres with arbitrary radius) all atoms are drawn with displacement ellipsoids at the 40% probability level; longer Sn–O bonds expanding the coordination sphere of the tin(II) atom from three, trigonal-pyramidal, to four, seesaw, are drawn as dashed sticks.

dimensional coordination polymer along the *c* axis (Fig. 1) while there are two crystallographically different MePyNO molecules in **2**, resulting in a molecular structure (Fig. 2).

Distortion of the pyridine *N*-oxide ring system as a result of its coordination is established through the C–C [mean values: $d(C_{ortho}-C_{meta}) = 1.376(1) \text{ \AA}$, $d(C_{meta}-C_{para}) = 1.394(3) \text{ \AA}$] and the N–C bond lengths [mean value: $d(N-C) = 1.347(3) \text{ \AA}$], and through the endocyclic bond angles at the different carbon atoms [mean values: $C_{ortho} = 120.0(3)^\circ$, $C_{meta} = 120.8(2)^\circ$, $C_{para} = 117.1(2)^\circ$] of the almost planar ligand. While these structural parameters are almost identical in both compounds, the N–O bond lengths differ significantly in **1** [1.363(2) \AA] and **2** [1.333(3)/1.340(3) \AA] as do the endocyclic bond angles [121.9(1) $^\circ$, **1**; 120.9(1) $^\circ$, **2**] at the N atom. Both effects result from the different (μ_2 , μ_1) coordination modes of the ligands, which also affect the Sn–O bond lengths that are strongly unsymmetrical [2.280(1) to 2.733(2) \AA , μ_2] in **1**, and less unsymmetrical [2.308(2) to 2.423(2) \AA , μ_1] in **2**.

Irrespective of the controversial discussion on the hybridization ability of atomic orbitals in the case of the heavier *p*-block elements (Kutzelnigg, 1984), the formation of four-electron three-center bonds (Rundle, 1963), and on the functionality of the so-called 5*s* lone electron pair (Dénes *et al.*, 2013) in hypervalent (Musher, 1969) tin(II) compounds,

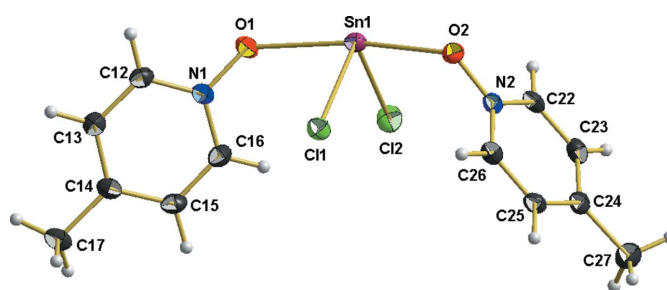


Figure 2
The asymmetric unit of SnCl₂·2MePyNO, **2**, with the atom-numbering scheme; with exception of the hydrogen atoms (which are shown as spheres with arbitrary radius) all atoms are drawn with displacement ellipsoids at the 40% probability level.

the fourfold coordination sphere around the tin(II) atoms of **1** and **2** can be expressed very well in terms of the VSEPR concept (Gillespie & Hargittai, 1991): its seesaw (*ss*) coordination results from two equatorially bonded chlorine atoms and two more electronegative and therefore axially located oxygen atoms of the Lewis base, MePyNO.

Differences in Sn–Cl distances are very small [2.4850 (4) and 2.4905 (4) Å, **1**; 2.4939 (6) and 2.5068 (6) Å, **2**, mean value: 2.494 (9) Å] as are the bond angles [95.73 (1)°, **1**; 94.59 (2)°, **2**] between them. Somewhat shorter Sn–Cl distances are found in the six crystallographically independent molecules of SnCl₂·DMF [$d(\text{Sn}-\text{Cl})_{\text{mean}} = 2.458$ (21) Å, $\langle(\text{Cl}-\text{Sn}-\text{Cl}) = 92.89$ (7)–89.09 (7)°] with a predominant trigonal–pyramidal coordination at tin, while the values in SnCl₂·2DMSO [$d(\text{Sn}-\text{Cl})_{\text{mean}} = 2.483$ (8) Å, $\langle(\text{Cl}-\text{Sn}-\text{Cl}) = 93.86$ (7)°] with a symmetrical seesaw coordination are in between.

Axes of the seesaws are bent [161.40 (6)°, **1**; 169.66 (6)°, **2**] towards the chlorine atoms properly due to electronic repulsion of the axial bonds through the 5s free-electron pairs. The corresponding Sn–O bonds are strongly different in both compounds, but differences are more expressed in **1** [2.280 (1) to 2.733 (1) Å, $\mu_2\text{-O}$] than in **2** [2.308 (2) to 2.430 (2) Å, $\mu_1\text{-O}$]. Because of the great [0.453 Å] difference between the two Sn–O bonds in **1**, one may suggest a threefold trigonal–pyramidal (*tpy*) tin(II) coordination instead of a fourfold seesaw (*ss*) coordination but valence-bond-sum calculations [parameters used: $r_o(\text{Sn}^{\text{II}}-\text{O}) = 1.984$ Å, $r_o(\text{Sn}^{\text{II}}-\text{Cl}) = 2.335$ Å, $b = 0.37$; Brese & O’Keefe (1991)] on the *tpy*

coordination result in a bond-valence sum of 1.78 v.u. while the longer Sn–O bond in the *ss*-coordination contributes 0.13 v.u. to the bond-valence sum (1.91 v.u.). Similar calculations for **2** result in a bond-valence sum of 2.00 v.u., fully consistent with the tin oxidation state of +II.

3. Supramolecular features

A common feature of many tin(II) compounds is the non-spherical ligand distribution around the divalent tin atom for which the term ‘hemidirected’ has been introduced (Shimoni-Livny *et al.*, 1998). The resulting void in the hemidirected coordination sphere often gives rise to additional more or less weak intermolecular (and intramolecular if appropriate Lewis base donor atoms are sterically available) interactions with interesting supramolecular features. In case of **1**, the formation of a one-dimensional coordination polymer *via* the $\mu_2\text{-O}$ -atom of the MePyNO molecule can be interpreted in terms of such supramolecular interactions: in this particular case, the hemidirected coordination sphere of a molecular, trigonal–pyramidal SnCl₂·MePyNO complex is partially filled through the oxygen atom of a MePyNO molecule of a neighboring building unit. The resulting coordination polymer forms a zigzag chain as all atoms are situated off the crystallographic glide plane at $x, 1/4, z$ (Fig. 3). Between the zigzag chains no further Lewis base/Lewis acid interactions below 3.5 Å are observed, but within the chains a very weak [3.460 (1) Å] attractive interaction is found between Cl2 and Sn1 of two neighboring building units (Fig. 3).

In case of **2** the tin atom of the SnCl₂·2MePyNO molecules shows a similar hemidirected coordination sphere. In the solid state, neighboring molecules form dimers *via* attractive but very weak [3.225 (2) Å] Sn–O interactions. Both molecules of these dimers are related to each other *via* a crystallographic twofold rotation axis (Fig. 4). Even if the coordination sphere of each tin atom remains unsymmetrical in these dimeric

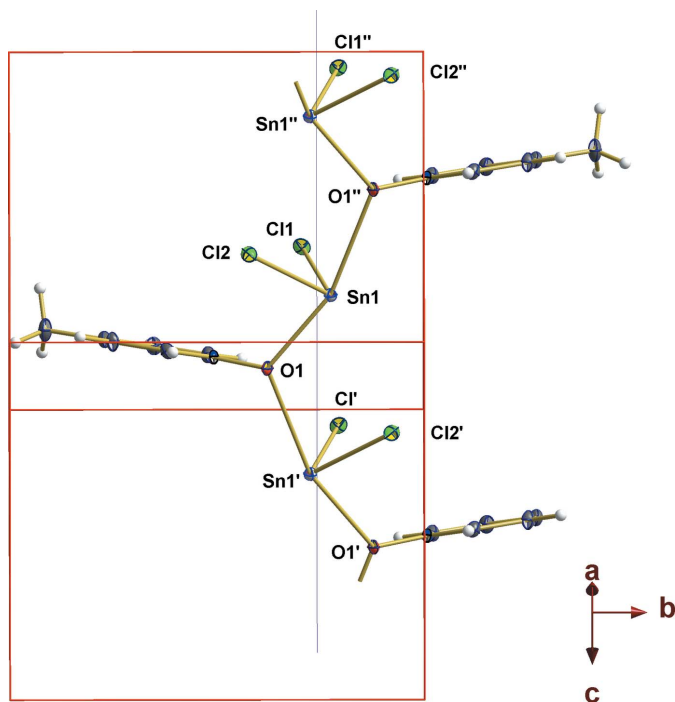


Figure 3

Ball-and-stick model of the one-dimensional coordination polymer of **1** viewed parallel to the glide plane (blue line); symmetry codes used to generate equivalent atoms: (') $x, \frac{3}{2} - y, \frac{1}{2} + z$; (')' $x, \frac{3}{2} - y, -\frac{1}{2} + z$.

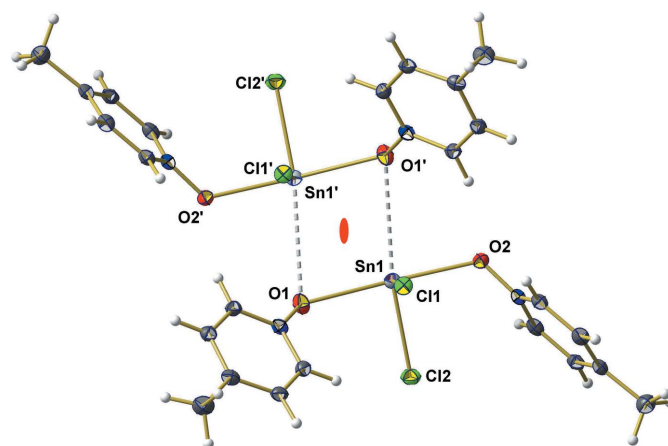


Figure 4

Ball-and-stick model of the dimeric aggregates found in the crystal structure of **2** looking down the crystallographic twofold rotation axis marked in red; additional Sn–O distances are indicated by dashed sticks in gray [symmetry codes used to generate equivalent atoms marked ' : $1 - x, y, \frac{3}{2} - z$].

Table 1
Experimental details.

	1	2
Crystal data		
Chemical formula	[SnCl ₂ (C ₆ H ₇ NO)]	[SnCl ₂ (C ₆ H ₇ NO) ₂]
<i>M_r</i>	298.72	407.84
Crystal system, space group	Monoclinic, <i>P</i> 2 ₁ / <i>c</i>	Orthorhombic, <i>Pbcn</i>
Temperature (K)	100	100
<i>a</i> , <i>b</i> , <i>c</i> (Å)	11.7934 (4), 9.4895 (3), 8.6170 (3)	19.9848 (8), 10.3723 (3), 14.4644 (5)
α , β , γ (°)	90, 106.455 (2), 90	90, 90, 90
<i>V</i> (Å ³)	924.86 (5)	2998.30 (18)
<i>Z</i>	4	8
Radiation type	Mo <i>K</i> α	Mo <i>K</i> α
μ (mm ⁻¹)	3.28	2.06
Crystal size (mm)	0.49 × 0.17 × 0.06	0.47 × 0.11 × 0.07
Data collection		
Diffractometer	Bruker APEXII CCD	Bruker APEXII CCD
Absorption correction	Multi-scan (<i>SADABS</i> ; Krause <i>et al.</i> , 2015)	Multi-scan (<i>SADABS</i> ; Krause <i>et al.</i> , 2015)
<i>T</i> _{min} , <i>T</i> _{max}	0.298, 0.817	0.442, 0.866
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	86401, 2232, 2132	136234, 3626, 3086
<i>R</i> _{int}	0.039	0.090
(<i>sin</i> θ / λ) _{max} (Å ⁻¹)	0.661	0.660
Refinement		
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.013, 0.032, 1.12	0.025, 0.064, 1.10
No. of reflections	2232	3626
No. of parameters	103	178
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å ⁻³)	0.41, -0.49	0.81, -0.33

Computer programs: *APEX2* and *SAINT* (Bruker, 2009), *SHELXS97* (Sheldrick, 2008), *SHELXL2014/7* (Sheldrick, 2015), *DIAMOND* (Brandenburg, 2006), *Mercury* (Macrae *et al.*, 2020) and *pubCIF* (Westrip, 2010).

aggregates (Fig. 5), no further intermolecular interactions could be observed below 3.5 Å.

4. Synthesis and crystallization

Both complexes are formed side by side on a reaction plate in the same micro-scale experiment when small amounts (about

100 mg) of SnCl₂ (Sigma–Aldrich) and an excess of 4-MePyNO (Alfa Aesar) are overlaid with a few drops of *N,N*-dimethylformamide (Fluka) as solvent. Compound **1** forms colorless, elongated plates, while **2** crystallizes in the form of small, colorless prisms.

5. Refinement

Crystal data, data collection and structure refinement details of **1** and **2** are summarized in Table 1. All H atoms were clearly identified in difference-Fourier syntheses but were refined with idealized positions and allowed to ride on their parent carbon atoms with 0.98 Å (–CH₃), and 0.95 Å (–CH_{arom}) and with common isotropic temperature factors for all hydrogen atoms of the aromatic rings and methyl groups.

Acknowledgements

We thank the Deutsche Forschungsgemeinschaft and the Government of Lower-Saxony for funding the diffractometer and acknowledge support by Deutsche Forschungsgemeinschaft (DFG) and Open Access Publishing Fund of Osnabrück University.

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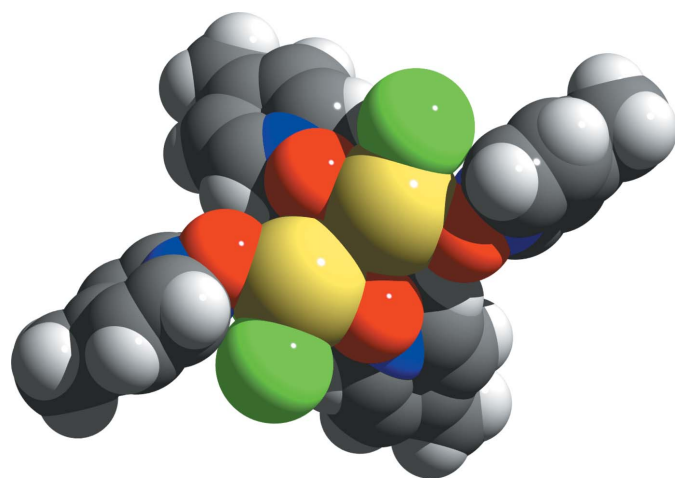


Figure 5
Space-filling model of the dimeric aggregates found in the crystal structure of **2** looking down into the voids on the backside of the tin atoms; color code used: Cl = green, O = red, N = blue, C = black, H = gray, Sn = yellow.

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supporting information

Acta Cryst. (2021). E77, 91-95 [https://doi.org/10.1107/S2056989021000025]

Two coordination compounds of SnCl₂ with 4-methylpyridine *N*-oxide

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Computing details

For both structures, data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINTE* (Bruker, 2009); data reduction: *SAINTE* (Bruker, 2009); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014/7* (Sheldrick, 2015); molecular graphics: *DIAMOND* (Brandenburg, 2006) and *Mercury* (Macrae *et al.*, 2020); software used to prepare material for publication: *publCIF* (Westrip, 2010).

catena-Poly[[dichloridotin(II)]- μ_2 -(4-methylpyridine *N*-oxide)- κ^2 O:O] (1)

Crystal data

[SnCl₂(C₆H₇NO)]

$M_r = 298.72$

Monoclinic, $P2_1/c$

$a = 11.7934$ (4) Å

$b = 9.4895$ (3) Å

$c = 8.6170$ (3) Å

$\beta = 106.455$ (2)°

$V = 924.86$ (5) Å³

$Z = 4$

$F(000) = 568$

$D_x = 2.145$ Mg m⁻³

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

Cell parameters from 9441 reflections

$\theta = 2.8$ – 28.3 °

$\mu = 3.28$ mm⁻¹

$T = 100$ K

Plate, colourless

$0.49 \times 0.17 \times 0.06$ mm

Data collection

Bruker APEXII CCD
diffractometer

φ and ω scans

Absorption correction: multi-scan
(SADABS; Krause *et al.*, 2015)

$T_{\min} = 0.298$, $T_{\max} = 0.817$

86401 measured reflections

2232 independent reflections

2132 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.039$

$\theta_{\max} = 28.0$ °, $\theta_{\min} = 3.3$ °

$h = -15 \rightarrow 15$

$k = -12 \rightarrow 12$

$l = -11 \rightarrow 11$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.013$

$wR(F^2) = 0.032$

$S = 1.12$

2232 reflections

103 parameters

0 restraints

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0134P)^2 + 0.6437P]$

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

$(\Delta/\sigma)_{\max} = 0.003$

$\Delta\rho_{\max} = 0.41$ e Å⁻³

$\Delta\rho_{\min} = -0.49$ e Å⁻³

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Sn1	0.65817 (2)	0.77494 (2)	0.40278 (2)	0.01249 (4)
Cl1	0.86766 (3)	0.70601 (4)	0.43743 (5)	0.01738 (8)
Cl2	0.57006 (3)	0.57820 (4)	0.21746 (4)	0.01829 (8)
O1	0.65673 (9)	0.62193 (11)	0.60614 (13)	0.0130 (2)
N1	0.70802 (11)	0.49228 (13)	0.61676 (15)	0.0114 (2)
C2	0.82254 (13)	0.47923 (17)	0.70246 (18)	0.0142 (3)
H2	0.8666	0.5598	0.7501	0.021 (2)*
C3	0.87519 (14)	0.34877 (17)	0.72050 (19)	0.0157 (3)
H3	0.9558	0.3397	0.7812	0.021 (2)*
C4	0.81191 (14)	0.22964 (16)	0.65083 (19)	0.0146 (3)
C5	0.69385 (14)	0.24895 (17)	0.5615 (2)	0.0166 (3)
H5	0.6482	0.1704	0.5111	0.021 (2)*
C6	0.64331 (13)	0.38104 (16)	0.54603 (18)	0.0143 (3)
H6	0.5630	0.3934	0.4856	0.021 (2)*
C7	0.86871 (15)	0.08689 (17)	0.6700 (2)	0.0215 (3)
H71	0.9108	0.0742	0.5880	0.051 (4)*
H72	0.8077	0.0141	0.6566	0.051 (4)*
H73	0.9247	0.0790	0.7781	0.051 (4)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sn1	0.01487 (6)	0.01142 (6)	0.01145 (6)	0.00176 (4)	0.00415 (4)	0.00048 (4)
Cl1	0.01430 (17)	0.01858 (18)	0.01902 (18)	0.00012 (14)	0.00435 (14)	0.00240 (14)
Cl2	0.02132 (18)	0.01852 (18)	0.01377 (17)	-0.00592 (15)	0.00295 (14)	-0.00073 (14)
O1	0.0161 (5)	0.0100 (5)	0.0138 (5)	0.0050 (4)	0.0055 (4)	0.0021 (4)
N1	0.0125 (6)	0.0099 (6)	0.0120 (6)	0.0021 (5)	0.0036 (5)	0.0018 (5)
C2	0.0134 (7)	0.0136 (7)	0.0142 (7)	-0.0013 (6)	0.0018 (6)	-0.0007 (6)
C3	0.0123 (7)	0.0157 (7)	0.0170 (7)	0.0016 (6)	0.0008 (6)	0.0017 (6)
C4	0.0157 (7)	0.0113 (7)	0.0163 (7)	0.0009 (6)	0.0038 (6)	0.0012 (6)
C5	0.0145 (7)	0.0133 (7)	0.0197 (8)	-0.0025 (6)	0.0011 (6)	-0.0009 (6)
C6	0.0122 (7)	0.0146 (7)	0.0152 (7)	-0.0018 (6)	0.0023 (6)	0.0008 (6)
C7	0.0194 (8)	0.0121 (7)	0.0300 (9)	0.0024 (6)	0.0022 (7)	0.0006 (7)

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

Sn1—O1	2.2795 (10)	C3—H3	0.9500
Sn1—Cl2	2.4850 (4)	C4—C5	1.399 (2)
Sn1—Cl1	2.4905 (4)	C4—C7	1.499 (2)

O1—N1	1.3626 (16)	C5—C6	1.378 (2)
N1—C6	1.343 (2)	C5—H5	0.9500
N1—C2	1.3490 (19)	C6—H6	0.9500
C2—C3	1.374 (2)	C7—H71	0.9800
C2—H2	0.9500	C7—H72	0.9800
C3—C4	1.393 (2)	C7—H73	0.9800
O1—Sn1—Cl2	85.56 (3)	C3—C4—C7	121.16 (14)
O1—Sn1—Cl1	87.92 (3)	C5—C4—C7	121.53 (14)
Cl2—Sn1—Cl1	95.726 (14)	C6—C5—C4	120.54 (15)
N1—O1—Sn1	121.80 (8)	C6—C5—H5	119.7
C6—N1—C2	121.88 (13)	C4—C5—H5	119.7
C6—N1—O1	119.66 (12)	N1—C6—C5	119.76 (14)
C2—N1—O1	118.44 (12)	N1—C6—H6	120.1
N1—C2—C3	119.67 (14)	C5—C6—H6	120.1
N1—C2—H2	120.2	C4—C7—H71	109.5
C3—C2—H2	120.2	C4—C7—H72	109.5
C2—C3—C4	120.83 (14)	H71—C7—H72	109.5
C2—C3—H3	119.6	C4—C7—H73	109.5
C4—C3—H3	119.6	H71—C7—H73	109.5
C3—C4—C5	117.31 (14)	H72—C7—H73	109.5

Dichloridobis(4-methylpyridine *N*-oxide- κ O)tin(II) (2)

Crystal data

[SnCl₂(C₆H₇NO)₂]

$M_r = 407.84$

Orthorhombic, *Pbcn*

$a = 19.9848$ (8) Å

$b = 10.3723$ (3) Å

$c = 14.4644$ (5) Å

$V = 2998.30$ (18) Å³

$Z = 8$

$F(000) = 1600$

Data collection

Bruker APEXII CCD
diffractometer

φ and ω scans

Absorption correction: multi-scan
(SADABS; Krause *et al.*, 2015)

$T_{\min} = 0.442$, $T_{\max} = 0.866$

136234 measured reflections

$D_x = 1.807$ Mg m⁻³

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

Cell parameters from 9991 reflections

$\theta = 2.6$ – 27.2°

$\mu = 2.06$ mm⁻¹

$T = 100$ K

Rod, colourless

$0.47 \times 0.11 \times 0.07$ mm

3626 independent reflections

3086 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.090$

$\theta_{\max} = 28.0^\circ$, $\theta_{\min} = 2.6^\circ$

$h = -26 \rightarrow 26$

$k = -13 \rightarrow 13$

$l = -19 \rightarrow 19$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.025$

$wR(F^2) = 0.064$

$S = 1.10$

3626 reflections

178 parameters

0 restraints

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0279P)^2 + 2.7839P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$

$$\Delta\rho_{\max} = 0.81 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.33 \text{ e } \text{\AA}^{-3}$$

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Sn1	0.55158 (2)	0.16754 (2)	0.64760 (2)	0.02085 (6)
Cl1	0.55438 (3)	0.40624 (6)	0.62730 (4)	0.02477 (13)
Cl2	0.66633 (3)	0.13226 (7)	0.58237 (5)	0.03087 (14)
O1	0.60299 (9)	0.21147 (17)	0.79652 (12)	0.0288 (4)
N1	0.63657 (10)	0.31475 (19)	0.82579 (14)	0.0218 (4)
C12	0.62539 (12)	0.3580 (2)	0.91192 (17)	0.0229 (5)
H12	0.5917	0.3191	0.9489	0.029 (4)*
C13	0.66263 (11)	0.4585 (2)	0.94668 (17)	0.0231 (5)
H13	0.6548	0.4874	1.0081	0.029 (4)*
C14	0.71145 (12)	0.5186 (2)	0.89344 (17)	0.0233 (5)
C15	0.71995 (11)	0.4734 (2)	0.80348 (17)	0.0242 (5)
H15	0.7518	0.5135	0.7641	0.029 (4)*
C16	0.68266 (12)	0.3714 (2)	0.77119 (17)	0.0243 (5)
H16	0.6894	0.3407	0.7100	0.029 (4)*
C17	0.75292 (14)	0.6270 (3)	0.9311 (2)	0.0335 (6)
H17A	0.7345	0.7095	0.9100	0.068 (7)*
H17B	0.7991	0.6183	0.9092	0.068 (7)*
H17C	0.7523	0.6241	0.9989	0.068 (7)*
O2	0.50452 (9)	0.16530 (16)	0.50191 (12)	0.0236 (4)
N2	0.53989 (9)	0.18673 (19)	0.42470 (13)	0.0193 (4)
C22	0.57330 (12)	0.0882 (2)	0.38493 (17)	0.0218 (5)
H22	0.5738	0.0058	0.4137	0.023 (3)*
C23	0.60658 (11)	0.1070 (2)	0.30287 (17)	0.0236 (5)
H23	0.6299	0.0371	0.2753	0.023 (3)*
C24	0.60656 (12)	0.2270 (2)	0.25955 (17)	0.0235 (5)
C25	0.57304 (13)	0.3268 (2)	0.30435 (17)	0.0230 (5)
H25	0.5728	0.4106	0.2778	0.023 (3)*
C26	0.54027 (12)	0.3055 (2)	0.38668 (18)	0.0214 (5)
H26	0.5179	0.3745	0.4168	0.023 (3)*
C27	0.64072 (15)	0.2477 (3)	0.16818 (19)	0.0350 (6)
H27A	0.6890	0.2567	0.1780	0.097 (10)*
H27B	0.6233	0.3261	0.1392	0.097 (10)*
H27C	0.6322	0.1736	0.1278	0.097 (10)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sn1	0.02042 (9)	0.01901 (10)	0.02311 (9)	-0.00041 (6)	0.00035 (6)	0.00205 (6)
C11	0.0277 (3)	0.0191 (3)	0.0275 (3)	0.0016 (2)	0.0005 (2)	0.0005 (2)
C12	0.0220 (3)	0.0358 (3)	0.0348 (3)	0.0041 (2)	0.0026 (2)	-0.0067 (3)
O1	0.0351 (10)	0.0244 (9)	0.0270 (9)	-0.0098 (8)	-0.0062 (8)	0.0017 (7)
N1	0.0213 (10)	0.0195 (10)	0.0247 (10)	-0.0023 (8)	-0.0028 (8)	0.0013 (8)
C12	0.0193 (11)	0.0250 (12)	0.0243 (12)	0.0016 (9)	0.0014 (9)	0.0039 (9)
C13	0.0218 (11)	0.0237 (12)	0.0240 (11)	0.0028 (9)	0.0002 (9)	0.0005 (9)
C14	0.0208 (11)	0.0191 (12)	0.0298 (12)	0.0008 (9)	-0.0008 (10)	0.0018 (10)
C15	0.0177 (10)	0.0270 (13)	0.0280 (12)	-0.0001 (9)	0.0013 (9)	0.0066 (10)
C16	0.0213 (11)	0.0289 (12)	0.0227 (12)	0.0016 (10)	0.0017 (9)	0.0031 (10)
C17	0.0350 (14)	0.0257 (13)	0.0398 (15)	-0.0099 (11)	0.0013 (12)	-0.0001 (12)
O2	0.0227 (8)	0.0262 (9)	0.0218 (8)	-0.0026 (7)	0.0007 (7)	0.0017 (7)
N2	0.0184 (9)	0.0193 (10)	0.0202 (10)	-0.0010 (7)	-0.0036 (7)	-0.0004 (7)
C22	0.0226 (11)	0.0149 (11)	0.0277 (12)	-0.0015 (9)	-0.0062 (9)	-0.0008 (9)
C23	0.0198 (11)	0.0231 (12)	0.0278 (12)	0.0021 (9)	-0.0037 (9)	-0.0066 (10)
C24	0.0189 (11)	0.0292 (13)	0.0225 (11)	-0.0031 (10)	-0.0038 (9)	-0.0023 (10)
C25	0.0249 (11)	0.0177 (11)	0.0262 (12)	-0.0030 (9)	-0.0031 (10)	0.0024 (9)
C26	0.0214 (11)	0.0183 (11)	0.0246 (11)	0.0022 (9)	-0.0033 (9)	-0.0007 (9)
C27	0.0352 (14)	0.0400 (16)	0.0298 (14)	-0.0019 (13)	0.0035 (11)	0.0004 (12)

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

Sn1—O2	2.3078 (17)	C17—H17B	0.9800
Sn1—O1	2.4296 (17)	C17—H17C	0.9800
Sn1—C11	2.4939 (6)	O2—N2	1.340 (3)
Sn1—C12	2.5068 (6)	N2—C26	1.349 (3)
O1—N1	1.333 (3)	N2—C22	1.350 (3)
N1—C12	1.343 (3)	C22—C23	1.375 (4)
N1—C16	1.348 (3)	C22—H22	0.9500
C12—C13	1.377 (3)	C23—C24	1.393 (4)
C12—H12	0.9500	C23—H23	0.9500
C13—C14	1.390 (3)	C24—C25	1.393 (3)
C13—H13	0.9500	C24—C27	1.503 (4)
C14—C15	1.393 (3)	C25—C26	1.377 (4)
C14—C17	1.500 (3)	C25—H25	0.9500
C15—C16	1.376 (3)	C26—H26	0.9500
C15—H15	0.9500	C27—H27A	0.9800
C16—H16	0.9500	C27—H27B	0.9800
C17—H17A	0.9800	C27—H27C	0.9800
O2—Sn1—O1	169.66 (6)	C14—C17—H17C	109.5
O2—Sn1—C11	84.93 (4)	H17A—C17—H17C	109.5
O1—Sn1—C11	84.76 (4)	H17B—C17—H17C	109.5
O2—Sn1—C12	91.58 (5)	N2—O2—Sn1	122.98 (13)
O1—Sn1—C12	88.52 (5)	O2—N2—C26	119.6 (2)

C11—Sn1—C12	94.59 (2)	O2—N2—C22	119.37 (19)
N1—O1—Sn1	130.18 (14)	C26—N2—C22	121.0 (2)
O1—N1—C12	118.6 (2)	N2—C22—C23	120.0 (2)
O1—N1—C16	120.5 (2)	N2—C22—H22	120.0
C12—N1—C16	120.8 (2)	C23—C22—H22	120.0
N1—C12—C13	120.1 (2)	C22—C23—C24	121.0 (2)
N1—C12—H12	119.9	C22—C23—H23	119.5
C13—C12—H12	119.9	C24—C23—H23	119.5
C12—C13—C14	121.1 (2)	C25—C24—C23	117.1 (2)
C12—C13—H13	119.4	C25—C24—C27	121.4 (2)
C14—C13—H13	119.4	C23—C24—C27	121.5 (2)
C13—C14—C15	116.9 (2)	C26—C25—C24	120.7 (2)
C13—C14—C17	121.5 (2)	C26—C25—H25	119.6
C15—C14—C17	121.6 (2)	C24—C25—H25	119.6
C16—C15—C14	120.6 (2)	N2—C26—C25	120.2 (2)
C16—C15—H15	119.7	N2—C26—H26	119.9
C14—C15—H15	119.7	C25—C26—H26	119.9
N1—C16—C15	120.4 (2)	C24—C27—H27A	109.5
N1—C16—H16	119.8	C24—C27—H27B	109.5
C15—C16—H16	119.8	H27A—C27—H27B	109.5
C14—C17—H17A	109.5	C24—C27—H27C	109.5
C14—C17—H17B	109.5	H27A—C27—H27C	109.5
H17A—C17—H17B	109.5	H27B—C27—H27C	109.5
