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# Synthesis, structure determination and characterization by UV-Vis and IR spectroscopy of bis(diisopropylammonium) cis-dichloridobis-(oxalato-k ${ }^{2} \mathrm{O}^{1}, \mathrm{O}^{2}$ )stannate(IV) 

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The organic-inorganic title salt, $\left(\mathrm{C}_{6} \mathrm{H}_{16} \mathrm{~N}\right)_{2}\left[\mathrm{Sn}\left(\mathrm{C}_{2} \mathrm{O}_{4}\right)_{2} \mathrm{Cl}_{2}\right]$ or $\left({ }^{i} \mathrm{Pr}_{2} \mathrm{NH}_{2}\right)_{2}$ [ $\mathrm{Sn}\left(\mathrm{C}_{2} \mathrm{O}_{4}\right)_{2} \mathrm{Cl}_{2}$ ], was obtained by reacting bis(diisopropylammonium) oxalate with $\operatorname{tin}(\mathrm{IV})$ chloride dihydrate in methanol. The $\mathrm{Sn}^{\mathrm{IV}}$ atom is coordinated by two chelating oxalate ligands and two chloride ions in cis positions, giving rise to an $\left[\mathrm{Sn}\left(\mathrm{C}_{2} \mathrm{O}_{4}\right)_{2} \mathrm{Cl}_{2}\right]^{2-}$ anion (point group symmetry 2), with the $\mathrm{Sn}^{\mathrm{IV}}$ atom in a slightly distorted octahedral coordination. The cohesion of the crystal structure is ensured by the formation of $\mathrm{N}-\mathrm{H} \cdots \mathrm{O}$ hydrogen bonding between $\left({ }^{\mathrm{i}} \mathrm{Pr}_{2} \mathrm{NH}_{2}\right)^{+}$cations and $\left[\mathrm{SnCl}_{2}\left(\mathrm{C}_{2} \mathrm{O}_{4}\right)_{2}\right]^{2-}$ anions. This gives rise to an infinite chain structure extending parallel to [101]. The main inter-chain interactions are van der Waals forces. The electronic spectrum of the title compound displays only one high intensity band in the UV region assignable to ligand-metal ion charge-transfer (LMCT) transitions. An IR spectrum was also recorded and is discussed.

## 1. Chemical context

As a result of their numerous applications in medicine, industry and agriculture (Kapoor et al., 2005), tin(IV) carboxylate compounds have attracted the attention of several research groups, resulting in the preparation and characterization of new compounds (Christie et al., 1979; Ng \& Kumar Das, 1993; Rocamora-Reverte et al., 2012; Reichelt \& Reuter, 2014). Derivatives of $\operatorname{tin}($ IV ) oxalate are a subclass of the tin(IV) carboxylate family and have likewise been studied extensively because oxalate ions, $\mathrm{C}_{2} \mathrm{O}_{4}{ }^{2-}$, play an important role as counter-ions or complex ligands in inorganic as well as in organometallic chemistry. One of the motivations to study these compounds is related to the rich coordinating behaviour of the oxalato ligand, which can adopt a monodentate, a bridging monodentate, a bridging bidentate, a monochelating, bidentate or a bichelating mode (Miskelly et al., 1983; Sow et al., 2012; Świtlicka-Olszewska et al., 2014). In this context, our group has previously published the syntheses and crystal structure determinations of some $\operatorname{tin}(I V)$ oxalate derivatives (Sarr et al., 2013, 2018). As a continuation of this work, we have studied the interaction between bis(diisopropylammonium) oxalate with $\operatorname{tin}(\mathrm{IV})$ chloride dihydrate, which yielded the title salt $\left(\mathrm{C}_{6} \mathrm{H}_{16} \mathrm{~N}\right)_{2}\left[\mathrm{Sn}\left(\mathrm{C}_{2} \mathrm{O}_{4}\right)_{2} \mathrm{Cl}_{2}\right]$ or $\left({ }^{i} \mathrm{Pr}_{2} \mathrm{NH}_{2}\right)_{2}\left[\mathrm{Sn}\left(\mathrm{C}_{2} \mathrm{O}_{4}\right)_{2} \mathrm{Cl}_{2}\right]$. Its crystal structure was deter-
mined by single crystal X-ray diffraction and was confirmed by infrared and UV-visible spectroscopic studies.


## 2. Structural commentary

The asymmetric unit of $\left({ }^{i} \mathrm{Pr}_{2} \mathrm{NH}_{2}\right)_{2}\left[\mathrm{Sn}\left(\mathrm{C}_{2} \mathrm{O}_{4}\right)_{2} \mathrm{Cl}_{2}\right]$ comprises one diisopropylammonium cation and one half of an $\left[\mathrm{Sn}\left(\mathrm{C}_{2} \mathrm{O}_{4}\right)_{2} \mathrm{Cl}_{2}\right]^{2-}$ anion, the other half being completed by the application of twofold rotation symmetry (Fig. 1) with the rotation axis running through the central $\mathrm{Sn}^{\mathrm{IV}}$ atom. The latter is chelated by two oxalate ligands and is additionally ligated by two Cl atoms in cis positions within a distorted octahedral coordination sphere $\left[\mathrm{Cl} 1{ }^{\mathrm{i}}-\mathrm{Sn} 1-\mathrm{Cl} 1=97.26(4)^{\circ}\right.$; $\mathrm{O} 1-\mathrm{Sn} 1-$ $\mathrm{O} 4=\mathrm{O} 1^{\mathrm{i}}-\mathrm{Sn} 1-\mathrm{O} 4^{\mathrm{i}}=79.27(6)^{\circ}, \mathrm{O} 1-\mathrm{Sn} 1-\mathrm{O} 4^{\mathrm{i}}=90.21(6)^{\circ}$; symmetry code: (i) $\left.x+\frac{1}{2},-y+\frac{3}{2}, z-\frac{1}{2}\right]$. Atoms $\mathrm{Cl1}^{\mathrm{i}}, \mathrm{O} 1, \mathrm{O} 1^{\mathrm{i}}$ and O4 define the equatorial plane [with a slight shift of Sn1 from this plane by 0.1163 (5) $\AA$; rms $=0.0687 \AA$ ] while Cl 1 and $\mathrm{O} 4^{\mathrm{i}}$ occupy the axial positions. The $\mathrm{O} 4^{\mathrm{i}}-\mathrm{Sn} 1-\mathrm{Cl} 1$ angle of $168.50(4)^{\circ}$ indicates a considerable deviation from linearity, which might be explained by the difference in size of the Cl and O atoms and by the small bite angle of $79.27(6)^{\circ}$ between the central $\mathrm{Sn}^{\mathrm{IV}}$ atom and the chelating O 1 and O 4 atoms.

As in the related structure of $\left({ }^{i} \mathrm{Pr}_{2} \mathrm{NH}_{2}\right)_{2}\left[\mathrm{Sn}\left(\mathrm{C}_{2} \mathrm{O}_{4}\right)_{2} \mathrm{I}_{2}\right]$ (Sarr et al., 2018), the lengths of the $\mathrm{C}-\mathrm{O}$ bonds within the oxalate ligands vary slightly because of the different functions of the oxygen atoms involved in the coordination of $\mathrm{Sn}^{\mathrm{IV}}$. The $\mathrm{C}-\mathrm{O}$ bond lengths of coordinating O atoms $[\mathrm{O} 1-\mathrm{C} 7=1.289$ (2) $\AA$; $\mathrm{O} 4-\mathrm{C} 8=1.289(2) \AA$ A $]$ are significantly longer than those of non-coordinating O atoms $[\mathrm{O} 2-\mathrm{C} 7=1.222(2) \AA, \mathrm{O} 3-\mathrm{C} 8=$ 1.223 (2) $\AA$ ]. The Sn1-C11 distance of 2.3422 (9) $\AA$ as well as


Figure 1
The molecular entities in the organic-inorganic title salt drawn with displacement ellipsoids at the $50 \%$ probability level; hydrogen atoms are depicted as spheres of arbitrary radius

Table 1
Hydrogen-bond geometry ( $\AA{ }^{\circ},{ }^{\circ}$ ).

| $D-\mathrm{H} \cdots A$ | $D-\mathrm{H}$ | $\mathrm{H} \cdots A$ | $D \cdots A$ | $D-\mathrm{H} \cdots A$ |
| :--- | :--- | :--- | :--- | :--- |
| $\mathrm{~N} 1-\mathrm{H} 1 A \cdots \mathrm{O}^{\mathrm{i}}$ | 0.91 | 2.05 | $2.943(2)$ | 167 |
| $\mathrm{~N} 1-\mathrm{H} 1 B \cdots \mathrm{O}^{\mathrm{ii}}$ | 0.91 | 1.95 | $2.855(2)$ | 177 |

Symmetry codes: (i) $x+\frac{1}{2},-y+\frac{3}{2}, z-\frac{1}{2}$; (ii) $-x+1, y,-z+\frac{3}{2}$.
the $\mathrm{Sn}-\mathrm{O}$ distances of $2.0710(16) \AA$ (O1) and of 2.1057 (15) $\AA$ (O4) are slightly shorter than corresponding bonds reported previously (Reichelt \& Reuter, 2014; Sarr et al., 2013; Diop et al., 2011; Sow et al., 2013; Skapski et al., 1974).

## 3. Supramolecular features

Each anionic complex $\left[\mathrm{Sn}\left(\mathrm{C}_{2} \mathrm{O}_{4}\right)_{2} \mathrm{Cl}_{2}\right]^{2-}$ is linked to two neighbours via four diisopropylammonium cations through $\mathrm{N}-\mathrm{H} \cdots \mathrm{O}$ hydrogen bonds, leading to infinite chains parallel to [101] (Table 1, Fig. 2). In a chain, the two non-coordinating oxygen atoms ( O 2 and O 3 ) of each oxalate ligand are involved as acceptors in hydrogen-bonding interactions (Table 1). The chains are arranged into layers extending parallel to (010), mainly interconnected by van der Waals forces (Fig. 3).

## 4. Database survey

A search in the Cambridge Structural Database (CSD, version 5.40, update Nov. 2018; Groom et al., 2016) resulted in 226 hits dealing with diisopropylammonium cations while only one hit deals with the $\left[\mathrm{Sn}\left(\mathrm{C}_{2} \mathrm{O}_{4}\right)_{2} \mathrm{Cl}_{2}\right]^{2-}$ anion (Sarr et al., 2013).


Figure 2
An infinite chain in the title structure, showing the $\mathrm{N}-\mathrm{H} \cdots \mathrm{O}$ hydrogen bonds as light-blue dashed lines. C-bound H atoms have been omitted for clarity.


Figure 3
The crystal packing of the title compound in a view down [001]. C-bound H atoms have been omitted for clarity.


The IR spectrum of the title compound.

## 5. Synthesis and crystallization

The title salt was obtained by mixing bis(diisopropylammonium) oxalate ( $\left.\left.{ }^{i} \mathrm{Pr}_{2} \mathrm{NH}_{2}\right)_{2} \mathrm{C}_{2} \mathrm{O}_{4} ; 0.30 \mathrm{~g} ; 15.50 \mathrm{mmol}\right)$ and $\operatorname{tin}(\mathrm{IV})$ chloride dihydrate $\left(\mathrm{SnCl}_{2} \cdot 2 \mathrm{H}_{2} \mathrm{O} ;(0.34 \mathrm{~g} ; 15.50 \mathrm{mmol})\right.$ in a 1:1 molar ratio in methanol. The obtained yellow solution was stirred for one $h$ and then filtered. Colourless prism-like crystals were obtained by slow evaporation of the filtrate over a period of ten days.

The IR spectrum confirms the presence of oxalate and diisopropylammonium groups in the title salt. In addition, the appearance of valence vibrations $\left(-\mathrm{CO}_{2}\right)$ in the form of three bands shows that the oxalate ligands are not centrosymmetric, in agreement with the difference in the $\mathrm{C}-\mathrm{O}$ bond lengths revealed by the X-ray study. Attributions of the vibrational bands of the title compound were made by comparison with previous studies (Sarr et al., 2018; Marinescu et al., 2002; Li et al., 2008). The vibrational bands at 3061 and $1579 \mathrm{~cm}^{-1}$ in the IR spectrum (Fig. 4) are assigned to the stretching and deformation modes $\nu(\mathrm{N}-\mathrm{H})$ and $\delta(\mathrm{N}-\mathrm{H})$, respectively, of $\mathrm{NH}_{2}$ - in the ammonium group. The bands at 1675,1375 and $1251 \mathrm{~cm}^{-1}$ are attributed to the asymmetric and symmetric vibrations of the oxalate $-\mathrm{CO}_{2}$ moiety while that at $795 \mathrm{~cm}^{-1}$ corresponds to the deformation vibrations $\delta(\mathrm{C}-\mathrm{O})$ (Sarr et


Figure 5
Wavelength (nm)
The electronic spectrum of the title compound.

Table 2
Experimental details.
Crystal data
Chemical formula
$M_{\mathrm{r}}$
Crystal system, space group
Temperature (K)
$a, b, c(\AA)$
$\beta\left({ }^{\circ}\right)$
$V\left(\mathrm{~A}^{3}\right)$
$V\left(\mathrm{~A}^{3}\right)$
Z
Radiation type
$\mu\left(\mathrm{mm}^{-1}\right)$
Crystal size (mm)
Data collection
Diffractometer
Absorption correction
$T_{\text {min }}, T_{\text {max }}$
No. of measured, independent and observed $[I>2 \sigma(I)]$ reflections
$R_{\text {int }}$
$(\sin \theta / \lambda)_{\max }\left(\AA^{-1}\right)$
Refinement
$R\left[F^{2}>2 \sigma\left(F^{2}\right)\right], w R\left(F^{2}\right), S$
No. of reflections
No. of parameters
H -atom treatment
$\Delta \rho_{\max }, \Delta \rho_{\text {min }}\left(\mathrm{e} \AA^{-3}\right)$
$\left(\mathrm{C}_{6} \mathrm{H}_{16} \mathrm{~N}\right)_{2}\left[\mathrm{Sn}\left(\mathrm{C}_{2} \mathrm{ClO}_{4}\right)_{2}\right]$
570.02

Monoclinic, $C 2 / c$
100
16.275 (8), 13.581 (6), 11.116 (4)
98.40 (3)
2430.7 (18)

4
Mo $K \alpha$
1.31
$0.56 \times 0.30 \times 0.22$

Bruker D8 VENTURE
Multi-scan (SADABS; Bruker, 2015)
0.626, 0.746

14360, 2800, 2597
0.023
0.650
$0.023,0.055,1.28$
2800
136
H-atom parameters constrained
$1.01,-0.59$

Computer programs: APEX3 and SAINT (Bruker, 2015), SHELXT (Sheldrick, 2015a), SHELXL (Sheldrick, 2015b), and OLEX2 (Dolomanov et al., 2009).
al., 2018; Marinescu et al., 2002; Li et al., 2008). The frequencies of stretching vibrations of the oxalate group often show slight deviations owing to the different coordination modes. The bands at $2882 \mathrm{~cm}^{-1}$ are assigned to the valence vibrations $v(\mathrm{C}-\mathrm{H})$ and those at $1486 \mathrm{~cm}^{-1}$ to the deformation vibrations $\delta(\mathrm{C}-\mathrm{H})$.

The electronic spectrum of the title compound is shown in Fig. 5. In the ultraviolet region, only one strong absorption band with a shoulder is observed. Generally, only $\pi \rightarrow \pi^{*}$, $\mathrm{n} \rightarrow \pi^{*}$ and LMCT transitions can be observed in the ultra-violet-visible (UV-Vis) region. The $\sigma \rightarrow \sigma^{*}$ transition requires an absorption of a photon with a wavelength which does not fall in the UV-Vis range. Thus, this strong absorption band at 320 nm (Fig. 5) may be assigned to ligand-metal ion charge transfer (LMCT) (Kane et al., 2016). However, as the ligandbased $\pi \rightarrow \pi^{*} / n \rightarrow \pi^{*}$ transitions absorb in the same area as the LMCT transitions, we cannot exclude a possibility of superposition of these transitions (ligand-based and LMCT) owing to the form of the absorption band (Ford \& Vogler, 1993; Filho et al., 2000). In the title compound, the $\mathrm{Sn}^{\mathrm{IV}}$ atom is surrounded by electron rich ligands (chlorido and oxalato), and $\pi \rightarrow \pi^{*}$ and $n \rightarrow \pi^{*}$ transitions may result from the nonbinding electron pairs present on chlorine atoms or oxygen atoms of oxalate (Wojciechowska et al., 2016).

## 6. Refinement details

Crystal data, data collection and structure refinement details are summarized in Table 2. All H atoms were placed in
geometrically idealized positions and constrained to ride on their parent atoms, with a $\mathrm{N}-\mathrm{H}$ distance of $0.91 \AA, \mathrm{C}_{\text {methyl }}-\mathrm{H}$ $=0.98 \AA$ and $\mathrm{C}_{\text {methine }}=1.0 \AA$, and with $U_{\text {iso }}(\mathrm{H})=1.2 U_{\text {eq }}(\mathrm{C}, \mathrm{N})$ or $1.5 U_{\text {eq }}\left(\mathrm{C}_{\text {methyl }}\right)$. Three reflections were omitted from refinement because they were obstructed by the beam stop.

## Acknowledgements

The authors thank the Université Cheikh Anta Diop DakarSénégal, the Laboratoire de Chimie et de Physique des Matériaux (LCPM) de l'Université Assane Seck de Ziguinchor, Sénégal and the ICMUB-UMR 6302, 9, avenue Alain Savary 21000 Dijon-France for financial support. All measurements were performed in the institutes quoted above. The authors also acknowledge the CNRS-Université de Strasbourg, France, through collaboration with Frederic Melin.

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## supporting information

Synthesis, structure determination and characterization by UV-Vis and IR spectroscopy of bis(diisopropylammonium) cis-dichloridobis(oxalato$\kappa^{2} O^{1}, O^{2}$ )stannate(IV)

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## Computing details

Data collection: APEX3 (Bruker, 2015); cell refinement: SAINT (Bruker, 2015); data reduction: SAINT (Bruker, 2015); program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: SHELXL (Sheldrick, 2015b); molecular graphics: OLEX2 (Dolomanov et al., 2009); software used to prepare material for publication: OLEX2 (Dolomanov et al., 2009).

Bis(diisopropylammonium) cis-dichloridobis(oxalato- $\kappa^{2} O^{1}, O^{2}$ )stannate(IV)

## Crystal data

$\left(\mathrm{C}_{6} \mathrm{H}_{16} \mathrm{~N}\right)_{2}\left[\mathrm{Sn}\left(\mathrm{C}_{2} \mathrm{ClO}_{4}\right)_{2}\right]$
$M_{r}=570.02$
Monoclinic, $C 2 / c$
$a=16.275$ (8) $\AA$
$b=13.581$ (6) $\AA$
$c=11.116$ (4) $\AA$
$\beta=98.40(3)^{\circ}$
$V=2430.7(18) \AA^{3}$
$Z=4$

## Data collection

Bruker D8 VENTURE
diffractometer
Radiation source: X-ray tube, Siemens KFF Mo 2K-90C
TRIUMPH curved crystal monochromator
Detector resolution: 1024 pixels $\mathrm{mm}^{-1}$
$\varphi$ and $\omega$ scans'
Absorption correction: multi-scan
(SADABS; Bruker, 2015)

## Refinement

Refinement on $F^{2}$
Least-squares matrix: full
$R\left[F^{2}>2 \sigma\left(F^{2}\right)\right]=0.023$
$w R\left(F^{2}\right)=0.055$
$S=1.28$
2800 reflections
136 parameters
$F(000)=1160$
$D_{\mathrm{x}}=1.558 \mathrm{Mg} \mathrm{m}^{-3}$
Mo $K \alpha$ radiation, $\lambda=0.71073 \AA$
Cell parameters from 7290 reflections
$\theta=3.0-27.5^{\circ}$
$\mu=1.31 \mathrm{~mm}^{-1}$
$T=100 \mathrm{~K}$
Plate, clear light colourless
$0.56 \times 0.30 \times 0.22 \mathrm{~mm}$
$T_{\min }=0.626, T_{\text {max }}=0.746$
14360 measured reflections
2800 independent reflections
2597 reflections with $I>2 \sigma(I)$
$R_{\text {int }}=0.023$
$\theta_{\text {max }}=27.5^{\circ}, \theta_{\text {min }}=3.0^{\circ}$
$h=-20 \rightarrow 21$
$k=-17 \rightarrow 17$
$l=-14 \rightarrow 14$

## 0 restraints

Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites
H -atom parameters constrained
$w=1 /\left[\sigma^{2}\left(F_{\mathrm{o}}{ }^{2}\right)+(0.011 P)^{2}+6.1885 P\right]$
where $P=\left(F_{o}^{2}+2 F_{\mathrm{c}}^{2}\right) / 3$

# supporting information 

$(\Delta / \sigma)_{\max }=0.002$
$\Delta \rho_{\max }=1.01 \mathrm{e}^{-3}$
$\Delta \rho_{\text {min }}=-0.59 \mathrm{e}_{\AA^{-3}}$

## 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 ( $\AA^{2}$ )

|  | $x$ | $y$ | $z$ | $U_{\mathrm{iso}}{ }^{*} / U_{\mathrm{eq}}$ |
| :--- | :--- | :--- | :--- | :--- |
| Sn1 | 0.5000 | $0.67465(2)$ | 0.7500 | $0.01307(6)$ |
| C11 | $0.55290(4)$ | $0.56066(4)$ | $0.89962(5)$ | $0.02556(12)$ |
| O1 | $0.38232(9)$ | $0.69379(11)$ | $0.79635(12)$ | $0.0158(3)$ |
| O2 | $0.31479(8)$ | $0.77413(11)$ | $0.92602(12)$ | $0.0165(3)$ |
| O3 | $0.46345(9)$ | $0.85537(11)$ | $1.03026(12)$ | $0.0159(3)$ |
| O4 | $0.52285(9)$ | $0.78744(10)$ | $0.88059(12)$ | $0.0147(3)$ |
| C7 | $0.37835(11)$ | $0.75429(14)$ | $0.88463(16)$ | $0.0117(3)$ |
| C8 | $0.46144(11)$ | $0.80483(14)$ | $0.93878(17)$ | $0.0121(4)$ |
| N1 | $0.82801(10)$ | $0.66470(12)$ | $0.67738(14)$ | $0.0117(3)$ |
| H1A | 0.8640 | 0.6618 | 0.6220 | $0.014^{*}$ |
| H1B | 0.7824 | 0.6983 | 0.6421 | $0.014^{*}$ |
| C1 | $0.80130(13)$ | $0.56088(15)$ | $0.70045(19)$ | $0.0180(4)$ |
| H1 | 0.7608 | 0.5626 | 0.7599 | $0.022^{*}$ |
| C2 | $0.87571(16)$ | $0.49960(18)$ | $0.7530(2)$ | $0.0317(6)$ |
| H2A | 0.8580 | 0.4317 | 0.7643 | $0.048^{*}$ |
| H2B | 0.9003 | 0.5271 | 0.8316 | $0.048^{*}$ |
| H2C | 0.9170 | 0.5004 | 0.6970 | $0.048^{*}$ |
| C3 | $0.75809(16)$ | $0.52045(17)$ | $0.5802(2)$ | $0.0268(5)$ |
| H3A | 0.7113 | 0.5632 | 0.5490 | $0.040^{*}$ |
| H3B | 0.7376 | 0.4539 | 0.5926 | $0.040^{*}$ |
| H3C | 0.7975 | 0.5181 | 0.5215 | $0.040^{*}$ |
| C4 | $0.86854(12)$ | $0.72450(16)$ | $0.78415(18)$ | $0.0163(4)$ |
| H4 | 0.9228 | 0.6934 | 0.8176 | $0.020^{*}$ |
| C5 | $0.88477(14)$ | $0.82664(16)$ | $0.7363(2)$ | $0.034^{*}$ |
| H5A | 0.9179 | 0.8209 | 0.6698 | $0.034^{*}$ |
| H5B | 0.9151 | 0.8662 | 0.8020 | $0.034^{*}$ |
| H5C | 0.8317 | 0.8586 | 0.7063 | $0.0231(5)$ |
| C6 | $0.81368(14)$ | $0.72851(18)$ | $0.88350(19)$ | $0.035^{*}$ |
| H6A | 0.7582 | 0.7518 | 0.8491 | $0.035^{*}$ |
| H6B | 0.8380 | 0.7738 | 0.9476 | 0.9179 |
| H6C | 0.8094 | 0.6626 |  | 0 |

Atomic displacement parameters $\left(\AA^{2}\right)$

|  | $U^{11}$ | $U^{22}$ | $U^{\beta 3}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
| :--- | :--- | :--- | :--- | :--- | :--- | :--- |
| Sn1 | $0.01120(10)$ | $0.01596(10)$ | $0.01279(10)$ | 0.000 | $0.00420(6)$ | 0.000 |
| C11 | $0.0335(3)$ | $0.0221(3)$ | $0.0217(2)$ | $0.0028(2)$ | $0.0062(2)$ | $0.0098(2)$ |


| O1 | $0.0111(6)$ | $0.0235(7)$ | $0.0134(6)$ | $-0.0049(6)$ | $0.0037(5)$ | $-0.0063(6)$ |
| :--- | :--- | :--- | :--- | :--- | :--- | :--- |
| O2 | $0.0099(6)$ | $0.0262(8)$ | $0.0139(7)$ | $-0.0022(6)$ | $0.0035(5)$ | $-0.0046(6)$ |
| O3 | $0.0147(7)$ | $0.0193(7)$ | $0.0130(6)$ | $-0.0013(6)$ | $0.0001(5)$ | $-0.0048(5)$ |
| O4 | $0.0138(7)$ | $0.0166(7)$ | $0.0146(7)$ | $-0.0011(5)$ | $0.0056(5)$ | $-0.0016(5)$ |
| C7 | $0.0112(8)$ | $0.0143(9)$ | $0.0095(8)$ | $-0.0013(7)$ | $0.0009(7)$ | $0.0026(7)$ |
| C8 | $0.0088(8)$ | $0.0143(9)$ | $0.0125(8)$ | $-0.0005(7)$ | $-0.0008(7)$ | $0.0032(7)$ |
| N1 | $0.0115(7)$ | $0.0134(8)$ | $0.0100(7)$ | $0.0003(6)$ | $0.0013(6)$ | $0.0021(6)$ |
| C1 | $0.0200(10)$ | $0.0132(9)$ | $0.0211(10)$ | $-0.0016(8)$ | $0.0040(8)$ | $0.0049(8)$ |
| C2 | $0.0323(13)$ | $0.0207(11)$ | $0.0403(14)$ | $0.0066(10)$ | $-0.0010(11)$ | $0.0129(10)$ |
| C3 | $0.0327(13)$ | $0.0165(10)$ | $0.0312(12)$ | $-0.0058(9)$ | $0.0042(10)$ | $-0.0029(9)$ |
| C4 | $0.0118(9)$ | $0.0227(10)$ | $0.0131(9)$ | $-0.0002(8)$ | $-0.0030(7)$ | $-0.0018(8)$ |
| C5 | $0.0213(11)$ | $0.0208(10)$ | $0.0246(11)$ | $-0.0058(9)$ | $0.0006(8)$ | $-0.0019(9)$ |
| C6 | $0.0250(11)$ | $0.0294(12)$ | $0.0144(10)$ | $0.0001(9)$ | $0.0016(8)$ | $-0.0015(8)$ |

Geometric parameters $\left(\AA,{ }^{\circ}\right)$

| Sn1-Cl1 | 2.3422 (9) | C1-C3 | 1.519 (3) |
| :---: | :---: | :---: | :---: |
| $\mathrm{Sn} 1-\mathrm{Cl1}{ }^{\text {i }}$ | 2.3422 (9) | C2-H2A | 0.9800 |
| Sn1-O1 | 2.0710 (16) | C2-H2B | 0.9800 |
| Sn1-O1 ${ }^{\text {i }}$ | 2.0711 (16) | $\mathrm{C} 2-\mathrm{H} 2 \mathrm{C}$ | 0.9800 |
| $\mathrm{Sn} 1-\mathrm{O} 4^{\text {i }}$ | 2.1057 (15) | C3-H3A | 0.9800 |
| Sn1-O4 | 2.1058 (15) | C3-H3B | 0.9800 |
| O1-C7 | 1.289 (2) | $\mathrm{C} 3-\mathrm{H} 3 \mathrm{C}$ | 0.9800 |
| O2-C7 | 1.222 (2) | C4-H4 | 1.0000 |
| O3-C8 | 1.223 (2) | C4-C5 | 1.523 (3) |
| O4-C8 | 1.289 (2) | C4-C6 | 1.519 (3) |
| C7-C8 | 1.557 (3) | C5-H5A | 0.9800 |
| N1-H1A | 0.9100 | C5-H5B | 0.9800 |
| N1-H1B | 0.9100 | C5-H5C | 0.9800 |
| N1-C1 | 1.508 (3) | C6-H6A | 0.9800 |
| N1-C4 | 1.508 (2) | C6-H6B | 0.9800 |
| $\mathrm{C} 1-\mathrm{H} 1$ | 1.0000 | C6-H6C | 0.9800 |
| $\mathrm{C} 1-\mathrm{C} 2$ | 1.514 (3) |  |  |
| $\mathrm{Cl1}{ }^{\text {i }}$ - $\mathrm{Sn} 1-\mathrm{Cl1}$ | 97.26 (4) | C2-C1-C3 | 112.4 (2) |
| $\mathrm{O} 1-\mathrm{Sn} 1-\mathrm{Cl1}$ | 99.42 (5) | $\mathrm{C} 3-\mathrm{C} 1-\mathrm{H} 1$ | 109.0 |
| $\mathrm{O} 1-\mathrm{Sn} 1-\mathrm{Cl1}^{\text {i }}$ | 90.13 (5) | $\mathrm{C} 1-\mathrm{C} 2-\mathrm{H} 2 \mathrm{~A}$ | 109.5 |
| O1- ${ }^{\text {i }} \mathrm{Sn} 1-\mathrm{Cl} 1$ | 90.13 (5) | $\mathrm{C} 1-\mathrm{C} 2-\mathrm{H} 2 \mathrm{~B}$ | 109.5 |
| $\mathrm{O} 1-\mathrm{Sn} 1-\mathrm{Cl}^{\mathrm{i}}$ | 99.42 (5) | $\mathrm{C} 1-\mathrm{C} 2-\mathrm{H} 2 \mathrm{C}$ | 109.5 |
| $\mathrm{O} 1-\mathrm{Sn} 1-\mathrm{Ol}^{\text {i }}$ | 165.59 (8) | $\mathrm{H} 2 \mathrm{~A}-\mathrm{C} 2-\mathrm{H} 2 \mathrm{~B}$ | 109.5 |
| $\mathrm{O} 1-\mathrm{Sn} 1-\mathrm{O} 4$ | 79.27 (6) | $\mathrm{H} 2 \mathrm{~A}-\mathrm{C} 2-\mathrm{H} 2 \mathrm{C}$ | 109.5 |
| $\mathrm{O} 1-\mathrm{Sn} 1-\mathrm{O} 4^{\text {i }}$ | 90.21 (6) | $\mathrm{H} 2 \mathrm{~B}-\mathrm{C} 2-\mathrm{H} 2 \mathrm{C}$ | 109.5 |
| $\mathrm{O} 1^{\mathrm{i}}-\mathrm{Sn} 1-\mathrm{O} 4^{\mathrm{i}}$ | 79.27 (6) | $\mathrm{C} 1-\mathrm{C} 3-\mathrm{H} 3 \mathrm{~A}$ | 109.5 |
| $\mathrm{O} 1{ }^{\mathrm{i}}-\mathrm{Sn} 1-\mathrm{O} 4$ | 90.21 (6) | C1-C3-H3B | 109.5 |
| $\mathrm{O} 4-\mathrm{Sn} 1-\mathrm{Cl} 1$ | 168.50 (4) | $\mathrm{C} 1-\mathrm{C} 3-\mathrm{H} 3 \mathrm{C}$ | 109.5 |
| $\mathrm{O} 4-\mathrm{Sn} 1-\mathrm{Cl1}^{\text {i }}$ | 168.49 (4) | H3A-C3-H3B | 109.5 |
| $\mathrm{O} 4-\mathrm{Sn} 1-\mathrm{Cl}^{\mathrm{i}}$ | 88.95 (5) | $\mathrm{H} 3 \mathrm{~A}-\mathrm{C} 3-\mathrm{H} 3 \mathrm{C}$ | 109.5 |
| $\mathrm{O} 4-\mathrm{Sn} 1-\mathrm{Cl1}$ | 88.95 (5) | $\mathrm{H} 3 \mathrm{~B}-\mathrm{C} 3-\mathrm{H} 3 \mathrm{C}$ | 109.5 |


| $\mathrm{O} 4-\mathrm{Sn} 1-\mathrm{O} 4$ | $86.66(8)$ |
| :--- | :--- |
| $\mathrm{C} 7-\mathrm{O} 1-\mathrm{Sn} 1$ | $114.87(12)$ |
| $\mathrm{C} 8-\mathrm{O} 4-\mathrm{Sn} 1$ | $114.04(12)$ |
| $\mathrm{O} 1-\mathrm{C} 7-\mathrm{C} 8$ | $115.98(16)$ |
| $\mathrm{O} 2-\mathrm{C} 7-\mathrm{O} 1$ | $124.51(18)$ |
| $\mathrm{O} 2-\mathrm{C} 7-\mathrm{C} 8$ | $119.50(17)$ |
| $\mathrm{O} 3-\mathrm{C} 8-\mathrm{O} 4$ | $126.25(18)$ |
| $\mathrm{O} 3-\mathrm{C} 8-\mathrm{C} 7$ | $119.01(17)$ |
| $\mathrm{O} 4-\mathrm{C} 8-\mathrm{C} 7$ | $114.74(16)$ |
| $\mathrm{H} 1 \mathrm{~A}-\mathrm{N} 1-\mathrm{H} 1 \mathrm{~B}$ | 107.1 |
| $\mathrm{C} 1-\mathrm{N} 1-\mathrm{H} 1 \mathrm{~A}$ | 107.7 |
| $\mathrm{C} 1-\mathrm{N} 1-\mathrm{H} 1 \mathrm{~B}$ | 107.7 |
| $\mathrm{C} 4-\mathrm{N} 1-\mathrm{H} 1 \mathrm{~A}$ | 107.7 |
| $\mathrm{C} 4-\mathrm{N} 1-\mathrm{H} 1 \mathrm{~B}$ | 107.7 |
| $\mathrm{C} 4-\mathrm{N} 1-\mathrm{C} 1$ | $118.25(15)$ |
| $\mathrm{N} 1-\mathrm{C} 1-\mathrm{H} 1$ | 109.0 |
| $\mathrm{~N} 1-\mathrm{C} 1-\mathrm{C} 2$ | $110.20(18)$ |
| $\mathrm{N} 1-\mathrm{C} 1-\mathrm{C} 3$ | $107.23(16)$ |
| $\mathrm{C} 2-\mathrm{C} 1-\mathrm{H} 1$ | 109.0 |
| $\mathrm{Sn} 1-\mathrm{O} 1-\mathrm{C} 7-\mathrm{O} 2$ | $-178.95(15)$ |
| $\mathrm{Sn} 1-\mathrm{O} 1-\mathrm{C} 7-\mathrm{C} 8$ | $1.7(2)$ |
| $\mathrm{Sn} 1-\mathrm{O} 4-\mathrm{C} 8-\mathrm{O} 3$ | $168.18(16)$ |
| $\mathrm{Sn} 1-\mathrm{O} 4-\mathrm{C} 8-\mathrm{C} 7$ | $-11.23(19)$ |
| $\mathrm{O} 1-\mathrm{C} 7-\mathrm{C} 8-\mathrm{O} 3$ | $-172.83(17)$ |
| $\mathrm{O} 1-\mathrm{C} 7-\mathrm{C} 8-\mathrm{O} 4$ | $6.6(2)$ |
|  |  |


| $\mathrm{N} 1-\mathrm{C} 4-\mathrm{H} 4$ | 109.0 |
| :--- | :--- |
| $\mathrm{~N} 1-\mathrm{C} 4-\mathrm{C} 5$ | $107.09(16)$ |
| $\mathrm{N} 1-\mathrm{C} 4-\mathrm{C} 6$ | $110.83(17)$ |
| $\mathrm{C} 5-\mathrm{C} 4-\mathrm{H} 4$ | 109.0 |
| $\mathrm{C} 6-\mathrm{C} 4-\mathrm{H} 4$ | 109.0 |
| $\mathrm{C} 6-\mathrm{C} 4-\mathrm{C} 5$ | $111.83(18)$ |
| $\mathrm{C} 4-\mathrm{C} 5-\mathrm{H} 5 \mathrm{~A}$ | 109.5 |
| C4-C5-H5B | 109.5 |
| C4-C5-H5C | 109.5 |
| H5A-C5-H5B | 109.5 |
| H5A-C5-H5C | 109.5 |
| H5B-C5-H5C | 109.5 |
| C4-C6-H6A | 109.5 |
| C4-C6-H6B | 109.5 |
| C4-C6-H6C | 109.5 |
| H6A-C6-H6B | 109.5 |
| H6A-C6-H6C | 109.5 |
| H6B-C6-H6C | 109.5 |


| $\mathrm{O} 2-\mathrm{C} 7-\mathrm{C} 8-\mathrm{O} 3$ | $7.8(3)$ |
| :--- | :--- |
| $\mathrm{O} 2-\mathrm{C} 7-\mathrm{C} 8-\mathrm{O} 4$ | $-172.75(17)$ |
| $\mathrm{C} 1-\mathrm{N} 1-\mathrm{C} 4-\mathrm{C} 5$ | $-177.16(17)$ |
| $\mathrm{C} 1-\mathrm{N} 1-\mathrm{C} 4-\mathrm{C} 6$ | $-54.9(2)$ |
| $\mathrm{C} 4-\mathrm{N} 1-\mathrm{C} 1-\mathrm{C} 2$ | $-60.8(2)$ |
| $\mathrm{C} 4-\mathrm{N} 1-\mathrm{C} 1-\mathrm{C} 3$ | $176.56(17)$ |

Symmetry code: (i) $-x+1, y,-z+3 / 2$.

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

| $D — \mathrm{H} \cdots A$ | $D-\mathrm{H}$ | $\mathrm{H} \cdots A$ | $D \cdots A$ | $D-\mathrm{H} \cdots A$ |
| :--- | :--- | :--- | :--- | :--- |
| $\mathrm{~N} 1 — \mathrm{H} 1 A \cdots 3^{3 i}$ | 0.91 | 2.05 | $2.943(2)$ | 167 |
| $\mathrm{~N} 1 — \mathrm{H} 1 B \cdots \mathrm{O}^{\mathrm{i}}$ | 0.91 | 1.95 | $2.855(2)$ | 177 |

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

