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## Structure Reports

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## Diacetatodi-tert-butyltin(IV)

Martin Reichelt and Hans Reuter*

Institut für Chemie neuer Materialien, Strukturchemie, Universität Osnabrück, Barbarastr. 7, D-49069 Osnabrück, Germany
Correspondence e-mail: hreuter@uos.de

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Key indicators: single-crystal X-ray study; $T=100 \mathrm{~K}$; mean $\sigma(\mathrm{C}-\mathrm{C})=0.003 \AA$; $R$ factor $=0.023 ; w R$ factor $=0.046 ;$ data-to-parameter ratio $=21.5$.

The title compound, $\left[\mathrm{Sn}\left(\mathrm{C}_{4} \mathrm{H}_{9}\right)_{2}\left(\mathrm{CH}_{3} \mathrm{COO}\right)_{2}\right]$, was synthesized in order to study the influence of large organic groups on the molecular structure of diorganotin diacetates. The title compound exhibits the same structure type as other diorganotin(IV) diacetates characterized by an unsymmetrical bidentate bonding mode of the two acetate groups to tin. The influence of the $t$-butyl groups on this molecular structure is expressed in two significant differences: tin-carbon bond lengths are much more longer than in the other diacetates, as are the additional interactions of the acetate groups with the tin atom. Intermolecular interactions are restricted to C $\mathrm{H} \cdots \mathrm{O}$ ones similar to those in the other diacetates, giving rise to a chain-like arrangement of the molecules with the tin atoms and acetate groups in the propagation plane.

## Related literature

For background to diorganotin(IV) carboxylates, see: Tiekink (1991) and to diorganotin(IV) diacetates, see: Alcock et al. (1992); Lockhart et al. (1987); Mistry et al. (1995).


## Experimental

## Crystal data

$\left[\mathrm{Sn}\left(\mathrm{C}_{4} \mathrm{H}_{9}\right)_{2}\left(\mathrm{C}_{2} \mathrm{H}_{3} \mathrm{O}_{2}\right)_{2}\right]$
$M_{r}=351.00$
Monoclinic, $P 2_{b} / n$
$a=6.1039$ (3) A
$b=15.3928$ (7) $\AA$
$c=15.9601$ ( 8 ) $\AA$
$\beta=95.462(2)^{\circ}$

## Data collection

## Bruker APEXII CCD

diffractometer
Absorption correction: multi-scan
(SADABS; Bruker, 2009)
$T_{\text {min }}=0.791, T_{\text {max }}=0.939$
76636 measured reflections 3590 independent reflections 2980 reflections with $I>2 \sigma(I)$ $R_{\text {int }}=0.081$

## Refinement

$R\left[F^{2}>2 \sigma\left(F^{2}\right)\right]=0.023 \quad 167$ parameters
$w R\left(F^{2}\right)=0.046 \quad \mathrm{H}$-atom parameters constrained
$S=1.04$
3590 reflections
$\Delta \rho_{\text {max }}=0.59 \mathrm{e}^{\AA^{-3}}$
$\Delta \rho_{\text {min }}=-0.51 \mathrm{e}^{-3}$

Table 1
Selected bond lengths ( $\AA$ ).

| Sn1-O21 | $2.1001(14)$ | O11-C15 | $1.304(3)$ |
| :--- | :--- | :--- | :--- |
| Sn1-O11 | $2.1002(14)$ | O12-C15 | $1.235(3)$ |
| Sn1-C11 | $2.175(2)$ | O21-C25 | $1.304(3)$ |
| Sn1-C21 | $2.176(2)$ | O22-C25 | $1.239(3)$ |

Table 2
Hydrogen-bond geometry ( $\AA^{\circ}{ }^{\circ}$ ).

| $D-\mathrm{H} \cdots A$ | $D-\mathrm{H}$ | $\mathrm{H} \cdots A$ | $D \cdots A$ | $D-\mathrm{H} \cdots A$ |
| :---: | :---: | :---: | :---: | :---: |
| $\mathrm{C} 12-\mathrm{H} 12 \mathrm{C} \cdots \mathrm{O} 21^{\text {i }}$ | 0.98 | 2.54 | 3.362 (3) | 142 |
| $\mathrm{C} 22-\mathrm{H} 22 \mathrm{C} \cdots \mathrm{O} 11^{\text {i }}$ | 0.98 | 2.65 | 3.584 (3) | 160 |
| C16-H16C . ${ }^{\text {O } 222^{\text {ii }}}$ | 0.98 | 2.69 | 3.636 (3) | 163 |
| C16-H16A $\cdots$ O22 $2^{\text {iii }}$ | 0.98 | 2.60 | 3.518 (3) | 155 |

Data collection: APEX2 (Bruker, 2009); cell refinement: SAINT (Bruker, 2009); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: DIAMOND (Brandenburg, 2006) and Mercury (Macrae et al., 2008); software used to prepare material for publication: SHELXTL (Sheldrick, 2008).

We thank the Deutsche Forschungsgemeinschaft and the Government of Lower Saxony for their financial support in the acquisition of the diffractometer.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AA2086).

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## supporting information

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## Diacetatodi-tert-butyltin(IV)

## Martin Reichelt and Hans Reuter

## S1. Comment

Diorganotin(IV) dicarboxylates, $R_{2} \operatorname{Sn}\left(\mathrm{O}_{2} \mathrm{CR}^{\prime}\right)_{2}$, belong to the class of diorganotin compounds, $R_{2} \operatorname{Sn} X_{2}$, with univalent anions $X$, such as the halides ( $X=\mathrm{F}, \mathrm{Cl}, \mathrm{Br}, \mathrm{I}$ ) or alkoxides ( $X=\mathrm{OR}^{\prime}$ ). In contrast to these anions, that behave as unidentate substitutents, the carboxylate groups, $\mathrm{O}_{2} \mathrm{CR}^{\prime}$, can act via its two oxygen atoms as a bidentate ligand in a more or less symmetrical or unsymmetrical coordination mode giving rise to additional intra- or intermolecular interactions (Tiekink, 1991). In the case of diacetates, $R^{\prime}=\mathrm{Me}$, this was formerly shown for $R=$ phenyl (Alcock et al. 1992) and $R=$ methyl (Lockhart et al. 1987, $\alpha$-modification; Mistry et al. 1995, $\beta$-modification) both revealing in solid state the same molecular structure type with a strongly unsymmetrical bonding mode of the acetate groups. On this background it was interesting to see whether the larger $t$-butyl groups are compatible with that structure type or not.
The asymmetric unit of the title compound (Fig. 1) consists of one formula unit with all atoms in general positions although the molecule displays a pseudo twofold rotation axis [midway $\mathrm{C} 11 / \mathrm{C} 21-\mathrm{Sn}$ - midway $\mathrm{O} 11 / \mathrm{O} 21$ ]. In a first approximation, the tin atom is fourfold coordinated by the two $t$-butyl groups and an oxygen atom of each acetate group. Both $\mathrm{Sn}-\mathrm{C}$ bond lengths are almost equal as are both $\mathrm{Sn}-\mathrm{O}$ bond lengths, too (see Table 1). The last ones are comparable with $\mathrm{Sn}-\mathrm{O}$ bond lengths in the other diacetates $[d(\mathrm{Sn}-\mathrm{O})=2.076$ (4), 2.079 (4), $R=\mathrm{Ph} ; 2 \times 2.106$ (2), $R=$ $\alpha$-Me, 2.098 (2), 2.094 (2), $R=\beta$-Me]. In comparison to the methyl compounds with $\mathrm{Sn}-\mathrm{C}$ bond lengths of $2 \times 2.098$ (3) $\AA[\alpha]$, respectively 2.096 (3), 2.088 (3) $\AA[\beta]$ and the phenyl compound $[2.119$ (5), 2.110 (6) $\AA] \mathrm{Sn}-\mathrm{C}$ bonds of the tertbutyl groups are considerable longer. From the bond angles of 138.97 (7) ${ }^{\circ}$ between the $t$-butyl groups and 79.93 (6) ${ }^{\circ}$ between the two oxygen atoms, the coordination polyhedron is compressed to a tetragonal disphenoid (Fig. 2). Obviously, this distortion is typical for diacetates [C-Sn-C $=131.4^{\circ}, R=\mathrm{Ph} ; 135.9(2)^{\circ}(1)^{\circ}, R=\alpha$-Me; $133.8(2)^{\circ}, R=$ $\beta$-Me; O-Sn-O $\left.=82.0(3)^{\circ}, R=\mathrm{Ph} ; 79.5(1)^{\circ}, R=\alpha-\mathrm{Me} ; 80.1(1)^{\circ}, R=\beta-\mathrm{Me}\right]$.
The coordination sphere of the tin atom is completed by the other two oxygen atoms of the acetate groups that undergo a much more weaker interaction with the tin atom $[d(\mathrm{Sn} \cdots \mathrm{O})=2.689$ (1) $\AA$ to $\mathrm{O} 22,2.647$ (2) $\AA$ to O12], resulting in a very unsymmetrical bidentate coordination mode of the acetate groups (Fig. 2). This is also reflected in two different CO distances for each acetate group (Table 1). Again, within the molecular structures of the other diacetates a similar coordination behaviour is observed but $\mathrm{Sn} \cdots \mathrm{O}$ interactions are considerably stronger [2.583 (4), 2.527 (5) $\AA, R=\mathrm{Ph} ; 2 x$ 2.539 (2) $\AA, R=\alpha$-Me; 2.563 (2), 2.595 (2) $\AA, R=\beta$-Me].

Both $t$-butyl groups are well ordered with a mean value for the $\mathrm{C}-\mathrm{C}$ bonds of 1.527 (3) $\AA$ [range: 1.522 (3) - 1.530 (3) $\AA$ ] and a mean $\mathrm{C}-\mathrm{C}-\mathrm{C}$ bond angle of $110.2(4)^{\circ}\left[\right.$ range: $\left.109.65(3)^{\circ}-110.65(3)^{\circ}\right]$. With respect to $\mathrm{Sn}-\mathrm{C}-\mathrm{C}$ bond angles, both $t$-butyl groups show similar effects: two angles are around the ideal tetrahedral value of $109.5^{\circ}$, whereas the third one is significantly smaller [107.09 (3) ${ }^{\circ}$ for $\mathrm{C} 12 ; 106.07(2)^{\circ}$ for C22].
In the solid, molecules are arranged in chains with the tin atoms and acetate groups defining the propagation plane (Fig. 3). This arrangement is also characteristic for both modifications of the methyl compounds but not for the phenyl one. In the present case, the intermolecular $\mathrm{O} \cdots \mathrm{Sn}$ distances of 4.692 (1) $\AA\left[\mathrm{O} 21 \cdots \mathrm{Sn} 1^{1}\right]$ and 4.694 (1) $\AA\left[\mathrm{O} 11 \cdots \mathrm{Sn} 1^{1}\right]$, however,
are so long that seems impossible that these interactions are responsible for the supra-molecular architecture. There are, however, $\mathrm{O} \cdots \mathrm{HC}$ interactions between the acetate groups and $t$-butyl groups of neighbouring molecules (Fig. 4) that are much more attractive (Table 2). Similar interactions are found in all other diorganotin(IV) diacetates and even in the phenyl compound.

## S2. Experimental

## Synthesis:

$0.51 \mathrm{~g}(0.68 \mathrm{mmol})$ di-t-butyltin oxide are dissolved in $0.25 \mathrm{~g}(4.16 \mathrm{mmol})$ of acetic acid (Fluka). The solution is stirred for 6 h at ambient temperature before the solvent is allowed to evaporate slowly. After about 1 week colourless block-like crystals are grown.

## Spectroscopic studies:

Elemental analysis calcd (\%) for $\mathrm{C}_{5} \mathrm{H}_{4} \mathrm{O}_{3}$ : C, 41.06; H, 6.89. Found: C, 40.99; H, 6.53. ${ }^{1} \mathrm{H}-\mathrm{NMR}\left(\mathrm{CDCl}_{3}\right.$, p.p.m.): 2.09
$(\mathrm{s}, \mathrm{OAc}, 3 \mathrm{H}), 1.35\left(\mathrm{~s}, \mathrm{tBu}, 9 \mathrm{H},{ }^{3}\left({ }^{1} \mathrm{H}-{ }^{119 / 117} \mathrm{Sn}=119.1 / 114.1 \mathrm{~Hz}\right) .\left\{{ }^{1} \mathrm{H}\right\}-{ }^{13} \mathrm{C}-\mathrm{NMR}\left(\mathrm{CDCl}_{3}\right.\right.$, p.p.m. $): 180.59\left(\mathrm{O}_{2} \mathrm{CCH}_{3}\right), 45.29$
$\left(\mathrm{C}_{\alpha}-\mathrm{tBu},{ }^{1} \mathrm{~J}\left({ }^{13} \mathrm{C}-{ }^{-199 / 17} \mathrm{Sn}\right)=515.3 / 492.4 \mathrm{~Hz}\right), 29.67\left(\mathrm{C}_{\beta}-\mathrm{tBu}\right), 20.37\left(\mathrm{O}_{2} \mathrm{CCH}_{3}\right)$. IR $\left(\mathrm{ATR}, \mathrm{cm}^{-1}\right): 2853.6,1601.0,1438.3$,
1367.9, 1323.9, 1160.4, 1053.5, 1018.2, 942.5, 807.9, 688.4, 620.1, 499.2.

## Crystallographic studies:

A suitable single-crystal was selected under a polarization microscope and mounted on a $50 \mu \mathrm{~m}$ MicroMesh MiTeGen Micromount ${ }^{\text {TM }}$ using FROMBLIN Y perfluoropolyether (LVAC 16/6, Aldrich).

## S3. Refinement

Hydrogen atoms were clearly identified in difference Fourier syntheses. Their positions were idealized and refined at calculated positions riding on the carbon atoms with $\mathrm{C}-\mathrm{H}=0.98 \AA$.


Figure 1
Molecular structure of the title compound, with $50 \%$ probability level displacement ellipsoids for non-H atoms.


Figure 2
Polyhedron model of the coordination sphere of the tin atom; $t$-butyl groups have been omitted for clarity, weak $\mathrm{Sn} \cdots \mathrm{O}$ interactions are indicated by dashed sticks. Displacement ellipsoids for non-H atoms are shown with $50 \%$ probability level


Figure 3
Perspective view of the crystal structure parallel to the crystallographic $a$ axis, showing the chain-like arrangement of acetate groups and tin atoms.


Figure 4
Intermolecular oxygen hydrogen interactions within the chain-like arrangement of the diacetate molecules [Symmetry operator: (1) $1+x, y, z]$.

## Diacetatodi-tert-butyltin(IV)

## Crystal data

$\left[\mathrm{Sn}\left(\mathrm{C}_{4} \mathrm{H}_{9}\right)_{2}\left(\mathrm{C}_{2} \mathrm{H}_{3} \mathrm{O}_{2}\right)_{2}\right]$
$M_{r}=351.00$
Monoclinic, $P 2_{1} / n$
Hall symbol: -P 2 yn
$a=6.1039$ (3) $\AA$
$b=15.3928$ (7) $\AA$
$c=15.9601$ (8) $\AA$
$\beta=95.462$ (2) ${ }^{\circ}$
$V=1492.74(12) \AA^{3}$
$Z=4$

## Data collection

Bruker APEXII CCD
diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan
(SADABS; Bruker, 2009)
$T_{\min }=0.791, T_{\max }=0.939$

## Refinement

Refinement on $F^{2}$
Least-squares matrix: full
$R\left[F^{2}>2 \sigma\left(F^{2}\right)\right]=0.023$
$w R\left(F^{2}\right)=0.046$
$S=1.04$
3590 reflections
167 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
$F(000)=712$
$D_{\mathrm{x}}=1.562 \mathrm{Mg} \mathrm{m}^{-3}$
Mo $K \alpha$ radiation, $\lambda=0.71073 \AA$
Cell parameters from 6480 reflections
$\theta=2.6-25.7^{\circ}$
$\mu=1.71 \mathrm{~mm}^{-1}$
$T=100 \mathrm{~K}$
Block, colourless
$0.14 \times 0.06 \times 0.04 \mathrm{~mm}$

76636 measured reflections
3590 independent reflections
2980 reflections with $I>2 \sigma(I)$
$R_{\text {int }}=0.081$
$\theta_{\text {max }}=28.0^{\circ}, \theta_{\text {min }}=1.8^{\circ}$
$h=-8 \rightarrow 8$
$k=-20 \rightarrow 19$
$l=-21 \rightarrow 21$

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H -atom parameters constrained
$w=1 /\left[\sigma^{2}\left(F_{0}^{2}\right)+(0.0133 P)^{2}+1.0511 P\right]$
where $P=\left(F_{\mathrm{o}}{ }^{2}+2 F_{\mathrm{c}}{ }^{2}\right) / 3$
$(\Delta / \sigma)_{\text {max }}=0.002$
$\Delta \rho_{\text {max }}=0.59 \mathrm{e}^{-3} \AA^{-3}$
$\Delta \rho_{\text {min }}=-0.51 \mathrm{e}^{-3}$

Extinction correction: SHELXL,
$\mathrm{Fc}^{*}=\mathrm{kFc}\left[1+0.001 \mathrm{xFc}^{2} \lambda^{3} / \sin (2 \theta)\right]^{-1 / 4}$
Extinction coefficient: 0.00201 (16)

## Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two 1.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.
Refinement. Refinement of $F^{2}$ against ALL reflections. The weighted $R$-factor $w R$ and goodness of fit $S$ are based on $F^{2}$, conventional $R$-factors $R$ are based on $F$, with $F$ set to zero for negative $F^{2}$. The threshold expression of $F^{2}>\sigma\left(F^{2}\right)$ is used only for calculating $R$-factors $(\mathrm{gt})$ etc. and is not relevant to the choice of reflections for refinement. $R$-factors based on $F^{2}$ are statistically about twice as large as those based on $F$, and $R$ - factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\hat{A}^{2}$ )

|  | $x$ | $y$ | $z$ | $U_{\text {iso }} * / U_{\text {eq }}$ |
| :---: | :---: | :---: | :---: | :---: |
| Sn1 | 0.19140 (2) | 0.295768 (9) | 0.285075 (8) | 0.01333 (6) |
| C11 | 0.0432 (3) | 0.20445 (14) | 0.19231 (13) | 0.0162 (4) |
| C12 | -0.1709 (3) | 0.24192 (16) | 0.14968 (14) | 0.0257 (5) |
| H12A | -0.2374 | 0.2001 | 0.1085 | 0.031 (2)* |
| H12B | -0.1396 | 0.2962 | 0.1211 | 0.031 (2)* |
| H12C | -0.2730 | 0.2534 | 0.1922 | 0.031 (2)* |
| C13 | 0.2095 (4) | 0.18975 (15) | 0.12781 (13) | 0.0216 (5) |
| H13A | 0.3485 | 0.1689 | 0.1566 | 0.031 (2)* |
| H13B | 0.2350 | 0.2445 | 0.0990 | 0.031 (2)* |
| H13C | 0.1515 | 0.1464 | 0.0865 | 0.031 (2)* |
| C14 | -0.0036 (4) | 0.11881 (15) | 0.23553 (14) | 0.0259 (5) |
| H14A | -0.1128 | 0.1285 | 0.2758 | 0.031 (2)* |
| H14B | 0.1327 | 0.0966 | 0.2654 | 0.031 (2)* |
| H14C | -0.0609 | 0.0764 | 0.1932 | 0.031 (2)* |
| C21 | 0.0894 (3) | 0.38924 (14) | 0.37595 (12) | 0.0161 (4) |
| C22 | -0.0723 (4) | 0.34722 (15) | 0.43092 (14) | 0.0245 (5) |
| H22A | -0.1180 | 0.3900 | 0.4713 | 0.030 (2)* |
| H22B | -0.0011 | 0.2980 | 0.4614 | 0.030 (2)* |
| H22C | -0.2016 | 0.3267 | 0.3953 | 0.030 (2)* |
| C23 | 0.2995 (4) | 0.41692 (15) | 0.42919 (14) | 0.0243 (5) |
| H23A | 0.2626 | 0.4591 | 0.4717 | 0.030 (2)* |
| H23B | 0.4014 | 0.4435 | 0.3928 | 0.030 (2)* |
| H23C | 0.3689 | 0.3659 | 0.4572 | 0.030 (2)* |
| C24 | -0.0185 (4) | 0.46712 (15) | 0.33009 (14) | 0.0249 (5) |
| H24A | -0.1502 | 0.4481 | 0.2950 | 0.030 (2)* |
| H24B | 0.0852 | 0.4936 | 0.2944 | 0.030 (2)* |
| H24C | -0.0601 | 0.5098 | 0.3713 | 0.030 (2)* |
| O11 | 0.4677 (2) | 0.22581 (9) | 0.33641 (9) | 0.0198 (3) |
| O12 | 0.1995 (3) | 0.18113 (10) | 0.40892 (10) | 0.0251 (4) |
| C15 | 0.3940 (4) | 0.17649 (14) | 0.39399 (13) | 0.0203 (5) |
| C16 | 0.5546 (4) | 0.11407 (16) | 0.43777 (14) | 0.0265 (5) |
| H16A | 0.5282 | 0.1099 | 0.4973 | 0.037 (4)* |
| H16B | 0.7049 | 0.1347 | 0.4333 | 0.037 (4)* |


| H16C | 0.5357 | 0.0567 | 0.4114 | $0.037(4)^{*}$ |
| :--- | :--- | :--- | :--- | :--- |
| O21 | $0.4416(2)$ | $0.36282(9)$ | $0.23048(9)$ | $0.0178(3)$ |
| O22 | $0.1404(2)$ | $0.41038(10)$ | $0.15759(9)$ | $0.0231(3)$ |
| C25 | $0.3440(4)$ | $0.40733(13)$ | $0.16819(13)$ | $0.0185(5)$ |
| C26 | $0.4899(4)$ | $0.45081(15)$ | $0.11080(14)$ | $0.0240(5)$ |
| H26A | 0.5160 | 0.4114 | 0.0646 | $0.038(4)^{*}$ |
| H26B | 0.6306 | 0.4656 | 0.1423 | $0.038(4)^{*}$ |
| H26C | 0.4188 | 0.5039 | 0.0879 | $0.038(4)^{*}$ |

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

|  | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
| :--- | :--- | :--- | :--- | :--- | :--- | :--- |
| Sn1 | $0.01484(8)$ | $0.01307(8)$ | $0.01231(8)$ | $0.00027(6)$ | $0.00240(5)$ | $-0.00084(6)$ |
| C 11 | $0.0158(10)$ | $0.0177(11)$ | $0.0156(10)$ | $-0.0010(8)$ | $0.0038(8)$ | $-0.0048(8)$ |
| C 12 | $0.0162(11)$ | $0.0342(14)$ | $0.0260(12)$ | $0.0018(10)$ | $-0.0017(9)$ | $-0.0072(10)$ |
| C 13 | $0.0222(11)$ | $0.0235(13)$ | $0.0194(11)$ | $-0.0006(9)$ | $0.0037(9)$ | $-0.0078(9)$ |
| C 14 | $0.0294(13)$ | $0.0220(12)$ | $0.0276(12)$ | $-0.0095(10)$ | $0.0089(10)$ | $-0.0041(10)$ |
| C 21 | $0.0153(10)$ | $0.0166(11)$ | $0.0167(10)$ | $0.0006(8)$ | $0.0028(8)$ | $-0.0051(8)$ |
| C 22 | $0.0249(12)$ | $0.0239(13)$ | $0.0265(12)$ | $0.0005(10)$ | $0.0128(10)$ | $-0.0036(10)$ |
| C 23 | $0.0246(12)$ | $0.0266(13)$ | $0.0216(12)$ | $-0.0006(10)$ | $0.0018(9)$ | $-0.0093(9)$ |
| C24 | $0.0303(13)$ | $0.0204(12)$ | $0.0246(12)$ | $0.0065(10)$ | $0.0057(10)$ | $-0.0020(9)$ |
| O11 | $0.0211(8)$ | $0.0180(8)$ | $0.0199(8)$ | $0.0020(6)$ | $-0.0005(6)$ | $0.0035(6)$ |
| O12 | $0.0273(9)$ | $0.0222(9)$ | $0.0263(9)$ | $0.0039(7)$ | $0.0055(7)$ | $0.0017(7)$ |
| C15 | $0.0286(13)$ | $0.0147(11)$ | $0.0166(11)$ | $0.0014(9)$ | $-0.0025(9)$ | $-0.0031(8)$ |
| C16 | $0.0337(13)$ | $0.0229(12)$ | $0.0221(12)$ | $0.0072(10)$ | $-0.0024(10)$ | $0.0049(10)$ |
| O21 | $0.0192(8)$ | $0.0172(8)$ | $0.0174(7)$ | $-0.0003(6)$ | $0.0046(6)$ | $0.0027(6)$ |
| O22 | $0.0226(9)$ | $0.0267(9)$ | $0.0202(8)$ | $0.0019(7)$ | $0.0028(6)$ | $0.0013(6)$ |
| C25 | $0.0279(12)$ | $0.0137(11)$ | $0.0147(10)$ | $-0.0021(9)$ | $0.0055(9)$ | $-0.0036(8)$ |
| C26 | $0.0304(13)$ | $0.0215(13)$ | $0.0209(12)$ | $-0.0012(10)$ | $0.0076(10)$ | $0.0030(9)$ |

Geometric parameters ( $A,{ }^{\circ}$ )

| Sn1-O21 | 2.1001 (14) | C22-H22B | 0.9800 |
| :---: | :---: | :---: | :---: |
| Sn1-O11 | 2.1002 (14) | C22-H22C | 0.9800 |
| Sn1-C11 | 2.175 (2) | $\mathrm{C} 23-\mathrm{H} 23 \mathrm{~A}$ | 0.9800 |
| Sn1-C21 | 2.176 (2) | C23-H23B | 0.9800 |
| C11-C14 | 1.527 (3) | $\mathrm{C} 23-\mathrm{H} 23 \mathrm{C}$ | 0.9800 |
| C11-C12 | 1.528 (3) | C24-H24A | 0.9800 |
| C11-C13 | 1.530 (3) | C24-H24B | 0.9800 |
| C12-H12A | 0.9800 | C24-H24C | 0.9800 |
| C12-H12B | 0.9800 | O11-C15 | 1.304 (3) |
| C12-H12C | 0.9800 | O12-C15 | 1.235 (3) |
| C13-H13A | 0.9800 | C15-C16 | 1.497 (3) |
| C13-H13B | 0.9800 | C16-H16A | 0.9800 |
| C13-H13C | 0.9800 | C16-H16B | 0.9800 |
| C14-H14A | 0.9800 | C16-H16C | 0.9800 |
| C14-H14B | 0.9800 | O21-C25 | 1.304 (3) |
| C14-H14C | 0.9800 | O22-C25 | 1.239 (3) |


| C21-C24 | 1.522 (3) |
| :---: | :---: |
| C21-C22 | 1.525 (3) |
| $\mathrm{C} 21-\mathrm{C} 23$ | 1.530 (3) |
| C22-H22A | 0.9800 |
| O21-Sn1-O11 | 79.93 (6) |
| $\mathrm{O} 21-\mathrm{Sn} 1-\mathrm{C} 11$ | 107.88 (7) |
| O11-Sn1-C11 | 101.58 (7) |
| O21-Sn1-C21 | 102.54 (7) |
| O11-Sn1-C21 | 110.43 (7) |
| C11-Sn1-C21 | 138.97 (7) |
| C14-C11-C12 | 109.77 (18) |
| C14-C11-C13 | 109.93 (18) |
| C12-C11-C13 | 110.51 (17) |
| C14-C11-Sn1 | 109.51 (14) |
| C12-C11-Sn1 | 109.99 (14) |
| C13-C11-Sn1 | 107.09 (13) |
| $\mathrm{C} 11-\mathrm{C} 12-\mathrm{H} 12 \mathrm{~A}$ | 109.5 |
| C11-C12-H12B | 109.5 |
| $\mathrm{H} 12 \mathrm{~A}-\mathrm{C} 12-\mathrm{H} 12 \mathrm{~B}$ | 109.5 |
| $\mathrm{C} 11-\mathrm{C} 12-\mathrm{H} 12 \mathrm{C}$ | 109.5 |
| $\mathrm{H} 12 \mathrm{~A}-\mathrm{C} 12-\mathrm{H} 12 \mathrm{C}$ | 109.5 |
| $\mathrm{H} 12 \mathrm{~B}-\mathrm{C} 12-\mathrm{H} 12 \mathrm{C}$ | 109.5 |
| $\mathrm{C} 11-\mathrm{C} 13-\mathrm{H} 13 \mathrm{~A}$ | 109.5 |
| C11-C13-H13B | 109.5 |
| H13A-C13-H13B | 109.5 |
| C11-C13-H13C | 109.5 |
| H13A-C13-H13C | 109.5 |
| $\mathrm{H} 13 \mathrm{~B}-\mathrm{C} 13-\mathrm{H} 13 \mathrm{C}$ | 109.5 |
| C11-C14-H14A | 109.5 |
| C11-C14-H14B | 109.5 |
| H14A-C14-H14B | 109.5 |
| C11-C14-H14C | 109.5 |
| H14A-C14-H14C | 109.5 |
| H14B-C14-H14C | 109.5 |
| C24-C21-C22 | 109.65 (17) |
| C24-C21-C23 | 110.46 (18) |
| C22-C21-C23 | 110.65 (17) |
| C24-C21-Sn1 | 109.79 (13) |
| C22-C21-Sn1 | 110.17 (14) |
| C23-C21-Sn1 | 106.07 (13) |
| C21-C22-H22A | 109.5 |


| C25-C26 | 1.495 (3) |
| :---: | :---: |
| C26-H26A | 0.9800 |
| C26-H26B | 0.9800 |
| C26-H26C | 0.9800 |
| $\mathrm{C} 21-\mathrm{C} 22-\mathrm{H} 22 \mathrm{~B}$ | 109.5 |
| $\mathrm{H} 22 \mathrm{~A}-\mathrm{C} 22-\mathrm{H} 22 \mathrm{~B}$ | 109.5 |
| $\mathrm{C} 21-\mathrm{C} 22-\mathrm{H} 22 \mathrm{C}$ | 109.5 |
| $\mathrm{H} 22 \mathrm{~A}-\mathrm{C} 22-\mathrm{H} 22 \mathrm{C}$ | 109.5 |
| $\mathrm{H} 22 \mathrm{~B}-\mathrm{C} 22-\mathrm{H} 22 \mathrm{C}$ | 109.5 |
| $\mathrm{C} 21-\mathrm{C} 23-\mathrm{H} 23 \mathrm{~A}$ | 109.5 |
| $\mathrm{C} 21-\mathrm{C} 23-\mathrm{H} 23 \mathrm{~B}$ | 109.5 |
| $\mathrm{H} 23 \mathrm{~A}-\mathrm{C} 23-\mathrm{H} 23 \mathrm{~B}$ | 109.5 |
| C21-C23-H23C | 109.5 |
| $\mathrm{H} 23 \mathrm{~A}-\mathrm{C} 23-\mathrm{H} 23 \mathrm{C}$ | 109.5 |
| H23B-C23-H23C | 109.5 |
| C21-C24-H24A | 109.5 |
| C21-C24-H24B | 109.5 |
| $\mathrm{H} 24 \mathrm{~A}-\mathrm{C} 24-\mathrm{H} 24 \mathrm{~B}$ | 109.5 |
| C21-C24-H24C | 109.5 |
| $\mathrm{H} 24 \mathrm{~A}-\mathrm{C} 24-\mathrm{H} 24 \mathrm{C}$ | 109.5 |
| H24B-C24-H24C | 109.5 |
| C15-O11-Sn1 | 104.81 (13) |
| O12-C15-O11 | 120.3 (2) |
| O12-C15-C16 | 123.1 (2) |
| O11-C15-C16 | 116.6 (2) |
| C15-C16-H16A | 109.5 |
| C15-C16-H16B | 109.5 |
| H16A-C16-H16B | 109.5 |
| C15-C16-H16C | 109.5 |
| H16A-C16-H16C | 109.5 |
| H16B-C16-H16C | 109.5 |
| C25-O21-Sn1 | 106.07 (13) |
| $\mathrm{O} 22-\mathrm{C} 25-\mathrm{O} 21$ | 120.25 (19) |
| O22-C25-C26 | 123.21 (19) |
| O21-C25-C26 | 116.51 (19) |
| C25-C26-H26A | 109.5 |
| C25-C26-H26B | 109.5 |
| H26A-C26-H26B | 109.5 |
| C25-C26-H26C | 109.5 |
| H26A-C26-H26C | 109.5 |
| H26B-C26-H26C | 109.5 |

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

| $D-\mathrm{H} \cdots A$ | $D-\mathrm{H}$ | $\mathrm{H} \cdots A$ | $D \cdots A$ | $D-\mathrm{H} \cdots A$ |
| :--- | :--- | :--- | :--- | :--- |
| $\mathrm{C} 12 — \mathrm{H} 12 C \cdots \mathrm{O} 21^{\mathrm{i}}$ | 0.98 | 2.54 | $3.362(3)$ | 142 |
| $\mathrm{C} 22 — \mathrm{H} 22 C \cdots \mathrm{O} 11^{\mathrm{i}}$ | 0.98 | 2.65 | $3.584(3)$ | 160 |

## supporting information

| $\mathrm{C} 16-\mathrm{H} 16 C \cdots \mathrm{O} 22^{\mathrm{ii}}$ | 0.98 | 2.69 | $3.636(3)$ | 163 |
| :--- | :--- | :--- | :--- | :--- |
| $\mathrm{C} 16-\mathrm{H} 16 A \cdots \mathrm{O} 22^{\mathrm{iii}}$ | 0.98 | 2.60 | $3.518(3)$ | 155 |

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

