

Dichloridobis(di-*tert*-butylmethylphosphine oxide- κ O)diphenyltin(IV)

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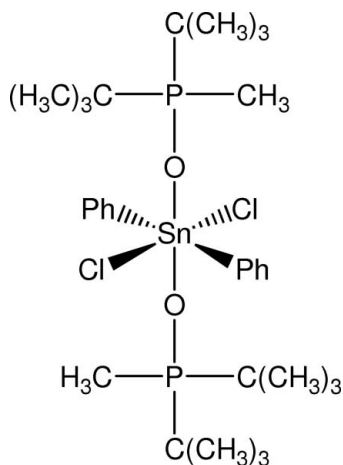
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Key indicators: single-crystal X-ray study; $T = 173$ K; mean $\sigma(\text{C}-\text{C}) = 0.012$ Å; R factor = 0.058; wR factor = 0.099; data-to-parameter ratio = 18.6.

The complete molecule of the title compound, $[\text{Sn}(\text{C}_6\text{H}_5)_2\text{Cl}_2(\text{C}_9\text{H}_{21}\text{OP})_2]$, is generated by crystallographic inversion symmetry, the Sn atom is located on a special position of site symmetry $\bar{1}$. The Sn atom adopts an all-*trans* $\text{SnC}_2\text{O}_2\text{Cl}_2$ octahedral geometry. As a consequence of the bulky substituents at the O atom, the P—O—Sn bond angle is 163.9 (3)°.

Related literature

For related literature, see: Lerner *et al.* (2005); Ruth *et al.* (2005, 2007).



Experimental

Crystal data

$[\text{Sn}(\text{C}_6\text{H}_5)_2\text{Cl}_2(\text{C}_9\text{H}_{21}\text{OP})_2]$
 $M_r = 696.25$

Monoclinic, $P2_1/c$
 $a = 12.1782$ (19) Å
 $b = 9.0866$ (8) Å
 $c = 16.339$ (2) Å
 $\beta = 111.518$ (11)°

$V = 1682.0$ (4) Å³
 $Z = 2$

Mo $K\alpha$ radiation
 $\mu = 1.04$ mm⁻¹
 $T = 173$ (2) K
 $0.13 \times 0.09 \times 0.07$ mm

Data collection

Stoe IPDSII two-circle diffractometer
 Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
 $T_{\min} = 0.877$, $T_{\max} = 0.931$

1731 measured reflections
 3145 independent reflections
 1754 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.087$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.058$
 $wR(F^2) = 0.098$
 $S = 0.88$
 3145 reflections

169 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.44$ e Å⁻³
 $\Delta\rho_{\min} = -0.83$ e Å⁻³

Table 1

Selected bond lengths (Å).

Sn1—C41	2.128 (7)	Sn1—Cl1	2.5567 (16)
Sn1—O1	2.232 (4)		

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2731).

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supporting information

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Dichloridobis(di-*tert*-butylmethylphosphine oxide- κ O)diphenyltin(IV)

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S1. Comment

We are interested in Lewis-acidic Sn(IV) compounds and their reactivity towards Lewis bases. Recently we have reported the synthesis and structure of $\{\text{Zn}[\text{Sn}(\text{CH}_2\text{SMe})_4]0.5\text{Cl}_2\}_n$ and $\text{Sn}(\text{CH}_2\text{PPh}_2)_4$ (Ruth *et al.*, 2007). In contrast to $[\text{SnCl}_4] \cdot [\text{CH}_3\text{SCH}_3]_2$ which forms an adduct in solid state with a six-coordinated Sn atom (Ruth *et al.*, 2005), the Sn(IV) centers in $\{\text{Zn}[\text{Sn}(\text{CH}_2\text{SMe})_4]0.5\text{Cl}_2\}_n$ and $\text{Sn}(\text{CH}_2\text{PPh}_2)_4$ are tetra-coordinated. However, Me_3SnCl forms an adduct with Me_3SnOH and H_2O in which the Sn atoms possess the coordination number five. It is interesting to note that this adduct represents an intermediate in Me_3SnCl hydrolysis (Lerner *et al.*, 2005). We report here the X-ray crystal structure analysis of the title adduct $[\text{Ph}_2\text{SnCl}_2] \cdot [t\text{Bu}_2\text{MePO}]_2$, (I). The synthesis of (I) was achieved by treatment of Ph_2SnCl_2 with two equivalents of $t\text{Bu}_2\text{MePO}$ as indicated in the equation below.

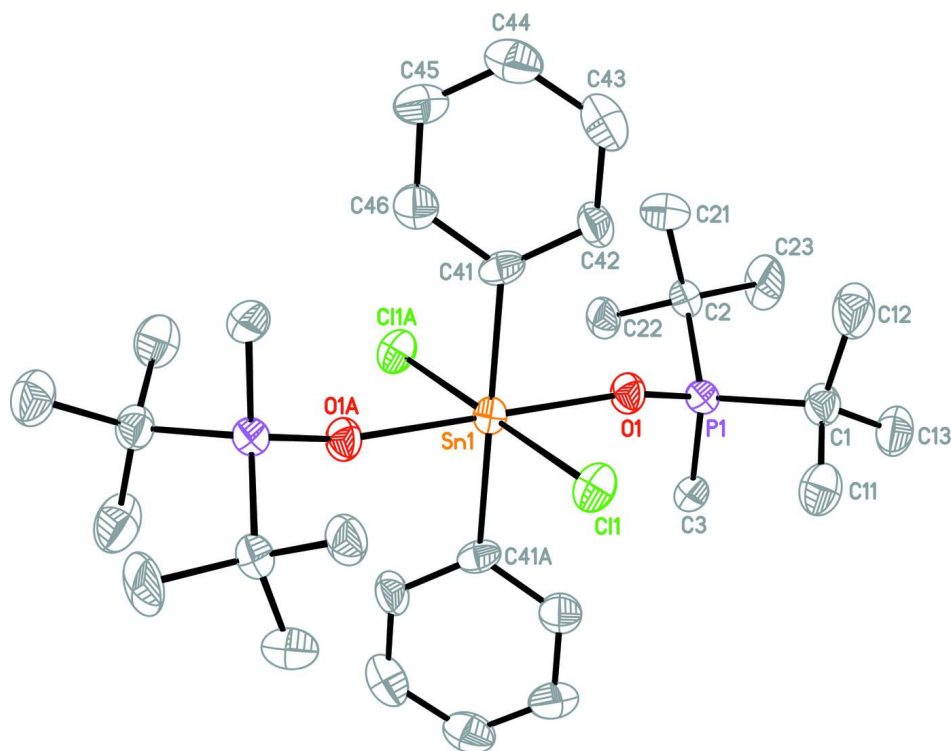
Compound (I) has crystallographic inversion symmetry with just half a molecule in the asymmetric unit. The Sn atom is hexacoordinated by three pairs of different ligands in an octahedral fashion (Table 1). All ligand pairs of the same kind are mutually *trans* at the Sn atom (Fig. 1). As a consequence of the bulky substituents at the O atom the P—O—Sn angle is enlarged to $163.9(3)^\circ$.

S2. Experimental

$t\text{Bu}_2\text{MePO}$ (2.05 mmol) was added with stirring at ambient temperature to a solution of Ph_2SnCl_2 (0.58 mmol) in 25 ml THF. Colourless blocks of (I) were grown by storing this solution at room temperature for several weeks.

S3. Refinement

The H atoms were geometrically positioned (C—H = 0.95–0.98 Å) and refined as riding with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{methyl C})$.


Figure 1

Perspective view of (I) with displacement ellipsoids drawn at the 50% probability level; H atoms are omitted for clarity. Symmetry operator for generating equivalent atoms: (A) $1 - x, 1 - y, 1 - z$.

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Crystal data

$[\text{Sn}(\text{C}_6\text{H}_5)_2\text{Cl}_2(\text{C}_9\text{H}_{21}\text{OP})_2]$

$M_r = 696.25$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2_1/c$

$a = 12.1782\ (19)\ \text{\AA}$

$b = 9.0866\ (8)\ \text{\AA}$

$c = 16.339\ (2)\ \text{\AA}$

$\beta = 111.518\ (11)^\circ$

$V = 1682.0\ (4)\ \text{\AA}^3$

$Z = 2$

$F(000) = 724$

$D_x = 1.375\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 4261 reflections

$\theta = 3.5\text{--}25.4^\circ$

$\mu = 1.04\ \text{mm}^{-1}$

$T = 173\ \text{K}$

Block, colourless

$0.13 \times 0.09 \times 0.07\ \text{mm}$

Data collection

Stoe IPDSII two-circle
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

ω scans

Absorption correction: multi-scan

(*MULABS*; Spek, 2003; Blessing, 1995)

$T_{\min} = 0.877, T_{\max} = 0.931$

11731 measured reflections

3145 independent reflections

1754 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.087$

$\theta_{\max} = 25.6^\circ, \theta_{\min} = 3.4^\circ$

$h = -14 \rightarrow 14$

$k = -10 \rightarrow 11$

$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.058$
 $wR(F^2) = 0.098$
 $S = 0.88$
 3145 reflections
 169 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: inferred from
 neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0102P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.44 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.83 \text{ e } \text{Å}^{-3}$

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.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 wR 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 $F^2 > \sigma(F^2)$ is used only for calculating R -factors(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 (Å^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Sn1	0.5000	0.5000	0.5000	0.02649 (18)
Cl1	0.46725 (18)	0.3097 (2)	0.60405 (11)	0.0361 (5)
P1	0.31774 (17)	0.7748 (2)	0.56073 (11)	0.0271 (4)
O1	0.3737 (4)	0.6468 (5)	0.5312 (3)	0.0300 (11)
C1	0.2236 (7)	0.7021 (8)	0.6188 (4)	0.0340 (17)
C2	0.2381 (6)	0.8878 (8)	0.4633 (4)	0.0337 (17)
C3	0.4234 (7)	0.8947 (8)	0.6369 (4)	0.0360 (18)
H3A	0.4684	0.8398	0.6903	0.054*
H3B	0.3822	0.9768	0.6521	0.054*
H3C	0.4774	0.9331	0.6100	0.054*
C11	0.3017 (7)	0.5921 (9)	0.6885 (5)	0.043 (2)
H11A	0.3721	0.6430	0.7282	0.064*
H11B	0.3257	0.5114	0.6589	0.064*
H11C	0.2568	0.5523	0.7225	0.064*
C12	0.1142 (8)	0.6241 (10)	0.5567 (5)	0.058 (3)
H12A	0.0656	0.6940	0.5126	0.086*
H12B	0.0686	0.5850	0.5903	0.086*
H12C	0.1380	0.5431	0.5273	0.086*
C13	0.1867 (8)	0.8238 (9)	0.6696 (5)	0.045 (2)
H13A	0.2574	0.8731	0.7101	0.068*
H13B	0.1428	0.7798	0.7031	0.068*
H13C	0.1366	0.8958	0.6279	0.068*
C21	0.1735 (8)	0.7857 (10)	0.3860 (5)	0.054 (2)
H21A	0.1137	0.7289	0.3994	0.081*
H21B	0.2303	0.7182	0.3762	0.081*

H21C	0.1351	0.8446	0.3329	0.081*
C22	0.3310 (6)	0.9731 (9)	0.4389 (4)	0.036 (2)
H22A	0.3744	1.0397	0.4871	0.054*
H22B	0.2916	1.0304	0.3853	0.054*
H22C	0.3862	0.9035	0.4288	0.054*
C23	0.1515 (7)	0.9993 (15)	0.4782 (5)	0.064 (3)
H23A	0.0918	0.9463	0.4938	0.096*
H23B	0.1128	1.0558	0.4242	0.096*
H23C	0.1950	1.0664	0.5261	0.096*
C41	0.3564 (7)	0.4098 (7)	0.3934 (4)	0.0276 (16)
C42	0.2454 (7)	0.3792 (8)	0.3993 (5)	0.0368 (18)
H42	0.2346	0.3983	0.4530	0.044*
C43	0.1523 (8)	0.3224 (9)	0.3291 (5)	0.049 (2)
H43	0.0771	0.3099	0.3334	0.059*
C44	0.1696 (8)	0.2832 (9)	0.2512 (5)	0.046 (2)
H44	0.1076	0.2388	0.2039	0.055*
C45	0.2763 (8)	0.3097 (9)	0.2440 (4)	0.042 (2)
H45	0.2876	0.2845	0.1912	0.051*
C46	0.3695 (7)	0.3738 (8)	0.3140 (4)	0.0347 (18)
H46	0.4424	0.3930	0.3074	0.042*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sn1	0.0255 (4)	0.0305 (4)	0.0254 (3)	0.0007 (6)	0.0116 (3)	-0.0013 (5)
C11	0.0442 (12)	0.0352 (11)	0.0335 (9)	-0.0015 (10)	0.0198 (9)	0.0010 (8)
P1	0.0226 (11)	0.0324 (11)	0.0262 (8)	0.0000 (9)	0.0086 (8)	-0.0023 (8)
O1	0.031 (3)	0.029 (3)	0.033 (2)	0.006 (2)	0.015 (2)	-0.002 (2)
C1	0.032 (5)	0.037 (4)	0.036 (4)	-0.005 (4)	0.017 (3)	-0.010 (3)
C2	0.024 (4)	0.045 (5)	0.034 (4)	0.001 (4)	0.013 (3)	-0.003 (3)
C3	0.032 (5)	0.052 (5)	0.025 (3)	-0.003 (4)	0.012 (3)	0.000 (3)
C11	0.051 (6)	0.039 (5)	0.046 (4)	-0.005 (4)	0.027 (4)	0.004 (4)
C12	0.055 (6)	0.072 (7)	0.058 (5)	-0.030 (5)	0.034 (5)	-0.027 (5)
C13	0.048 (6)	0.052 (5)	0.046 (4)	-0.006 (5)	0.029 (4)	-0.003 (4)
C21	0.038 (5)	0.078 (7)	0.036 (4)	0.003 (5)	0.002 (4)	0.001 (4)
C22	0.034 (4)	0.040 (6)	0.039 (3)	0.015 (4)	0.018 (3)	0.009 (4)
C23	0.057 (6)	0.087 (6)	0.059 (4)	0.056 (8)	0.033 (4)	0.026 (7)
C41	0.036 (5)	0.021 (4)	0.021 (3)	0.000 (3)	0.004 (3)	-0.004 (3)
C42	0.024 (4)	0.047 (5)	0.043 (4)	-0.009 (4)	0.017 (4)	-0.013 (4)
C43	0.033 (5)	0.052 (6)	0.066 (5)	0.000 (5)	0.022 (4)	0.000 (4)
C44	0.036 (5)	0.041 (5)	0.048 (4)	-0.002 (4)	0.003 (4)	-0.006 (4)
C45	0.043 (5)	0.047 (5)	0.029 (4)	-0.004 (4)	0.004 (4)	0.001 (3)
C46	0.036 (5)	0.032 (5)	0.035 (4)	0.007 (4)	0.012 (4)	0.008 (3)

Geometric parameters (Å, °)

Sn1—C41 ⁱ	2.128 (7)	C12—H12C	0.9800
Sn1—C41	2.128 (7)	C13—H13A	0.9800

Sn1—O1	2.232 (4)	C13—H13B	0.9800
Sn1—O1 ⁱ	2.232 (4)	C13—H13C	0.9800
Sn1—Cl1 ⁱ	2.5567 (16)	C21—H21A	0.9800
Sn1—Cl1	2.5567 (16)	C21—H21B	0.9800
P1—O1	1.513 (4)	C21—H21C	0.9800
P1—C3	1.794 (7)	C22—H22A	0.9800
P1—C2	1.842 (7)	C22—H22B	0.9800
P1—C1	1.858 (7)	C22—H22C	0.9800
C1—C12	1.522 (10)	C23—H23A	0.9800
C1—C11	1.552 (10)	C23—H23B	0.9800
C1—C13	1.545 (9)	C23—H23C	0.9800
C2—C22	1.541 (10)	C41—C46	1.402 (8)
C2—C21	1.533 (11)	C41—C42	1.418 (10)
C2—C23	1.544 (11)	C42—C43	1.383 (10)
C3—H3A	0.9800	C42—H42	0.9500
C3—H3B	0.9800	C43—C44	1.412 (10)
C3—H3C	0.9800	C43—H43	0.9500
C11—H11A	0.9800	C44—C45	1.367 (11)
C11—H11B	0.9800	C44—H44	0.9500
C11—H11C	0.9800	C45—C46	1.409 (10)
C12—H12A	0.9800	C45—H45	0.9500
C12—H12B	0.9800	C46—H46	0.9500
C41 ⁱ —Sn1—C41	180.0	C1—C12—H12B	109.5
C41 ⁱ —Sn1—O1	90.6 (2)	H12A—C12—H12B	109.5
C41—Sn1—O1	89.4 (2)	C1—C12—H12C	109.5
C41 ⁱ —Sn1—O1 ⁱ	89.4 (2)	H12A—C12—H12C	109.5
C41—Sn1—O1 ⁱ	90.6 (2)	H12B—C12—H12C	109.5
O1—Sn1—O1 ⁱ	180.0	C1—C13—H13A	109.5
C41 ⁱ —Sn1—Cl1 ⁱ	90.10 (18)	C1—C13—H13B	109.5
C41—Sn1—Cl1 ⁱ	89.90 (18)	H13A—C13—H13B	109.5
O1—Sn1—Cl1 ⁱ	92.09 (12)	C1—C13—H13C	109.5
O1 ⁱ —Sn1—Cl1 ⁱ	87.91 (12)	H13A—C13—H13C	109.5
C41 ⁱ —Sn1—Cl1	89.90 (18)	H13B—C13—H13C	109.5
C41—Sn1—Cl1	90.10 (18)	C2—C21—H21A	109.5
O1—Sn1—Cl1	87.91 (12)	C2—C21—H21B	109.5
O1 ⁱ —Sn1—Cl1	92.09 (12)	H21A—C21—H21B	109.5
Cl1 ⁱ —Sn1—Cl1	180.0	C2—C21—H21C	109.5
O1—P1—C3	113.3 (3)	H21A—C21—H21C	109.5
O1—P1—C2	108.0 (3)	H21B—C21—H21C	109.5
C3—P1—C2	106.1 (3)	C2—C22—H22A	109.5
O1—P1—C1	108.9 (3)	C2—C22—H22B	109.5
C3—P1—C1	106.4 (3)	H22A—C22—H22B	109.5
C2—P1—C1	114.3 (3)	C2—C22—H22C	109.5
P1—O1—Sn1	163.9 (3)	H22A—C22—H22C	109.5
C12—C1—C11	109.9 (7)	H22B—C22—H22C	109.5
C12—C1—C13	109.6 (6)	C2—C23—H23A	109.5
C11—C1—C13	106.7 (6)	C2—C23—H23B	109.5

C12—C1—P1	112.3 (4)	H23A—C23—H23B	109.5
C11—C1—P1	106.1 (5)	C2—C23—H23C	109.5
C13—C1—P1	112.0 (5)	H23A—C23—H23C	109.5
C22—C2—C21	107.1 (6)	H23B—C23—H23C	109.5
C22—C2—C23	108.7 (7)	C46—C41—C42	116.8 (7)
C21—C2—C23	110.7 (7)	C46—C41—Sn1	120.6 (5)
C22—C2—P1	107.5 (5)	C42—C41—Sn1	122.6 (5)
C21—C2—P1	108.9 (5)	C43—C42—C41	122.0 (6)
C23—C2—P1	113.8 (4)	C43—C42—H42	119.0
P1—C3—H3A	109.5	C41—C42—H42	119.0
P1—C3—H3B	109.5	C42—C43—C44	119.6 (7)
H3A—C3—H3B	109.5	C42—C43—H43	120.2
P1—C3—H3C	109.5	C44—C43—H43	120.2
H3A—C3—H3C	109.5	C45—C44—C43	119.6 (8)
H3B—C3—H3C	109.5	C45—C44—H44	120.2
C1—C11—H11A	109.5	C43—C44—H44	120.2
C1—C11—H11B	109.5	C44—C45—C46	120.7 (7)
H11A—C11—H11B	109.5	C44—C45—H45	119.6
C1—C11—H11C	109.5	C46—C45—H45	119.6
H11A—C11—H11C	109.5	C41—C46—C45	121.1 (7)
H11B—C11—H11C	109.5	C41—C46—H46	119.4
C1—C12—H12A	109.5	C45—C46—H46	119.4
C3—P1—O1—Sn1	-21.3 (11)	C1—P1—C2—C21	-79.8 (6)
C2—P1—O1—Sn1	95.9 (10)	O1—P1—C2—C23	165.5 (7)
C1—P1—O1—Sn1	-139.5 (9)	C3—P1—C2—C23	-72.7 (7)
C41 ⁱ —Sn1—O1—P1	31.6 (10)	C1—P1—C2—C23	44.2 (8)
C41—Sn1—O1—P1	-148.4 (10)	O1—Sn1—C41—C46	142.0 (5)
Cl1 ⁱ —Sn1—O1—P1	-58.5 (10)	O1 ⁱ —Sn1—C41—C46	-38.0 (5)
Cl1—Sn1—O1—P1	121.5 (10)	Cl1 ⁱ —Sn1—C41—C46	49.9 (5)
O1—P1—C1—C12	-69.5 (6)	Cl1—Sn1—C41—C46	-130.1 (5)
C3—P1—C1—C12	168.1 (6)	O1—Sn1—C41—C42	-39.9 (6)
C2—P1—C1—C12	51.4 (7)	O1 ⁱ —Sn1—C41—C42	140.1 (6)
O1—P1—C1—C11	50.5 (5)	Cl1 ⁱ —Sn1—C41—C42	-132.0 (6)
C3—P1—C1—C11	-71.9 (5)	Cl1—Sn1—C41—C42	48.0 (6)
C2—P1—C1—C11	171.4 (5)	C46—C41—C42—C43	-2.7 (11)
O1—P1—C1—C13	166.6 (5)	Sn1—C41—C42—C43	179.1 (6)
C3—P1—C1—C13	44.2 (6)	C41—C42—C43—C44	4.7 (12)
C2—P1—C1—C13	-72.5 (6)	C42—C43—C44—C45	-3.8 (12)
O1—P1—C2—C22	-74.1 (5)	C43—C44—C45—C46	0.8 (12)
C3—P1—C2—C22	47.7 (5)	C42—C41—C46—C45	-0.3 (10)
C1—P1—C2—C22	164.5 (5)	Sn1—C41—C46—C45	178.0 (6)
O1—P1—C2—C21	41.6 (6)	C44—C45—C46—C41	1.2 (12)
C3—P1—C2—C21	163.4 (5)		

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