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# Dicyclohexylammonium bis(chlorodifluoroacetato-κO)cyclopentyldiphenylstannate(IV)

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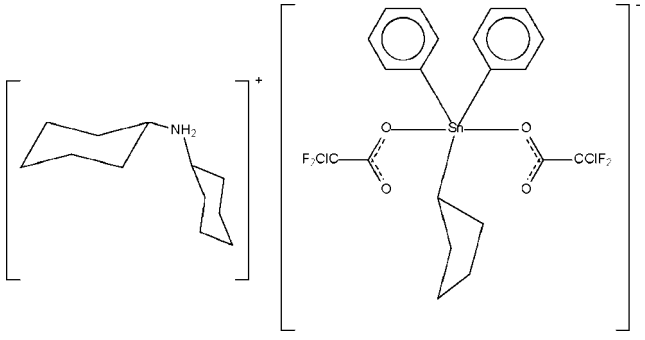
Key indicators: single-crystal X-ray study; $T$ = 100 K; mean $\sigma$(C–C) = 0.010 Å; disorder in main residue; $R$ factor = 0.050; $wR$ factor = 0.133; data-to-parameter ratio = 18.6.

The five-coordinate Sn atom in the title mixed organyl stannate compound, $(C_{12}H_{24}N)[Sn(C_5H_9)(C_6H_5)_2(C_2ClF_2O_2)]$, is in a *trans*-$C_3SnO_2$ trigonal–bipyramidal coordination environment. The $NH_2$ groups of the cations act as hydrogen-bond donors to two symmetry-related anions, resulting in the formation of linear chains. One of the phenyl rings is disordered over two sites with equal occupancies.

## Related literature

For details of the crystal structure of dicyclohexylammonium bis(chlorodifluoroacetato)cyclohexyldiphenylstannate(IV), see Teo *et al.* (2008). For a review of the structural chemistry of organotin carboxylates, see: Tiekink (1991, 1994).




## Experimental

### Crystal data

$(C_{12}H_{24}N)[Sn(C_5H_9)(C_6H_5)_2(C_2ClF_2O_2)]$
$M_r$ = 783.27
Monoclinic, $P2_1$
$a$ = 8.8610 (2) Å
$b$ = 19.3132 (3) Å
$c$ = 10.6823 (2) Å
$\beta$ = 109.385 (1)°
$V$ = 1724.47 (6) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.95 mm$^{-1}$
$T$ = 100 (2) K
0.30 × 0.20 × 0.15 mm

### Data collection

Bruker SMART APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.679, $T_{max}$ = 0.870
18017 measured reflections
7831 independent reflections
6637 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.038

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.132
$S$ = 1.04
7831 reflections
421 parameters
41 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.11 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.71 e Å$^{-3}$
Absolute structure: Flack (1983), 3766 Friedel pairs
Flack parameter: −0.03 (3)

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| Sn1—C1 | 2.147 (5) | Sn1—O1 | 2.287 (4) |
| Sn1—C7 | 2.136 (6) | Sn1—O3 | 2.249 (4) |
| Sn1—C13 | 2.117 (6) | | |
| C1—Sn1—C7 | 119.2 (2) | C7—Sn1—O1 | 90.6 (2) |
| C1—Sn1—C13 | 121.7 (2) | C7—Sn1—O3 | 87.8 (2) |
| C1—Sn1—O1 | 91.3 (2) | C13—Sn1—O1 | 82.8 (3) |
| C1—Sn1—O3 | 90.9 (2) | C13—Sn1—O3 | 96.5 (3) |
| C7—Sn1—C13 | 118.9 (2) | O1—Sn1—O3 | 177.7 (2) |

**Table 2**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1*N*1···O2 | 0.88 | 1.88 | 2.758 (6) | 173 |
| N1—H1*N*2···O4$^{i}$ | 0.88 | 1.93 | 2.804 (6) | 169 |

Symmetry code: (i) $-x+1, y+\frac{1}{2}, -z+1$.

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2611).

## References

Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Bruker (2007). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Teo, Y. Y., Lo, K. M. & Ng, S. W. (2008). *Acta Cryst.* E**64** (tk2258).
Tiekink, E. R. T. (1991). *Appl. Organomet. Chem.* **5**, 1–23.
Tiekink, E. R. T. (1994). *Trends Organomet. Chem.* **1**, 71–116.
Westrip, S. P. (2008). *publCIF*. In preparation.

# supporting information

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# Dicyclohexylammonium bis(chlorodifluoroacetato-$\kappa O$)cyclopentyldiphenylstannate(IV)

**Yin Yin Teo, Kong Mun Lo and Seik Weng Ng**

## S1. Comment

The preceding study reports the crystal structure of dicyclohexylammonium bis(chlorodifluroacetato)cyclohexyldiphenylstannate (Teo *et al.*, 2008).

Replacing the cyclohexyl ligand on tin by the cyclopentyl ligand furnishes a similar stannate compound(I), (Fig. 1). The cation engages in hydrogen bonding to the anion to give rise to a chain structure.

## S2. Experimental

Cyclopentyldiphenyltin hydroxide (0.36 g, 1 mmol) was added to an ethanol solution (50 ml) of dicyclohexylamine (0.20 ml, 2 mmol) and chlorodifluoroacetic acid (0.1 ml, 2 mmol). The solution was heated to dissolve the reactants completely; the filtered solution yielded the salt in 70% yield.

## S3. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.95 to 0.99 Å) and were included in the refinement in the riding model approximation, with $U$(H) set to 1.2$U_{eq}$(C). The ammonium H atoms (N–H 0.88 Å) were similarly treated.

For the cyclopentyl ligand, the 1,2-relatd C–C distances were restrained to 1.54±0.01 Å and the 1,3-related one to 2.51 + 0.01 Å. One of the aromatic rings is disordered over two sites; the occupancies could not be refined, and was assumed to be 1:1. For this ring, the 1,2-related distances were restrained to 1.39±0.01 Å and the 1,4-related ones to 2.78±0.01 Å. The temperature factors of the primed atoms were set to those of the unprimed ones; the six atoms of each ring were restrained to be approximately flat.

There is minor disorder in the chlorodifluoromethyl groups; the C–Cl distance was restrained to 1.75±0.01 Å and the C–F distances to 1.35±0.01 Å. The final difference Fourier map had a large peak in the vicinity of Sn1.

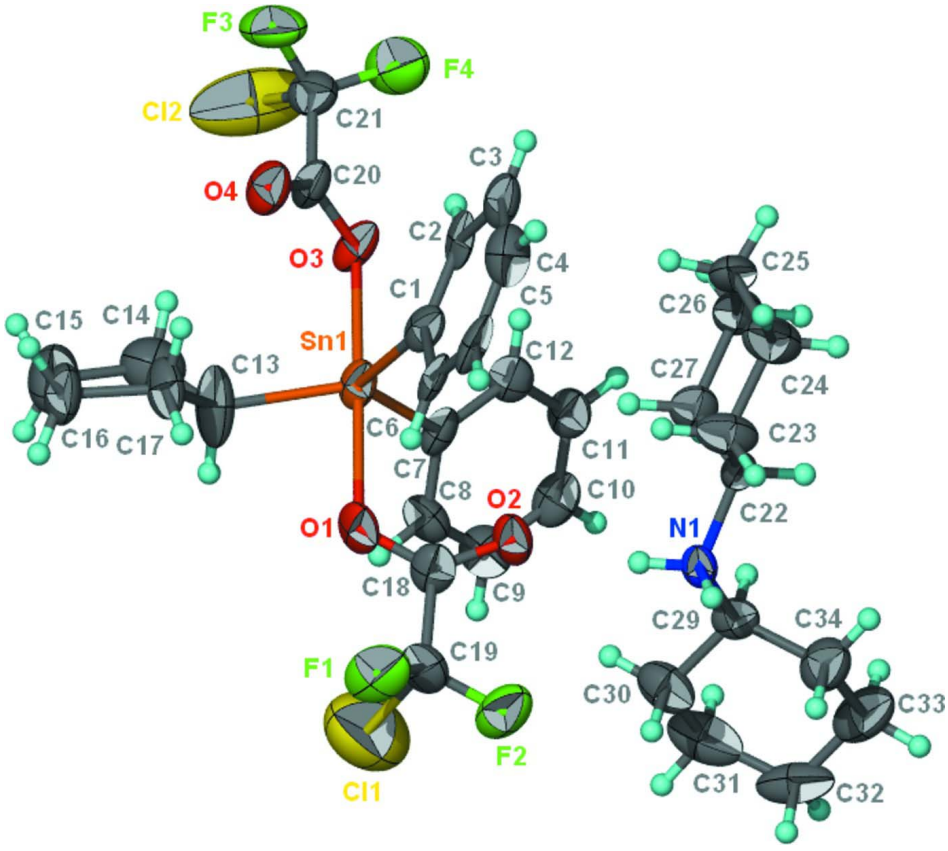


**Figure 1**

50% Probability thermal ellipsoid plot of the two independent formula units of $[(C_6H_{11})_2NH_2)]$ $[Sn(C_5H_9)(C_6H_5)_2(O_2CClF_2)_2]$. Hydrogen atoms are drawn as spheres of arbitrary radius. The disorder is not shown.

### Dicyclohexylammonium bis(chlorodifluoroacetato-$\kappa O$)cyclopentylhexyldiphenylstannate(IV)]

*Crystal data*

$(C_{12}H_{24}N)[Sn(C_5H_9)(C_6H_5)_2(C_2ClF_2O_2)]$
$M_r = 783.27$
Monoclinic, $P2_1$
Hall symbol: P 2yb
$a = 8.8610$ (2) Å
$b = 19.3132$ (3) Å
$c = 10.6823$ (2) Å
$\beta = 109.385$ (1)°
$V = 1724.47$ (6) Å$^3$
$Z = 2$
$F(000) = 800$
$D_x = 1.508$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4999 reflections
$\theta = 2.3$–24.4°
$\mu = 0.95$ mm$^{-1}$
$T = 100$ K
Irregular block, colorless
0.30 × 0.20 × 0.15 mm

*Data collection*

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.679$, $T_{max} = 0.870$
18017 measured reflections
7831 independent reflections
6637 reflections with $I > 2\sigma(I)$
$R_{int} = 0.038$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.0°$
$h = -11 \rightarrow 10$
$k = -25 \rightarrow 25$
$l = -13 \rightarrow 13$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.132$
$S = 1.04$
7831 reflections
421 parameters
41 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
$w = 1/[\sigma^2(F_o^2) + (0.0717P)^2 + 1.4264P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.11$ e Å$^{-3}$
$\Delta\rho_{min} = -0.71$ e Å$^{-3}$
Absolute structure: Flack (1983), 3766 Friedel pairs
Absolute structure parameter: −0.03 (3)

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| Sn1 | 0.41483 (4) | 0.50001 (7) | 0.27785 (3) | 0.03273 (11) | |
| Cl1 | 0.5652 (5) | 0.7213 (2) | 0.0894 (3) | 0.1213 (13) | |
| Cl2 | 0.1779 (5) | 0.25792 (18) | 0.2051 (3) | 0.1310 (16) | |
| F1 | 0.7544 (5) | 0.7106 (2) | 0.3078 (5) | 0.0684 (13) | |
| F2 | 0.5523 (5) | 0.7777 (2) | 0.2839 (5) | 0.0622 (12) | |
| F3 | 0.3331 (6) | 0.2399 (2) | 0.4277 (5) | 0.0721 (14) | |
| F4 | 0.1352 (6) | 0.3071 (3) | 0.4035 (6) | 0.0776 (15) | |
| O1 | 0.5515 (5) | 0.5970 (2) | 0.2551 (5) | 0.0389 (10) | |
| O2 | 0.4078 (5) | 0.6683 (2) | 0.3391 (5) | 0.0372 (10) | |
| O3 | 0.2746 (5) | 0.4049 (2) | 0.2913 (4) | 0.0366 (9) | |
| O4 | 0.4990 (6) | 0.3475 (2) | 0.4043 (5) | 0.0419 (10) | |
| N1 | 0.2491 (6) | 0.7638 (2) | 0.4417 (4) | 0.0264 (9) | |
| H1N1 | 0.2976 | 0.7356 | 0.4026 | 0.032* | |
| H1N2 | 0.3196 | 0.7947 | 0.4861 | 0.032* | |
| C1 | 0.5036 (5) | 0.5092 (2) | 0.4901 (5) | 0.0335 (12) | |
| C2 | 0.445 (4) | 0.4703 (16) | 0.574 (4) | 0.034 (3) | 0.50 |
| H2 | 0.3604 | 0.4389 | 0.5336 | 0.040* | 0.50 |
| C3 | 0.502 (6) | 0.474 (2) | 0.711 (4) | 0.040 (5) | 0.50 |
| H3 | 0.4581 | 0.4462 | 0.7645 | 0.047* | 0.50 |
| C4 | 0.625 (6) | 0.520 (3) | 0.7657 (10) | 0.0475 (18) | 0.50 |
| H4 | 0.6666 | 0.5237 | 0.8597 | 0.057* | 0.50 |
| C5 | 0.693 (5) | 0.561 (2) | 0.692 (4) | 0.037 (4) | 0.50 |
| H5 | 0.7779 | 0.5926 | 0.7332 | 0.045* | 0.50 |
| C6 | 0.6282 (18) | 0.5547 (12) | 0.555 (4) | 0.033 (3) | 0.50 |
| H6 | 0.6714 | 0.5826 | 0.5018 | 0.039* | 0.50 |
| C2′ | 0.420 (4) | 0.4798 (14) | 0.567 (4) | 0.034 (3) | 0.50 |
| H2′ | 0.3212 | 0.4565 | 0.5261 | 0.040* | 0.50 |
| C3′ | 0.484 (6) | 0.486 (2) | 0.703 (4) | 0.040 (5) | 0.50 |
| H3′ | 0.4279 | 0.4657 | 0.7564 | 0.047* | 0.50 |
| C4′ | 0.627 (6) | 0.519 (3) | 0.7660 (10) | 0.0475 (18) | 0.50 |
| H4′ | 0.6697 | 0.5224 | 0.8599 | 0.057* | 0.50 |
| C5′ | 0.707 (5) | 0.548 (2) | 0.686 (4) | 0.037 (4) | 0.50 |

| | | | | | |
|---|---|---|---|---|---|
| H5′ | 0.8061 | 0.5706 | 0.7278 | 0.045* | 0.50 |
| C6′ | 0.6483 (16) | 0.5438 (12) | 0.549 (4) | 0.033 (3) | 0.50 |
| H6′ | 0.7046 | 0.5640 | 0.4959 | 0.039* | 0.50 |
| C7 | 0.1931 (7) | 0.5482 (3) | 0.1691 (6) | 0.0315 (12) | |
| C8 | 0.1848 (8) | 0.5921 (3) | 0.0642 (6) | 0.0373 (13) | |
| H8 | 0.2746 | 0.5977 | 0.0355 | 0.045* | |
| C9 | 0.0415 (8) | 0.6283 (4) | 0.0009 (7) | 0.0447 (15) | |
| H9 | 0.0348 | 0.6580 | −0.0718 | 0.054* | |
| C10 | −0.0874 (8) | 0.6213 (3) | 0.0426 (6) | 0.0409 (14) | |
| H10 | −0.1823 | 0.6469 | 0.0000 | 0.049* | |
| C11 | −0.0815 (7) | 0.5781 (3) | 0.1447 (7) | 0.0373 (13) | |
| H11 | −0.1720 | 0.5731 | 0.1728 | 0.045* | |
| C12 | 0.0584 (7) | 0.5410 (3) | 0.2081 (6) | 0.0340 (12) | |
| H12 | 0.0619 | 0.5105 | 0.2788 | 0.041* | |
| C13 | 0.5521 (7) | 0.4533 (4) | 0.1721 (7) | 0.056 (2) | |
| H13 | 0.5677 | 0.4912 | 0.1139 | 0.068* | |
| C14 | 0.4920 (7) | 0.3925 (4) | 0.0773 (8) | 0.062 (2) | |
| H14A | 0.4110 | 0.4081 | −0.0064 | 0.075* | |
| H14B | 0.4437 | 0.3565 | 0.1182 | 0.075* | |
| C15 | 0.6441 (9) | 0.3636 (4) | 0.0507 (7) | 0.074 (3) | |
| H15A | 0.6432 | 0.3123 | 0.0501 | 0.089* | |
| H15B | 0.6480 | 0.3802 | −0.0358 | 0.089* | |
| C16 | 0.7884 (7) | 0.3909 (4) | 0.1650 (7) | 0.0494 (17) | |
| H16A | 0.8564 | 0.4204 | 0.1297 | 0.059* | |
| H16B | 0.8539 | 0.3519 | 0.2142 | 0.059* | |
| C17 | 0.7225 (7) | 0.4334 (4) | 0.2575 (6) | 0.0433 (15) | |
| H17A | 0.7215 | 0.4054 | 0.3348 | 0.052* | |
| H17B | 0.7883 | 0.4753 | 0.2898 | 0.052* | |
| C18 | 0.5096 (7) | 0.6545 (3) | 0.2852 (6) | 0.0358 (13) | |
| C19 | 0.5981 (7) | 0.7165 (3) | 0.2514 (6) | 0.0458 (16) | |
| C20 | 0.3529 (8) | 0.3533 (3) | 0.3501 (7) | 0.0358 (14) | |
| C21 | 0.2525 (7) | 0.2913 (3) | 0.3561 (5) | 0.0415 (14) | |
| C22 | 0.1928 (7) | 0.7229 (3) | 0.5380 (6) | 0.0306 (12) | |
| H22 | 0.1109 | 0.7509 | 0.5611 | 0.037* | |
| C23 | 0.3314 (9) | 0.7076 (3) | 0.6660 (6) | 0.0437 (15) | |
| H23A | 0.4127 | 0.6786 | 0.6458 | 0.052* | |
| H23B | 0.3830 | 0.7514 | 0.7063 | 0.052* | |
| C24 | 0.2667 (10) | 0.6697 (4) | 0.7623 (7) | 0.0514 (18) | |
| H24A | 0.1915 | 0.7003 | 0.7870 | 0.062* | |
| H24B | 0.3563 | 0.6585 | 0.8441 | 0.062* | |
| C25 | 0.1806 (9) | 0.6031 (3) | 0.7024 (7) | 0.0423 (15) | |
| H25A | 0.2590 | 0.5698 | 0.6893 | 0.051* | |
| H25B | 0.1325 | 0.5820 | 0.7648 | 0.051* | |
| C26 | 0.0522 (8) | 0.6162 (3) | 0.5720 (7) | 0.0433 (15) | |
| H26A | −0.0349 | 0.6433 | 0.5874 | 0.052* | |
| H26B | 0.0069 | 0.5714 | 0.5318 | 0.052* | |
| C27 | 0.1156 (8) | 0.6560 (3) | 0.4746 (6) | 0.0361 (13) | |
| H27A | 0.1950 | 0.6272 | 0.4514 | 0.043* | |

| | | | | |
|---|---|---|---|---|
| H27B | 0.0263 | 0.6662 | 0.3920 | 0.043* |
| C29 | 0.1158 (7) | 0.8005 (3) | 0.3367 (6) | 0.0317 (12) |
| H29 | 0.0177 | 0.7711 | 0.3160 | 0.038* |
| C30 | 0.1550 (8) | 0.8094 (4) | 0.2104 (7) | 0.0463 (16) |
| H30A | 0.1732 | 0.7635 | 0.1766 | 0.056* |
| H30B | 0.2541 | 0.8370 | 0.2286 | 0.056* |
| C31 | 0.0151 (9) | 0.8464 (5) | 0.1056 (8) | 0.061 (2) |
| H31A | 0.0441 | 0.8545 | 0.0249 | 0.074* |
| H31B | −0.0802 | 0.8160 | 0.0809 | 0.074* |
| C32 | −0.0264 (9) | 0.9144 (4) | 0.1539 (8) | 0.057 (2) |
| H32A | −0.1222 | 0.9340 | 0.0862 | 0.069* |
| H32B | 0.0632 | 0.9473 | 0.1667 | 0.069* |
| C33 | −0.0591 (9) | 0.9056 (4) | 0.2842 (8) | 0.057 (2) |
| H33A | −0.1579 | 0.8780 | 0.2686 | 0.069* |
| H33B | −0.0765 | 0.9517 | 0.3178 | 0.069* |
| C34 | 0.0797 (9) | 0.8696 (4) | 0.3879 (7) | 0.0471 (16) |
| H34A | 0.1758 | 0.8994 | 0.4106 | 0.057* |
| H34B | 0.0525 | 0.8622 | 0.4696 | 0.057* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Sn1 | 0.01949 (15) | 0.02969 (16) | 0.0492 (2) | 0.0012 (2) | 0.01167 (12) | −0.0076 (2) |
| Cl1 | 0.153 (4) | 0.124 (3) | 0.103 (2) | −0.027 (2) | 0.065 (2) | −0.0111 (19) |
| Cl2 | 0.164 (3) | 0.085 (2) | 0.0910 (19) | −0.061 (2) | −0.029 (2) | 0.0079 (15) |
| F1 | 0.031 (2) | 0.050 (2) | 0.128 (4) | −0.0105 (18) | 0.031 (2) | 0.006 (2) |
| F2 | 0.045 (3) | 0.035 (2) | 0.115 (4) | −0.0008 (18) | 0.037 (3) | 0.002 (2) |
| F3 | 0.062 (3) | 0.057 (3) | 0.110 (4) | 0.019 (2) | 0.045 (3) | 0.048 (3) |
| F4 | 0.049 (3) | 0.063 (3) | 0.134 (5) | 0.010 (2) | 0.049 (3) | 0.024 (3) |
| O1 | 0.021 (2) | 0.045 (2) | 0.058 (3) | −0.0021 (18) | 0.022 (2) | −0.004 (2) |
| O2 | 0.027 (2) | 0.040 (2) | 0.050 (3) | −0.0052 (19) | 0.020 (2) | −0.010 (2) |
| O3 | 0.024 (2) | 0.0246 (19) | 0.053 (3) | 0.0051 (16) | 0.0032 (18) | −0.0040 (17) |
| O4 | 0.031 (2) | 0.039 (2) | 0.054 (3) | 0.0095 (19) | 0.013 (2) | −0.0006 (19) |
| N1 | 0.021 (2) | 0.029 (2) | 0.031 (2) | −0.0031 (18) | 0.0116 (19) | −0.0055 (17) |
| C1 | 0.020 (2) | 0.027 (3) | 0.052 (3) | 0.006 (2) | 0.011 (2) | 0.001 (2) |
| C2 | 0.011 (8) | 0.037 (7) | 0.049 (5) | 0.011 (5) | 0.007 (6) | −0.012 (4) |
| C3 | 0.029 (9) | 0.038 (14) | 0.054 (5) | 0.010 (8) | 0.016 (5) | −0.005 (5) |
| C4 | 0.043 (4) | 0.053 (5) | 0.046 (3) | 0.007 (3) | 0.013 (3) | −0.012 (3) |
| C5 | 0.029 (6) | 0.023 (13) | 0.053 (5) | 0.008 (8) | 0.003 (5) | −0.018 (7) |
| C6 | 0.024 (4) | 0.019 (6) | 0.054 (5) | 0.008 (5) | 0.012 (5) | −0.009 (6) |
| C2′ | 0.011 (8) | 0.037 (7) | 0.049 (5) | 0.011 (5) | 0.007 (6) | −0.012 (4) |
| C3′ | 0.029 (9) | 0.038 (14) | 0.054 (5) | 0.010 (8) | 0.016 (5) | −0.005 (5) |
| C4′ | 0.043 (4) | 0.053 (5) | 0.046 (3) | 0.007 (3) | 0.013 (3) | −0.012 (3) |
| C5′ | 0.029 (6) | 0.023 (13) | 0.053 (5) | 0.008 (8) | 0.003 (5) | −0.018 (7) |
| C6′ | 0.024 (4) | 0.019 (6) | 0.054 (5) | 0.008 (5) | 0.012 (5) | −0.009 (6) |
| C7 | 0.028 (3) | 0.022 (2) | 0.042 (3) | 0.005 (2) | 0.009 (2) | −0.007 (2) |
| C8 | 0.032 (3) | 0.047 (3) | 0.036 (3) | −0.006 (3) | 0.016 (3) | −0.001 (2) |
| C9 | 0.038 (4) | 0.053 (4) | 0.040 (4) | 0.002 (3) | 0.010 (3) | 0.004 (3) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C10 | 0.030 (3) | 0.039 (3) | 0.046 (4) | 0.006 (3) | 0.003 (3) | −0.004 (3) |
| C11 | 0.023 (3) | 0.038 (3) | 0.053 (4) | −0.001 (2) | 0.014 (3) | −0.004 (3) |
| C12 | 0.024 (3) | 0.033 (3) | 0.043 (3) | −0.006 (2) | 0.010 (2) | −0.006 (2) |
| C13 | 0.028 (3) | 0.084 (6) | 0.050 (4) | 0.015 (4) | 0.004 (3) | −0.030 (4) |
| C14 | 0.061 (5) | 0.066 (5) | 0.059 (5) | −0.006 (4) | 0.019 (4) | 0.005 (4) |
| C15 | 0.098 (8) | 0.067 (5) | 0.070 (6) | 0.022 (5) | 0.045 (5) | −0.003 (4) |
| C16 | 0.039 (4) | 0.049 (4) | 0.068 (5) | 0.013 (3) | 0.027 (3) | −0.008 (3) |
| C17 | 0.026 (3) | 0.059 (4) | 0.047 (4) | 0.014 (3) | 0.015 (3) | −0.004 (3) |
| C18 | 0.022 (3) | 0.037 (3) | 0.046 (3) | −0.005 (2) | 0.009 (2) | −0.008 (3) |
| C19 | 0.031 (3) | 0.049 (4) | 0.066 (5) | −0.001 (3) | 0.027 (3) | 0.005 (3) |
| C20 | 0.032 (4) | 0.026 (3) | 0.050 (4) | 0.008 (2) | 0.014 (3) | −0.003 (2) |
| C21 | 0.035 (3) | 0.037 (3) | 0.054 (4) | 0.005 (3) | 0.016 (3) | 0.010 (3) |
| C22 | 0.032 (3) | 0.021 (2) | 0.045 (3) | 0.001 (2) | 0.022 (3) | −0.001 (2) |
| C23 | 0.052 (4) | 0.042 (3) | 0.034 (3) | −0.021 (3) | 0.011 (3) | 0.000 (3) |
| C24 | 0.065 (5) | 0.047 (4) | 0.047 (4) | −0.019 (3) | 0.026 (4) | −0.004 (3) |
| C25 | 0.058 (4) | 0.025 (3) | 0.055 (4) | −0.007 (3) | 0.033 (3) | 0.002 (3) |
| C26 | 0.040 (4) | 0.032 (3) | 0.064 (4) | −0.009 (3) | 0.025 (3) | −0.003 (3) |
| C27 | 0.031 (3) | 0.030 (3) | 0.047 (3) | −0.009 (2) | 0.013 (3) | −0.005 (3) |
| C29 | 0.023 (3) | 0.031 (3) | 0.039 (3) | −0.006 (2) | 0.008 (2) | 0.001 (2) |
| C30 | 0.031 (3) | 0.066 (5) | 0.043 (4) | −0.005 (3) | 0.012 (3) | 0.008 (3) |
| C31 | 0.034 (4) | 0.093 (6) | 0.050 (4) | −0.012 (4) | 0.004 (3) | 0.025 (4) |
| C32 | 0.034 (4) | 0.050 (4) | 0.073 (5) | −0.010 (3) | −0.003 (3) | 0.030 (4) |
| C33 | 0.046 (5) | 0.043 (4) | 0.075 (5) | 0.011 (3) | 0.009 (4) | 0.011 (3) |
| C34 | 0.041 (4) | 0.043 (4) | 0.055 (4) | 0.008 (3) | 0.014 (3) | 0.003 (3) |

*Geometric parameters (Å, º)*

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|---|---|---|---|
| Sn1—C1 | 2.147 (5) | C12—H12 | 0.9500 |
| Sn1—C7 | 2.136 (6) | C13—C14 | 1.527 (7) |
| Sn1—C13 | 2.117 (6) | C13—C17 | 1.530 (7) |
| Sn1—O1 | 2.287 (4) | C13—H13 | 1.0000 |
| Sn1—O3 | 2.249 (4) | C14—C15 | 1.569 (7) |
| Cl1—C19 | 1.660 (6) | C14—H14A | 0.9900 |
| Cl2—C21 | 1.657 (6) | C14—H14B | 0.9900 |
| F1—C19 | 1.319 (6) | C15—C16 | 1.539 (7) |
| F2—C19 | 1.331 (6) | C15—H15A | 0.9900 |
| F3—C21 | 1.311 (6) | C15—H15B | 0.9900 |
| F4—C21 | 1.334 (6) | C16—C17 | 1.541 (6) |
| O1—C18 | 1.246 (7) | C16—H16A | 0.9900 |
| O2—C18 | 1.249 (7) | C16—H16B | 0.9900 |
| O3—C20 | 1.257 (7) | C17—H17A | 0.9900 |
| O4—C20 | 1.235 (8) | C17—H17B | 0.9900 |
| N1—C29 | 1.508 (7) | C18—C19 | 1.540 (9) |
| N1—C22 | 1.508 (7) | C20—C21 | 1.506 (9) |
| N1—H1N1 | 0.8800 | C22—C27 | 1.512 (8) |
| N1—H1N2 | 0.8800 | C22—C23 | 1.534 (9) |
| C1—C2 | 1.391 (9) | C22—H22 | 1.0000 |
| C1—C2′ | 1.394 (9) | C23—C24 | 1.522 (9) |

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|---|---|---|---|
| C1—C6′ | 1.396 (9) | C23—H23A | 0.9900 |
| C1—C6 | 1.401 (9) | C23—H23B | 0.9900 |
| C2—C3 | 1.387 (9) | C24—C25 | 1.524 (9) |
| C2—H2 | 0.9500 | C24—H24A | 0.9900 |
| C3—C4 | 1.383 (9) | C24—H24B | 0.9900 |
| C3—H3 | 0.9500 | C25—C26 | 1.499 (10) |
| C4—C5 | 1.388 (9) | C25—H25A | 0.9900 |
| C4—H4 | 0.9500 | C25—H25B | 0.9900 |
| C5—C6 | 1.393 (9) | C26—C27 | 1.542 (9) |
| C5—H5 | 0.9500 | C26—H26A | 0.9900 |
| C6—H6 | 0.9500 | C26—H26B | 0.9900 |
| C2′—C3′ | 1.386 (9) | C27—H27A | 0.9900 |
| C2′—H2′ | 0.9500 | C27—H27B | 0.9900 |
| C3′—C4′ | 1.382 (9) | C29—C30 | 1.510 (8) |
| C3′—H3′ | 0.9500 | C29—C34 | 1.516 (9) |
| C4′—C5′ | 1.387 (9) | C29—H29 | 1.0000 |
| C4′—H4′ | 0.9500 | C30—C31 | 1.543 (10) |
| C5′—C6′ | 1.393 (9) | C30—H30A | 0.9900 |
| C5′—H5′ | 0.9500 | C30—H30B | 0.9900 |
| C6′—H6′ | 0.9500 | C31—C32 | 1.499 (12) |
| C7—C8 | 1.388 (9) | C31—H31A | 0.9900 |
| C7—C12 | 1.396 (8) | C31—H31B | 0.9900 |
| C8—C9 | 1.410 (9) | C32—C33 | 1.523 (11) |
| C8—H8 | 0.9500 | C32—H32A | 0.9900 |
| C9—C10 | 1.365 (10) | C32—H32B | 0.9900 |
| C9—H9 | 0.9500 | C33—C34 | 1.523 (10) |
| C10—C11 | 1.361 (9) | C33—H33A | 0.9900 |
| C10—H10 | 0.9500 | C33—H33B | 0.9900 |
| C11—C12 | 1.396 (9) | C34—H34A | 0.9900 |
| C11—H11 | 0.9500 | C34—H34B | 0.9900 |

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| C1—Sn1—C7 | 119.2 (2) | C13—C17—C16 | 105.1 (4) |
| C1—Sn1—C13 | 121.7 (2) | C13—C17—H17A | 110.7 |
| C1—Sn1—O1 | 91.3 (2) | C16—C17—H17A | 110.7 |
| C1—Sn1—O3 | 90.9 (2) | C13—C17—H17B | 110.7 |
| C7—Sn1—C13 | 118.9 (2) | C16—C17—H17B | 110.7 |
| C7—Sn1—O1 | 90.6 (2) | H17A—C17—H17B | 108.8 |
| C7—Sn1—O3 | 87.8 (2) | O1—C18—O2 | 129.2 (6) |
| C13—Sn1—O1 | 82.8 (3) | O1—C18—C19 | 114.5 (5) |
| C13—Sn1—O3 | 96.5 (3) | O2—C18—C19 | 116.3 (5) |
| O1—Sn1—O3 | 177.7 (2) | F1—C19—F2 | 108.8 (5) |
| C18—O1—Sn1 | 119.3 (4) | F1—C19—C18 | 111.3 (5) |
| C20—O3—Sn1 | 117.0 (4) | F2—C19—C18 | 114.0 (5) |
| C29—N1—C22 | 113.5 (4) | F1—C19—Cl1 | 106.0 (4) |
| C29—N1—H1N1 | 108.9 | F2—C19—Cl1 | 105.0 (5) |
| C29—N1—H1N1 | 108.9 | C18—C19—Cl1 | 111.2 (5) |
| C22—N1—H1N2 | 108.9 | O4—C20—O3 | 128.7 (6) |
| C22—N1—H1N2 | 108.9 | O4—C20—C21 | 116.7 (5) |

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| H1N1—N1—H1N2 | 107.7 | O3—C20—C21 | 114.6 (6) |
| C2′—C1—C6′ | 121 (3) | F3—C21—F4 | 107.1 (5) |
| C2—C1—C6 | 115 (3) | F3—C21—C20 | 114.3 (5) |
| C2—C1—Sn1 | 123.3 (19) | F4—C21—C20 | 112.3 (5) |
| C2′—C1—Sn1 | 120.4 (18) | F3—C21—Cl2 | 103.7 (5) |
| C6′—C1—Sn1 | 118 (2) | F4—C21—Cl2 | 110.4 (5) |
| C6—C1—Sn1 | 122 (2) | C20—C21—Cl2 | 108.6 (4) |
| C3—C2—C1 | 125 (3) | N1—C22—C27 | 110.2 (5) |
| C3—C2—H2 | 117.5 | N1—C22—C23 | 111.1 (5) |
| C1—C2—H2 | 117.5 | C27—C22—C23 | 110.1 (5) |
| C4—C3—C2 | 116 (3) | N1—C22—H22 | 108.4 |
| C4—C3—H3 | 122.1 | C27—C22—H22 | 108.4 |
| C2—C3—H3 | 122.1 | C23—C22—H22 | 108.4 |
| C3—C4—C5 | 124 (3) | C24—C23—C22 | 109.0 (6) |
| C3—C4—H4 | 117.8 | C24—C23—H23A | 109.9 |
| C5—C4—H4 | 117.8 | C22—C23—H23A | 109.9 |
| C4—C5—C6 | 116 (3) | C24—C23—H23B | 109.9 |
| C4—C5—H5 | 122.0 | C22—C23—H23B | 109.9 |
| C6—C5—H5 | 122.0 | H23A—C23—H23B | 108.3 |
| C5—C6—C1 | 124 (3) | C23—C24—C25 | 111.7 (5) |
| C5—C6—H6 | 118.0 | C23—C24—H24A | 109.3 |
| C1—C6—H6 | 118.0 | C25—C24—H24A | 109.3 |
| C3′—C2′—C1 | 118 (3) | C23—C24—H24B | 109.3 |
| C3′—C2′—H2′ | 120.9 | C25—C24—H24B | 109.3 |
| C1—C2′—H2′ | 120.9 | H24A—C24—H24B | 107.9 |
| C4′—C3′—C2′ | 122 (3) | C26—C25—C24 | 111.5 (5) |
| C4′—C3′—H3′ | 118.8 | C26—C25—H25A | 109.3 |
| C2′—C3′—H3′ | 118.8 | C24—C25—H25A | 109.3 |
| C3′—C4′—C5′ | 118 (3) | C26—C25—H25B | 109.3 |
| C3′—C4′—H4′ | 121.2 | C24—C25—H25B | 109.3 |
| C5′—C4′—H4′ | 121.2 | H25A—C25—H25B | 108.0 |
| C4′—C5′—C6′ | 123 (3) | C25—C26—C27 | 112.1 (5) |
| C4′—C5′—H5′ | 118.7 | C25—C26—H26A | 109.2 |
| C6′—C5′—H5′ | 118.7 | C27—C26—H26A | 109.2 |
| C5′—C6′—C1 | 118 (3) | C25—C26—H26B | 109.2 |
| C5′—C6′—H6′ | 121.2 | C27—C26—H26B | 109.2 |
| C1—C6′—H6′ | 121.2 | H26A—C26—H26B | 107.9 |
| C8—C7—C12 | 118.7 (5) | C22—C27—C26 | 109.6 (5) |
| C8—C7—Sn1 | 119.9 (4) | C22—C27—H27A | 109.8 |
| C12—C7—Sn1 | 121.2 (4) | C26—C27—H27A | 109.8 |
| C7—C8—C9 | 119.3 (6) | C22—C27—H27B | 109.8 |
| C7—C8—H8 | 120.4 | C26—C27—H27B | 109.8 |
| C9—C8—H8 | 120.4 | H27A—C27—H27B | 108.2 |
| C10—C9—C8 | 120.8 (6) | N1—C29—C30 | 111.2 (5) |
| C10—C9—H9 | 119.6 | N1—C29—C34 | 110.6 (5) |
| C8—C9—H9 | 119.6 | C30—C29—C34 | 111.2 (5) |
| C11—C10—C9 | 120.6 (6) | N1—C29—H29 | 107.9 |
| C11—C10—H10 | 119.7 | C30—C29—H29 | 107.9 |

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| C9—C10—H10 | 119.7 | C34—C29—H29 | 107.9 |
| C10—C11—C12 | 119.7 (6) | C29—C30—C31 | 109.5 (6) |
| C10—C11—H11 | 120.1 | C29—C30—H30A | 109.8 |
| C12—C11—H11 | 120.1 | C31—C30—H30A | 109.8 |
| C7—C12—C11 | 120.9 (6) | C29—C30—H30B | 109.8 |
| C7—C12—H12 | 119.5 | C31—C30—H30B | 109.8 |
| C11—C12—H12 | 119.5 | H30A—C30—H30B | 108.2 |
| C14—C13—C17 | 105.1 (5) | C32—C31—C30 | 112.7 (7) |
| C14—C13—Sn1 | 123.1 (5) | C32—C31—H31A | 109.1 |
| C17—C13—Sn1 | 114.6 (4) | C30—C31—H31A | 109.1 |
| C14—C13—H13 | 104.0 | C32—C31—H31B | 109.1 |
| C17—C13—H13 | 104.0 | C30—C31—H31B | 109.1 |
| Sn1—C13—H13 | 104.0 | H31A—C31—H31B | 107.8 |
| C13—C14—C15 | 105.2 (5) | C31—C32—C33 | 111.1 (6) |
| C13—C14—H14A | 110.7 | C31—C32—H32A | 109.4 |
| C15—C14—H14A | 110.7 | C33—C32—H32A | 109.4 |
| C13—C14—H14B | 110.7 | C31—C32—H32B | 109.4 |
| C15—C14—H14B | 110.7 | C33—C32—H32B | 109.4 |
| H14A—C14—H14B | 108.8 | H32A—C32—H32B | 108.0 |
| C16—C15—C14 | 105.8 (4) | C32—C33—C34 | 111.3 (6) |
| C16—C15—H15A | 110.6 | C32—C33—H33A | 109.4 |
| C14—C15—H15A | 110.6 | C34—C33—H33A | 109.4 |
| C16—C15—H15B | 110.6 | C32—C33—H33B | 109.4 |
| C14—C15—H15B | 110.6 | C34—C33—H33B | 109.4 |
| H15A—C15—H15B | 108.7 | H33A—C33—H33B | 108.0 |
| C15—C16—C17 | 107.4 (4) | C29—C34—C33 | 110.9 (6) |
| C15—C16—H16A | 110.2 | C29—C34—H34A | 109.5 |
| C17—C16—H16A | 110.2 | C33—C34—H34A | 109.5 |
| C15—C16—H16B | 110.2 | C29—C34—H34B | 109.5 |
| C17—C16—H16B | 110.2 | C33—C34—H34B | 109.5 |
| H16A—C16—H16B | 108.5 | H34A—C34—H34B | 108.1 |
| | | | |
| C13—Sn1—O1—C18 | 169.9 (5) | C8—C9—C10—C11 | −1.3 (10) |
| C7—Sn1—O1—C18 | 50.8 (5) | C9—C10—C11—C12 | 0.6 (10) |
| C1—Sn1—O1—C18 | −68.4 (5) | C8—C7—C12—C11 | −1.0 (9) |
| C13—Sn1—O3—C20 | 60.0 (5) | Sn1—C7—C12—C11 | 173.6 (4) |
| C7—Sn1—O3—C20 | 178.9 (5) | C10—C11—C12—C7 | 0.6 (9) |
| C1—Sn1—O3—C20 | −62.0 (5) | C7—Sn1—C13—C14 | −61.9 (8) |
| C13—Sn1—C1—C2 | −108.5 (13) | C1—Sn1—C13—C14 | 124.4 (6) |
| C7—Sn1—C1—C2 | 77.8 (13) | O3—Sn1—C13—C14 | 29.2 (7) |
| O3—Sn1—C1—C2 | −10.2 (13) | O1—Sn1—C13—C14 | −148.5 (7) |
| O1—Sn1—C1—C2 | 169.2 (13) | C7—Sn1—C13—C17 | 168.2 (5) |
| C13—Sn1—C1—C2′ | −121.7 (13) | C1—Sn1—C13—C17 | −5.5 (7) |
| C7—Sn1—C1—C2′ | 64.6 (13) | O3—Sn1—C13—C17 | −100.6 (6) |
| O3—Sn1—C1—C2′ | −23.5 (13) | O1—Sn1—C13—C17 | 81.6 (6) |
| O1—Sn1—C1—C2′ | 155.9 (13) | C17—C13—C14—C15 | −32.5 (8) |
| C13—Sn1—C1—C6′ | 56.8 (11) | Sn1—C13—C14—C15 | −166.2 (6) |
| C7—Sn1—C1—C6′ | −116.9 (11) | C13—C14—C15—C16 | 19.2 (9) |

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| O3—Sn1—C1—C6′ | 155.1 (11) | C14—C15—C16—C17 | 1.1 (9) |
| O1—Sn1—C1—C6′ | −25.5 (11) | C14—C13—C17—C16 | 33.2 (8) |
| C13—Sn1—C1—C6 | 70.4 (11) | Sn1—C13—C17—C16 | 171.5 (5) |
| C7—Sn1—C1—C6 | −103.4 (11) | C15—C16—C17—C13 | −21.0 (8) |
| O3—Sn1—C1—C6 | 168.6 (11) | Sn1—O1—C18—O2 | 6.9 (9) |
| O1—Sn1—C1—C6 | −12.0 (11) | Sn1—O1—C18—C19 | −173.6 (4) |
| C2′—C1—C2—C3 | −102 (18) | O1—C18—C19—F1 | −56.4 (7) |
| C6′—C1—C2—C3 | 13.2 (19) | O2—C18—C19—F1 | 123.1 (6) |
| C6—C1—C2—C3 | −0.2 (3) | O1—C18—C19—F2 | 180.0 (5) |
| Sn1—C1—C2—C3 | 178.7 (11) | O2—C18—C19—F2 | −0.5 (8) |
| C1—C2—C3—C4 | 0.0 (3) | O1—C18—C19—Cl1 | 61.5 (6) |
| C2—C3—C4—C5 | 0.0 (7) | O2—C18—C19—Cl1 | −118.9 (5) |
| C3—C4—C5—C6 | 0.2 (9) | Sn1—O3—C20—O4 | 1.0 (9) |
| C4—C5—C6—C1 | −0.5 (9) | Sn1—O3—C20—C21 | 179.9 (4) |
| C2—C1—C6—C5 | 0.5 (7) | O4—C20—C21—F3 | 4.9 (8) |
| C2′—C1—C6—C5 | 13 (2) | O3—C20—C21—F3 | −174.1 (6) |
| C6′—C1—C6—C5 | −102 (19) | O4—C20—C21—F4 | 127.2 (6) |
| Sn1—C1—C6—C5 | −178.4 (12) | O3—C20—C21—F4 | −51.8 (7) |
| C2—C1—C2′—C3′ | 70 (18) | O4—C20—C21—Cl2 | −110.4 (6) |
| C6′—C1—C2′—C3′ | −0.2 (3) | O3—C20—C21—Cl2 | 70.6 (6) |
| C6—C1—C2′—C3′ | −13 (2) | C29—N1—C22—C27 | 76.9 (6) |
| Sn1—C1—C2′—C3′ | 178.3 (11) | C29—N1—C22—C23 | −160.8 (5) |
| C1—C2′—C3′—C4′ | 0.0 (3) | N1—C22—C23—C24 | 177.0 (5) |
| C2′—C3′—C4′—C5′ | 0.0 (6) | C27—C22—C23—C24 | −60.6 (7) |
| C3′—C4′—C5′—C6′ | 0.2 (8) | C22—C23—C24—C25 | 57.4 (8) |
| C4′—C5′—C6′—C1 | −0.5 (8) | C23—C24—C25—C26 | −54.2 (9) |
| C2—C1—C6′—C5′ | −12 (3) | C24—C25—C26—C27 | 53.0 (7) |
| C2′—C1—C6′—C5′ | 0.5 (6) | N1—C22—C27—C26 | −177.6 (5) |
| C6—C1—C6′—C5′ | 72 (18) | C23—C22—C27—C26 | 59.5 (7) |
| Sn1—C1—C6′—C5′ | −178.1 (11) | C25—C26—C27—C22 | −56.0 (7) |
| C13—Sn1—C7—C8 | −46.3 (6) | C22—N1—C29—C30 | −151.5 (5) |
| C1—Sn1—C7—C8 | 127.6 (4) | C22—N1—C29—C34 | 84.5 (6) |
| O3—Sn1—C7—C8 | −142.5 (5) | N1—C29—C30—C31 | 179.5 (6) |
| O1—Sn1—C7—C8 | 35.8 (5) | C34—C29—C30—C31 | −56.8 (7) |
| C13—Sn1—C7—C12 | 139.2 (5) | C29—C30—C31—C32 | 55.6 (8) |
| C1—Sn1—C7—C12 | −46.9 (5) | C30—C31—C32—C33 | −54.3 (8) |
| O3—Sn1—C7—C12 | 42.9 (4) | C31—C32—C33—C34 | 54.0 (9) |
| O1—Sn1—C7—C12 | −138.8 (4) | N1—C29—C34—C33 | −177.8 (5) |
| C12—C7—C8—C9 | 0.3 (9) | C30—C29—C34—C33 | 58.1 (8) |
| Sn1—C7—C8—C9 | −174.4 (5) | C32—C33—C34—C29 | −55.9 (8) |
| C7—C8—C9—C10 | 0.9 (10) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1*N*1···O2 | 0.88 | 1.88 | 2.758 (6) | 173 |

| | | | | |
|---|---|---|---|---|
| N1—H1*N*2···O4$^{i}$ | 0.88 | 1.93 | 2.804 (6) | 169 |

Symmetry code: (i) −*x*+1, *y*+1/2, −*z*+1.