

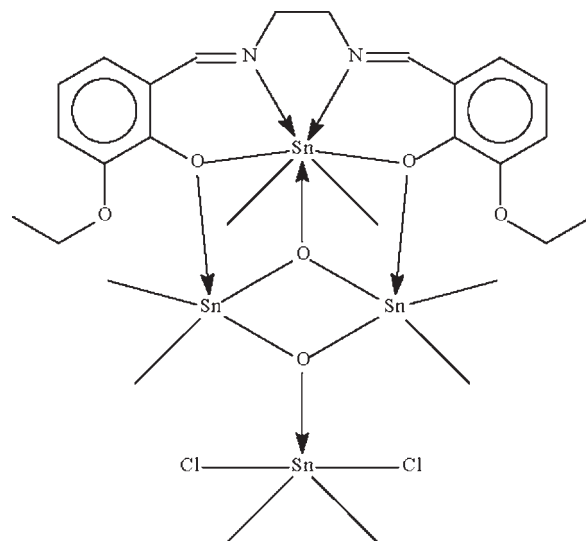
# Dichlorido{ $\mu_3$ -6,6'-diethoxy-2,2'-[ethane-1,2-diylbis(nitrilomethylidyne)]-diphenolato}octamethyldi- $\mu_3$ -oxido-tetratin(IV)

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Key indicators: single-crystal X-ray study;  $T = 100$  K; mean  $\sigma(\text{C}-\text{C}) = 0.007$  Å; disorder in main residue;  $R$  factor = 0.027;  $wR$  factor = 0.072; data-to-parameter ratio = 21.5.



## Experimental

### Crystal data

$[\text{Sn}_4(\text{CH}_3)_8(\text{C}_{20}\text{H}_{22}\text{N}_2\text{O}_4)\text{Cl}_2\text{O}_2]$   
 $M_r = 1052.33$   
Tetragonal,  $P4_32_12$   
 $a = 9.8723$  (1) Å  
 $c = 38.0217$  (5) Å  
 $V = 3705.68$  (6) Å<sup>3</sup>

$Z = 4$   
Mo  $K\alpha$  radiation  
 $\mu = 2.85$  mm<sup>-1</sup>  
 $T = 100$  K  
 $0.20 \times 0.18 \times 0.15$  mm

### Data collection

Bruker SMART APEX  
diffractometer  
Absorption correction: multi-scan  
(*SADABS*; Sheldrick, 1996)  
 $T_{\min} = 0.863$ ,  $T_{\max} = 1.000$   
(expected range = 0.563–0.652)

68779 measured reflections  
4259 independent reflections  
4089 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.029$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.027$   
 $wR(F^2) = 0.072$   
 $S = 1.07$   
4259 reflections  
198 parameters  
10 restraints

H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.42$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.69$  e Å<sup>-3</sup>  
Absolute structure: Flack (1983),  
1694 Friedel pairs  
Flack parameter: 0.00 (4)

In the title tetranuclear tin(IV) complex,  $[\text{Sn}_4(\text{CH}_3)_8(\text{C}_{20}\text{H}_{22}\text{N}_2\text{O}_4)\text{Cl}_2\text{O}_2]$ , there are three completely different tin-atom coordinations. One metal atom (site symmetry 2) adopts a distorted pentagonal-bipyramidal  $\text{SnC}_2\text{N}_2\text{O}_3$  coordination arising from the  $N,N',O,O'$ -tetradentate deprotonated Schiff base, two methyl groups in the axial sites and a  $\mu_3$ -O atom that also bonds to two further Sn atoms. Two symmetry-equivalent Sn atoms adopt very distorted  $\text{SnC}_2\text{O}_4$  arrangements that could be described as pentagonal-bipyramidal with one equatorial vertex missing and the C atoms in the axial site. The final Sn atom (site symmetry 2) adopts an  $\text{SnC}_2\text{Cl}_2\text{O}$  trigonal-bipyramidal arrangement, with Cl atoms in the axial sites. As well as the two Sn atoms, one O atom lies on a twofold rotation axis, and another is disordered about the axis. The terminal ethoxy group is disordered over two sets of sites with equal occupancy.

## Related literature

For other organotin derivatives of 6,6'-dialkoxy-2,2'-[ethane-1,2-diylbis(nitrilomethylidyne)]diphenol, see: Cunningham *et al.* (2004). For the crystal structure of 6,6'-diethoxy-2,2'-[ethane-1,2-diylbis(nitrilomethylidyne)]diphenol, see: Bermejo *et al.* (2007).

**Table 1**

Selected geometric parameters (Å, °).

|                         |           |                        |             |
|-------------------------|-----------|------------------------|-------------|
| Sn1—O3                  | 2.072 (4) | Sn2—C2                 | 2.091 (5)   |
| Sn1—C1                  | 2.112 (5) | Sn2—C3                 | 2.100 (5)   |
| Sn1—O1                  | 2.410 (3) | Sn2—O4                 | 2.125 (17)  |
| Sn1—N1                  | 2.426 (4) | Sn3—O4                 | 1.964 (5)   |
| Sn2—O1                  | 2.463 (3) | Sn3—C4                 | 2.114 (5)   |
| Sn2—O2                  | 2.791 (4) | Sn3—Cl1                | 2.5829 (15) |
| Sn2—O3                  | 2.006 (2) |                        |             |
| Cl <sup>i</sup> —Sn1—Cl | 173.9 (3) | C4—Sn3—C4 <sup>i</sup> | 132.6 (3)   |
| C2—Sn2—C3               | 147.8 (2) |                        |             |

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

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); 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: *pubCIF* (Westrip, 2009).

We thank the University of Malaya (RG020/09AFR) for supporting this study.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5035).

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## References

- Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
- Bermejo, M. R., Fernández, M. I., Gómez-Fórneas, E., González-Noya, A., Maneiro, M., Pedrido, R. & Rodríguez, M. J. (2007). *Eur. J. Inorg. Chem.* pp. 3789–3797.
- Bruker (2008). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Cunningham, D., Gilligan, K., Hannon, M., Kelly, C., McArdle, P. & O'Malley, A. (2004). *Organometallics*, **23**, 984–994.
- Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
- Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Westrip, S. P. (2009). *pubCIF*. In preparation.

**supplementary materials**

*Acta Cryst.* (2009). E65, m1103-m1104 [ doi:10.1107/S1600536809032255 ]

**Dichlorido{ $\mu_3$ -6,6'-diethoxy-2,2'-[ethane-1,2-diylbis(nitrilomethylidene)]diphenolato}octamethyldi- $\mu_3$ -oxido-tetratin(IV)**

**S. M. Lee, K. M. Lo and S. W. Ng**

**Experimental**

One mmol (0.36 g) of the Schiff base was synthesized in toluene according to a literature procedure (Bermejo *et al.*, 2007) from 3-ethoxysalicylaldehyde and ethylenediamine. To the solution was added an excess of triethylamine (0.5 ml). A toluene solution of dimethyltin dichloride (0.20 g, 1 mmol) was added and the mixture heated. Yellow blocks of (I) were isolated from the cool filtered solution.

**Refinement**

Hydrogen atoms were placed at calculated positions (C–H 0.95–0.99 Å) and were treated as riding on their parent atoms, with  $U(H)$  set to 1.2–1.5 times  $U_{eq}(C)$ .

The ethoxy group is disordered over two positions in respect of the carbon atoms. The occupancy could not be refined, and was arbitrarily regarded as 0.5 each. The C–O distances were restrained to  $1.45 \pm 0.01$  Å and the C–C distances to  $1.50 \pm 0.01$  Å. The displacement factors of the primed atoms were set of those of the unprimed ones, and the anisotropic temperature factors were restrained to be nearly isotropic by tight restraints.

**Figures**

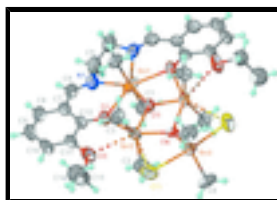


Fig. 1. View of (I) at the 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius. The disorder in the ethyl groups are not shown. Unlabelled atoms are generated by the symmetry operation ( $y, x, -z$ ).

**Dichlorido{ $\mu_3$ -6,6'-diethoxy-2,2'-[ethane-1,2-diylbis(nitrilomethylidene)]diphenolato}octamethyldi- $\mu_3$ -oxido-tetratin(IV)**

*Crystal data*

[Sn<sub>4</sub>(CH<sub>3</sub>)<sub>8</sub>(C<sub>20</sub>H<sub>22</sub>N<sub>2</sub>O<sub>4</sub>)Cl<sub>2</sub>O<sub>2</sub>]

$M_r = 1052.33$

Tetragonal,  $P4_32_12$

Hall symbol: P4 nw 2abw

$a = 9.8723$  (1) Å

$b = 9.8723$  Å

$c = 38.0217$  (5) Å

$Z = 4$

$F_{000} = 2040$

$D_x = 1.886$  Mg m<sup>-3</sup>

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

Cell parameters from 9779 reflections

$\theta = 2.1$ – $26.5^\circ$

$\mu = 2.85$  mm<sup>-1</sup>

# supplementary materials

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$\alpha = 90^\circ$   
 $\beta = 90^\circ$   
 $\gamma = 90^\circ$   
 $V = 3705.68 (6) \text{ \AA}^3$

$T = 100 \text{ K}$   
Block, yellow  
 $0.20 \times 0.18 \times 0.15 \text{ mm}$

## Data collection

Bruker SMART APEX diffractometer  
Radiation source: fine-focus sealed tube  
Monochromator: graphite  
 $T = 100 \text{ K}$   
 $\omega$  scans  
Absorption correction: Multi-scan (SADABS; Sheldrick, 1996)  
 $T_{\min} = 0.863, T_{\max} = 1.000$   
68779 measured reflections

4259 independent reflections  
4089 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.029$   
 $\theta_{\max} = 27.5^\circ$   
 $\theta_{\min} = 2.1^\circ$   
 $h = -12 \rightarrow 12$   
 $k = -12 \rightarrow 12$   
 $l = -49 \rightarrow 48$

## Refinement

Refinement on  $F^2$   
Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.027$   
 $wR(F^2) = 0.072$   
 $S = 1.07$   
4259 reflections  
198 parameters  
10 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  
 $w = 1/[\sigma^2(F_o^2) + (0.0361P)^2 + 5.6973P]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 0.42 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -0.69 \text{ e \AA}^{-3}$   
Extinction correction: none  
Absolute structure: Flack (1983), 1694 Friedel pairs  
Flack parameter: 0.00 (4)

## Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

|     | <i>x</i>     | <i>y</i>     | <i>z</i>     | $U_{\text{iso}}^*/U_{\text{eq}}$ | Occ. (<1) |
|-----|--------------|--------------|--------------|----------------------------------|-----------|
| Sn1 | 0.17065 (3)  | 0.17065 (3)  | 0.0000       | 0.05087 (13)                     |           |
| Sn2 | 0.33507 (3)  | 0.46713 (3)  | 0.035901 (8) | 0.04062 (9)                      |           |
| Sn3 | 0.64067 (3)  | 0.64067 (3)  | 0.0000       | 0.03517 (10)                     |           |
| Cl1 | 0.51581 (18) | 0.74108 (17) | 0.05370 (4)  | 0.0693 (4)                       |           |
| O1  | 0.1449 (3)   | 0.3159 (3)   | 0.05051 (8)  | 0.0476 (8)                       |           |
| O2  | 0.1753 (4)   | 0.5180 (4)   | 0.09499 (11) | 0.0663 (11)                      |           |
| O3  | 0.3190 (3)   | 0.3190 (3)   | 0.0000       | 0.0383 (9)                       |           |
| O4  | 0.5075 (16)  | 0.4933 (15)  | 0.0032 (5)   | 0.027 (2)                        | 0.50      |
| N1  | -0.0212 (4)  | 0.0806 (4)   | 0.03231 (12) | 0.0486 (10)                      |           |
| C1  | 0.2924 (5)   | 0.0328 (5)   | 0.02834 (14) | 0.0505 (12)                      |           |
| H1A | 0.3582       | -0.0091      | 0.0124       | 0.076*                           |           |

|      |             |             |              |             |      |
|------|-------------|-------------|--------------|-------------|------|
| H1B  | 0.2350      | -0.0376     | 0.0387       | 0.076*      |      |
| H1C  | 0.3406      | 0.0811      | 0.0471       | 0.076*      |      |
| C2   | 0.4546 (6)  | 0.3956 (6)  | 0.07732 (13) | 0.0564 (13) |      |
| H2A  | 0.5248      | 0.3352      | 0.0680       | 0.085*      |      |
| H2B  | 0.3977      | 0.3457      | 0.0940       | 0.085*      |      |
| H2C  | 0.4974      | 0.4723      | 0.0893       | 0.085*      |      |
| C3   | 0.2074 (5)  | 0.6200 (5)  | 0.01652 (15) | 0.0509 (12) |      |
| H3A  | 0.2616      | 0.7000      | 0.0105       | 0.076*      |      |
| H3B  | 0.1406      | 0.6441      | 0.0345       | 0.076*      |      |
| H3C  | 0.1606      | 0.5873      | -0.0045      | 0.076*      |      |
| C4   | 0.8115 (6)  | 0.5917 (6)  | 0.03104 (17) | 0.0613 (15) |      |
| H4A  | 0.8022      | 0.4991      | 0.0400       | 0.092*      |      |
| H4B  | 0.8177      | 0.6549      | 0.0508       | 0.092*      |      |
| H4C  | 0.8936      | 0.5983      | 0.0167       | 0.092*      |      |
| C5   | -0.0612 (6) | -0.0545 (6) | 0.01965 (15) | 0.0645 (16) |      |
| H5A  | -0.1547     | -0.0747     | 0.0275       | 0.077*      |      |
| H5B  | -0.0002     | -0.1237     | 0.0299       | 0.077*      |      |
| C6   | -0.0948 (5) | 0.1346 (5)  | 0.05572 (13) | 0.0465 (11) |      |
| H6   | -0.1747     | 0.0862      | 0.0617       | 0.056*      |      |
| C7   | -0.0736 (5) | 0.2594 (5)  | 0.07448 (12) | 0.0418 (10) |      |
| C8   | -0.1778 (5) | 0.2942 (6)  | 0.09830 (14) | 0.0509 (12) |      |
| H8   | -0.2577     | 0.2406      | 0.0992       | 0.061*      |      |
| C9   | -0.1654 (6) | 0.4048 (6)  | 0.12023 (14) | 0.0575 (14) |      |
| H9   | -0.2372     | 0.4286      | 0.1357       | 0.069*      |      |
| C10  | -0.0475 (6) | 0.4810 (6)  | 0.11964 (12) | 0.0551 (14) |      |
| H10  | -0.0369     | 0.5548      | 0.1354       | 0.066*      |      |
| C11  | 0.0529 (5)  | 0.4504 (5)  | 0.09661 (12) | 0.0452 (11) |      |
| C12  | 0.0447 (5)  | 0.3385 (5)  | 0.07272 (11) | 0.0399 (9)  |      |
| C13  | 0.2125 (16) | 0.6125 (10) | 0.1229 (3)   | 0.061 (2)*  | 0.50 |
| H13A | 0.3109      | 0.6070      | 0.1276       | 0.073*      | 0.50 |
| H13B | 0.1633      | 0.5900      | 0.1448       | 0.073*      | 0.50 |
| C14  | 0.1759 (17) | 0.7520 (13) | 0.1109 (4)   | 0.080 (3)*  | 0.50 |
| H14A | 0.0782      | 0.7565      | 0.1065       | 0.120*      | 0.50 |
| H14B | 0.2252      | 0.7734      | 0.0893       | 0.120*      | 0.50 |
| H14C | 0.2001      | 0.8174      | 0.1293       | 0.120*      | 0.50 |
| C13' | 0.1933 (14) | 0.6412 (9)  | 0.1157 (3)   | 0.061 (2)*  | 0.50 |
| H13C | 0.2913      | 0.6628      | 0.1162       | 0.073*      | 0.50 |
| H13D | 0.1656      | 0.6214      | 0.1402       | 0.073*      | 0.50 |
| C14' | 0.1189 (16) | 0.7656 (14) | 0.1039 (5)   | 0.080 (3)*  | 0.50 |
| H14D | 0.1808      | 0.8246      | 0.0908       | 0.120*      | 0.50 |
| H14E | 0.0841      | 0.8141      | 0.1245       | 0.120*      | 0.50 |
| H14F | 0.0432      | 0.7393      | 0.0887       | 0.120*      | 0.50 |

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

|     | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$      | $U^{13}$      | $U^{23}$      |
|-----|--------------|--------------|--------------|---------------|---------------|---------------|
| Sn1 | 0.03415 (14) | 0.03415 (14) | 0.0843 (3)   | -0.00937 (17) | -0.01282 (18) | 0.01282 (18)  |
| Sn2 | 0.04630 (18) | 0.03956 (16) | 0.03599 (14) | 0.01171 (13)  | 0.00052 (13)  | -0.00805 (12) |

## supplementary materials

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|     |              |              |             |               |               |              |
|-----|--------------|--------------|-------------|---------------|---------------|--------------|
| Sn3 | 0.03041 (12) | 0.03041 (12) | 0.0447 (2)  | -0.00586 (15) | -0.00722 (12) | 0.00722 (12) |
| Cl1 | 0.0783 (10)  | 0.0730 (10)  | 0.0567 (8)  | 0.0163 (8)    | -0.0115 (7)   | -0.0226 (7)  |
| O1  | 0.0442 (18)  | 0.0470 (19)  | 0.0516 (18) | -0.0007 (16)  | 0.0139 (15)   | -0.0160 (15) |
| O2  | 0.066 (3)    | 0.068 (3)    | 0.065 (2)   | -0.014 (2)    | 0.014 (2)     | -0.035 (2)   |
| O3  | 0.0332 (13)  | 0.0332 (13)  | 0.049 (2)   | -0.0092 (17)  | 0.0115 (14)   | -0.0115 (14) |
| O4  | 0.031 (4)    | 0.022 (3)    | 0.029 (5)   | -0.004 (3)    | -0.006 (3)    | 0.005 (3)    |
| N1  | 0.048 (2)    | 0.039 (2)    | 0.059 (2)   | -0.0105 (17)  | 0.012 (2)     | -0.0063 (19) |
| C1  | 0.052 (3)    | 0.039 (2)    | 0.061 (3)   | 0.004 (2)     | -0.007 (2)    | 0.008 (2)    |
| C2  | 0.058 (3)    | 0.067 (3)    | 0.044 (2)   | 0.004 (3)     | -0.007 (2)    | 0.013 (2)    |
| C3  | 0.042 (3)    | 0.041 (3)    | 0.070 (3)   | 0.009 (2)     | -0.003 (2)    | -0.001 (2)   |
| C4  | 0.044 (3)    | 0.061 (3)    | 0.079 (4)   | -0.004 (2)    | -0.027 (3)    | 0.011 (3)    |
| C5  | 0.054 (3)    | 0.050 (3)    | 0.090 (4)   | -0.023 (2)    | 0.024 (3)     | -0.014 (3)   |
| C6  | 0.040 (2)    | 0.049 (3)    | 0.051 (3)   | -0.004 (2)    | 0.008 (2)     | 0.006 (2)    |
| C7  | 0.046 (3)    | 0.044 (3)    | 0.035 (2)   | 0.0085 (19)   | 0.0034 (19)   | 0.0063 (19)  |
| C8  | 0.044 (3)    | 0.058 (3)    | 0.051 (3)   | 0.007 (2)     | 0.008 (2)     | 0.008 (2)    |
| C9  | 0.060 (3)    | 0.070 (4)    | 0.043 (3)   | 0.017 (3)     | 0.015 (3)     | 0.003 (2)    |
| C10 | 0.070 (4)    | 0.059 (3)    | 0.036 (2)   | 0.018 (3)     | 0.006 (2)     | -0.007 (2)   |
| C11 | 0.051 (3)    | 0.049 (3)    | 0.035 (2)   | 0.009 (2)     | 0.006 (2)     | -0.004 (2)   |
| C12 | 0.041 (2)    | 0.048 (3)    | 0.0302 (19) | 0.009 (2)     | 0.0041 (17)   | 0.0026 (19)  |

### *Geometric parameters (Å, °)*

|                      |             |                    |            |
|----------------------|-------------|--------------------|------------|
| Sn1—O3               | 2.072 (4)   | C2—H2B             | 0.9800     |
| Sn1—C1 <sup>i</sup>  | 2.112 (5)   | C2—H2C             | 0.9800     |
| Sn1—C1               | 2.112 (5)   | C3—H3A             | 0.9800     |
| Sn1—O1               | 2.410 (3)   | C3—H3B             | 0.9800     |
| Sn1—O1 <sup>i</sup>  | 2.410 (3)   | C3—H3C             | 0.9800     |
| Sn1—N1 <sup>i</sup>  | 2.426 (4)   | C4—H4A             | 0.9800     |
| Sn1—N1               | 2.426 (4)   | C4—H4B             | 0.9800     |
| Sn2—Cl1              | 3.310 (2)   | C4—H4C             | 0.9800     |
| Sn2—O1               | 2.463 (3)   | C5—C5 <sup>i</sup> | 1.497 (12) |
| Sn2—O2               | 2.791 (4)   | C5—H5A             | 0.9900     |
| Sn2—O3               | 2.006 (2)   | C5—H5B             | 0.9900     |
| Sn2—C2               | 2.091 (5)   | C6—C7              | 1.439 (7)  |
| Sn2—C3               | 2.100 (5)   | C6—H6              | 0.9500     |
| Sn2—O4               | 2.125 (17)  | C7—C12             | 1.406 (7)  |
| Sn2—O4 <sup>i</sup>  | 2.192 (16)  | C7—C8              | 1.414 (7)  |
| Sn2—Sn2 <sup>i</sup> | 3.2943 (6)  | C8—C9              | 1.379 (8)  |
| Sn3—O4               | 1.964 (5)   | C8—H8              | 0.9500     |
| Sn3—O4 <sup>i</sup>  | 1.964 (5)   | C9—C10             | 1.386 (9)  |
| Sn3—C4               | 2.114 (5)   | C9—H9              | 0.9500     |
| Sn3—C4 <sup>i</sup>  | 2.114 (5)   | C10—C11            | 1.356 (7)  |
| Sn3—Cl1              | 2.5829 (15) | C10—H10            | 0.9500     |
| Sn3—Cl1 <sup>i</sup> | 2.5829 (15) | C11—C12            | 1.432 (7)  |
| O1—C12               | 1.320 (5)   | C13—C14            | 1.493 (9)  |
| O2—C11               | 1.381 (7)   | C13—H13A           | 0.9900     |
| O2—C13               | 1.459 (8)   | C13—H13B           | 0.9900     |

|                                      |             |                         |           |
|--------------------------------------|-------------|-------------------------|-----------|
| O2—C13'                              | 1.460 (8)   | C14—H14A                | 0.9800    |
| O3—Sn2 <sup>i</sup>                  | 2.006 (2)   | C14—H14B                | 0.9800    |
| O4—O4 <sup>i</sup>                   | 0.31 (4)    | C14—H14C                | 0.9800    |
| O4—Sn2 <sup>i</sup>                  | 2.192 (16)  | C13'—C14'               | 1.499 (9) |
| N1—C6                                | 1.267 (6)   | C13'—H13C               | 0.9900    |
| N1—C5                                | 1.472 (6)   | C13'—H13D               | 0.9900    |
| C1—H1A                               | 0.9800      | C14'—H14D               | 0.9800    |
| C1—H1B                               | 0.9800      | C14'—H14E               | 0.9800    |
| C1—H1C                               | 0.9800      | C14'—H14F               | 0.9800    |
| C2—H2A                               | 0.9800      |                         |           |
| O3—Sn1—C1 <sup>i</sup>               | 93.05 (15)  | C6—N1—Sn1               | 130.5 (3) |
| O3—Sn1—C1                            | 93.04 (15)  | C5—N1—Sn1               | 112.1 (3) |
| C1 <sup>i</sup> —Sn1—C1              | 173.9 (3)   | Sn1—C1—H1A              | 109.5     |
| O3—Sn1—O1                            | 69.76 (8)   | Sn1—C1—H1B              | 109.5     |
| C1 <sup>i</sup> —Sn1—O1              | 89.99 (17)  | H1A—C1—H1B              | 109.5     |
| C1—Sn1—O1                            | 92.12 (17)  | Sn1—C1—H1C              | 109.5     |
| O3—Sn1—O1 <sup>i</sup>               | 69.76 (8)   | H1A—C1—H1C              | 109.5     |
| C1 <sup>i</sup> —Sn1—O1 <sup>i</sup> | 92.12 (17)  | H1B—C1—H1C              | 109.5     |
| C1—Sn1—O1 <sup>i</sup>               | 89.99 (17)  | Sn2—C2—H2A              | 109.5     |
| O1—Sn1—O1 <sup>i</sup>               | 139.52 (16) | Sn2—C2—H2B              | 109.5     |
| O3—Sn1—N1 <sup>i</sup>               | 144.20 (10) | H2A—C2—H2B              | 109.5     |
| C1 <sup>i</sup> —Sn1—N1 <sup>i</sup> | 87.14 (19)  | Sn2—C2—H2C              | 109.5     |
| C1—Sn1—N1 <sup>i</sup>               | 87.92 (18)  | H2A—C2—H2C              | 109.5     |
| O1—Sn1—N1 <sup>i</sup>               | 146.01 (13) | H2B—C2—H2C              | 109.5     |
| O1 <sup>i</sup> —Sn1—N1 <sup>i</sup> | 74.46 (13)  | Sn2—C3—H3A              | 109.5     |
| O3—Sn1—N1                            | 144.20 (10) | Sn2—C3—H3B              | 109.5     |
| C1 <sup>i</sup> —Sn1—N1              | 87.92 (18)  | H3A—C3—H3B              | 109.5     |
| C1—Sn1—N1                            | 87.14 (19)  | Sn2—C3—H3C              | 109.5     |
| O1—Sn1—N1                            | 74.46 (13)  | H3A—C3—H3C              | 109.5     |
| O1 <sup>i</sup> —Sn1—N1              | 146.01 (13) | H3B—C3—H3C              | 109.5     |
| N1 <sup>i</sup> —Sn1—N1              | 71.6 (2)    | Sn3—C4—H4A              | 109.5     |
| O3—Sn2—C2                            | 108.10 (17) | Sn3—C4—H4B              | 109.5     |
| O3—Sn2—C3                            | 103.75 (16) | H4A—C4—H4B              | 109.5     |
| C2—Sn2—C3                            | 147.8 (2)   | Sn3—C4—H4C              | 109.5     |
| O3—Sn2—O4                            | 75.7 (4)    | H4A—C4—H4C              | 109.5     |
| C2—Sn2—O4                            | 91.7 (5)    | H4B—C4—H4C              | 109.5     |
| C3—Sn2—O4                            | 100.8 (5)   | N1—C5—C5 <sup>i</sup>   | 110.7 (4) |
| O3—Sn2—O4 <sup>i</sup>               | 74.2 (4)    | N1—C5—H5A               | 109.5     |
| C2—Sn2—O4 <sup>i</sup>               | 99.8 (5)    | C5 <sup>i</sup> —C5—H5A | 109.5     |
| C3—Sn2—O4 <sup>i</sup>               | 93.4 (5)    | N1—C5—H5B               | 109.5     |
| O4—Sn2—O4 <sup>i</sup>               | 8.1 (10)    | C5 <sup>i</sup> —C5—H5B | 109.5     |
| O3—Sn2—O1                            | 69.60 (11)  | H5A—C5—H5B              | 108.1     |
| C2—Sn2—O1                            | 93.19 (18)  | N1—C6—C7                | 128.7 (5) |
| C3—Sn2—O1                            | 93.30 (17)  | N1—C6—H6                | 115.7     |



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|                                       |             |                           |            |
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| O4—Sn2—O1                             | 144.8 (4)   | C7—C6—H6                  | 115.7      |
| O4 <sup>i</sup> —Sn2—O1               | 143.8 (4)   | C12—C7—C8                 | 120.0 (5)  |
| O3—Sn2—Sn2 <sup>i</sup>               | 34.82 (9)   | C12—C7—C6                 | 125.0 (4)  |
| C2—Sn2—Sn2 <sup>i</sup>               | 105.49 (17) | C8—C7—C6                  | 114.8 (5)  |
| C3—Sn2—Sn2 <sup>i</sup>               | 103.37 (16) | C9—C8—C7                  | 121.0 (5)  |
| O4—Sn2—Sn2 <sup>i</sup>               | 41.0 (4)    | C9—C8—H8                  | 119.5      |
| O4 <sup>i</sup> —Sn2—Sn2 <sup>i</sup> | 39.5 (4)    | C7—C8—H8                  | 119.5      |
| O1—Sn2—Sn2 <sup>i</sup>               | 104.40 (7)  | C8—C9—C10                 | 119.6 (5)  |
| O4—Sn3—O4 <sup>i</sup>                | 9.1 (10)    | C8—C9—H9                  | 120.2      |
| O4—Sn3—C4                             | 109.3 (6)   | C10—C9—H9                 | 120.2      |
| O4 <sup>i</sup> —Sn3—C4               | 118.2 (6)   | C11—C10—C9                | 120.2 (5)  |
| O4—Sn3—C4 <sup>i</sup>                | 118.2 (6)   | C11—C10—H10               | 119.9      |
| O4 <sup>i</sup> —Sn3—C4 <sup>i</sup>  | 109.3 (6)   | C9—C10—H10                | 119.9      |
| C4—Sn3—C4 <sup>i</sup>                | 132.6 (3)   | C10—C11—O2                | 124.1 (5)  |
| O4—Sn3—C11                            | 85.2 (6)    | C10—C11—C12               | 122.6 (5)  |
| O4 <sup>i</sup> —Sn3—C11              | 87.2 (6)    | O2—C11—C12                | 113.2 (4)  |
| C4—Sn3—C11                            | 91.55 (19)  | O1—C12—C7                 | 124.0 (4)  |
| C4 <sup>i</sup> —Sn3—C11              | 91.49 (19)  | O1—C12—C11                | 119.6 (4)  |
| O4—Sn3—C11 <sup>i</sup>               | 87.2 (6)    | C7—C12—C11                | 116.4 (4)  |
| O4 <sup>i</sup> —Sn3—C11 <sup>i</sup> | 85.2 (6)    | O2—C13—C14                | 108.0 (10) |
| C4—Sn3—C11 <sup>i</sup>               | 91.49 (19)  | O2—C13—H13A               | 110.1      |
| C4 <sup>i</sup> —Sn3—C11 <sup>i</sup> | 91.55 (19)  | C14—C13—H13A              | 110.1      |
| C11—Sn3—C11 <sup>i</sup>              | 172.42 (8)  | O2—C13—H13B               | 110.1      |
| C12—O1—Sn1                            | 133.6 (3)   | C14—C13—H13B              | 110.1      |
| C12—O1—Sn2                            | 127.8 (3)   | H13A—C13—H13B             | 108.4      |
| Sn1—O1—Sn2                            | 95.76 (11)  | O2—C13'—C14'              | 117.5 (10) |
| C11—O2—C13                            | 119.8 (7)   | O2—C13'—H13C              | 107.9      |
| C11—O2—C13'                           | 119.0 (7)   | C14'—C13'—H13C            | 107.9      |
| Sn2—O3—Sn2 <sup>i</sup>               | 110.36 (18) | O2—C13'—H13D              | 107.9      |
| Sn2—O3—Sn1                            | 124.82 (9)  | C14'—C13'—H13D            | 107.9      |
| Sn2 <sup>i</sup> —O3—Sn1              | 124.82 (9)  | H13C—C13'—H13D            | 107.2      |
| O4 <sup>i</sup> —O4—Sn3               | 85.4 (5)    | C13'—C14'—H14D            | 109.5      |
| O4 <sup>i</sup> —O4—Sn2               | 98 (6)      | C13'—C14'—H14E            | 109.5      |
| Sn3—O4—Sn2                            | 131.5 (10)  | H14D—C14'—H14E            | 109.5      |
| O4 <sup>i</sup> —O4—Sn2 <sup>i</sup>  | 73 (6)      | C13'—C14'—H14F            | 109.5      |
| Sn3—O4—Sn2 <sup>i</sup>               | 127.4 (9)   | H14D—C14'—H14F            | 109.5      |
| Sn2—O4—Sn2 <sup>i</sup>               | 99.5 (2)    | H14E—C14'—H14F            | 109.5      |
| C6—N1—C5                              | 117.2 (4)   |                           |            |
| O3—Sn1—O1—C12                         | 162.8 (4)   | O3—Sn2—O4—O4 <sup>i</sup> | 78 (5)     |
| C1 <sup>i</sup> —Sn1—O1—C12           | 69.6 (4)    | C2—Sn2—O4—O4 <sup>i</sup> | -174 (6)   |
| C1—Sn1—O1—C12                         | -104.7 (4)  | C3—Sn2—O4—O4 <sup>i</sup> | -23 (6)    |
| O1 <sup>i</sup> —Sn1—O1—C12           | 162.8 (4)   | O1—Sn2—O4—O4 <sup>i</sup> | 88 (6)     |

|  |              |  |            |
|--|--------------|--|------------|
| N1 <sup>i</sup> —Sn1—O1—C12              | -15.3 (6)    | Sn2 <sup>i</sup> —Sn2—O4—O4 <sup>i</sup> | 75 (5)     |
| N1—Sn1—O1—C12                            | -18.3 (4)    | O3—Sn2—O4—Sn3                            | 169.5 (13) |
| O3—Sn1—O1—Sn2                            | 1.46 (6)     | C2—Sn2—O4—Sn3                            | -82.3 (12) |
| C1 <sup>i</sup> —Sn1—O1—Sn2              | -91.79 (18)  | C3—Sn2—O4—Sn3                            | 67.9 (12)  |
| C1—Sn1—O1—Sn2                            | 93.93 (18)   | O4 <sup>i</sup> —Sn2—O4—Sn3              | 91 (6)     |
| O1 <sup>i</sup> —Sn1—O1—Sn2              | 1.46 (6)     | O1—Sn2—O4—Sn3                            | 179.8 (5)  |
| N1 <sup>i</sup> —Sn1—O1—Sn2              | -176.7 (2)   | Sn2 <sup>i</sup> —Sn2—O4—Sn3             | 165.9 (17) |
| N1—Sn1—O1—Sn2                            | -179.63 (16) | O3—Sn2—O4—Sn2 <sup>i</sup>               | 3.6 (5)    |
| O3—Sn2—O1—C12                            | -164.5 (4)   | C2—Sn2—O4—Sn2 <sup>i</sup>               | 111.9 (6)  |
| C2—Sn2—O1—C12                            | 87.4 (4)     | C3—Sn2—O4—Sn2 <sup>i</sup>               | -98.0 (6)  |
| C3—Sn2—O1—C12                            | -61.0 (4)    | O4 <sup>i</sup> —Sn2—O4—Sn2 <sup>i</sup> | -75 (5)    |
| O4—Sn2—O1—C12                            | -175.1 (9)   | O1—Sn2—O4—Sn2 <sup>i</sup>               | 13.9 (13)  |
| O4 <sup>i</sup> —Sn2—O1—C12              | -161.3 (9)   | O3—Sn1—N1—C6                             | 19.7 (6)   |
| Sn2 <sup>i</sup> —Sn2—O1—C12             | -165.7 (3)   | C1 <sup>i</sup> —Sn1—N1—C6               | -72.6 (5)  |
| O3—Sn2—O1—Sn1                            | -1.51 (7)    | C1—Sn1—N1—C6                             | 110.9 (5)  |
| C2—Sn2—O1—Sn1                            | -109.65 (19) | O1—Sn1—N1—C6                             | 17.9 (5)   |
| C3—Sn2—O1—Sn1                            | 101.95 (18)  | O1 <sup>i</sup> —Sn1—N1—C6               | -163.3 (4) |
| O4—Sn2—O1—Sn1                            | -12.1 (9)    | N1 <sup>i</sup> —Sn1—N1—C6               | -160.3 (6) |
| O4 <sup>i</sup> —Sn2—O1—Sn1              | 1.7 (9)      | O3—Sn1—N1—C5                             | -165.8 (3) |
| Sn2 <sup>i</sup> —Sn2—O1—Sn1             | -2.76 (12)   | C1 <sup>i</sup> —Sn1—N1—C5               | 101.8 (4)  |
| C2—Sn2—O3—Sn2 <sup>i</sup>               | -91.27 (17)  | C1—Sn1—N1—C5                             | -74.6 (4)  |
| C3—Sn2—O3—Sn2 <sup>i</sup>               | 93.72 (16)   | O1—Sn1—N1—C5                             | -167.6 (4) |
| O4—Sn2—O3—Sn2 <sup>i</sup>               | -4.2 (5)     | O1 <sup>i</sup> —Sn1—N1—C5               | 11.1 (5)   |
| O4 <sup>i</sup> —Sn2—O3—Sn2 <sup>i</sup> | 4.1 (5)      | N1 <sup>i</sup> —Sn1—N1—C5               | 14.2 (3)   |
| O1—Sn2—O3—Sn2 <sup>i</sup>               | -177.87 (9)  | C6—N1—C5—C5 <sup>i</sup>                 | 134.5 (6)  |
| C2—Sn2—O3—Sn1                            | 88.73 (17)   | Sn1—N1—C5—C5 <sup>i</sup>                | -40.7 (7)  |
| C3—Sn2—O3—Sn1                            | -86.28 (16)  | C5—N1—C6—C7                              | 174.7 (5)  |
| O4—Sn2—O3—Sn1                            | 175.8 (5)    | Sn1—N1—C6—C7                             | -11.1 (8)  |
| O4 <sup>i</sup> —Sn2—O3—Sn1              | -175.9 (5)   | N1—C6—C7—C12                             | -7.7 (8)   |
| O1—Sn2—O3—Sn1                            | 2.13 (9)     | N1—C6—C7—C8                              | 177.8 (5)  |
| Sn2 <sup>i</sup> —Sn2—O3—Sn1             | 180.0        | C12—C7—C8—C9                             | -0.4 (7)   |
| C1 <sup>i</sup> —Sn1—O3—Sn2              | 86.70 (15)   | C6—C7—C8—C9                              | 174.3 (5)  |
| C1—Sn1—O3—Sn2                            | -93.30 (15)  | C7—C8—C9—C10                             | -1.8 (8)   |
| O1—Sn1—O3—Sn2                            | -2.17 (9)    | C8—C9—C10—C11                            | 2.7 (8)    |
| O1 <sup>i</sup> —Sn1—O3—Sn2              | 177.83 (9)   | C9—C10—C11—O2                            | -177.8 (5) |
| N1 <sup>i</sup> —Sn1—O3—Sn2              | 176.03 (19)  | C9—C10—C11—C12                           | -1.5 (8)   |
| N1—Sn1—O3—Sn2                            | -3.97 (19)   | C13—O2—C11—C10                           | 11.1 (9)   |
| C1 <sup>i</sup> —Sn1—O3—Sn2 <sup>i</sup> | -93.30 (15)  | C13 <sup>i</sup> —O2—C11—C10             | -8.6 (9)   |
| C1—Sn1—O3—Sn2 <sup>i</sup>               | 86.70 (15)   | C13—O2—C11—C12                           | -165.5 (7) |
| O1—Sn1—O3—Sn2 <sup>i</sup>               | 177.83 (9)   | C13 <sup>i</sup> —O2—C11—C12             | 174.7 (6)  |
| O1 <sup>i</sup> —Sn1—O3—Sn2 <sup>i</sup> | -2.17 (9)    | Sn1—O1—C12—C7                            | 10.6 (7)   |
| N1 <sup>i</sup> —Sn1—O3—Sn2 <sup>i</sup> | -3.97 (19)   | Sn2—O1—C12—C7                            | 166.8 (3)  |

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| N1—Sn1—O3—Sn2 <sup>i</sup>                | 176.03 (19) | Sn1—O1—C12—C11                            | -168.7 (3) |
| C4—Sn3—O4—O4 <sup>i</sup>                 | -167 (8)    | Sn2—O1—C12—C11                            | -12.5 (6)  |
| C4 <sup>i</sup> —Sn3—O4—O4 <sup>i</sup>   | 13 (9)      | C8—C7—C12—O1                              | -177.7 (4) |
| Cl1—Sn3—O4—O4 <sup>i</sup>                | 103 (8)     | C6—C7—C12—O1                              | 8.1 (7)    |
| Cl1 <sup>i</sup> —Sn3—O4—O4 <sup>i</sup>  | -77 (8)     | C8—C7—C12—C11                             | 1.6 (7)    |
| O4 <sup>i</sup> —Sn3—O4—Sn2               | -97 (9)     | C6—C7—C12—C11                             | -172.6 (5) |
| C4—Sn3—O4—Sn2                             | 95.4 (12)   | C10—C11—C12—O1                            | 178.7 (5)  |
| C4 <sup>i</sup> —Sn3—O4—Sn2               | -83.7 (12)  | O2—C11—C12—O1                             | -4.6 (7)   |
| Cl1—Sn3—O4—Sn2                            | 5.4 (11)    | C10—C11—C12—C7                            | -0.7 (7)   |
| Cl1 <sup>i</sup> —Sn3—O4—Sn2              | -174.0 (11) | O2—C11—C12—C7                             | 176.0 (4)  |
| O4 <sup>i</sup> —Sn3—O4—Sn2 <sup>i</sup>  | 65 (8)      | C11—O2—C13—C14                            | -96.5 (12) |
| C4—Sn3—O4—Sn2 <sup>i</sup>                | -102.3 (10) | C13 <sup>i</sup> —O2—C13—C14              | -4(3)      |
| C4 <sup>i</sup> —Sn3—O4—Sn2 <sup>i</sup>  | 78.6 (13)   | C11—O2—C13 <sup>i</sup> —C14 <sup>i</sup> | -71.7 (14) |
| Cl1—Sn3—O4—Sn2 <sup>i</sup>               | 167.7 (11)  | C13—O2—C13 <sup>i</sup> —C14 <sup>i</sup> | -169 (5)   |
| Cl1 <sup>i</sup> —Sn3—O4—Sn2 <sup>i</sup> | -11.7 (11)  |   |            |

Symmetry codes: (i)  $y, x, -z$ .

Fig. 1

