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Di-μ_2-ethanolato-octamethylbis(μ-4-methyl-5-sulfanylidene-4,5-dihydro-1*H*-1,2,4-triazol-1-ido-κ^2N^1:N^2)di-μ_3-oxido-tetratin(IV)

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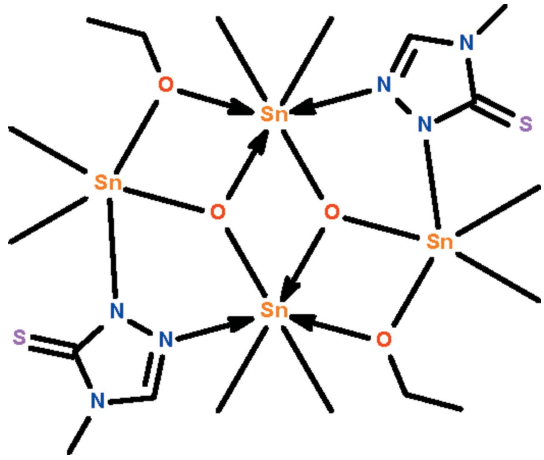
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.005 Å; R factor = 0.026; wR factor = 0.065; data-to-parameter ratio = 24.1.

The tetranuclear title compound, $[Sn_4(CH_3)_8(C_2H_5O)_2O_2(C_3H_4N_3S)_2]$, lies about a center of inversion; the molecule features a three-rung-staircase Sn_4O_4 core in which two Sn^{IV} atoms are bridged by the 4-methyl-5-sulfanylidene-4,5-dihydro-1*H*-1,2,4-triazol-1-ide group. The negatively charged N atom of the group binds to the terminal Sn^{IV} atom at a shorter distance [Sn—N = 2.240 (3) Å] compared with the neutral N atom that binds to the central Sn^{IV} atom [Sn← N = 2.641 (3) Å]. The terminal Sn^{IV} atom is five-coordinate in a *cis*-C_2SnNO_2 trigonal–bipyramidal geometry [C—Sn—C = 127.5 (2)°], whereas the central Sn^{IV} atom is six-coordinate in a C_2SnNO_3 skew-trazepoidal bipyramidal geometry [C—Sn—C = 145.0 (2)°].

Related literature

For the $[Sn_2O(CH_3)_4(CH_3O)(C_3H_4N_3S)]_2$ homolog, see: Najafi *et al.* (2011).



Experimental

Crystal data

$[Sn_4(CH_3)_8(C_2H_5O)_2O_2(C_3H_4N_3S)_2]$
M_r = 945.46
Monoclinic, $P2_1/n$
a = 9.3965 (4) Å
b = 17.8939 (7) Å
c = 9.9084 (4) Å
β = 103.036 (4)°
V = 1623.06 (11) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 3.20 mm^{-1}
T = 100 K
0.30 × 0.25 × 0.20 mm

Data collection

Agilent SuperNova (Dual, Cu at zero, Atlas) diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2012)
T_{min} = 0.447, T_{max} = 0.567
15728 measured reflections
3743 independent reflections
3280 reflections with $I > 2\sigma(I)$
R_{int} = 0.037

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.065
S = 1.04
3743 reflections
155 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.75 e Å^{-3}
$\Delta\rho_{min}$ = −0.67 e Å^{-3}

Data collection: *CrysAlis PRO* (Agilent, 2012); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; 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, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT6863).

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supporting information

Acta Cryst. (2012). E**68**, m1545 [doi:10.1107/S1600536812047708]

Di-μ_2-ethanolato-octamethylbis(μ-4-methyl-5-sulfanylidene-4,5-dihydro-1*H*-1,2,4-triazol-1-ido-κ^2N^1:N^2)di-μ_3-oxido-tetratin(IV)

Ezzatollah Najafi, Mostafa M. Amini and Seik Weng Ng

S1. Comment

The title compound (Scheme I, Fig. 1), a distannoxane, was the unexpected product from an attempt at synthesizing a dimethyltin 4-methyl-4*H*-1,2,4-triazol-3-thiolate that possesses a tin-sulfur linkage. In the reaction of diorganotin oxides with organic acids (particularly carboxylic acids), tetranuclear distannoxanes are sometimes formed; these compounds have four organic groups. In the present reaction, two of the four organic groups are replaced by ethoxide groups.

Tetranuclear $Sn_4O_2(CH_3)_8(C_2H_5O)_2(C_3H_4N_3S)_2$ lies about a center-of-inversion; the molecule features a three-rung-staircase Sn_4O_4 core in which two Sn atoms are bridged by the $C_3H_4N_3S$ triazolate group. The negatively-charged N atom of the group binds to the terminal Sn atom at a shorter distance [Sn–N 2.240 (3) Å] compared with the neutral N atom that binds to the central Sn atom [Sn←N 2.641 (3) Å]. The terminal Sn atom is five-coordinate in a *cis*-C_3SnNO trigonal bipyramid whereas the central Sn atom is six-coordinate in a C_2SnNO_3 skew-trazepoidal bipyramidal geometry.

S2. Experimental

Dimethyltin diisothiocyanate (1 mmol), 4-methyl-4*H*-1,2,4-triazole-3-thiol (1 mmol) and 1,10-phenanthroline (1 mmol) were loaded into a convection tube; several drops of triethylamine were added. The tube was filled with dry ethanol and kept at 333 K. Colorless crystals were collected from the side arm after several days.

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions [C—H 0.95 to 0.98 Å, U_{iso}(H) 1.2 to 1.5U_{eq}(C)] and were included in the refinement in the riding model approximation.

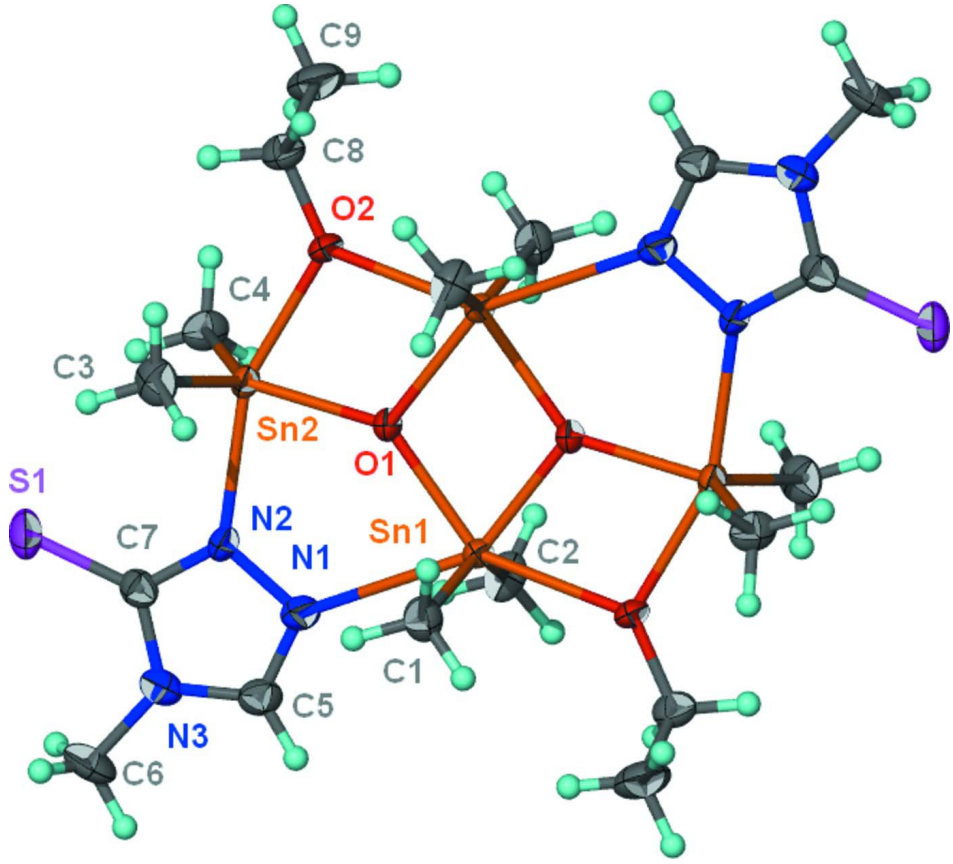

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $Sn_4O_2(CH_3)_8(C_2H_5O)_2(C_3H_4N_3S)_2$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

Di-μ_2-ethanolato-octamethylbis(μ-4-methyl-5-sulfanylidene-4,5-dihydro- 1*H*-1,2,4-triazolido-κ^2N^1:N^2)di-μ_3-oxido- tetratin(IV)

Crystal data

$[Sn_4(CH_3)_8(C_2H_5O)_2O_2(C_3H_4N_3S)_2]$
M_r = 945.46
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 9.3965 (4) Å
b = 17.8939 (7) Å
c = 9.9084 (4) Å
β = 103.036 (4)°
V = 1623.06 (11) Å^3
Z = 2
$F(000)$ = 912
D_x = 1.935 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 6388 reflections
θ = 2.9–27.5°
μ = 3.20 mm^{-1}
T = 100 K
Prism, colorless
0.30 × 0.25 × 0.20 mm

Data collection

Agilent SuperNova (Dual, Cu at zero, Atlas) diffractometer
Radiation source: SuperNova (Mo) X-ray Source
Mirror monochromator
Detector resolution: 10.4041 pixels mm^{-1}
ω scan
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2012)
T_{min} = 0.447, T_{max} = 0.567
15728 measured reflections
3743 independent reflections

3280 reflections with $I > 2\sigma(I)$
$R_{int} = 0.037$
$\theta_{max} = 27.6°$, $\theta_{min} = 2.9°$
$h = -11 \rightarrow 12$
$k = -23 \rightarrow 21$
$l = -11 \rightarrow 12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.026$
$wR(F^2) = 0.065$
$S = 1.04$
3743 reflections
155 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
$w = 1/[\sigma^2(F_o^2) + (0.0302P)^2 + 2.3203P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.75$ e Å^{-3}
$\Delta\rho_{min} = -0.67$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Sn1	0.37077 (2)	0.436391 (12)	0.49959 (2)	0.01497 (7)
Sn2	0.70638 (2)	0.467990 (13)	0.80539 (2)	0.01507 (7)
S1	0.69104 (11)	0.33447 (6)	1.09067 (10)	0.0342 (2)
O1	0.5639 (2)	0.48179 (13)	0.6221 (2)	0.0182 (5)
O2	0.7909 (3)	0.55849 (14)	0.7058 (2)	0.0200 (5)
N1	0.4168 (3)	0.36557 (17)	0.7393 (3)	0.0224 (6)
N2	0.5475 (3)	0.37949 (17)	0.8347 (3)	0.0199 (6)
N3	0.4324 (3)	0.29205 (17)	0.9189 (3)	0.0233 (6)
C1	0.4547 (4)	0.3342 (2)	0.4438 (4)	0.0253 (8)
H1A	0.5373	0.3442	0.4012	0.038*
H1B	0.3782	0.3076	0.3777	0.038*
H1C	0.4877	0.3034	0.5268	0.038*
C2	0.2100 (4)	0.4864 (2)	0.5902 (4)	0.0265 (8)
H2A	0.2210	0.5408	0.5902	0.040*
H2B	0.2221	0.4686	0.6857	0.040*
H2C	0.1125	0.4727	0.5366	0.040*
C3	0.8781 (4)	0.3928 (2)	0.7987 (4)	0.0276 (8)
H3A	0.9637	0.4208	0.7859	0.041*
H3B	0.8469	0.3579	0.7214	0.041*
H3C	0.9034	0.3647	0.8858	0.041*
C4	0.6810 (4)	0.5344 (2)	0.9736 (3)	0.0247 (8)
H4A	0.7441	0.5785	0.9803	0.037*
H4B	0.7081	0.5054	1.0594	0.037*
H4C	0.5789	0.5504	0.9595	0.037*
C5	0.3523 (4)	0.3131 (2)	0.7935 (3)	0.0236 (7)
H5	0.2603	0.2921	0.7505	0.028*
C6	0.4001 (5)	0.2340 (2)	1.0099 (4)	0.0343 (9)
H6A	0.3039	0.2124	0.9699	0.051*
H6B	0.3999	0.2555	1.1008	0.051*
H6C	0.4747	0.1948	1.0204	0.051*
C7	0.5556 (4)	0.3356 (2)	0.9450 (3)	0.0212 (7)

C8	0.9254 (4)	0.5956 (2)	0.7578 (4)	0.0239 (7)
H8A	0.9841	0.5950	0.6864	0.029*
H8B	0.9809	0.5685	0.8400	0.029*
C9	0.9025 (5)	0.6751 (2)	0.7972 (5)	0.0381 (10)
H9A	0.9974	0.6990	0.8327	0.057*
H9B	0.8458	0.6758	0.8689	0.057*
H9C	0.8493	0.7024	0.7155	0.057*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sn1	0.01475 (12)	0.01498 (13)	0.01399 (11)	−0.00248 (8)	0.00072 (8)	0.00005 (8)
Sn2	0.01482 (12)	0.01626 (13)	0.01265 (11)	0.00072 (8)	−0.00002 (8)	−0.00025 (8)
S1	0.0269 (5)	0.0421 (6)	0.0294 (5)	0.0007 (4)	−0.0028 (4)	0.0170 (4)
O1	0.0186 (12)	0.0190 (12)	0.0146 (11)	−0.0030 (10)	−0.0011 (9)	0.0042 (9)
O2	0.0177 (12)	0.0203 (13)	0.0190 (12)	−0.0075 (10)	−0.0018 (9)	0.0009 (9)
N1	0.0239 (16)	0.0221 (16)	0.0198 (14)	−0.0062 (13)	0.0017 (11)	−0.0017 (11)
N2	0.0179 (14)	0.0235 (16)	0.0154 (13)	−0.0038 (12)	−0.0021 (10)	0.0026 (11)
N3	0.0275 (17)	0.0184 (16)	0.0251 (15)	−0.0027 (13)	0.0083 (12)	0.0011 (12)
C1	0.031 (2)	0.022 (2)	0.0214 (17)	0.0031 (16)	0.0031 (14)	−0.0010 (14)
C2	0.0229 (19)	0.036 (2)	0.0215 (17)	0.0055 (16)	0.0056 (14)	−0.0044 (15)
C3	0.0238 (19)	0.025 (2)	0.034 (2)	0.0090 (16)	0.0073 (15)	0.0025 (15)
C4	0.029 (2)	0.027 (2)	0.0198 (17)	−0.0050 (16)	0.0076 (14)	−0.0073 (14)
C5	0.0273 (19)	0.0212 (19)	0.0222 (17)	−0.0050 (15)	0.0056 (14)	−0.0048 (13)
C6	0.043 (2)	0.024 (2)	0.039 (2)	−0.0082 (18)	0.0167 (18)	0.0084 (16)
C7	0.0218 (18)	0.0180 (18)	0.0242 (17)	0.0023 (14)	0.0062 (13)	0.0017 (13)
C8	0.0184 (17)	0.025 (2)	0.0285 (18)	−0.0033 (15)	0.0049 (14)	−0.0017 (14)
C9	0.030 (2)	0.030 (2)	0.050 (3)	−0.0106 (18)	0.0016 (18)	−0.0093 (18)

Geometric parameters (Å, º)

Sn1—O1^{i}	2.076 (2)	C1—H1B	0.9800
Sn1—O1	2.106 (2)	C1—H1C	0.9800
Sn1—C1	2.114 (4)	C2—H2A	0.9800
Sn1—C2	2.121 (4)	C2—H2B	0.9800
Sn1—O2^{i}	2.250 (2)	C2—H2C	0.9800
Sn1—N1	2.641 (3)	C3—H3A	0.9800
Sn2—O1	2.013 (2)	C3—H3B	0.9800
Sn2—C4	2.105 (3)	C3—H3C	0.9800
Sn2—C3	2.114 (4)	C4—H4A	0.9800
Sn2—O2	2.140 (2)	C4—H4B	0.9800
Sn2—N2	2.240 (3)	C4—H4C	0.9800
S1—C7	1.695 (4)	C5—H5	0.9500
O1—Sn1^{i}	2.076 (2)	C6—H6A	0.9800
O2—C8	1.418 (4)	C6—H6B	0.9800
O2—Sn1^{i}	2.250 (2)	C6—H6C	0.9800
N1—C5	1.297 (5)	C8—C9	1.503 (6)
N1—N2	1.393 (4)	C8—H8A	0.9900

N2—C7	1.334 (4)	C8—H8B	0.9900
N3—C5	1.353 (4)	C9—H9A	0.9800
N3—C7	1.371 (5)	C9—H9B	0.9800
N3—C6	1.452 (5)	C9—H9C	0.9800
C1—H1A	0.9800		

O1^{i}—Sn1—O1	74.55 (9)	H1A—C1—H1C	109.5
O1^{i}—Sn1—C1	106.36 (12)	H1B—C1—H1C	109.5
O1—Sn1—C1	99.22 (12)	Sn1—C2—H2A	109.5
O1^{i}—Sn1—C2	106.31 (13)	Sn1—C2—H2B	109.5
O1—Sn1—C2	101.36 (12)	H2A—C2—H2B	109.5
C1—Sn1—C2	144.96 (16)	Sn1—C2—H2C	109.5
O1^{i}—Sn1—O2^{i}	70.95 (8)	H2A—C2—H2C	109.5
O1—Sn1—O2^{i}	145.49 (9)	H2B—C2—H2C	109.5
C1—Sn1—O2^{i}	90.83 (12)	Sn2—C3—H3A	109.5
C2—Sn1—O2^{i}	88.04 (12)	Sn2—C3—H3B	109.5
O1^{i}—Sn1—N1	148.36 (9)	H3A—C3—H3B	109.5
O1—Sn1—N1	73.83 (9)	Sn2—C3—H3C	109.5
C1—Sn1—N1	79.92 (12)	H3A—C3—H3C	109.5
C2—Sn1—N1	79.00 (12)	H3B—C3—H3C	109.5
O2^{i}—Sn1—N1	140.66 (9)	Sn2—C4—H4A	109.5
O1—Sn2—C4	118.29 (13)	Sn2—C4—H4B	109.5
O1—Sn2—C3	114.01 (12)	H4A—C4—H4B	109.5
C4—Sn2—C3	127.46 (15)	Sn2—C4—H4C	109.5
O1—Sn2—O2	74.44 (9)	H4A—C4—H4C	109.5
C4—Sn2—O2	93.32 (12)	H4B—C4—H4C	109.5
C3—Sn2—O2	95.86 (13)	N1—C5—N3	111.5 (3)
O1—Sn2—N2	82.92 (9)	N1—C5—H5	124.3
C4—Sn2—N2	95.66 (13)	N3—C5—H5	124.3
C3—Sn2—N2	95.15 (14)	N3—C6—H6A	109.5
O2—Sn2—N2	157.27 (9)	N3—C6—H6B	109.5
Sn2—O1—Sn1^{i}	112.77 (11)	H6A—C6—H6B	109.5
Sn2—O1—Sn1	141.62 (12)	N3—C6—H6C	109.5
Sn1^{i}—O1—Sn1	105.45 (9)	H6A—C6—H6C	109.5
C8—O2—Sn2	125.4 (2)	H6B—C6—H6C	109.5
C8—O2—Sn1^{i}	132.4 (2)	N2—C7—N3	106.8 (3)
Sn2—O2—Sn1^{i}	101.70 (9)	N2—C7—S1	126.7 (3)
C5—N1—N2	105.8 (3)	N3—C7—S1	126.4 (3)
C5—N1—Sn1	136.2 (2)	O2—C8—C9	111.7 (3)
N2—N1—Sn1	117.7 (2)	O2—C8—H8A	109.3
C7—N2—N1	109.2 (3)	C9—C8—H8A	109.3
C7—N2—Sn2	127.3 (2)	O2—C8—H8B	109.3
N1—N2—Sn2	123.4 (2)	C9—C8—H8B	109.3
C5—N3—C7	106.7 (3)	H8A—C8—H8B	108.0
C5—N3—C6	128.2 (3)	C8—C9—H9A	109.5
C7—N3—C6	125.1 (3)	C8—C9—H9B	109.5
Sn1—C1—H1A	109.5	H9A—C9—H9B	109.5
Sn1—C1—H1B	109.5	C8—C9—H9C	109.5

H1A—C1—H1B	109.5	H9A—C9—H9C	109.5
Sn1—C1—H1C	109.5	H9B—C9—H9C	109.5
C4—Sn2—O1—Sn1^{i}	88.55 (16)	O1^{i}—Sn1—N1—N2	−9.0 (3)
C3—Sn2—O1—Sn1^{i}	−86.31 (16)	O1—Sn1—N1—N2	−6.9 (2)
O2—Sn2—O1—Sn1^{i}	3.23 (10)	C1—Sn1—N1—N2	95.9 (3)
N2—Sn2—O1—Sn1^{i}	−178.81 (13)	C2—Sn1—N1—N2	−112.3 (3)
C4—Sn2—O1—Sn1	−96.9 (2)	O2^{i}—Sn1—N1—N2	174.7 (2)
C3—Sn2—O1—Sn1	88.2 (2)	C5—N1—N2—C7	−1.2 (4)
O2—Sn2—O1—Sn1	177.8 (2)	Sn1—N1—N2—C7	−176.1 (2)
N2—Sn2—O1—Sn1	−4.3 (2)	C5—N1—N2—Sn2	−177.7 (2)
O1^{i}—Sn1—O1—Sn2	−174.8 (3)	Sn1—N1—N2—Sn2	7.5 (3)
C1—Sn1—O1—Sn2	−70.2 (2)	O1—Sn2—N2—C7	−179.2 (3)
C2—Sn1—O1—Sn2	81.3 (2)	C4—Sn2—N2—C7	−61.3 (3)
O2^{i}—Sn1—O1—Sn2	−175.41 (15)	C3—Sn2—N2—C7	67.2 (3)
N1—Sn1—O1—Sn2	6.38 (19)	O2—Sn2—N2—C7	−174.1 (3)
O1^{i}—Sn1—O1—Sn1^{i}	0.0	O1—Sn2—N2—N1	−3.4 (3)
C1—Sn1—O1—Sn1^{i}	104.54 (13)	C4—Sn2—N2—N1	114.5 (3)
C2—Sn1—O1—Sn1^{i}	−103.98 (14)	C3—Sn2—N2—N1	−117.0 (3)
O2^{i}—Sn1—O1—Sn1^{i}	−0.7 (2)	O2—Sn2—N2—N1	1.7 (4)
N1—Sn1—O1—Sn1^{i}	−178.86 (13)	N2—N1—C5—N3	0.3 (4)
O1—Sn2—O2—C8	−175.8 (3)	Sn1—N1—C5—N3	173.6 (2)
C4—Sn2—O2—C8	65.7 (3)	C7—N3—C5—N1	0.7 (4)
C3—Sn2—O2—C8	−62.5 (3)	C6—N3—C5—N1	−178.0 (4)
N2—Sn2—O2—C8	178.9 (3)	N1—N2—C7—N3	1.7 (4)
O1—Sn2—O2—Sn1^{i}	−2.81 (9)	Sn2—N2—C7—N3	177.9 (2)
C4—Sn2—O2—Sn1^{i}	−121.28 (13)	N1—N2—C7—S1	−177.6 (3)
C3—Sn2—O2—Sn1^{i}	110.52 (13)	Sn2—N2—C7—S1	−1.3 (5)
N2—Sn2—O2—Sn1^{i}	−8.1 (3)	C5—N3—C7—N2	−1.5 (4)
O1^{i}—Sn1—N1—C5	178.2 (3)	C6—N3—C7—N2	177.3 (3)
O1—Sn1—N1—C5	−179.7 (4)	C5—N3—C7—S1	177.8 (3)
C1—Sn1—N1—C5	−76.9 (4)	C6—N3—C7—S1	−3.4 (5)
C2—Sn1—N1—C5	75.0 (4)	Sn2—O2—C8—C9	−112.7 (3)
O2^{i}—Sn1—N1—C5	1.9 (4)	Sn1^{i}—O2—C8—C9	76.5 (4)

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