Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

[4-Chloro-*N*′-(3-methoxy-2-oxidobenzylidene)benzohydrazidato]dimethyltin(IV)

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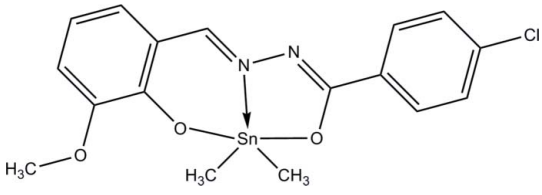
Received 15 November 2008; accepted 3 December 2008

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.006 Å; R factor = 0.029; wR factor = 0.080; data-to-parameter ratio = 14.1.

In the title molecule, $[Sn(CH_3)_2(C_{15}H_{11}ClN_2O_3)]$, the two benzene rings form a dihedral angle of 6.37 (2)°. The Sn atom is coordinated by one N [Sn—N = 2.187 (3) Å], two O [Sn—O = 2.123 (3) and 2.174 (3) Å] and two C [Sn—C = 2.096 (4) and 2.101 (4) Å] atoms in a distorted trigonal-bipyramidal geometry. The crystal packing exhibits weak intermolecular C—H···O hydrogen bonds, which link the molecules into centrosymmetric dimers with an Sn···Sn separation of 4.330 (6) Å, and π–π interactions [centroid–centroid distance of 3.690 (5) Å between the benzene rings of neighbouring molecules].

Related literature

For a related crystal structure, see Hong *et al.* (2006).



Experimental

Crystal data

$[Sn(CH_3)_2(C_{15}H_{11}ClN_2O_3)]$
M_r = 451.47
Monoclinic, $C2/c$
a = 30.015 (3) Å
b = 9.5039 (10) Å
c = 13.5615 (18) Å
β = 113.189 (2)°
V = 3556.0 (7) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 1.60 mm^{-1}
T = 298 (2) K
0.50 × 0.20 × 0.08 mm

Data collection

Siemens SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.501, T_{max} = 0.882
8568 measured reflections
3113 independent reflections
2392 reflections with $I > 2\sigma(I)$
R_{int} = 0.061

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.080
S = 1.00
3113 reflections
220 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.66 e Å^{-3}
$\Delta\rho_{min}$ = −0.57 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C15—H15B···O1^{i}	0.96	2.53	3.290 (6)	136

Symmetry code: (i) $-x, -y+2, -z$.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

We acknowledge the financial support of the Shandong Provincial Science Foundation and the State Key Laboratory of Crystalline Materials, Shandong University.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2482).

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supplementary materials

Acta Cryst. (2009). E**65**, m36 [**doi:10.1107/S1600536808040786**]

[4-Chloro-*N*'-(3-methoxy-2-oxidobenzylidene)benzohydrazidato]dimethyltin(IV)

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Comment

The molecular structure of the title compound, (I), is shown in Fig. 1. The Sn atom has distorted trigonal-bipyramidal coordination geometry, with atoms O1 and O2 in axial positions [O1—Sn1—O2 = 153.92 (11)°] and the atoms C16, C17 and N2 in equatorial positions. The sum of the equatorial angles C16—Sn1—C17, C16—Sn1—N2 and C17—Sn—N2 is 359.4 (1) °, indicating approximate coplanarity for these atoms. The Sn1—N2 bond length is 2.187 (3) Å close to the sum of the non-polar covalent radii 2.15 Å, indicating a strong Sn—N interaction. The Sn-O coordinating bond lengths are close to those in the reported compound $[Sn(C_6H_5)_2(C_{14}H_{10}N_2O_3)].C_2H_6O$ (Hong *et al.*, 2006). The crystal packing exhibits weak intermolecular C—H···O hydrogen bonds (Table 2), which link the molecules into centrosymmetric dimers with Sn···Sn separation of 4.330 (6) Å, and π–π interactions proved by short distance of 3.690 (5) between the centroids of benzene rings from the neighbouring molecules (Table 1).

Experimental

The reaction was carried out under nitrogen atmosphere. *o*-vanillin 4-chlorobenzhydrazone(1 mmol) and sodium ethoxide (1.2 mmol) were added to the solution of benzene(30 ml) in a Schlenk flask and stirred for 0.5 h. Dimethyltin dichloride (1 mmol) was then added to the reactor and the reaction mixture was stirred for 4 h at 313 K.The resulting clear solution was evaporated under vacuum. The product was crystallized from a mixture of dichloromethane/methanol (1:1). Analysis calculated (75%) for $C_{17}H_{17}ClN_2O_3Sn$ (Mr = 451.47): C, 45.22; H, 3.80; N, 6.20, found: C, 45.09; H, 3.76; N, 6.35.

Refinement

All H atoms attached to C atoms were fixed geometrically and treated as riding with, with aromatic C—H distances of 0.93 Å, methyl C—H distances of 0.96 Å. The U_{iso}(H) values were set at 1.5U_{iso}(C) for the methyl H atoms, and at 1.2U_{iso}(C) for the other H atoms.

Figures

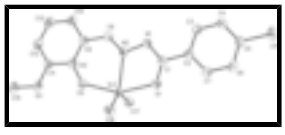


Fig. 1. The molecular structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme. H atoms have been omitted for clarity.

[4-Chloro-*N*'-(3-methoxy-2- oxidobenzylidene)benzohydrazidato]dimethyltin(IV)

Crystal data

$[Sn(CH_3)_2(C_{15}H_{11}ClN_2O_3)]$ F_{000} = 1792

M_r = 451.47	D_x = 1.687 Mg m^{-3}
Monoclinic, $C2/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 30.015 (3) Å	Cell parameters from 4052 reflections
b = 9.5039 (10) Å	θ = 2.3–28.1º
c = 13.5615 (18) Å	μ = 1.60 mm^{-1}
β = 113.189 (2)º	T = 298 (2) K
V = 3556.0 (7) Å^3	Block, orange
Z = 8	0.50 × 0.20 × 0.08 mm

Data collection

Siemens SMART CCD area-detector diffractometer	3113 independent reflections
Radiation source: fine-focus sealed tube	2392 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.061
T = 298(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 2.3º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −35→35
T_{min} = 0.501, T_{max} = 0.882	k = −10→11
8568 measured reflections	l = −8→16

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.029	H-atom parameters constrained
$wR(F^2)$ = 0.080	$w = 1/[\sigma^2(F_o^2) + (0.0351P)^2 + 0.1164P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.00	$(\Delta/\sigma)_{max}$ = 0.001
3113 reflections	$\Delta\rho_{max}$ = 0.66 e Å^{-3}
220 parameters	$\Delta\rho_{min}$ = −0.56 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Sn1	0.032667 (8)	0.79772 (3)	0.059696 (18)	0.03884 (12)
Cl1	0.23989 (4)	0.12286 (15)	0.38025 (11)	0.0824 (4)
N1	0.03655 (10)	0.5011 (3)	0.1544 (2)	0.0386 (7)
N2	0.00348 (9)	0.6097 (3)	0.1058 (2)	0.0343 (7)
O1	0.08954 (9)	0.6394 (3)	0.1128 (2)	0.0516 (7)
O2	−0.04042 (8)	0.8667 (3)	0.0035 (2)	0.0481 (7)
O3	−0.11949 (8)	1.0208 (3)	−0.0695 (2)	0.0593 (8)

C1	0.07925 (12)	0.5289 (4)	0.1536 (3)	0.0375 (9)
C2	0.11826 (12)	0.4249 (4)	0.2063 (3)	0.0373 (9)
C3	0.11002 (14)	0.3030 (4)	0.2523 (3)	0.0452 (10)
H3	0.0788	0.2837	0.2472	0.054*
C4	0.14663 (14)	0.2095 (4)	0.3054 (3)	0.0484 (10)
H4	0.1405	0.1282	0.3361	0.058*
C5	0.19244 (14)	0.2392 (5)	0.3119 (3)	0.0515 (11)
C6	0.20167 (14)	0.3569 (5)	0.2656 (4)	0.0706 (14)
H6	0.2328	0.3747	0.2696	0.085*
C7	0.16426 (14)	0.4496 (5)	0.2123 (4)	0.0603 (12)
H7	0.1704	0.5297	0.1803	0.072*
C8	−0.03937 (12)	0.5902 (4)	0.1040 (3)	0.0394 (9)
H8	−0.0445	0.5060	0.1330	0.047*
C9	−0.07999 (13)	0.6841 (4)	0.0622 (3)	0.0392 (9)
C10	−0.07884 (13)	0.8150 (4)	0.0152 (3)	0.0396 (9)
C11	−0.12244 (12)	0.8960 (4)	−0.0225 (3)	0.0446 (10)
C12	−0.16337 (14)	0.8486 (5)	−0.0114 (3)	0.0570 (12)
H12	−0.1911	0.9040	−0.0351	0.068*
C13	−0.16372 (15)	0.7186 (5)	0.0348 (4)	0.0655 (14)
H13	−0.1917	0.6872	0.0417	0.079*
C14	−0.12297 (13)	0.6366 (5)	0.0703 (3)	0.0529 (11)
H14	−0.1236	0.5488	0.1001	0.063*
C15	−0.16273 (14)	1.1007 (5)	−0.1172 (4)	0.0785 (16)
H15A	−0.1874	1.0434	−0.1683	0.118*
H15B	−0.1566	1.1810	−0.1529	0.118*
H15C	−0.1733	1.1315	−0.0626	0.118*
C16	0.03486 (14)	0.8041 (4)	−0.0928 (3)	0.0450 (10)
H16A	0.0310	0.8996	−0.1180	0.068*
H16B	0.0091	0.7475	−0.1416	0.068*
H16C	0.0654	0.7683	−0.0887	0.068*
C17	0.05918 (14)	0.9110 (4)	0.2044 (3)	0.0508 (11)
H17A	0.0896	0.8717	0.2515	0.076*
H17B	0.0363	0.9053	0.2378	0.076*
H17C	0.0637	1.0077	0.1901	0.076*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sn1	0.03741 (16)	0.0408 (2)	0.04022 (17)	0.00259 (12)	0.01728 (12)	0.00166 (12)
Cl1	0.0532 (6)	0.0705 (9)	0.1054 (10)	0.0202 (6)	0.0120 (7)	0.0138 (8)
N1	0.0381 (16)	0.0376 (19)	0.0420 (17)	0.0032 (14)	0.0179 (13)	0.0047 (15)
N2	0.0349 (15)	0.0350 (18)	0.0347 (15)	−0.0004 (14)	0.0156 (13)	−0.0008 (14)
O1	0.0453 (15)	0.0475 (18)	0.0701 (18)	0.0051 (13)	0.0315 (14)	0.0148 (16)
O2	0.0381 (13)	0.0485 (18)	0.0603 (16)	0.0043 (12)	0.0222 (13)	0.0120 (14)
O3	0.0347 (14)	0.0504 (19)	0.086 (2)	0.0068 (13)	0.0167 (14)	0.0133 (17)
C1	0.041 (2)	0.040 (2)	0.0346 (19)	0.0022 (17)	0.0176 (16)	−0.0034 (17)
C2	0.0383 (19)	0.036 (2)	0.040 (2)	−0.0003 (16)	0.0178 (16)	−0.0031 (17)
C3	0.042 (2)	0.045 (3)	0.048 (2)	−0.0001 (19)	0.0167 (18)	−0.002 (2)

C4	0.048 (2)	0.041 (3)	0.054 (2)	0.0014 (19)	0.0167 (19)	0.008 (2)
C5	0.043 (2)	0.048 (3)	0.055 (2)	0.008 (2)	0.0104 (19)	−0.002 (2)
C6	0.033 (2)	0.073 (3)	0.101 (4)	0.002 (2)	0.022 (2)	0.019 (3)
C7	0.047 (2)	0.055 (3)	0.082 (3)	0.001 (2)	0.029 (2)	0.018 (3)
C8	0.045 (2)	0.041 (2)	0.0353 (19)	−0.0024 (18)	0.0191 (16)	−0.0051 (17)
C9	0.0394 (19)	0.048 (3)	0.0317 (18)	0.0020 (18)	0.0152 (16)	−0.0009 (17)
C10	0.0358 (19)	0.047 (3)	0.0346 (19)	−0.0015 (17)	0.0128 (16)	−0.0051 (18)
C11	0.039 (2)	0.045 (3)	0.047 (2)	0.0023 (19)	0.0141 (17)	−0.001 (2)
C12	0.038 (2)	0.063 (3)	0.071 (3)	0.006 (2)	0.022 (2)	0.011 (3)
C13	0.039 (2)	0.087 (4)	0.076 (3)	0.002 (2)	0.029 (2)	0.019 (3)
C14	0.042 (2)	0.064 (3)	0.056 (2)	−0.001 (2)	0.0234 (19)	0.011 (2)
C15	0.041 (2)	0.067 (3)	0.113 (4)	0.011 (2)	0.014 (3)	0.023 (3)
C16	0.053 (2)	0.042 (2)	0.045 (2)	0.0016 (19)	0.0245 (18)	0.0022 (19)
C17	0.056 (2)	0.047 (3)	0.044 (2)	−0.005 (2)	0.0149 (19)	−0.002 (2)

Geometric parameters (Å, °)

Sn1—C16	2.096 (4)	C6—H6	0.9300
Sn1—C17	2.101 (4)	C7—H7	0.9300
Sn1—O2	2.123 (2)	C8—C9	1.436 (5)
Sn1—O1	2.174 (3)	C8—H8	0.9300
Sn1—N2	2.187 (3)	C9—C10	1.404 (5)
Cl1—C5	1.752 (4)	C9—C14	1.412 (5)
N1—C1	1.313 (4)	C10—C11	1.428 (5)
N1—N2	1.403 (4)	C11—C12	1.371 (5)
N2—C8	1.290 (4)	C12—C13	1.387 (6)
O1—C1	1.281 (4)	C12—H12	0.9300
O2—C10	1.319 (4)	C13—C14	1.368 (6)
O3—C11	1.365 (5)	C13—H13	0.9300
O3—C15	1.421 (5)	C14—H14	0.9300
C1—C2	1.483 (5)	C15—H15A	0.9600
C2—C7	1.371 (5)	C15—H15B	0.9600
C2—C3	1.383 (5)	C15—H15C	0.9600
C3—C4	1.376 (5)	C16—H16A	0.9600
C3—H3	0.9300	C16—H16B	0.9600
C4—C5	1.372 (6)	C16—H16C	0.9600
C4—H4	0.9300	C17—H17A	0.9600
C5—C6	1.364 (6)	C17—H17B	0.9600
C6—C7	1.386 (6)	C17—H17C	0.9600
Sn1···Sn1^{i}	4.3302 (6)	Cg1···Cg2^{ii}	3.690 (5)
C16—Sn1—C17	139.41 (15)	C14—C13—H13	119.9
C16—Sn1—O2	93.81 (13)	C12—C13—H13	119.9
C17—Sn1—O2	97.74 (13)	C13—C14—C9	120.5 (4)
C16—Sn1—O1	91.21 (13)	C13—C14—H14	119.7
C17—Sn1—O1	94.93 (13)	C9—C14—H14	119.7
O2—Sn1—O1	153.92 (11)	O3—C15—H15A	109.5
C16—Sn1—N2	118.54 (12)	O3—C15—H15B	109.5
C17—Sn1—N2	101.45 (14)	H15A—C15—H15B	109.5

O2—Sn1—N2	83.18 (10)	O3—C15—H15C	109.5
O1—Sn1—N2	71.99 (10)	H15A—C15—H15C	109.5
C16—Sn1—Sn1^{i}	77.92 (10)	H15B—C15—H15C	109.5
C17—Sn1—Sn1^{i}	81.00 (11)	Sn1—C16—H16A	109.5
O2—Sn1—Sn1^{i}	48.68 (7)	Sn1—C16—H16B	109.5
O1—Sn1—Sn1^{i}	156.79 (7)	H16A—C16—H16B	109.5
N2—Sn1—Sn1^{i}	131.22 (7)	Sn1—C16—H16C	109.5
C1—N1—N2	111.2 (3)	H16A—C16—H16C	109.5
C8—N2—N1	114.8 (3)	H16B—C16—H16C	109.5
C8—N2—Sn1	128.4 (3)	Sn1—C17—H17A	109.5
N1—N2—Sn1	116.5 (2)	Sn1—C17—H17B	109.5
C1—O1—Sn1	114.8 (2)	H17A—C17—H17B	109.5
C10—O2—Sn1	132.1 (2)	Sn1—C17—H17C	109.5
C11—O3—C15	117.4 (3)	H17A—C17—H17C	109.5
O1—C1—N1	124.8 (3)	H17B—C17—H17C	109.5
O1—C1—C2	118.4 (3)	C3—CG1—C5	119.9 (3)
N1—C1—C2	116.7 (3)	C3—CG1—C7	119.8 (2)
C7—C2—C3	118.1 (4)	C5—CG1—C7	120.3 (2)
C7—C2—C1	120.0 (4)	C3—CG1—C6	179.4 (3)
C3—C2—C1	121.9 (3)	C5—CG1—C6	59.6 (3)
C2—C3—C4	121.9 (4)	C7—CG1—C6	60.6 (2)
C2—C3—H3	119.0	C3—CG1—C4	60.1 (2)
C4—C3—H3	119.0	C5—CG1—C4	59.8 (2)
C5—C4—C3	118.3 (4)	C7—CG1—C4	179.6 (3)
C5—C4—H4	120.8	C6—CG1—C4	119.4 (3)
C3—C4—H4	120.8	C3—CG1—C2	60.4 (2)
C6—C5—C4	121.3 (4)	C5—CG1—C2	179.2 (2)
C6—C5—Cl1	119.4 (3)	C7—CG1—C2	59.5 (2)
C4—C5—Cl1	119.3 (4)	C6—CG1—C2	120.1 (3)
C5—C6—C7	119.5 (4)	C4—CG1—C2	120.5 (2)
C5—C6—H6	120.3	C3—CG1—CG2^{ii}	91.56 (16)
C7—C6—H6	120.3	C5—CG1—CG2^{ii}	102.81 (18)
C2—C7—C6	120.8 (4)	C7—CG1—CG2^{ii}	75.46 (19)
C2—C7—H7	119.6	C6—CG1—CG2^{ii}	88.2 (2)
C6—C7—H7	119.6	C4—CG1—CG2^{ii}	104.11 (17)
N2—C8—C9	127.1 (4)	C2—CG1—CG2^{ii}	77.95 (14)
N2—C8—H8	116.4	C11—CG2—C12	59.4 (2)
C9—C8—H8	116.4	C11—CG2—C14	178.1 (2)
C10—C9—C14	120.4 (3)	C12—CG2—C14	118.9 (2)
C10—C9—C8	124.2 (4)	C11—CG2—C13	119.3 (2)
C14—C9—C8	115.5 (4)	C12—CG2—C13	59.9 (2)
O2—C10—C9	124.3 (3)	C14—CG2—C13	59.0 (2)
O2—C10—C11	118.5 (4)	C11—CG2—C9	120.8 (2)
C9—C10—C11	117.2 (3)	C12—CG2—C9	179.5 (2)
O3—C11—C12	124.2 (3)	C14—CG2—C9	61.0 (2)
O3—C11—C10	114.6 (3)	C13—CG2—C9	120.0 (2)
C12—C11—C10	121.2 (4)	C11—CG2—C10	61.1 (2)

C11—C12—C13	120.6 (4)	C12—CG2—C10	120.4 (2)
C11—C12—H12	119.7	C14—CG2—C10	120.6 (2)
C13—C12—H12	119.7	C13—CG2—C10	179.6 (3)
C14—C13—C12	120.1 (4)	C9—CG2—C10	59.7 (2)

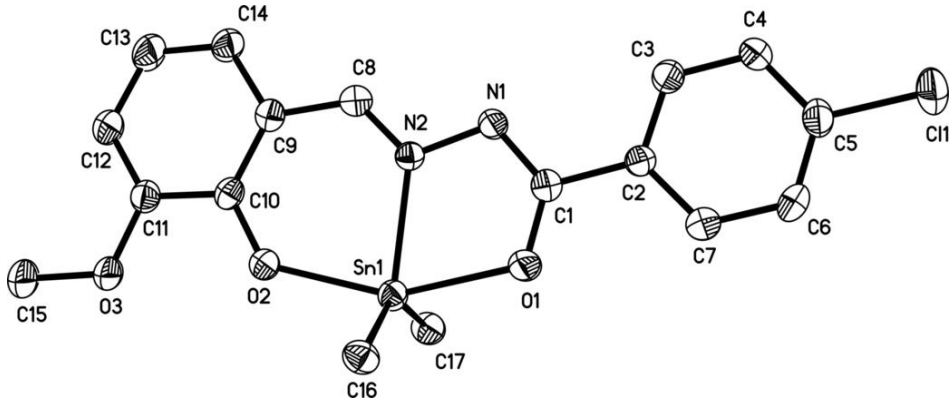
Symmetry codes: (i) −*x*, −*y*+2, −*z*; (ii) −*x*, −*y*+1, −*z*.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C15—H15B···O1^{i}	0.96	2.53	3.290 (6)	136

Symmetry codes: (i) −*x*, −*y*+2, −*z*.

Fig. 1


C14
C13
C8
N1
C4
C3
N2
C9
C5
Cl1
C12
C10
C1
C2
C11
C6
C7
Sn1
O2
O1
C15
O3
C17
C16