

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

Bis(acetohydroxamato- κ^2O,O')diphenyltin(IV)

Caihong Yue, Qingkun Wu and Handong Yin*

College of Chemistry and Chemical Engineering, Liaocheng University, Shandong 252059, People's Republic of China

Correspondence e-mail: handongyin@163.com

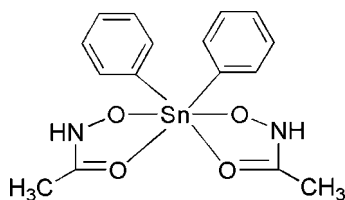
Received 10 June 2011; accepted 29 June 2011

Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(C-C) = 0.008$ Å; R factor = 0.030; wR factor = 0.077; data-to-parameter ratio = 14.5.

The complex molecule of the title compound, $[\text{Sn}(\text{C}_6\text{H}_5)_2(\text{C}_2\text{H}_4\text{NO}_2)_2]$, has crystallographically imposed twofold symmetry. The Sn atom is coordinated by four O atoms from two acetohydroxamate ligands and by two C atoms from phenyl groups in a distorted octahedral geometry. In the crystal, molecules are connected by $\text{N}-\text{H}\cdots\text{O}$ hydrogen-bonding interactions, forming a chain structure along the c axis

Related literature

For the biological activity of diorganotin(IV) complexes with hydroxamates, see: Shang *et al.* (2007). For a related structure, see: Harrison *et al.* (1976). For van der Waals radii, see: Bondi (1964).



Experimental

Crystal data

 $[\text{Sn}(\text{C}_6\text{H}_5)_2(\text{C}_2\text{H}_4\text{NO}_2)_2]$ $M_r = 421.01$

Monoclinic, $C2/c$
 $a = 18.7713$ (17) Å
 $b = 10.2683$ (8) Å
 $c = 9.8326$ (6) Å
 $\beta = 112.842$ (1)°
 $V = 1746.6$ (2) Å³

$Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 1.48$ mm⁻¹
 $T = 298$ K
 $0.38 \times 0.33 \times 0.19$ mm

Data collection

Siemens SMART CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.603$, $T_{\max} = 0.766$

4295 measured reflections
 1542 independent reflections
 1367 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.048$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.030$
 $wR(F^2) = 0.077$
 $S = 1.00$
 1542 reflections

106 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.72$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.94$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1}\cdots\text{O1}^i$	0.86	2.03	2.847 (4)	159

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

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 National Natural Science Foundation of China (20771053), the National Basic Research Program (No. 2010CB234601) and the Natural Science Foundation of Shandong Province (Y2008B48) for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2612).

References

- Bondi, A. (1964). *J. Phys. Chem.* **68**, 441–443.
 Harrison, P. G., King, T. J. & Phillips, R. C. (1976). *J. Chem. Soc. Dalton Trans.* pp. 2317–2321.
 Shang, X., Wu, J., Pombeiro, A. J. L. & Li, Q. (2007). *Appl. Organomet. Chem.* **21**, 919–925.
 Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Siemens (1996). *SMART and SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

supporting information

Acta Cryst. (2011). E67, m1034 [doi:10.1107/S160053681102558X]

Bis(acetohydroxamato- κ^2O,O')diphenyltin(IV)

Caihong Yue, Qingkun Wu and Handong Yin

S1. Comment

Among many multidentate organic ligands, hydroxamic acids are of particular importance, because of their remarkable structural diversity and biological applications (Shang *et al.*, 2007). As a continuation of our interest in this area, we have synthesized the title compound and report its crystal structure herein.

The molecular structure of the compound is depicted in Fig. 1. The complex molecule has crystallographically imposed two-fold symmetry. The tin atom is six-coordinated in a distorted octahedral geometry. The Sn–O bond distances (Sn1–O2 = 2.103 (2) Å; Sn1–O = 2.275 (3) Å) are close to the sum of the covalent radii (2.13 Å; Bondi, 1964), and the Sn–C distance (Sn1–C3 = 2.147 (4) Å) are in the range observed in a related compound (2.14–2.18 Å; Harrison *et al.*, 1976). In the crystal packing, the molecules are linked by N—H \cdots O hydrogen bonds (Table 1) into a one-dimensional chains parallel to the *c* axis (Fig. 2).

S2. Experimental

The reaction was carried out under nitrogen atmosphere. Acetohydroxamic acid (0.4 mmol) and KOH (0.4 mmol) in methanol (30 ml) were added to a Schlenk flask and stirred for 30 min. Diphenyltin dichloride (0.2 mmol) was then added to the reactor. The reaction mixture was stirred for 8 h at room temperature and then filtrated. The filtrate was evaporated *in vacuo* to dryness. The obtained solid was recrystallized from a dichloromethane-petroleum ether (3:1 v/v) solution (yield 86%; m. p. 431 K). Anal. Calcd (%) for C₁₆H₁₈N₂O₄Sn (Mr = 421.01): C, 45.64; H, 4.31; N, 6.65; O, 15.20. Found (%): C, 45.60; H, 4.25; N, 6.62; O, 15.16.

S3. Refinement

All H atoms were positioned with idealized geometry (C—H = 0.83–0.96 Å; N—H = 0.86 Å) and were refined isotropically with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C}, \text{N})$ or $1.5 U_{\text{eq}}(\text{C})$ for methyl H atoms.

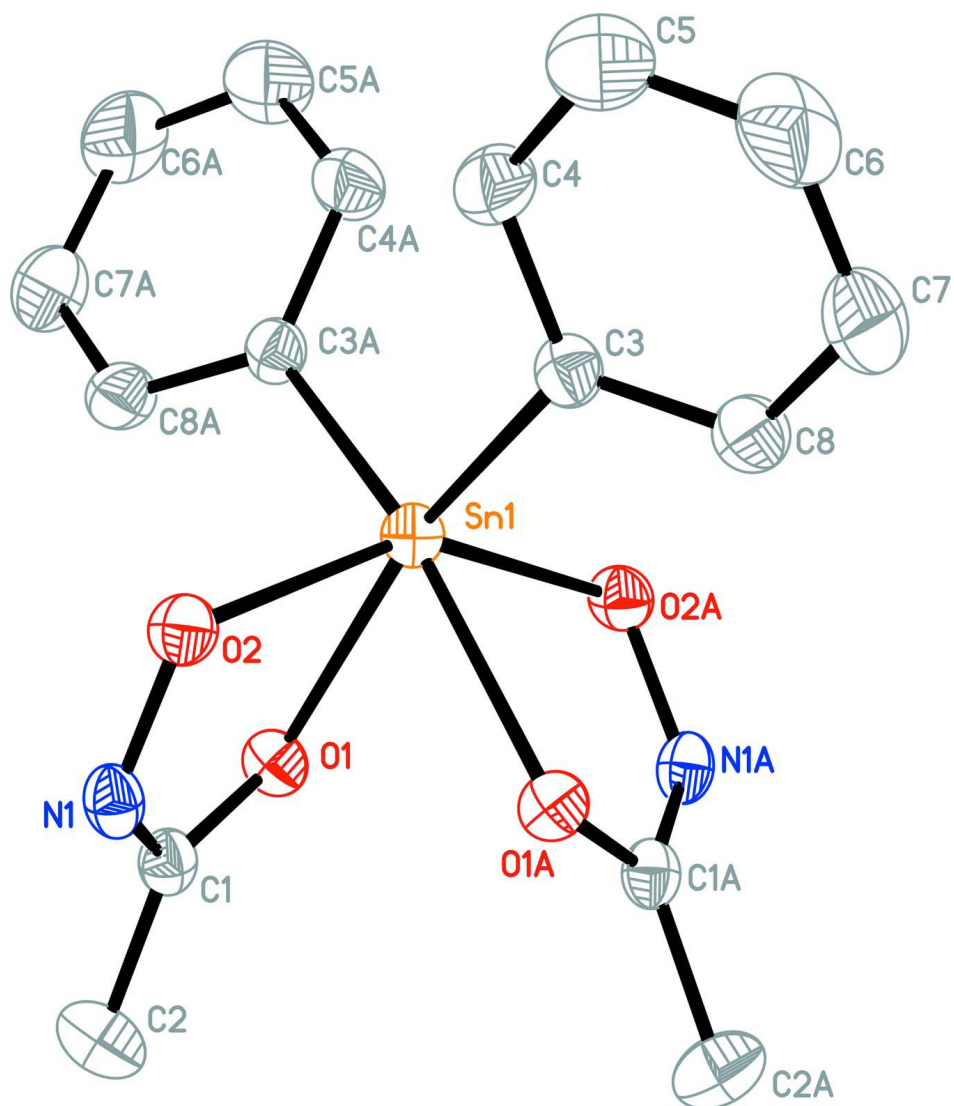


Figure 1

The molecular structure of the compound, showing 50% probability displacement ellipsoids. H atoms are omitted for clarity. Symmetry code: (A) = $-x, y, -z + 1/2$.

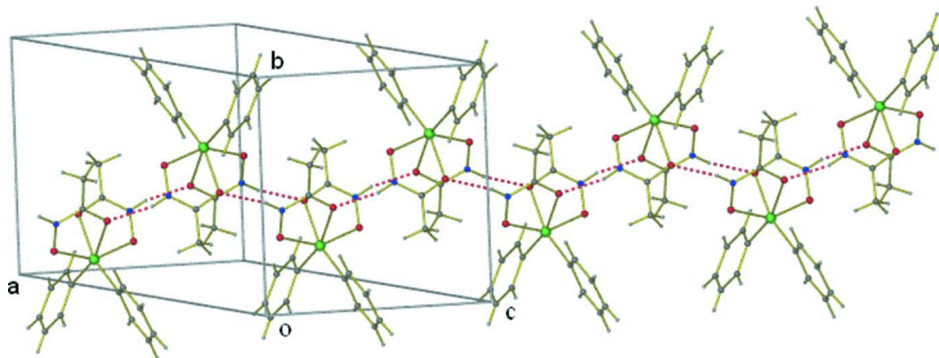


Figure 2

View of the one-dimensional linear chain structure in the title compound.

Bis(acetohydroxamato- κ^2O,O')diphenyltin(IV)*Crystal data*[Sn(C₆H₅)₂(C₂H₄NO₂)₂] $M_r = 421.01$ Monoclinic, $C2/c$ Hall symbol: $-C\ 2yc$ $a = 18.7713\ (17)\ \text{\AA}$ $b = 10.2683\ (8)\ \text{\AA}$ $c = 9.8326\ (6)\ \text{\AA}$ $\beta = 112.842\ (1)^\circ$ $V = 1746.6\ (2)\ \text{\AA}^3$ $Z = 4$ $F(000) = 840$ $D_x = 1.601\ \text{Mg m}^{-3}$ Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 2602 reflections

 $\theta = 2.3\text{--}26.2^\circ$ $\mu = 1.48\ \text{mm}^{-1}$ $T = 298\ \text{K}$

Block, colourless

 $0.38 \times 0.33 \times 0.19\ \text{mm}$ *Data collection*Siemens SMART CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

phi and ω scans

Absorption correction: multi-scan

(SADABS; Sheldrick, 1996)

 $T_{\min} = 0.603$, $T_{\max} = 0.766$

4295 measured reflections

1542 independent reflections

1367 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.048$ $\theta_{\max} = 25.0^\circ$, $\theta_{\min} = 2.3^\circ$ $h = -22 \rightarrow 14$ $k = -12 \rightarrow 11$ $l = -11 \rightarrow 11$ *Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.030$ $wR(F^2) = 0.077$ $S = 1.00$

1542 reflections

106 parameters

0 restraints

Primary atom site location: structure-invariant

direct methods

Secondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0322P)^2 + 6.1631P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.72\ \text{e \AA}^{-3}$ $\Delta\rho_{\min} = -0.94\ \text{e \AA}^{-3}$ *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Sn1	0.0000	0.27983 (4)	0.2500	0.03426 (15)
O1	-0.07784 (14)	0.4550 (3)	0.2287 (3)	0.0377 (6)
O2	-0.04374 (15)	0.3345 (3)	0.0264 (3)	0.0399 (6)
N1	-0.07653 (16)	0.4552 (3)	0.0036 (3)	0.0371 (7)
H1	-0.0874	0.4929	-0.0802	0.045*
C1	-0.0919 (2)	0.5148 (4)	0.1067 (4)	0.0356 (8)
C2	-0.1242 (3)	0.6484 (5)	0.0813 (5)	0.0599 (12)
H2A	-0.1762	0.6467	0.0766	0.090*
H2B	-0.1241	0.6812	-0.0101	0.090*
H2C	-0.0932	0.7039	0.1608	0.090*

C3	0.0918 (2)	0.1575 (4)	0.2454 (4)	0.0393 (9)
C4	0.0752 (3)	0.0355 (5)	0.1751 (5)	0.0591 (12)
H4	0.0244	0.0061	0.1339	0.071*
C5	0.1343 (4)	-0.0411 (6)	0.1669 (7)	0.0833 (19)
H5	0.1228	-0.1216	0.1200	0.100*
C6	0.2094 (3)	0.0010 (7)	0.2273 (7)	0.0827 (18)
H6	0.2485	-0.0494	0.2182	0.099*
C7	0.2269 (3)	0.1177 (6)	0.3013 (6)	0.0710 (15)
H7	0.2781	0.1450	0.3452	0.085*
C8	0.1685 (2)	0.1946 (5)	0.3103 (5)	0.0506 (11)
H8	0.1811	0.2731	0.3612	0.061*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sn1	0.0343 (2)	0.0369 (2)	0.0340 (2)	0.000	0.01590 (15)	0.000
O1	0.0429 (14)	0.0422 (16)	0.0321 (13)	0.0038 (12)	0.0190 (11)	0.0018 (11)
O2	0.0462 (15)	0.0414 (16)	0.0324 (13)	-0.0006 (13)	0.0158 (11)	-0.0028 (12)
N1	0.0322 (16)	0.049 (2)	0.0299 (16)	-0.0002 (14)	0.0117 (13)	0.0062 (14)
C1	0.0300 (18)	0.043 (2)	0.0332 (19)	-0.0028 (16)	0.0122 (15)	0.0030 (16)
C2	0.073 (3)	0.055 (3)	0.061 (3)	0.019 (3)	0.036 (2)	0.015 (2)
C3	0.040 (2)	0.043 (2)	0.0341 (19)	0.0034 (18)	0.0137 (16)	-0.0027 (17)
C4	0.057 (3)	0.051 (3)	0.061 (3)	0.003 (2)	0.014 (2)	-0.017 (2)
C5	0.089 (4)	0.069 (4)	0.087 (4)	0.020 (3)	0.029 (3)	-0.031 (3)
C6	0.068 (4)	0.091 (5)	0.091 (4)	0.033 (3)	0.034 (3)	-0.012 (4)
C7	0.043 (3)	0.080 (4)	0.087 (4)	0.014 (3)	0.022 (2)	0.002 (3)
C8	0.042 (2)	0.046 (3)	0.063 (3)	0.0045 (19)	0.019 (2)	-0.003 (2)

Geometric parameters (Å, °)

Sn1—O2 ⁱ	2.103 (2)	C2—H2C	0.9600
Sn1—O2	2.103 (2)	C3—C8	1.383 (6)
Sn1—C3 ⁱ	2.147 (4)	C3—C4	1.406 (6)
Sn1—C3	2.147 (4)	C4—C5	1.388 (7)
Sn1—O1 ⁱ	2.275 (3)	C4—H4	0.9300
Sn1—O1	2.275 (3)	C5—C6	1.371 (9)
O1—C1	1.281 (4)	C5—H5	0.9300
O2—N1	1.363 (4)	C6—C7	1.374 (8)
N1—C1	1.308 (5)	C6—H6	0.9300
N1—H1	0.8600	C7—C8	1.381 (7)
C1—C2	1.482 (6)	C7—H7	0.9300
C2—H2A	0.9600	C8—H8	0.9300
C2—H2B	0.9600		
O2 ⁱ —Sn1—O2	149.02 (16)	C1—C2—H2B	109.5
O2 ⁱ —Sn1—C3 ⁱ	97.18 (12)	H2A—C2—H2B	109.5
O2—Sn1—C3 ⁱ	100.81 (12)	C1—C2—H2C	109.5
O2 ⁱ —Sn1—C3	100.81 (12)	H2A—C2—H2C	109.5

O2—Sn1—C3	97.18 (12)	H2B—C2—H2C	109.5
C3 ⁱ —Sn1—C3	108.4 (2)	C8—C3—C4	117.7 (4)
O2 ⁱ —Sn1—O1 ⁱ	73.53 (10)	C8—C3—Sn1	122.0 (3)
O2—Sn1—O1 ⁱ	82.02 (10)	C4—C3—Sn1	120.3 (3)
C3 ⁱ —Sn1—O1 ⁱ	162.29 (13)	C5—C4—C3	120.2 (5)
C3—Sn1—O1 ⁱ	88.41 (13)	C5—C4—H4	119.9
O2 ⁱ —Sn1—O1	82.02 (10)	C3—C4—H4	119.9
O2—Sn1—O1	73.53 (10)	C6—C5—C4	120.5 (5)
C3 ⁱ —Sn1—O1	88.41 (13)	C6—C5—H5	119.7
C3—Sn1—O1	162.29 (13)	C4—C5—H5	119.7
O1 ⁱ —Sn1—O1	75.54 (13)	C5—C6—C7	119.9 (5)
C1—O1—Sn1	110.9 (2)	C5—C6—H6	120.1
N1—O2—Sn1	112.5 (2)	C7—C6—H6	120.1
C1—N1—O2	121.3 (3)	C6—C7—C8	120.0 (5)
C1—N1—H1	119.4	C6—C7—H7	120.0
O2—N1—H1	119.4	C8—C7—H7	120.0
O1—C1—N1	118.3 (4)	C7—C8—C3	121.5 (5)
O1—C1—C2	121.6 (4)	C7—C8—H8	119.2
N1—C1—C2	120.1 (3)	C3—C8—H8	119.2
C1—C2—H2A	109.5		
O2 ⁱ —Sn1—O1—C1	144.7 (2)	C3 ⁱ —Sn1—C3—C8	-148.3 (4)
O2—Sn1—O1—C1	-16.0 (2)	O1 ⁱ —Sn1—C3—C8	26.0 (4)
C3 ⁱ —Sn1—O1—C1	-117.8 (3)	O1—Sn1—C3—C8	50.8 (6)
C3—Sn1—O1—C1	44.1 (5)	O2 ⁱ —Sn1—C3—C4	133.1 (4)
O1 ⁱ —Sn1—O1—C1	69.8 (2)	O2—Sn1—C3—C4	-72.2 (4)
O2 ⁱ —Sn1—O2—N1	-24.99 (19)	C3 ⁱ —Sn1—C3—C4	31.7 (3)
C3 ⁱ —Sn1—O2—N1	99.4 (2)	O1 ⁱ —Sn1—C3—C4	-154.0 (4)
C3—Sn1—O2—N1	-150.2 (2)	O1—Sn1—C3—C4	-129.2 (4)
O1 ⁱ —Sn1—O2—N1	-62.9 (2)	C8—C3—C4—C5	-2.7 (7)
O1—Sn1—O2—N1	14.3 (2)	Sn1—C3—C4—C5	177.3 (4)
Sn1—O2—N1—C1	-12.8 (4)	C3—C4—C5—C6	0.1 (9)
Sn1—O1—C1—N1	15.1 (4)	C4—C5—C6—C7	2.3 (10)
Sn1—O1—C1—C2	-164.6 (3)	C5—C6—C7—C8	-2.2 (10)
O2—N1—C1—O1	-2.5 (5)	C6—C7—C8—C3	-0.4 (9)
O2—N1—C1—C2	177.2 (3)	C4—C3—C8—C7	2.8 (7)
O2 ⁱ —Sn1—C3—C8	-46.9 (4)	Sn1—C3—C8—C7	-177.2 (4)
O2—Sn1—C3—C8	107.8 (4)		

Symmetry code: (i) $-x, y, -z+1/2$.*Hydrogen-bond geometry* (\AA , $^\circ$)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1 \cdots O1 ⁱⁱ	0.86	2.03	2.847 (4)	159

Symmetry code: (ii) $x, -y+1, z-1/2$.