

Bis(μ -2-pyridine-2,6-dicarboxylato)bis-[aquadimethyltin(IV)]

 Mingqing Yang,^a Rufen Zhang^{a*} and Yuying Sun^b
^aDepartment of Chemistry, Liaocheng University, Liaocheng 252059, People's Republic of China, and ^bAnalytical and Testing Center of Beihua University, Jilin 132031, People's Republic of China

Correspondence e-mail: macl@lcu.edu.cn

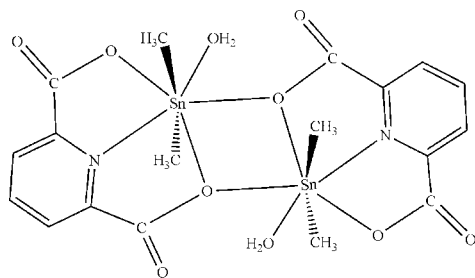
Received 27 October 2007; accepted 31 October 2007

 Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.009$ Å; R factor = 0.046; wR factor = 0.123; data-to-parameter ratio = 13.6.

The title dinuclear complex, $[\text{Sn}_2(\text{CH}_3)_4(\text{C}_7\text{H}_3\text{NO}_4)_2(\text{H}_2\text{O})_2]$, lies on a crystallographic inversion center. The unique Sn^{IV} atom is coordinated in a slightly distorted pentagonal-bipyramidal geometry. The two symmetry-related Sn^{IV} atoms are connected *via* two asymmetric Sn—O—Sn bridges (Sn—O = 2.473 and 2.634 Å), such that an exactly planar Sn—O—Sn—O ring is formed.

Related literature

For related literature, see: Aizawa *et al.* (1996); Dubey & Roy (2003); Ma *et al.* (2005).



Experimental

Crystal data

$[\text{Sn}_2(\text{CH}_3)_4(\text{C}_7\text{H}_3\text{NO}_4)_2(\text{H}_2\text{O})_2]$	$V = 2272.9$ (16) Å ³
$M_r = 663.76$	$Z = 4$
Monoclinic, $C2/c$	Mo $K\alpha$ radiation
$a = 11.319$ (5) Å	$\mu = 2.25$ mm ⁻¹
$b = 10.705$ (4) Å	$T = 298$ (2) K
$c = 18.768$ (8) Å	$0.23 \times 0.17 \times 0.14$ mm
$\beta = 91.864$ (5)°	

Data collection

Bruker SMART CCD area detector diffractometer	5571 measured reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	1998 independent reflections
$T_{\text{min}} = 0.625$, $T_{\text{max}} = 0.743$	1645 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.059$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.046$	4 restraints
$wR(F^2) = 0.123$	H-atom parameters constrained
$S = 1.00$	$\Delta\rho_{\text{max}} = 0.99$ e Å ⁻³
1998 reflections	$\Delta\rho_{\text{min}} = -1.94$ e Å ⁻³
147 parameters	

Data collection: SMART (Bruker, 1996); cell refinement: SAINT (Bruker, 1996); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997a); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997a); molecular graphics: SHELXTL (Sheldrick, 1997b); software used to prepare material for publication: SHELXTL.

The authors thank the National Natural Science Foundation of China (20271025) for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2542).

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supplementary materials

Acta Cryst. (2007). E63, m2924 [doi:10.1107/S1600536807054980]

Bis(μ -2-pyridine-2,6-dicarboxylato)bis[aquadimethyltin(IV)]

M. Yang, R. Zhang and Y. Sun

Comment

In recent years, organotin complexes have been attracting more and more attention due to their wide industrial applications and biological activities (Dubey & Roy, 2003). Organotin(IV) dicarboxylates have been studied in considerable detail, and in general the reported organotin dicarboxylates exist as dinuclear, one-dimensional zigzag chain and cyclic structures (Ma *et al.*, 2005). Herein we report the structure of the title complex (Fig. 1). For the coordination of the Sn^{IV} atom, two C atoms of the methyl groups are in the apical positions [C—Sn—C = 167.1 (3)°], and four O atoms and one N atom are in the equatorial positions giving a slightly distorted pentagonal-bipyramidal environment. The Sn—O, Sn—N bond distances are comparable to those reported in the literature (Aizawa *et al.*, 1996). The longest Sn—O distance is 2.634 (5) Å for Sn—Oⁱ [symmetry code: (i) 1 - x, -y, 1 - z].

Experimental

The reaction was carried out under N₂ atmosphere. 2,6-pyridinedicarboxylic acid (0.167 g, 1 mmol) was added to a solution of benzene (30 ml) with sodium ethoxide (0.136 g, 2 mmol) in a Schlenk flask. After stirring for 10 min, dimethyltin dichloride (0.220 g, 1 mmol) was added to the mixture. The mixture was kept at 313 K for 12 h. After cooling to room temperature, the solution was filtered. The solvent of the filtrate was gradually removed by evaporation under vacuum until a solid product was obtained. The solid was then recrystallized from diethyl ether. Colorless single crystals of the title complex were obtained after one week. Yield, 81%. Analysis calculated for C₁₈H₂₂N₂O₁₀Sn₂: C 32.57, H 3.34, N, 4.22; found: C 32.36, H 3.22, N, 4.31. The elemental analyses were performed with PERKIN ELMER MODEL 2400 SERIES II.

Refinement

All H atoms were placed geometrically idealized positions and treated as riding on their parent atoms, with aromatic C—H distances of 0.93 Å and methyl C—H distances of 0.96 Å. The $U_{\text{iso}}(\text{H})$ values were set at 1.2 $U_{\text{eq}}(\text{C})$ for the aromatic H atoms and 1.5 $U_{\text{eq}}(\text{C})$ for methyl H atoms.

Figures

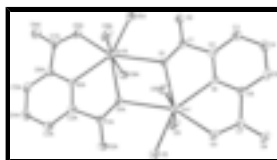


Fig. 1. The molecular structure of the title complex, showing 30% probability displacement ellipsoids and the atom-numbering scheme for non-H atoms [symmetry code: (A) 1 - x, -y, 1 - z].

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Crystal data

$[\text{Sn}_2(\text{CH}_3)_4(\text{C}_7\text{H}_3\text{NO}_4)_2(\text{H}_2\text{O})_2]$	$F_{000} = 1296$
$M_r = 663.76$	$D_x = 1.940 \text{ Mg m}^{-3}$
Monoclinic, $C2/c$	Mo $K\alpha$ radiation
Hall symbol: $-C 2yc$	$\lambda = 0.71073 \text{ \AA}$
$a = 11.319 (5) \text{ \AA}$	Cell parameters from 2820 reflections
$b = 10.705 (4) \text{ \AA}$	$\theta = 2.6\text{--}28.2^\circ$
$c = 18.768 (8) \text{ \AA}$	$\mu = 2.25 \text{ mm}^{-1}$
$\beta = 91.864 (5)^\circ$	$T = 298 (2) \text{ K}$
$V = 2272.9 (16) \text{ \AA}^3$	Block, colorless
$Z = 4$	$0.23 \times 0.17 \times 0.14 \text{ mm}$

Data collection

Bruker SMART CCD area detector diffractometer	1998 independent reflections
Radiation source: fine-focus sealed tube	1645 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.059$
$T = 298(2) \text{ K}$	$\theta_{\text{max}} = 25.0^\circ$
φ and ω scans	$\theta_{\text{min}} = 2.2^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -13 \rightarrow 13$
$T_{\text{min}} = 0.625$, $T_{\text{max}} = 0.743$	$k = -12 \rightarrow 12$
5571 measured reflections	$l = -19 \rightarrow 22$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.046$	H-atom parameters constrained
$wR(F^2) = 0.123$	$w = 1/[\sigma^2(F_o^2) + (0.077P)^2]$
$S = 1.00$	where $P = (F_o^2 + 2F_c^2)/3$
1998 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
147 parameters	$\Delta\rho_{\text{max}} = 0.99 \text{ e \AA}^{-3}$
4 restraints	$\Delta\rho_{\text{min}} = -1.93 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

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	0.39712 (4)	0.01350 (4)	0.59215 (2)	0.0274 (2)
N1	0.4229 (4)	0.2188 (4)	0.6236 (3)	0.0236 (11)
O1	0.5332 (4)	0.1262 (4)	0.5141 (2)	0.0270 (10)
O2	0.5885 (5)	0.3132 (4)	0.4773 (3)	0.0440 (13)
O3	0.2964 (5)	0.0520 (5)	0.6883 (3)	0.0435 (12)
O4	0.2345 (6)	0.1879 (5)	0.7685 (3)	0.0642 (18)
O5	0.2951 (4)	-0.1711 (4)	0.6116 (3)	0.0415 (12)
H1	0.2831	-0.1981	0.6534	0.062*
H2	0.3360	-0.2263	0.5911	0.062*
C1	0.5411 (5)	0.2409 (6)	0.5194 (3)	0.0261 (14)
C2	0.4850 (5)	0.2990 (6)	0.5842 (3)	0.0255 (14)
C3	0.4904 (5)	0.4248 (6)	0.6008 (4)	0.0329 (15)
H3	0.5341	0.4793	0.5735	0.039*
C4	0.4294 (7)	0.4685 (7)	0.6589 (4)	0.0393 (17)
H4	0.4324	0.5527	0.6711	0.047*
C5	0.3639 (6)	0.3850 (6)	0.6986 (4)	0.0315 (15)
H5A	0.3221	0.4127	0.7374	0.038*
C6	0.3620 (5)	0.2609 (6)	0.6794 (3)	0.0290 (14)
C7	0.2929 (6)	0.1611 (6)	0.7156 (4)	0.0373 (17)
C8	0.2604 (5)	0.0637 (7)	0.5183 (4)	0.0348 (16)
H8A	0.2927	0.1119	0.4804	0.052*
H8B	0.2022	0.1124	0.5419	0.052*
H8C	0.2241	-0.0105	0.4989	0.052*
C9	0.5481 (6)	-0.0590 (8)	0.6463 (4)	0.0439 (18)
H9A	0.5806	-0.1257	0.6188	0.066*
H9B	0.5265	-0.0904	0.6920	0.066*
H9C	0.6060	0.0059	0.6527	0.066*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sn1	0.0322 (3)	0.0288 (3)	0.0212 (3)	-0.00140 (17)	0.0044 (2)	-0.00003 (18)

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N1	0.026 (3)	0.029 (3)	0.016 (3)	0.000 (2)	0.003 (2)	0.000 (2)
O1	0.034 (2)	0.029 (3)	0.018 (2)	-0.0059 (18)	0.0012 (19)	-0.0028 (18)
O2	0.063 (3)	0.032 (3)	0.038 (3)	-0.013 (2)	0.027 (3)	-0.005 (2)
O3	0.062 (3)	0.039 (3)	0.030 (3)	-0.009 (3)	0.023 (2)	-0.004 (2)
O4	0.104 (5)	0.049 (3)	0.043 (4)	-0.015 (3)	0.048 (3)	-0.011 (3)
O5	0.062 (3)	0.030 (3)	0.034 (3)	-0.001 (2)	0.019 (2)	0.001 (2)
C1	0.028 (3)	0.028 (4)	0.022 (3)	-0.004 (3)	0.001 (3)	0.001 (3)
C2	0.020 (3)	0.034 (4)	0.022 (4)	0.000 (2)	0.000 (3)	0.000 (3)
C3	0.031 (3)	0.034 (4)	0.034 (4)	-0.006 (3)	0.007 (3)	-0.001 (3)
C4	0.048 (4)	0.031 (4)	0.039 (5)	-0.002 (3)	0.007 (4)	-0.009 (3)
C5	0.032 (4)	0.038 (4)	0.025 (4)	0.004 (3)	0.003 (3)	-0.007 (3)
C6	0.032 (3)	0.033 (4)	0.022 (3)	-0.003 (3)	0.002 (3)	0.000 (3)
C7	0.050 (4)	0.032 (4)	0.031 (4)	-0.004 (3)	0.011 (3)	-0.006 (3)
C8	0.030 (3)	0.036 (4)	0.039 (4)	-0.004 (3)	0.003 (3)	0.006 (3)
C9	0.045 (4)	0.064 (5)	0.023 (4)	0.007 (4)	-0.005 (3)	0.002 (4)

Geometric parameters (Å, °)

Sn1—C9	2.108 (7)	C1—C2	1.523 (8)
Sn1—C8	2.114 (6)	C2—C3	1.383 (9)
Sn1—O3	2.204 (5)	C3—C4	1.390 (10)
Sn1—N1	2.292 (5)	C3—H3	0.9300
Sn1—O5	2.324 (5)	C4—C5	1.394 (10)
Sn1—O1	2.473 (4)	C4—H4	0.9300
Sn1—O1 ⁱ	2.634 (5)	C5—C6	1.376 (9)
N1—C2	1.347 (7)	C5—H5A	0.9300
N1—C6	1.350 (8)	C6—C7	1.499 (9)
O1—C1	1.235 (7)	C8—H8A	0.9600
O2—C1	1.241 (7)	C8—H8B	0.9600
O3—C7	1.277 (8)	C8—H8C	0.9600
O4—C7	1.245 (8)	C9—H9A	0.9600
O5—H1	0.8500	C9—H9B	0.9600
O5—H2	0.8500	C9—H9C	0.9600
C9—Sn1—C8	167.1 (3)	C3—C2—C1	124.2 (6)
C9—Sn1—O3	96.1 (2)	C2—C3—C4	119.1 (6)
C8—Sn1—O3	95.9 (2)	C2—C3—H3	120.5
C9—Sn1—N1	97.7 (3)	C4—C3—H3	120.5
C8—Sn1—N1	90.6 (2)	C3—C4—C5	119.3 (6)
O3—Sn1—N1	71.02 (17)	C3—C4—H4	120.3
C9—Sn1—O5	90.6 (3)	C5—C4—H4	120.3
C8—Sn1—O5	87.8 (2)	C6—C5—C4	118.9 (6)
O3—Sn1—O5	75.95 (17)	C6—C5—H5A	120.6
N1—Sn1—O5	146.58 (17)	C4—C5—H5A	120.6
C9—Sn1—O1	87.4 (2)	N1—C6—C5	121.4 (6)
C8—Sn1—O1	86.8 (2)	N1—C6—C7	113.5 (6)
O3—Sn1—O1	137.91 (16)	C5—C6—C7	125.0 (6)
N1—Sn1—O1	66.95 (15)	O4—C7—O3	123.7 (6)
O5—Sn1—O1	146.11 (15)	O4—C7—C6	119.7 (6)
C2—N1—C6	120.1 (5)	O3—C7—C6	116.6 (6)

C2—N1—Sn1	122.3 (4)	Sn1—C8—H8A	109.5
C6—N1—Sn1	117.2 (4)	Sn1—C8—H8B	109.5
C1—O1—Sn1	118.9 (4)	H8A—C8—H8B	109.5
C7—O3—Sn1	121.5 (4)	Sn1—C8—H8C	109.5
Sn1—O5—H1	121.9	H8A—C8—H8C	109.5
Sn1—O5—H2	103.8	H8B—C8—H8C	109.5
H1—O5—H2	106.7	Sn1—C9—H9A	109.5
O1—C1—O2	126.9 (6)	Sn1—C9—H9B	109.5
O1—C1—C2	116.0 (5)	H9A—C9—H9B	109.5
O2—C1—C2	117.0 (6)	Sn1—C9—H9C	109.5
N1—C2—C3	121.1 (6)	H9A—C9—H9C	109.5
N1—C2—C1	114.7 (5)	H9B—C9—H9C	109.5
C9—Sn1—N1—C2	89.9 (5)	Sn1—N1—C2—C3	174.1 (5)
C8—Sn1—N1—C2	-80.2 (5)	C6—N1—C2—C1	-175.2 (5)
O3—Sn1—N1—C2	-176.3 (5)	Sn1—N1—C2—C1	-2.8 (7)
O5—Sn1—N1—C2	-167.2 (4)	O1—C1—C2—N1	-6.6 (8)
O1—Sn1—N1—C2	6.1 (4)	O2—C1—C2—N1	172.5 (6)
C9—Sn1—N1—C6	-97.5 (5)	O1—C1—C2—C3	176.6 (6)
C8—Sn1—N1—C6	92.5 (5)	O2—C1—C2—C3	-4.3 (10)
O3—Sn1—N1—C6	-3.6 (4)	N1—C2—C3—C4	-0.6 (10)
O5—Sn1—N1—C6	5.5 (6)	C1—C2—C3—C4	176.0 (6)
O1—Sn1—N1—C6	178.8 (5)	C2—C3—C4—C5	-0.4 (11)
C9—Sn1—O1—C1	-109.6 (5)	C3—C4—C5—C6	0.4 (10)
C8—Sn1—O1—C1	81.9 (5)	C2—N1—C6—C5	-1.6 (9)
O3—Sn1—O1—C1	-13.4 (6)	Sn1—N1—C6—C5	-174.5 (5)
N1—Sn1—O1—C1	-10.1 (4)	C2—N1—C6—C7	177.2 (5)
O5—Sn1—O1—C1	163.3 (4)	Sn1—N1—C6—C7	4.3 (7)
C9—Sn1—O3—C7	98.4 (6)	C4—C5—C6—N1	0.6 (10)
C8—Sn1—O3—C7	-86.3 (6)	C4—C5—C6—C7	-178.1 (7)
N1—Sn1—O3—C7	2.3 (5)	Sn1—O3—C7—O4	177.9 (6)
O5—Sn1—O3—C7	-172.6 (6)	Sn1—O3—C7—C6	-0.8 (9)
O1—Sn1—O3—C7	5.5 (7)	N1—C6—C7—O4	178.8 (7)
Sn1—O1—C1—O2	-166.9 (6)	C5—C6—C7—O4	-2.4 (11)
Sn1—O1—C1—C2	12.2 (7)	N1—C6—C7—O3	-2.4 (9)
C6—N1—C2—C3	1.7 (9)	C5—C6—C7—O3	176.4 (7)

Symmetry codes: (i) $-x+1, -y, -z+1$.

Fig. 1

