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Key indicators

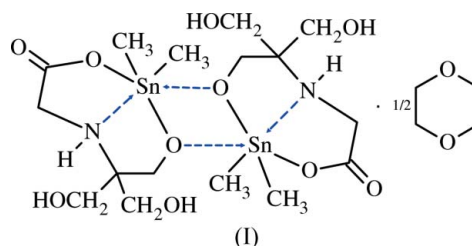
Single-crystal X-ray study
 $T = 296$ K
Mean $\sigma(\text{C}-\text{C}) = 0.005$ Å
Disorder in solvent or counterion
 R factor = 0.027
 wR factor = 0.088
Data-to-parameter ratio = 13.9For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Bis[μ -*N*-[bis(hydroxymethyl)(oxidomethyl)-
methyl]glycinato}bis[dimethyltin(IV)]
dioxane hemisolvate

The title complex, $[\text{Sn}_2(\text{CH}_3)_4(\text{C}_6\text{H}_{11}\text{NO}_5)_2] \cdot 0.5\text{C}_4\text{H}_8\text{O}_2$, comprises the centrosymmetric cyclic dimeric Sn^{IV} complex and an uncoordinated, partially occupied, centrosymmetric dioxane molecule. Each Sn^{IV} atom has a distorted octahedral six-coordinate configuration formed by the tridentate tricine ligand, one bridging O atom and two methyl groups. Within the cyclic dimer, the $\text{Sn} \cdots \text{Sn}$ separation is 3.6574 (4) Å. All H atoms of the NH and OH groups are involved in intermolecular hydrogen bonds with the O atoms as acceptors. There are also important weak intermolecular $\text{C}-\text{H} \cdots \text{O}$ hydrogen bonds, which link the molecules of the dimer and dioxane into an infinite three-dimensional network.

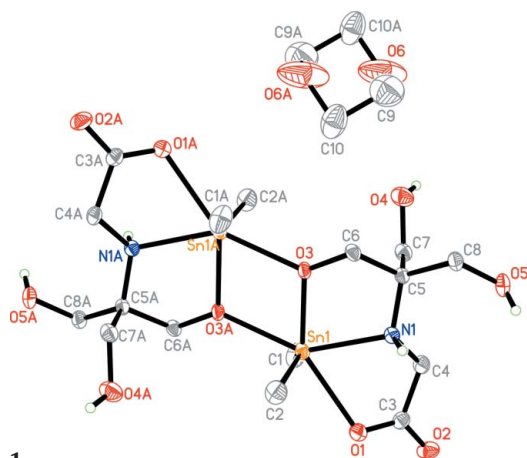
Received 17 October 2005
Accepted 8 November 2005
Online 16 November 2005

Comment

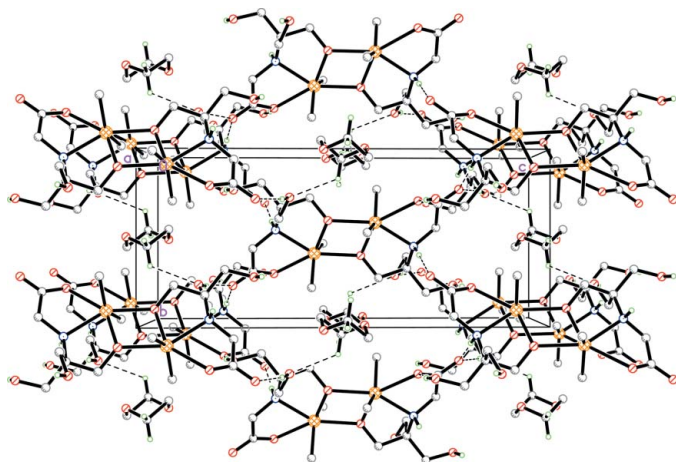
For the past decade, organotin complexes have been paid increasing attention, due to their established or potential pharmaceutical value, their versatile molecular structure and their supramolecular architecture (Ma *et al.*, 2005). Among organotin compounds, organotin carboxylates are of particular importance owing to their anticancer properties (Fang *et al.*, 2001; Sadiq-ur-Rehman *et al.*, 2005). Tricine, *N*-[tris-(hydroxymethyl)methyl]glycine, is a widely used reagent in biochemistry, especially for its buffer properties (Silva *et al.*, 2001). As part of our investigations into the structure and applications of organotin complexes of tricine, we report here the synthesis and X-ray crystal structure analysis of the title complex, (I), a new dimethyltin(IV) complex of tricine.



Complex (I) crystallizes in the space group $P2_1/n$, with one half-molecule of the dinuclear oxygen-bridged dimethyltin(IV) complex of tricine and one-half of the 50% occupied dioxane solvent molecule in the asymmetric unit. As shown in Fig. 1, each pair of Sn^{IV} atoms has the same coordination environment and is bridged by O to form a centrosymmetric dinuclear ring. The $\text{Sn} \cdots \text{Sn}$ separation is 3.6574 (4) Å, and the centre of symmetry is situated between the two Sn atoms. Each Sn^{IV} atom has a distorted octahedral six-coordinate configuration formed by the tridentate tricine ligand, one bridging O atom and two methyl groups. The tricine ligand is *mer*-coordinated, with its N atom and two O atoms in the

**Figure 1**

The structure of (I), showing 30% probability displacement ellipsoids. The solvent molecule also lies on an inversion centre (occupancy 50%). Atoms labelled with the suffix A are at the symmetry position $(2 - x, 2 - y, -z)$ for the dimer and $(1 - x, 1 - y, -z)$ for the dioxane.

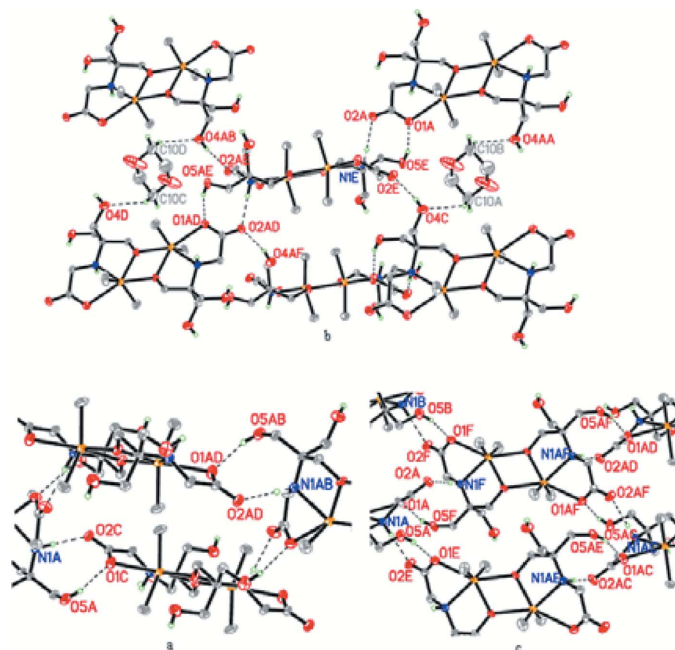
**Figure 2**

A packing diagram for (I), viewed along the *a* axis. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonds have been omitted for clarity.

equatorial positions. The fourth equatorial position is occupied by the bridging O atom. For the equatorial plane, the mean deviation is 0.0342 (3) Å. The axial positions are occupied by the two methyl groups bonded to the Sn atom.

The three Sn—O bond lengths show clear differences. The longest one is 2.489 (3) Å for Sn1—O1, which is due to the partial double-bond properties of the carbonyl O atom. The O3A—Sn1 bond length [2.337 (3) Å] is much longer than the O3—Sn1 bond [2.074 (3) Å], showing that the bond strength of atom O3A coordinated to the Sn atom is less than for atom O3. The Sn1—N1 bond length is 2.284 (3) Å, which is much shorter than values reported in the literature (Lo *et al.*, 1992; Yin *et al.*, 2004). This can be explained by the formation of the polycyclic system.

In the Sn1/O1/C3/C4/N1 ring, four atoms (O1, C3, C4 and N1) are nearly coplanar, with a mean deviation of 0.0437 (4) Å, with atom Sn1 (the flap) projecting out of the plane by 0.367 (2) Å; the dihedral angle between the four-atom plane and the N1/Sn1/O1 flap is 39.5 (4)°. In the other

**Figure 3**

The hydrogen bonds (dashed lines) in the packing of (I), viewed along (a) the *a* axis, (b) the *b* axis and (c) the *c* axis. H atoms, and most other atoms not involved in hydrogen bonds, have been omitted for clarity.

five-membered ring, atoms Sn1, O3, N1 and C5 are nearly coplanar, with a mean deviation of 0.0669 (3) Å. Atom C6 projects out of the plane by 0.033 (2) Å and the dihedral angle for the flap is 44.7 (3)°.

The dimers of (I) are held together by classical intermolecular hydrogen bonds (N1—H1...O2ⁱⁱ, O5—H5...O1ⁱⁱ and O4—H4...O2ⁱⁱⁱ; symmetry codes as in Table 2). The weak intermolecular C10—H10A...O4 hydrogen bond links the dimer and the dioxane solvent molecule. All these hydrogen bonds link the molecules of the dimer and dioxane molecules into an infinite three-dimensional network, as shown in Fig. 3.

Experimental

Tricine (2 mmol, 0.358 g) and dimethyltin oxide (1 mmol, 0.165 g) were dissolved in dry toluene (30 ml) and heated for 5 h under reflux. The solvent was removed in a rotatory evaporator and the residue (no obvious melting point) was recrystallized from a 1:1 mixture of ethanol–dioxane. Single crystals of (I) suitable for X-ray diffraction analysis precipitated after several days. Analysis, calculated for C₁₆H₃₄N₂O₁₀Sn₂: C 29.48, H 5.26, N 4.30%; found: C 29.19, H 5.32, N 4.33%. IR (KBr, ν , cm⁻¹): 3442 (OH), 3269 (NH), 1522 (COO), 1244, 1130 (C—O), 623 (Sn—C).

Crystal data

[Sn₂(CH₃)₄(C₆H₁₁NO₅)₂]
·0.5C₄H₈O₂
M_r = 695.88
Monoclinic, *P*2₁/*n*
a = 8.1343 (4) Å
b = 8.3520 (4) Å
c = 19.3060 (9) Å
 β = 92.615 (1)
V = 1310.24 (11) Å³
Z = 2

D_x = 1.764 Mg m⁻³
Mo *K* α radiation
Cell parameters from 11611
reflections
 θ = 2.1–25.1°
 μ = 1.96 mm⁻¹
T = 296 (2) K
Block, white
0.37 × 0.27 × 0.14 mm

Data collection

Bruker APEX-II area-detector diffractometer	2325 independent reflections
φ and ω scans	2198 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Bruker, 2004)	$R_{\text{int}} = 0.027$
$T_{\text{min}} = 0.476$, $T_{\text{max}} = 0.760$	$\theta_{\text{max}} = 25.1^\circ$
11611 measured reflections	$h = -9 \rightarrow 9$
	$k = -9 \rightarrow 9$
	$l = -23 \rightarrow 23$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.058P)^2 + 3.0289P]$
$R[F^2 > 2\sigma(F^2)] = 0.027$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.088$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.00$	$\Delta\rho_{\text{max}} = 0.94 \text{ e } \text{\AA}^{-3}$
2325 reflections	$\Delta\rho_{\text{min}} = -0.59 \text{ e } \text{\AA}^{-3}$
167 parameters	
H-atom parameters constrained	

Table 1

Selected geometric parameters (\AA , $^\circ$).

Sn1—C1	2.111 (4)	Sn1—O1	2.489 (3)
Sn1—C2	2.087 (4)	Sn1—O3	2.074 (3)
Sn1—N1	2.284 (3)	Sn1—O3 ⁱ	2.337 (3)
C2—Sn1—C1	141.7 (2)	O3—Sn1—N1	76.35 (10)
N1—Sn1—O1	67.12 (10)	O3 ⁱ —Sn1—O1	148.51 (9)
N1—Sn1—O3 ⁱ	144.34 (10)	O3—Sn1—O3 ⁱ	68.09 (10)
O3—Sn1—C1	108.46 (18)	Sn1—O3—Sn1 ⁱ	111.91 (10)

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

Table 2

Hydrogen-bond geometry (\AA , $^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1 \cdots O2 ⁱⁱ	0.91	2.01	2.924 (5)	178
O4—H4 \cdots O2 ⁱⁱⁱ	0.82	1.99	2.787 (5)	165
O5—H5 \cdots O1 ⁱⁱ	0.82	2.08	2.860 (5)	159
C10—H10A \cdots O4	0.97	2.49	3.097 (1)	120

Symmetry codes: (ii) $-x + \frac{5}{2}, y - \frac{1}{2}, -z + \frac{1}{2}$; (iii) $-x + \frac{3}{2}, y - \frac{1}{2}, -z + \frac{1}{2}$.

The H atoms were positioned geometrically and treated as riding on their parent atoms, with C—H distances of 0.97 (methylene) and 0.96 \AA (methyl), and with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl H atoms and $1.2U_{\text{eq}}(\text{C})$ for other H atoms. The solvent dioxane shows some apparent disorder and three distance restraints were applied. According to the published method (Bowes *et al.*, 2002; Hulme & Tocher, 2004), atoms O6, C9 and C10 were refined anisotropically with restraints of C9—C10 = 1.50 (1) \AA , and O6—C10 and O6—C10A = 1.45 (1) \AA . The dioxane molecule was assigned an occupancy of 0.5 in view of the high displacement parameters.

Data collection: APEX2 (Bruker, 2004); cell refinement: APEX2; data reduction: APEX2; program(s) used to solve structure: APEX2; program(s) used to refine structure: APEX2; molecular graphics: APEX2; software used to prepare material for publication: APEX2.

The authors gratefully acknowledge financial support from the Natural Science Foundation of JiangXi Province (grant No. 0320026) and the JiangXi Provincial Education Office (grant No. 2001387).

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