

Bis(diisopropylammonium) dimethyl-
dioxalatostannateTao Xu,^a Shi-Yao Yang,^a
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Key indicators

Single-crystal X-ray study

T = 298 K

Mean $\sigma(\text{C}-\text{C}) = 0.007 \text{ \AA}$

R factor = 0.041

wR factor = 0.098

Data-to-parameter ratio = 16.8

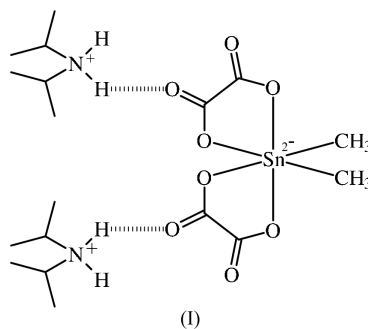
For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

The six-coordinate Sn atom in bis(diisopropylammonium) dimethyldioxalatostannate, $[(\text{C}_3\text{H}_7)_2\text{NH}_2]^+ [(\text{CH}_3)_2\text{Sn}(\text{O}_2\text{C}-\text{CO}_2)]^{2-}$, exists in a *cis*- C_2SnO_4 coordination sphere [$\text{Sn}-\text{C}$ 2.127 (5) Å; $\text{C}-\text{Sn}-\text{C}$ 109.2 (3) $^\circ$]. The anion lies on a special position of 2 symmetry. The ammonium cations and the stannate anions are linked by hydrogen bonds into a linear chain.

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Comment

Six-coordinate bis-chelated diorganotin compounds whose chelating groups themselves have only a small 'bite', adopt an $R_2\text{Sn}$ alignment that is approximately 135° (Ng *et al.*, 1987), *i.e.*, the skeleton is neither right-angled nor linear. An example of such a group is the oxalato group, two of which chelate to the dibutyltin entity in bis(dicyclohexylammonium) dioxalatodibutylstannate to give rise to a skew-trapezoidal C_2SnO_4 geometry for the tin atom (Ng *et al.*, 1992). The homologous dioxalatodimethylstannate is sufficiently Lewis acidic to permit the incorporation of an ethanol molecule into the coordination sphere, and the Sn atom in this stannate is seven-coordinate (Ng, 1996). On the other hand, the use of the sterically similar diisopropylammonium counterion in place of the dicyclohexylammonium cation led to the title compound (I), whose Sn atom shows a *cis*-alignment [$\text{C}-\text{Sn}-\text{C}$ 109.2 (3) Å] of the $(\text{CH}_3)_2\text{Sn}$ skeleton (Fig. 1).



The chelation of the oxalato group is relatively symmetrical [$\text{Sn}1-\text{O}1$ 2.185 (3) and $\text{Sn}1-\text{O}3$ 2.138 (2) Å], unlike that found in the skew-trapezoidal dioxalatodibutylstannate ion, for which $\text{Sn}-\text{O}$ and $\text{Sn}\leftarrow\text{O}$ bonds can be differentiated (Ng *et al.*, 1992). The counterion interacts with the uncoordinated O atoms (Table 2), the hydrogen bonding scheme giving rise to the formation of a linear chain that propagates along the $[10\bar{1}]$ direction (Fig. 2). The supramolecular chemistry of other ammonium organostannates has been reviewed (Haiduc & Edlmann, 1999).

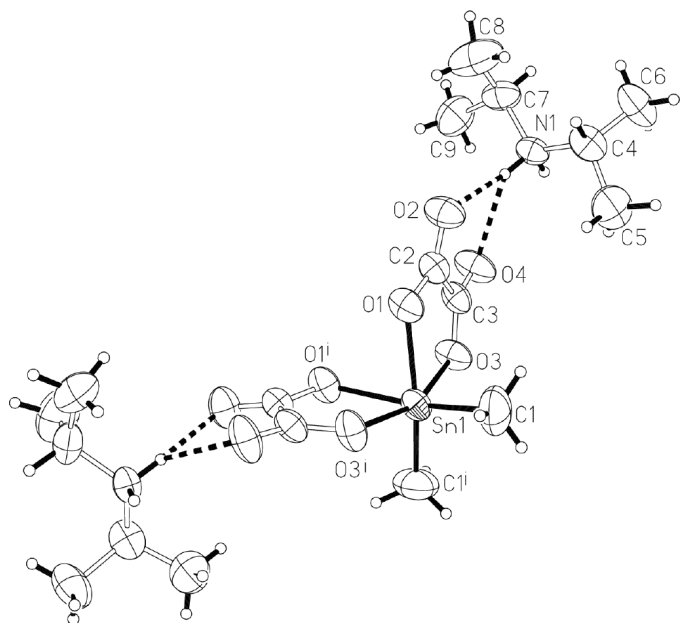


Figure 1
ORTEP (Johnson, 1976) plot of bis(diisopropylammonium) dimethyldioxalatoantimonate; displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as spheres of arbitrary radii. [Symmetry code (i): $1 - x, y, \frac{3}{2} - z$.]

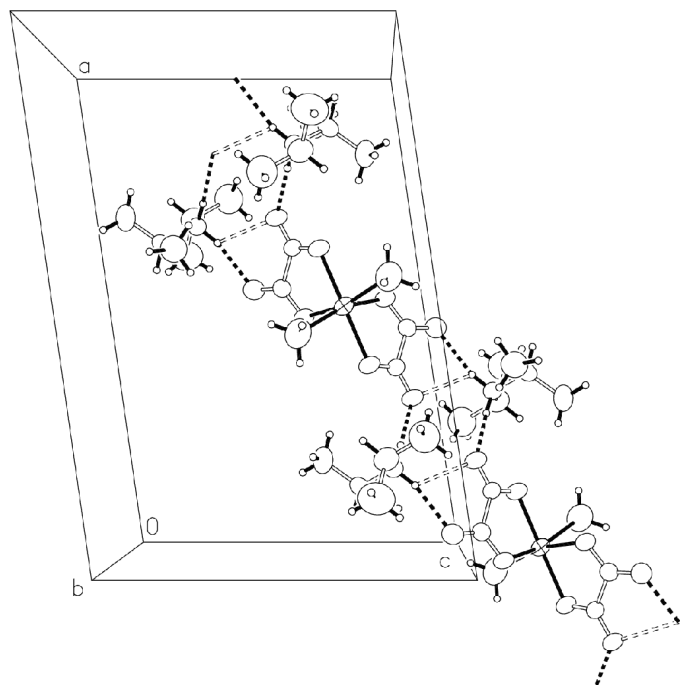


Figure 2
ORTEP (Johnson, 1976) plot of the hydrogen-bonded chain.

Experimental

Diisopropylamine (1.01 g, 10 mmol) and oxalic acid dihydrate (1.26 g, 10 mmol) were placed in a small volume of ethanol and the mixture heated to dissolve the resulting diisopropylammonium hydrogen oxalate. Dimethyltin oxide (0.83 g, 5 mmol) was added and the

mixture heated for several minutes until the oxide dissolved completely. The solution was filtered. Colorless crystals separated from the cool solution when the solvent was allowed to evaporate slowly.

Crystal data

$2C_6H_{16}N[Sn(C_6H_8O_8)_2]$
 $M_r = 529.19$
 Monoclinic, $C2/c$
 $a = 16.4714$ (8) Å
 $b = 14.0769$ (7) Å
 $c = 11.0443$ (5) Å
 $\beta = 98.128$ (1)°
 $V = 2535.1$ (2) Å³
 $Z = 4$

$D_x = 1.387$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 5928 reflections
 $\theta = 2.5$ – 27.6°
 $\mu = 1.05$ mm⁻¹
 $T = 298$ (2) K
 Parallelepiped, colorless
 $0.47 \times 0.40 \times 0.09$ mm

Data collection

Bruker SMART APEX area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.652, T_{\max} = 0.910$
 8978 measured reflections

2234 independent reflections
 2062 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.036$
 $\theta_{\max} = 25.0^\circ$
 $h = -19 \rightarrow 19$
 $k = -16 \rightarrow 16$
 $l = -13 \rightarrow 13$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.041$
 $wR(F^2) = 0.098$
 $S = 1.08$
 2234 reflections
 133 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0578P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.83$ e Å⁻³
 $\Delta\rho_{\min} = -0.45$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Sn1—C1	2.127 (5)	Sn1—O1	2.185 (3)
Sn1—O3	2.138 (2)		
C1—Sn1—C1 ⁱ	109.2 (3)	O1—Sn1—O1 ⁱ	79.6 (1)
C1—Sn1—O1	87.3 (2)	O1—Sn1—O3	75.3 (1)
C1—Sn1—O1 ⁱ	159.6 (2)	O1—Sn1—O3 ⁱ	88.8 (1)
C1—Sn1—O3	103.0 (2)	O3—Sn1—O3 ⁱ	159.4 (2)
C1—Sn1—O3 ⁱ	89.0 (2)		

Symmetry code: (i) $1 - x, y, \frac{3}{2} - z$.

Table 2

Hydrogen-bonding geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
N1—H1n1 \cdots O2	0.90	2.03	2.906 (4)	165.2
N1—H1n1 \cdots O4	0.90	2.40	2.941 (4)	118.6
N1—H1n2 \cdots O4 ⁱⁱ	0.90	1.97	2.864 (4)	173.7

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

H atoms were placed in calculated positions (N—H 0.90 and C—H 0.96 Å) and were allowed to ride on their parent atoms; $U(H) = 1.2U_{\text{eq}}(C, N)$ for the ammonium N and methine C atoms, and $1.5U_{\text{eq}}$ for the methyl C atoms. The torsional angle for the tin-bound methyl group was also refined.

Data collection: SMART (Bruker, 2001); cell refinement: SMART; data reduction: SAINT (Bruker, 2001); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEPII (Johnson, 1976); software used to prepare material for publication: SHELXL97.

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References

- Bruker (2001). *SAINTE* and *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Oak Ridge, Tennessee, USA.
- Haiduc, I. & Edelmann, F. T. (1999). *Supramolecular Organometallic Chemistry*. Weinheim: Wiley-VCH.
- Ng, S. W. (1996). *Acta Cryst.* **C52**, 2990–2992.
- Ng, S. W., Chen, W., Kumar Das, V. G. & Mak, T. C. W. (1987). *J. Organomet. Chem.* **334**, 295–305.
- Ng, S. W., Kumar Das, V. G., Gielen, M. & Tiekink, E. R. T. (1992). *Appl. Organomet. Chem.* **6**, 19–25.
- Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.