

Bis(diisopropylammonium) diphenyldioxalato-stannate(IV)

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Key indicators

Single-crystal X-ray study

T = 298 K

Mean $\sigma(\text{C}-\text{C}) = 0.014 \text{ \AA}$

R factor = 0.080

wR factor = 0.166

Data-to-parameter ratio = 15.8

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) diphenyldioxalato-stannate(IV), $[(\text{C}_3\text{H}_7)_2\text{NH}_2]^+_2 [(\text{C}_6\text{H}_5)_2\text{Sn}(\text{O}_2\text{CCO}_2)]^{2-}$, exists in a *cis*- C_2SnO_4 octahedral coordination sphere [Sn—C 2.139 (7) and 2.144 (7) Å; C—Sn—C 103.8 (3)°]. The ammonium cations and the stannate anions are linked by hydrogen bonds into a zigzag chain, running along the *c* axis of the monoclinic cell.

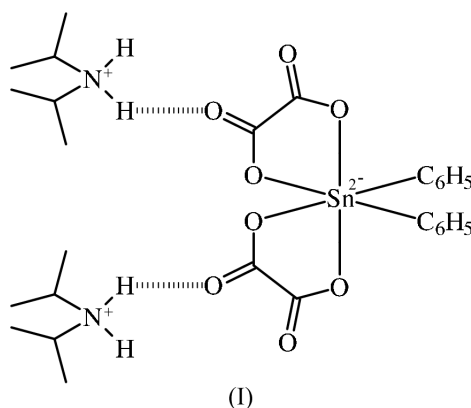
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Comment

A previous report describes the structure of bis(diisopropylammonium) dimethyldioxalato-stannate(IV), a six-coordinate *cis*- C_2SnO_4 octahedral ammonium stannate (Xu *et al.*, 2003). For six-coordinate diorganotin systems, the dialkyltin derivatives generally adopt a *trans* configuration and the diaryltin derivatives a *cis* configuration (Davies, 1997; Omae, 1989). Bis(diisopropylammonium) diphenyldioxalato-stannate(IV), (I), also adopts a *cis* configuration (Fig. 1), and the chelation by the oxalate is symmetrical [Sn1—O1 2.162 (5) and Sn1—O3 2.128 (5) Å; Sn1—O5 2.191 (5) and Sn1—O7 2.124 (4) Å]. The counter-ion interacts with the uncoordinated O atoms (Table 2), the hydrogen-bonding scheme giving rise to the formation of a linear chain which propagates by glide planes along the *c* direction (Fig. 2).



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. Diphenyltin oxide (1.40 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

$2\text{C}_6\text{H}_{16}\text{N}^+ \cdot [\text{Sn}(\text{C}_6\text{H}_5)_2(\text{C}_2\text{O}_4)_2]^{2-}$
 $M_r = 653.33$
 Monoclinic, $P2_1/n$
 $a = 9.4647$ (5) Å
 $b = 20.901$ (1) Å
 $c = 15.9940$ (8) Å
 $\beta = 93.364$ (1)°
 $V = 3158.6$ (3) Å³
 $Z = 4$

$D_x = 1.374$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 5007 reflections
 $\theta = 2.5\text{--}27.6^\circ$
 $\mu = 0.86$ mm⁻¹
 $T = 298$ (2) K
 Parallelepiped, colorless
 $0.29 \times 0.18 \times 0.12$ mm

Data collection

Bruker SMART APEX area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.705$, $T_{\max} = 0.905$
 23125 measured reflections

5563 independent reflections
 5505 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.039$
 $\theta_{\text{max}} = 25.0^\circ$
 $h = -11 \rightarrow 11$
 $k = -24 \rightarrow 24$
 $l = -19 \rightarrow 19$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.080$
 $wR(F^2) = 0.166$
 $S = 1.30$
 5563 reflections
 352 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0485P)^2 + 12.4071P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 1.14$ e Å⁻³
 $\Delta\rho_{\text{min}} = -1.40$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Sn1—C1	2.139 (7)	Sn1—O3	2.128 (5)
Sn1—C7	2.144 (7)	Sn1—O5	2.191 (5)
Sn1—O1	2.162 (5)	Sn1—O7	2.124 (4)
C1—Sn1—C7	103.8 (3)	C7—Sn1—O7	93.8 (2)
C1—Sn1—O1	164.4 (2)	O1—Sn1—O3	76.0 (2)
C1—Sn1—O3	95.5 (2)	O1—Sn1—O5	78.8 (2)
C1—Sn1—O5	87.6 (2)	O1—Sn1—O7	82.3 (2)
C1—Sn1—O7	101.8 (2)	O3—Sn1—O7	153.6 (2)
C7—Sn1—O1	90.9 (2)	O3—Sn1—O5	85.2 (2)
C7—Sn1—O3	101.3 (3)	O7—Sn1—O5	76.0 (2)
C7—Sn1—O5	166.2 (2)		

Table 2

Hydrogen-bonding geometry (Å, °).

$D\text{—}H\cdots A$	$D\text{—}H$	$H\cdots A$	$D\cdots A$	$D\text{—}H\cdots A$
N1—H1n2 \cdots O2	0.90	2.03	2.878 (8)	157
N1—H1n2 \cdots O4	0.90	2.39	3.026 (8)	128
N1—H1n1 \cdots O4 ⁱ	0.90	1.96	2.856 (8)	178
N2—H2n2 \cdots O6	0.90	2.34	2.938 (7)	124
N2—H2n1 \cdots O6 ⁱⁱ	0.90	1.91	2.812 (7)	178
N2—H2n2 \cdots O8	0.90	2.07	2.931 (7)	160

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

H atoms were placed in calculated positions and were allowed to ride on their parent atoms; $U(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$ for the ammonium N, methine and aromatic C atoms, and $1.5U_{\text{eq}}$ for the methyl C atoms. The largest peak and hole in the final difference Fourier map are about 1 Å from Sn1.

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.

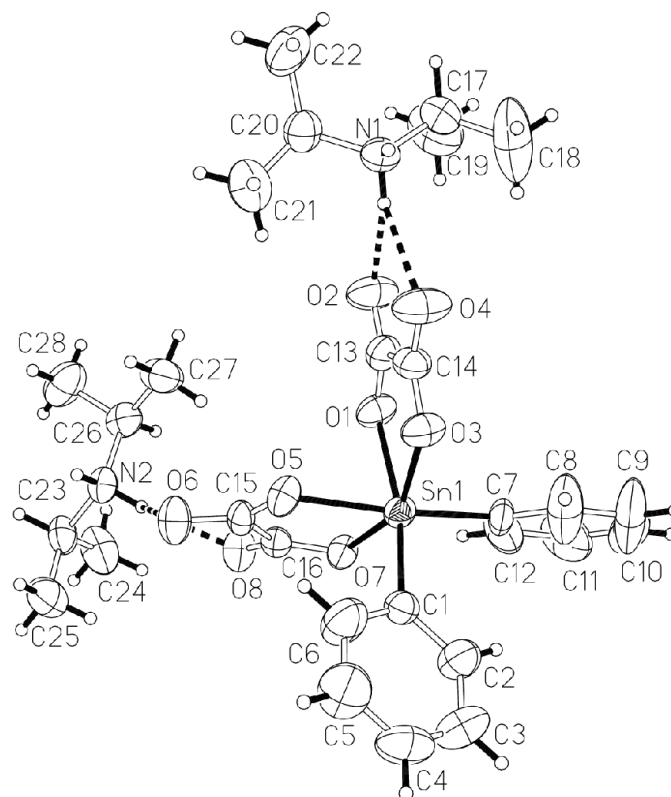


Figure 1

ORTEP (Johnson, 1976) view of bis(diisopropylammonium) diphenyldioxalatostannate(IV); displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as spheres of arbitrary radii.

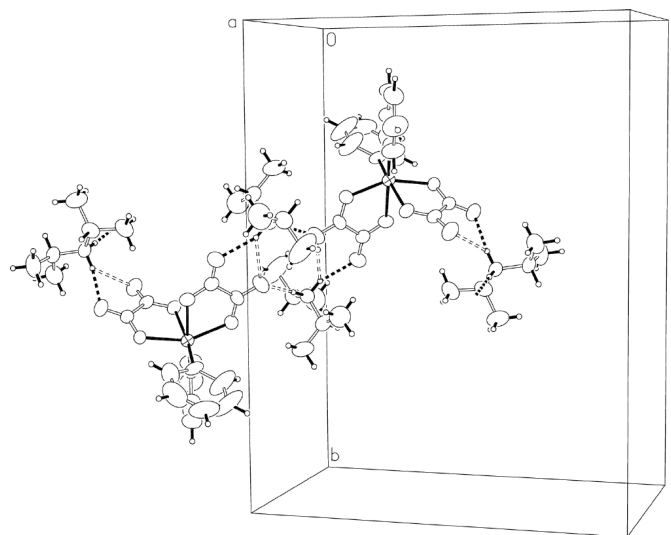


Figure 2

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

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