

Bis(diisopropylammonium) diphenyl-  
dioxalatostannate(IV)Tao Xu,<sup>a</sup> Shi-Yao Yang,<sup>a</sup>  
Zhao-Xiong Xie<sup>a</sup> and  
Seik Weng Ng<sup>b\*</sup><sup>a</sup>State Key Laboratory for Physical Chemistry of  
Solid Surfaces, Xiamen University, Xiamen  
361005, People's Republic of China, and<sup>b</sup>Department of Chemistry, University of  
Malaya, 50603 Kuala Lumpur, Malaysia

Correspondence e-mail: seikweng@um.edu.my

## 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) diphenyldioxalatostannate(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*- $\text{C}_2\text{SnO}_4$  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.

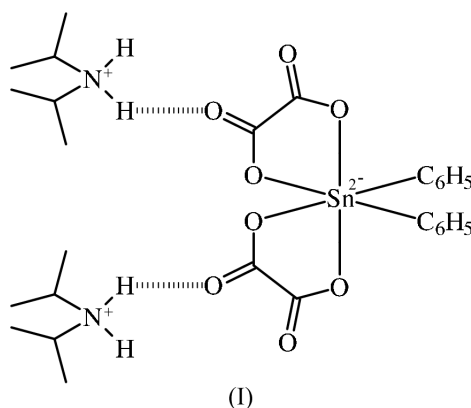
Received 29 August 2003

Accepted 3 September 2003

Online 11 September 2003

## Comment

A previous report describes the structure of bis(diisopropylammonium) dimethyldioxalatostannate(IV), a six-coordinate *cis*- $\text{C}_2\text{SnO}_4$  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) diphenyldioxalatostannate(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) Å<sup>3</sup>  
 $Z = 4$

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

## Data collection

Bruker SMART APEX area-detector diffractometer  
 $\varphi$  and  $\omega$  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<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -1.40$  e Å<sup>-3</sup>

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, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
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 <sup>i</sup>	0.90	1.96	2.856 (8)	178
N2—H2n2 $\cdots$ O6	0.90	2.34	2.938 (7)	124
N2—H2n1 $\cdots$ O6 <sup>ii</sup>	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.

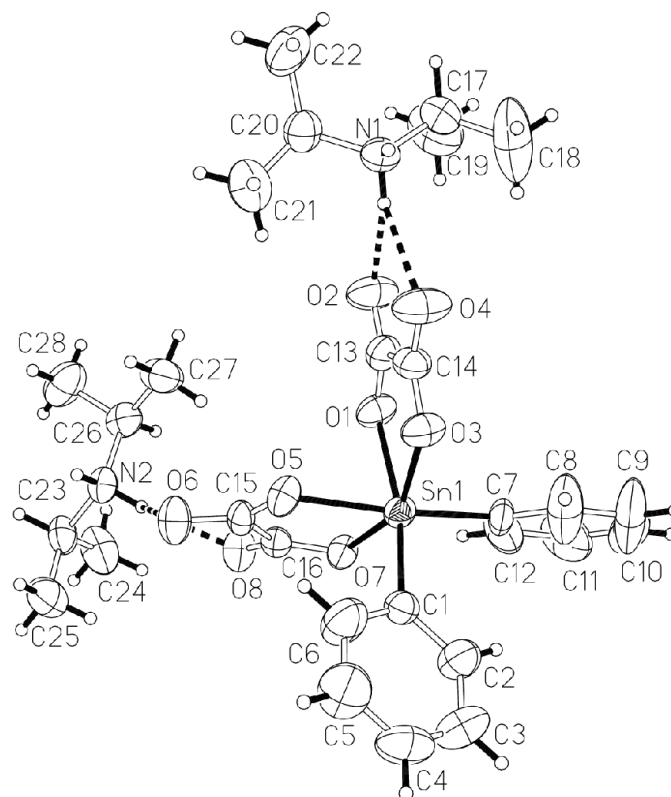


Figure 1

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.

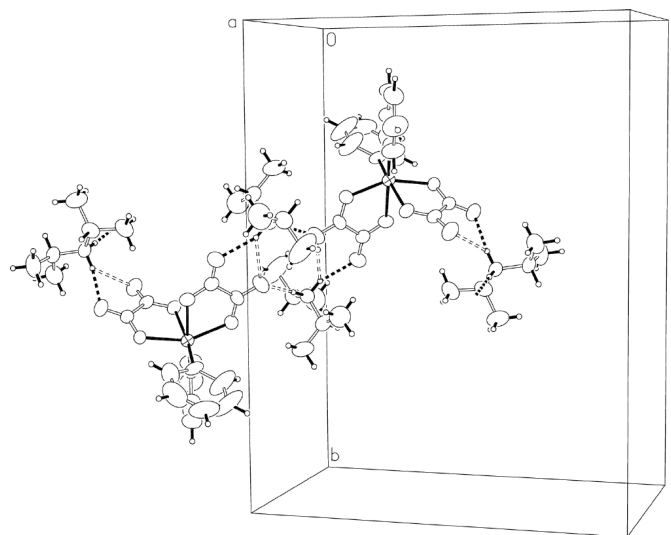


Figure 2

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

The authors thank the National Natural Science Foundation of China (No. 20173046), Xiamen University and the University of Malaya for supporting this work.

## References

Bruker (2001). SAINT and SMART. Bruker AXS Inc., Madison, Wisconsin, USA.

- Davies, A. G. (1997). *Organotin Chemistry*. New York: John Wiley and Sons.
- Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Oak Ridge, Tennessee, USA.
- Omae, I. (1989). *Organotin Chemistry. J. Organomet. Chem. Library*, Vol. 21. New York: Elsevier.
- Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Xu, T., Yang, S.-Y., Xie, Z.-X. & Ng, S. W. (2003). *Acta Cryst.* **E59**, m870–m872.