

Sanz, J. L., Cervera, B., Ruiz, R., Bois, C., Faus, J., Lloret, F. & Julve, M. (1996). *J. Chem. Soc. Dalton Trans.* pp. 1359–1366.  
 Sheldrick, G. M. (1985). *SHELXS86. Crystallographic Computing 3*, edited by G. M. Sheldrick, C. Krüger & R. Goddard, pp. 175–189. Oxford University Press.

*Acta Cryst.* (1999). **C55**, 875–876

## (2,2'-Bipyridyl-*N,N'*)di-*n*-butyldichlorotin(IV)

JENNIFER GILL,<sup>a</sup> HANS PARGE,<sup>b</sup> CHRISTINE J. CARDIN,<sup>c</sup>  
 CHRIS TSAMIS<sup>d</sup> AND CONSTANTIN KAVOUNIS<sup>a</sup>

<sup>a</sup>Department of Physics, Aristotle University of Thessaloniki, Gr-540 06 Thessaloniki, Greece, <sup>b</sup>Agouron Pharmaceuticals, 10350 North Torrey Pines Road, La Jolla, CA 92037-1020, USA, <sup>c</sup>Department of Chemistry, University of Reading, Whiteknights, Reading RG4 9BB, England, and <sup>d</sup>Department of Chemistry, Aristotle University of Thessaloniki, Gr-540 06 Thessaloniki, Greece. E-mail: kavounis@ccf.auth.gr

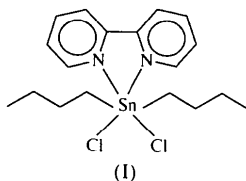
(Received 8 January 1999; accepted 23 February 1999)

### Abstract

The title compound, [SnCl<sub>2</sub>(C<sub>4</sub>H<sub>9</sub>)<sub>2</sub>(C<sub>10</sub>H<sub>8</sub>N<sub>2</sub>)], is the product of the reaction of 2,2'-bipyridyl with dibutyltin dichloride. The Sn<sup>IV</sup> centre is octahedrally coordinated by a bidentate 2,2'-bipyridyl ligand, two *cis* chlorides and two *trans* butyl groups. The extensively delocalized bipyridyl group is not absolutely planar, with the pyridyl rings twisted by 7.5 (3)°.

### Comment

The title compound is a six-coordinated organotin complex of the type R<sub>2</sub>SnX<sub>2</sub>L<sub>2</sub>, where R is an alkyl or aryl group, X is a halogen and L<sub>2</sub> is a bidentate nitrogen-donor ligand. The importance of this compound comes from its reported antitumour activity (Crowe *et al.*, 1984), which is probably due to the rather long Sn—N bond lengths. The structural investigation of the present compound, (I), is part of an ongoing study of a series of diorganotin dichelate complexes with nitrogenous bidentate ligands.



The Sn, the two Cl and the two N atoms of the bipyridyl group are coplanar, with an r.m.s. deviation of 0.013 Å, and this plane forms angles of 5.9 (2) and 4.1 (3)° with the pyridyl rings (Fig. 1). The two planar butyl groups (C11—C12—C13—C14 and C15—C16—C17—C18) are almost perpendicular to the above-mentioned basal plane of the molecule, with angles of 84.8 (8) and 81.7 (6)°, and form a dihedral angle of 69 (1)°. Each pyridyl ring is planar (r.m.s. deviations of 0.003 and 0.011 Å).

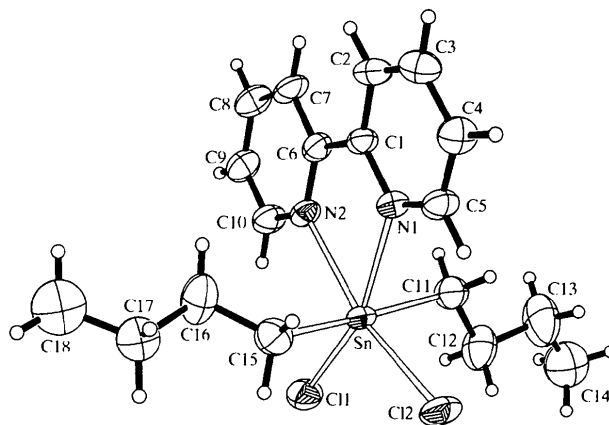


Fig. 1. View of the molecule of the title compound with the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. Only the major disorder component is shown, and one H atom on each of C16 and C18 is wholly obscured by the parent atom.

There are deviations from ideal octahedral geometry about the Sn atom, as was also observed in the case of 2,2'-bipyridyldichlorodiphenyltin (Harrison *et al.*, 1974). The C11—Sn—C15 angle is only 169.3 (4)°, while the bond angles subtended at tin lie in the range 84.5 (3)–96.7 (3)°. The C11—Sn—C12 angle of 104.28 (8)° is the result of the mutual repulsion of the two Cl atoms, while the N1—Sn—N2 angle of 67.8 (2)° is similar to values given in the literature (Harrison *et al.*, 1974; Kabanos *et al.*, 1992).

The difference in the Sn—C11 and Sn—C12 bond lengths is probably due to a specific intermolecular interaction involving the H atom attached to C2 and the C12 atom of a symmetry-related molecule; the C2···C12(−1 + x, y, z) separation is 3.598 (8) Å and the C2—H2···C12 angle has a value of 166°.

### Experimental

The title compound was prepared by addition of a benzene solution of 2,2'-bipyridyl to a benzene solution of dibutyltin dichloride. Crystals suitable for X-ray analysis were obtained from hot benzene (Harrison *et al.*, 1974).

**Crystal data**

[SnCl<sub>2</sub>(C<sub>4</sub>H<sub>9</sub>)<sub>2</sub>(C<sub>10</sub>H<sub>8</sub>N<sub>2</sub>)]  
*M<sub>r</sub>* = 460.00  
 Monoclinic  
*P*2<sub>1</sub>/*n*  
*a* = 9.529 (2) Å  
*b* = 16.354 (5) Å  
*c* = 13.719 (5) Å  
 $\beta$  = 105.130 (10)°  
*V* = 2063.8 (11) Å<sup>3</sup>  
*Z* = 4  
*D<sub>x</sub>* = 1.480 Mg m<sup>-3</sup>  
*D<sub>m</sub>* not measured

Mo *K*α radiation  
 $\lambda$  = 0.71073 Å  
 Cell parameters from 56 reflections  
 $\theta$  = 12.38–19.62°  
 $\mu$  = 1.498 mm<sup>-1</sup>  
*T* = 293 (2) K  
 Parallelepiped  
 0.8 × 0.2 × 0.2 mm  
 Colourless

**Data collection**

Philips PW1100 diffractometer (updated by Stoe)  
 $\omega$ -2 $\theta$  scans  
 Absorption correction:  
 $\psi$  scan (North *et al.*, 1968)  
*T<sub>min</sub>* = 0.469, *T<sub>max</sub>* = 0.741  
 3575 measured reflections  
 3456 independent reflections

2873 reflections with *I* > 2σ(*I*)  
 $R_{\text{int}}$  = 0.064  
 $\theta_{\text{max}}$  = 25.08°  
 $h$  = -10 → 8  
 $k$  = 0 → 17  
 $l$  = -8 → 16  
 3 standard reflections  
 frequency: 120 min  
 intensity decay: 6.1%

**Refinement**

Refinement on *F*<sup>2</sup>  
 $R[F^2 > 2\sigma(F^2)] = 0.054$   
 $wR(F^2) = 0.158$   
 $S = 1.119$   
 3456 reflections  
 207 parameters  
 H atoms treated by a mixture of independent and constrained refinement  
 $w = 1/[\sigma^2(F_o^2) + (0.0680P)^2 + 9.9655P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$

( $\Delta/\sigma$ )<sub>max</sub> = 0.001  
 $\Delta\rho_{\text{max}}$  = 1.21 e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}}$  = -1.09 e Å<sup>-3</sup>  
 Extinction correction: *SHELXL97* (Sheldrick, 1997)  
 Extinction coefficient: 5 (5) × 10<sup>-4</sup>  
 Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. Selected geometric parameters (Å, °)

Sn—C11	2.493 (2)	Sn—C15	2.142 (9)
Sn—C12	2.566 (2)	Sn—N1	2.412 (6)
Sn—C11	2.127 (9)	Sn—N2	2.405 (6)
C11—Sn—C11	96.7 (3)	C15—Sn—C12	85.3 (3)
C11—Sn—C12	89.3 (3)	N1—Sn—C11	159.73 (16)
C11—Sn—C15	169.3 (4)	N1—Sn—C12	95.96 (16)
C11—Sn—N1	84.5 (3)	N1—Sn—N2	67.8 (2)
C11—Sn—N2	88.8 (3)	N2—Sn—C11	91.98 (17)
C15—Sn—N1	86.8 (3)	N2—Sn—C12	163.74 (17)
C15—Sn—N2	93.8 (3)	C11—Sn—C12	104.28 (8)
C15—Sn—C11	93.6 (3)		

The rather high values of the isotropic displacement parameters of atoms C14, C17 and C18 of the two butyl groups were indicative of unmodelled disorder (Warda, 1998). The assumption of 'similar' *U<sup>ij</sup>* values (*SIMU*) for the adjacent atoms and the application of distance restraints to target values of 1.52 (1) Å proved to be very effective for all pairs of atoms separated by less than 0.8 Å on the two butyl chains. This type of refinement led to occupancies of 0.76 (3) and 0.24 (3) for C14 and C14', respectively, and values of 0.71 (3) for C17 and C18, and of 0.29 (2) for C17' and C18'. The final difference electron-density map revealed a highest peak of 1.21 e Å<sup>-3</sup>

0.97 Å from Sn and a deepest hole of -1.09 e Å<sup>-3</sup> 0.83 Å from Sn. The data have been corrected for absorption effects. Five low-resolution reflections suffering from extinction were omitted from the refinement. An extinction correction was applied. H atoms were placed in calculated positions and thereafter allowed to ride on their parent atoms, with *U<sub>iso</sub>*(H) values of 1.2*U<sub>eq</sub>*(C).

Data collection: *DIF4* (Stoe & Cie, 1987a). Cell refinement: *DIF4*. Data reduction: *REDU4* (Stoe & Cie, 1987b). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997). Molecular graphics: *PLATON* (Spek, 1990). Software used to prepare material for publication: *SHELXL97*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1319). Services for accessing these data are described at the back of the journal.

**References**

- Crowe, A. J., Smith, P. J., Cardin, C. J., Parge, H. E. & Smith, F. E. (1984). *Cancer Lett.* **24**, 45–48.  
 Harrison, G., King, T. J. & Richards, J. A. (1974). *J. Chem. Soc. Dalton Trans.* pp. 1723–1726.  
 Kabanos, T. A., Keramidas, A. D., Menzafos, D., Russo, U., Terzis, A. & Tsangaris, J. M. (1992). *J. Chem. Soc. Dalton Trans.* pp. 2729–2733.  
 North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.  
 Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.  
 Sheldrick, G. M. (1997). *SHELXL97. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.  
 Spek, A. L. (1990). *Acta Cryst.* **A46**, C-34.  
 Stoe & Cie (1987a). *DIF4. Diffractometer Control Program*. Version 6.2. Stoe & Cie, Darmstadt, Germany.  
 Stoe & Cie (1987b). *REDU4. Data Reduction Program*. Version 6.2. Stoe & Cie, Darmstadt, Germany.  
 Warda, S. A. (1998). *Acta Cryst.* **C54**, 460–462.

*Acta Cryst.* (1999). **C55**, 876–878

**Bis(2-methyl-8-quinolinolato-*N,O*)tin(II)**

CHITOSHI KITAMURA,<sup>a</sup> AKIO YONEDA,<sup>a</sup> KEN-ICHI SUGIURA<sup>b</sup>  
 AND YOSHITERU SAKATA<sup>b</sup>

<sup>a</sup>Department of Applied Chemistry, Himeji Institute of Technology, Shosha 2167, Himeji, Hyogo 671-2201, Japan, and <sup>b</sup>The Institute of Scientific and Industrial Research, Osaka University, Mihogaoka 8-1, Ibaraki, Osaka 567-0047, Japan. E-mail: kitamura@chem.eng.himeji-tech.ac.jp

(Received 4 January 1999; accepted 16 February 1999)

**Abstract**

The title compound, [Sn(C<sub>10</sub>H<sub>8</sub>NO)<sub>2</sub>], displays a unique geometry, looking like a two-bladed screw. The geometry about the Sn center is distorted pseudo-trigonal