

- Gerlach, G., Hönle, W. & Simon, A. (1982). *Z. Anorg. Allg. Chem.* **486**, 7–21.
- Ginzburg, A. G., Bokyi, N. G., Yanovsky, A. I., Struchkov, Yu. T., Setkina, V. N. & Kursanov, D. N. (1977). *J. Organomet. Chem.* **136**, 45–55.
- Harms, K. (1987). *XCAD-4. Programm zur Datenreduktion von CAD-4-Meßdaten*. Univ. Marburg, Deutschland.
- Hönle, W. & Simon, A. (1986). *Z. Naturforsch. Teil B*, **41**, 1391–1398.
- Keller, E. (1988). *SCHAKAL. Programm zur graphischen Repräsentation von molekularen und kristallographischen Modellen*. Univ. Freiburg, Deutschland.
- Khan, M. A., Peppe, C. & Tuck, D. G. (1984). *Can. J. Chem.* **62**, 601–605.
- Khan, M. A., Tuck, D. G., Taylor, M. J. & Rogers, D. A. (1986). *J. Crystallogr. Spectrosc. Res.* **16**, 895–905.
- McArdle, P. (1994). *J. Appl. Cryst.* **27**, 438–439.
- Müller, U. (1980). *Acta Cryst.* **B36**, 1075–1081.
- Müller, U., Klingelhöfer, P., Eicher, J. & Bohrer, R. (1984). *Z. Kristallogr.* **168**, 121–131.
- Müller, U. & Siekmann, J. (1995). *Acta Cryst.* In Vorbereitung.
- Peppe, C. & Tuck, D. G. (1984). *Can. J. Chem.* **62**, 2793–2797.
- Shamir, J., Luski, S., Bino, A., Cohen, S. & Gibson, D. (1985). *Inorg. Chem.* **24**, 2301–2309.
- Sheldrick, G. M. (1985). *SHELXS86. Programm zur Lösung von Kristallstrukturen*. Univ. Göttingen, Deutschland.
- Sheldrick, G. M. (1993). *SHELXL93. Programm zur Verfeinerung von Kristallstrukturen*. Univ. Göttingen, Deutschland.
- Staffel, T. & Meyer, G. (1987). *Z. Anorg. Allg. Chem.* **552**, 113–122.
- Tuck, D. G. (1993). *Chem. Soc. Rev.* pp. 269–276.
- Uhl, W. (1993). *Angew. Chem.* **105**, 1449–1461; *Angew. Chem. Int. Ed. Engl.* **32**, 1386–1397.
- Uhl, W., Layh, M. & Hiller, W. (1989). *J. Organomet. Chem.* **368**, 139–154.

Acta Cryst. (1995). **C51**, 1124–1125

Tetramethylammonium Dichlorotriphenylstannate

SEIK WENG NG

Institute of Advanced Studies, University of Malaya, 59100 Kuala Lumpur, Malaysia

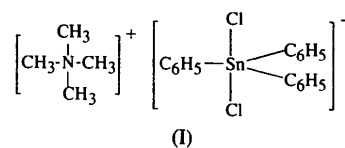
(Received 29 March 1994; accepted 16 December 1994)

Abstract

The crystal structure of tetramethylammonium dichlorotriphenylstannate, [(CH₃)₄N][SnCl₂(C₆H₅)₃], consists of non-interacting ammonium cations and *trans*-C₃SnCl₂ trigonal-bipyramidal stannate anions.

Comment

The Sn atom of the title compound (I) lies on the rotation axis and the Sn—Cl bond lengths are equal [Sn—Cl 2.598 (1) Å]; the bond is lengthened relative to the Sn—Cl bond in the parent triphenyltin chloride Lewis acid [Sn—Cl 2.354 (1) and 2.356 (1) Å (Tse, Lee & Gabe, 1986)].



The dichlorotriphenylstannate ion retains its stereochemical integrity in solution, as inferred from the solution NMR spectrum of the tetraethylammonium salt (Holecek, Nadvornik, Handlir & Lycka, 1983). The phenacyltriphenylarsonium salt shows two inequivalent Sn—Cl bond distances [2.573 (7) and 2.689 (6) Å (Harrison, Molloy, Phillips, Smith & Crowe, 1978)]. The dibromotriphenylstannate ion also displays *trans* trigonal-bipyramidal coordination at the Sn atom [Sn—Br 2.7512 (9) and 2.7908 (7) Å] in the tetraethylammonium salt (Wharf & Simard, 1991).

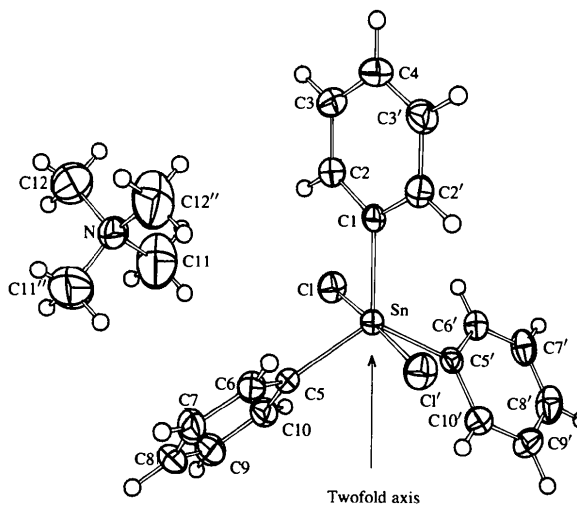


Fig. 1. Atomic labelling scheme for tetramethylammonium dichlorotriphenylstannate. Displacement ellipsoids are plotted at the 30% level.

Experimental

Tetramethylammonium chloride was heated with an equimolar amount of triphenyltin chloride in ethanol. Slow cooling of the filtered solution gave crystals of the title salt.

Crystal data

[(CH₃)₄N][SnCl₂(C₆H₅)₃]

M_r = 495.06

Monoclinic

*C*2

a = 14.648 (2) Å

b = 9.8802 (5) Å

c = 10.900 (1) Å

β = 134.984 (3)°

V = 1115.7 (2) Å³

Z = 2

D_x = 1.474 Mg m⁻³

Mo *Kα* radiation

λ = 0.71073 Å

Cell parameters from 25

reflections

θ = 10–12°

μ = 1.396 mm⁻¹

T = 300 K

Irregular

0.21 × 0.14 × 0.14 mm

(0.16 mm radius)

Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
 $\omega/2\theta$ scans
 Absorption correction: refined from ΔF (DIFABS; Walker & Stuart, 1983)
 $T_{\min} = 0.68$, $T_{\max} = 0.82$
 1093 measured reflections
 1007 independent reflections

1004 observed reflections [$I > 3\sigma(I)$]
 $R_{\text{int}} = 0.025$
 $\theta_{\max} = 25^\circ$
 $h = 0 \rightarrow 17$
 $k = 0 \rightarrow 11$
 $l = -12 \rightarrow 9$
 3 standard reflections
 frequency: 60 min
 intensity decay: none

Refinement

Refinement on F
 $R = 0.020$
 $wR = 0.025$
 $S = 0.389$
 1004 reflections
 119 parameters
 $w = 1/[\sigma^2(F) + 0.0004F^2 + 1]$

$(\Delta/\sigma)_{\max} = 0.01$
 $\Delta\rho_{\max} = 0.19(2) \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.07(2) \text{ e } \text{\AA}^{-3}$
 Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

	x	y	z	B_{eq}
Sn	0	1/4	0	2.445 (7)
Cl	0.23377 (7)	0.2433 (3)	0.1360 (1)	3.81 (2)
N	0	0.4305 (7)	-1/2	4.1 (2)
C1	0	0.4670 (7)	0	2.6 (1)
C2	0.0757 (4)	0.5402 (6)	-0.0102 (5)	3.3 (1)
C3	0.0748 (4)	0.6803 (6)	-0.0122 (5)	4.0 (1)
C4	0	0.751 (2)	0	4.4 (2)
C5	-0.0621 (3)	0.1451 (5)	-0.2199 (4)	2.6 (1)
C6	-0.1776 (4)	0.1804 (6)	-0.3865 (5)	3.2 (1)
C7	-0.2176 (4)	0.1150 (7)	-0.5311 (5)	4.1 (1)
C8	-0.1463 (4)	0.0117 (7)	-0.5128 (5)	4.7 (1)
C9	-0.0320 (4)	-0.0242 (6)	-0.3485 (5)	4.4 (1)
C10	0.0093 (4)	0.0428 (6)	-0.2051 (5)	3.7 (1)
C11	0.0915 (7)	0.344 (1)	-0.3486 (9)	9.9 (3)
C12	0.0621 (6)	0.5147 (9)	-0.5325 (8)	9.3 (2)

Table 2. Selected geometric parameters (\AA , $^\circ$)

Sn—C1	2.598 (1)	C3—C4	1.38 (1)
Sn—C1	2.144 (8)	C5—C6	1.392 (7)
Sn—C5	2.137 (5)	C5—C10	1.379 (8)
N—C11	1.46 (1)	C6—C7	1.392 (8)
N—C12	1.448 (9)	C7—C8	1.37 (1)
C1—C2	1.392 (7)	C8—C9	1.38 (1)
C2—C3	1.385 (8)	C9—C10	1.383 (8)
Cl—Sn—C1 ⁱ	177.1 (2)	Sn—C1—C2	121.3 (4)
Cl—Sn—C1	91.5 (1)	Sn—C1—C2 ⁱ	121.3 (4)
Cl—Sn—C5	88.4 (1)	C2—C1—C2	117.4 (7)
Cl—Sn—C5 ⁱ	90.2 (1)	C1—C2—C3	121.4 (6)
Cl ⁱ —Sn—C1	91.5 (1)	C2—C3—C4	120 (1)
Cl ⁱ —Sn—C5	90.2 (1)	C3—C4—C3	119 (2)
Cl ⁱ —Sn—C5 ⁱ	88.4 (1)	Sn—C5—C6	120.2 (4)
Cl ⁱ —Sn—C5 ^j	119.0 (1)	Sn—C5—C10	122.5 (4)
Cl—Sn—C5	119.0 (1)	C6—C5—C10	117.3 (5)
C5—Sn—C5 ⁱ	121.9 (3)	C5—C6—C7	120.7 (6)
C11—N—C11 ⁱⁱ	108 (1)	C6—C7—C8	120.9 (6)
C11—N—C12	107.1 (6)	C7—C8—C9	118.8 (6)
C11 ⁱⁱ —N—C12	107.1 (6)	C8—C9—C10	120.4 (6)
C12—N—C12 ⁱⁱ	110 (1)	C5—C10—C9	121.9 (6)

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

Non-H atoms were refined anisotropically. H atoms were generated ($C-H = 0.95 \text{ \AA}$, $B = 5 \text{ \AA}^2$) and were allowed to ride on the parent C atoms.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *MolEN* (Fair, 1990). Structure solution was by direct and Patterson methods. Structure refinement used *MolEN*. Molecular graphics were produced using *ORTEPII* (Johnson, 1976). Preparation of material for publication: *MolEN*.

The author thanks the University of Malaya (PJP 152/91) for supporting this work.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: TA1012). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
 Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis*. Enraf–Nonius, Delft, The Netherlands.
 Harrison, P. G., Molloy, K., Phillips, R. B., Smith, P. J. & Crowe, A. J. (1978). *J. Organomet. Chem.* **160**, 421–434.
 Holecek, J., Nadvornik, M., Handlir, K. & Lycka, A. (1983). *J. Organomet. Chem.* **241**, 177–184.
 Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
 Tse, J. S., Lee, F. L. & Gabe, E. J. (1986). *Acta Cryst.* **C42**, 1876–1878.
 Walker, N. & Stuart, D. (1983). *Acta Cryst.* **A39**, 158–166.
 Wharf, I. & Simard, M. G. (1991). *Acta Cryst.* **C47**, 1605–1609.

Acta Cryst. (1995). **C51**, 1125–1128

Bis(phthalocyaninato)erbium ($\alpha 1$ Phase)

GUIDO OSTENDORP, JAN-PETER WERNER
 AND HEINER HOMBORG

*Institut für Anorganische Chemie, Universität Kiel,
 Olshausenstraße 40, D-24098 Kiel, Germany*

(Received 5 October 1994; accepted 3 January 1995)

Abstract

In the title compound, $\alpha 1$ -bis(phthalocyaninato)erbium $\{\alpha 1\text{-[Er(Pc)}_2]\}$, where $\text{Pc} = \text{C}_{32}\text{H}_{16}\text{N}_8^{2-/-}$, Er^{III} is eightfold coordinated by the isoindole N atoms (N_{iso}) of the two staggered (41.4°) saucer-shaped Pc ligands. The Er— N_{iso} distance is $2.41(1) \text{ \AA}$; the height of the slightly distorted ErN_8 square antiprism is 2.74 \AA . The $[\text{Er(Pc)}_2]$ molecules are packed in columns parallel to the tetragonal c axis. In this new tetragonal $\alpha 1$ phase, the disposition of the metal ion is ordered and the Pc rings are structurally equivalent.