

Sandra Mandolesi,^a Markus Schürmann,^b Hans Preut^{b*} and Terence Mitchell^b^aDepartamento de Química, Universidad Nacional del Sur, Avda. Alem 1253, 8000 Bahía Blanca, Argentina, and ^bFachbereich Chemie, Universität Dortmund, Otto-Hahn-Str. 6, 44221 Dortmund, Germany

Correspondence e-mail: uch002@uxp1.hrz.uni-dortmund.de

Key indicators

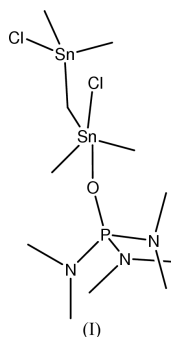
Single-crystal X-ray study
T = 291 K
Mean $\sigma(\text{N}-\text{C}) = 0.004 \text{ \AA}$
R factor = 0.027
wR factor = 0.063
Data-to-parameter ratio = 25.2For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

A 1:1 adduct between bis(chlorodimethylstannyl)methane and hexamethylphosphoric acid triamide (HMPA)

In the title compound, $[\text{Sn}_2(\text{CH}_2)(\text{CH}_3)_4\text{Cl}_2] \cdot \text{C}_6\text{H}_{18}\text{N}_3\text{OP}$, the molecule lies on a mirror plane. The two pentacoordinated Sn atoms are part of a planar four-membered $-\text{C}-\text{Sn} \cdots \text{Cl}-\text{Sn}-$ ring [Sn—C 2.103 (4) and 2.093 (4), Sn—Cl 2.6333 (11) and Sn \cdots Cl 3.0369 (12) Å; Sn—C—Sn 115.8 (2) $^\circ$].

Comment

Although bis(chlorodimethylstannyl)methane is a potential acceptor for either one or two monodentate ligands, only 1:1 adducts have so far been described. Two types of coordination are known: either both Sn atoms undergo bonding in such a way that the ligand bridges the two Sn atoms, as in the case of dimethyl sulfoxide (Mandolesi *et al.*, 2001), both Sn atoms thus increasing their coordination number to five, or only one Sn atom undergoes coordination, as in the case of pyridine (Austin *et al.*, 1987). In the title compound, HMPA bonds *via* O to give a structure completely analogous to the 1:1 adduct formed by the nitrogen donor pyridine. The ligand is complexed to one Sn atom site to form an almost perfect trigonal bipyramid. Cl1 does, however, interact with Sn2, the distance between these atoms being 3.0369 (12) Å [pyridine: 3.009 (3) Å], so that Sn2 also has a distorted trigonal bipyramidal geometry (angle Cl1—Sn2—Cl2 176.98 (4) $^\circ$ [pyridine 176.3 (1) $^\circ$]). The Sn—Cl bond lengths are very different {Sn2—Cl2 2.4403 (12) Å [pyridine 2.468 (2) Å] and Sn1—Cl1 2.6333 (11) Å [pyridine 2.638 (3) Å]}. A further similarity between (I) and the pyridine adduct is the bond angle O1—Sn1—Cl1 [176.27 (8) $^\circ$ in (I); 175.4 (2) $^\circ$ in pyridine]. Atoms Sn1, Cl1, C3, Sn2, Cl2, O1, P1 and N1 lie on a mirror plane.



Experimental

0.3 g (0.73 mmol) of 2,2-bis(chlorodimethylstannyl)propane (Austin *et al.*, 1987; Karol *et al.*, 1983) was dissolved in 2 ml of dry HMPA and the mixture stirred for 30 min. The solution was left overnight at 287–288 K. The crystals were separated and dried very carefully to remove the solvent from their surface; m.p. 407–410 K, yield 56%.

Crystal data

[Sn₂(CH₂)(CH₃)₄Cl₂]·C₆H₁₈N₃OP
M_r = 561.65
 Orthorhombic, *Pnma*
a = 12.7116 (3) Å
b = 12.4934 (3) Å
c = 14.3478 (3) Å
V = 2278.59 (9) Å³
Z = 4
D_x = 1.637 Mg m⁻³

Mo *K*α radiation
 Cell parameters from 14378 reflections
 θ = 3.2–27.5°
 μ = 2.50 mm⁻¹
T = 291 (1) K
 Block, colourless
 0.19 × 0.13 × 0.13 mm

Data collection

Nonius KappaCCD diffractometer
 ω scans
 Absorption correction: none
 14378 measured reflections
 2727 independent reflections
 1915 reflections with *I* > 2σ(*I*)

*R*_{int} = 0.031
 θ_{\max} = 27.5°
h = -16 → 16
k = -16 → 16
l = -18 → 18

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.028
wR(*F*²) = 0.063
S = 0.96
 2727 reflections
 108 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0298P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.40 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.40 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (Å, °).

| | | | |
|-------------------------|-------------|-------------------------|-------------|
| Sn1—C3 | 2.103 (4) | Sn2—C3 | 2.093 (4) |
| Sn1—C1 | 2.104 (4) | Sn2—Cl2 | 2.4403 (12) |
| Sn1—O1 | 2.220 (3) | Sn2—Cl1 | 3.0369 (12) |
| Sn1—Cl1 | 2.6333 (11) | | |
| Sn2—C2 | 2.092 (5) | | |
| C3—Sn1—C1 | 119.27 (15) | O1—Sn1—Cl1 | 176.27 (8) |
| C1 ⁱ —Sn1—C1 | 121.4 (3) | C2 ⁱ —Sn2—C2 | 118.9 (4) |
| C3—Sn1—O1 | 87.84 (13) | C2—Sn2—C3 | 117.9 (2) |
| C1—Sn1—O1 | 89.81 (12) | C2—Sn2—Cl2 | 97.17 (14) |
| C3—Sn1—Cl1 | 88.42 (12) | C3—Sn2—Cl2 | 98.54 (12) |
| C1—Sn1—Cl1 | 92.02 (12) | Sn2—C3—Sn1 | 115.8 (2) |

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

H atoms were placed in calculated positions, with *U*_{iso} constrained to be 1.5*U*_{eq} of the carrier atom for the methyl-H and 1.2*U*_{eq} for the remaining H atoms. The methyl groups were allowed to rotate but not to tip.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97*, *PARST95* (Nardelli, 1995), *PLATON* (Spek, 2001).

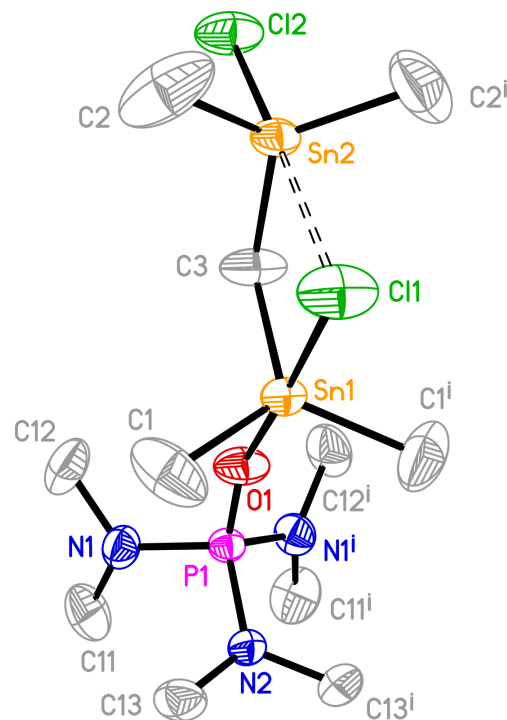


Figure 1

View of the title compound, showing the labelling scheme. Displacement ellipsoids are shown at the 30% probability level. H atoms have been omitted. [Symmetry code: (i) $x, \frac{1}{2} - y, z$]

SM thanks the Deutsche Akademische Austauschdienst DAAD for the award of a scholarship

References

- Austin, M., Gebreyes, K., Kuivila, H. G., Swami, K. & Zubieta, J. A. (1987). *Organometallics*, **6**, 834–842.
 Karol, T., Hutchinson, J., Hyde, J., Kuivila, H. G. & Zubieta, J. (1983). *Organometallics*, **2**, 106–114.
 Mandolesi, S., Studentkowski, M., Preut, H. & Mitchell, T. N. (2001). *Acta Cryst. E57*, m543–m544.
 Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
 Nonius (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
 Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr and R. M. Sweet, pp. 307–326. New York: Academic Press.
 Sheldrick, G. M. (1990). *Acta Cryst. A46*, 467–473.
 Sheldrick, G. M. (1991). *SHELXTL-Plus*. Release 4.1 Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
 Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
 Spek, A. L. (2001). *PLATON*. University of Utrecht, The Netherlands.