

Collection des données

Diffractomètre Enraf-Nonius CAD-4	$\theta_{\max} = 26^\circ$
Balayage θ -2 θ	$h = -9 \rightarrow 8$
Pas de correction d'absorption	$k = 0 \rightarrow 10$
1897 réflexions mesurées	$l = -10 \rightarrow 10$
1897 réflexions indépendantes	3 réflexions de référence
1654 réflexions observées [$I \geq 3\sigma(I)$]	fréquence: 120 min variation d'intensité: 4,4% (correction de décroissance)

Affinement

Affinement basé sur les F	$\Delta\rho_{\max} = 0,39$ (7) e \AA^{-3}
$R = 0,023$	$\Delta\rho_{\min} = -0,44$ (7) e \AA^{-3}
$wR = 0,029$	Correction d'extinction:
$S = 1,17$	Stout & Jensen (1968)
1654 réflexions	Coefficient d'extinction:
101 paramètres	$3,5$ (4) $\times 10^{-7}$
Les paramètres des atomes d'hydrogène en position théorique	Facteurs de diffusion des <i>International Tables for</i> <i>X-ray Crystallography</i> (1974, Tome IV)
$w = 1/(1 + 0,01F)$	
$(\Delta/\sigma)_{\max} < 0,01$	

Tableau 1. Coordonnées atomiques et facteurs d'agitation thermique isotrope équivalents (\AA^2)

$$U_{\text{éq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	$U_{\text{éq}}$
Pd	0	0	0	0,02983 (6)
Cl(1)	0,2450 (1)	0,30511 (9)	0,0112 (1)	0,0406 (2)
Cl(2)	0,2165 (1)	-0,0591 (1)	0,1419 (1)	0,0458 (2)
S	0,1129 (1)	-0,2365 (1)	0,6690 (1)	0,0503 (2)
N(12)	0,0308 (4)	-0,5200 (4)	0,7977 (4)	0,0603 (8)
N(13)	0,3553 (4)	-0,3116 (4)	0,8402 (3)	0,0480 (6)
C(1)	0,3310 (4)	-0,0129 (4)	0,6940 (4)	0,0397 (6)
C(2)	0,2807 (4)	0,1039 (4)	0,5984 (4)	0,0410 (6)
C(3)	0,4584 (4)	0,2920 (4)	0,6012 (4)	0,0413 (8)
C(4)	0,4111 (4)	0,4062 (4)	0,5000 (4)	0,0400 (6)
C(11)	0,1764 (4)	-0,3625 (4)	0,7807 (4)	0,0407 (6)

Tableau 2. Distances interatomiques (\AA), angles des liaisons ($^\circ$) et angles de torsion ($^\circ$), longueurs et angles des liaisons hydrogène (\AA , $^\circ$)

Pd—Cl(1)	2,3246 (6)	N(13)—C(11)	1,304 (4)
Pd—Cl(2)	2,3029 (9)	C(1)—C(2)	1,525 (5)
S—C(1)	1,804 (2)	C(2)—C(3)	1,522 (4)
S—C(11)	1,731 (4)	C(3)—C(4)	1,528 (5)
N(12)—C(11)	1,310 (4)	C(4)—C(4')	1,526 (4)
Cl(1)—Pd—Cl(2)	90,04 (3)	C(3)—C(4)—C(4')	112,3 (3)
C(1)—S—C(11)	104,8 (1)	S—C(11)—N(12)	115,1 (3)
S—C(1)—C(2)	106,6 (2)	S—C(11)—N(13)	123,7 (2)
C(1)—C(2)—C(3)	111,5 (3)	N(12)—C(11)—N(13)	121,2 (4)
C(2)—C(3)—C(4)	111,8 (3)		
C(11)—S—C(1)—C(2)	-179,5 (2)	S—C(1)—C(2)—C(3)	-176,0 (2)
C(1)—S—C(11)—N(12)	170,5 (3)	C(1)—C(2)—C(3)—C(4)	177,6 (3)
C(1)—S—C(11)—N(13)	-11,9 (3)	C(2)—C(3)—C(4)—C(4')	-179,2 (3)

D—H...A	D...A	D—H...A
N(12)—H(12)...Cl(1 ^v)	3,314 (4)	151
N(13)—H(13')...Cl(1 ⁱⁱⁱ)	3,274 (3)	115
N(12)—H(12)...Cl(2 ^{iv})	3,234 (3)	124
N(13)—H(13)...Cl(2 ⁱⁱⁱ)	3,300 (2)	166

Codes de symétrie: (i) $1 - x, 1 - y, 1 - z$; (iii) $1 - x, -y, 1 - z$;
(iv) $-x, -1 - y, 1 - z$; (v) $x, y - 1, 1 + z$.

La structure a été résolue par la méthode de l'atome lourd et la Fig. 1 réalisée avec le programme ORTEPII (Johnson, 1976). Tous les programmes utilisés appartiennent au système SDP (B. A. Frenz & Associates, Inc., 1982).

Les listes des facteurs de structure, des facteurs d'agitation thermique anisotrope, des coordonnées des atomes d'hydrogène, des distances des atomes d'hydrogène et des plans moyens ont été déposées au dépôt d'archives de l'UICr (Référence: PA1105). On peut en obtenir des copies en s'adressant à: The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, Angleterre.

Références

- B. A. Frenz & Associates, Inc. (1982). *SDP Structure Determination Package*. College Station, Texas, EU.
- Johnson, C. K. (1976). *ORTEPII*. Rapport ORNL-5138. Oak Ridge National Laboratory, Tennessee, EU.
- Kind, R., Plesko, S., Arend, H., Blinc, R., Zeks, B., Seliger, J., Lozar, B., Slak, J., Levstik, A., Filipic, C., Zagar, V., Lahajnar, G., Milia, F. & Chapuis, G. (1979). *J. Chem. Phys.* **71**, 2118–2130.
- Nguyen-Huy, D., Viossat, B. & Lancelot, J. C. (1994). *Acta Cryst.* **C50**, 1434–1437.
- Stout, G. H. & Jensen, L. H. (1968). *X-ray Structure Determination*, pp. 410–412. Londres: MacMillan.
- Viossat, B., Nguyen-Huy, D. & Lancelot, J. C. (1994). *Acta Cryst.* **C50**, 362–364.

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Triphenyltin Iodide

SEIK WENG NG

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

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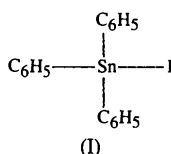
Abstract

The geometry at the Sn atom in iodotriphenyltin, $[\text{SnI}(\text{C}_6\text{H}_5)_3]$, is distorted tetrahedral.

Comment

The title structure completes the series of triphenyltin halide structures. Triphenyltin fluoride forms a polymeric chain structure [Sn—F 2.1458 (3) \AA] in which the five-coordinate Sn atom shows trigonal bipyramidal coordination (Tudela, Gutierrez-Puebla & Monge, 1992). Both triphenyltin chloride [Sn—Cl 2.354 (1) and 2.356 (1) \AA] (Tse, Lee & Gabe, 1986) and triphenyltin bromide [Sn—Br 2.495 (2) \AA] (Preut & Huber, 1979) adopt monomeric tetrahedral configurations. The iodide

[Sn—I 2.7081 (6) Å] also exists as a tetrahedral molecule and short intermolecular contacts are absent in the crystal structure. Bond dimensions in the present triphenyltin iodide, (I), do not differ significantly from those found in another triclinic modification [Sn—I 2.6916 (8) and 2.7060 (8) Å] in which there are two independent tetrahedral molecules in the unit cell (Simard & Wharf, 1994).



Although triphenyltin chloride forms a plethora of complexes with donor ligands, only a few complexes of the iodide have been reported and none has yet been structurally verified. The methyl-substituted iodide, tris(*p*-tolyl)tin iodide, forms a 1/2 adduct with 4,4'-bipyridine that displays an Sn—I bond distance of 2.830 (1) Å (Bajue, Bramwell, Charles, Cervantes-Lee & Pannell, 1992).

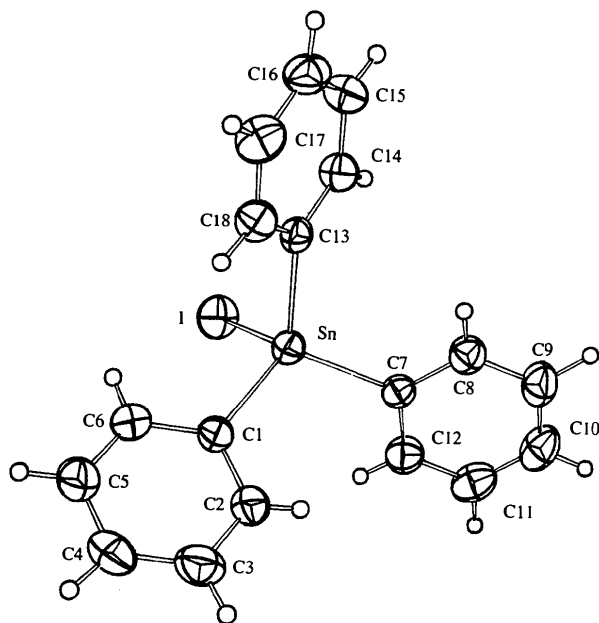


Fig. 1. ORTEP (Johnson, 1976) plot of triphenyltin iodide with displacement ellipsoids at the 30% probability level.

Experimental

Triphenyltin nicotinate (Ng, Kumar Das, van Meurs, Schagen & Straver, 1989) was treated with an excess of methyl iodide in ethanol in an attempt to synthesize the *N*-methylated nicotinate. Slow cooling of the filtered solution gave triphenyltin iodide instead. The compound is stable to light despite the presence of an Sn—I bond.

Crystal data

[Sn(C₆H₅)₃]
M_r = 476.91
 Triclinic
*P*1
a = 9.6093 (9) Å
b = 9.606 (1) Å
c = 10.118 (1) Å
 α = 81.581 (9)°
 β = 77.02 (1)°
 γ = 72.97 (1)°
V = 866.9 (2) Å³
Z = 2
D_x = 1.827 Mg m⁻³

Mo K α radiation

λ = 0.71073 Å
 Cell parameters from 25 reflections
 θ = 10–12°
 μ = 3.230 mm⁻¹
T = 300 K
 Irregular
 0.28 × 0.22 × 0.14 mm
 (0.13 mm radius)
 Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
 $\omega/2\theta$ scans
 Absorption correction: semi-empirical (North, Phillips & Mathews, 1968)
 T_{\min} = 0.9433, T_{\max} = 0.9994
 3250 measured reflections
 3053 independent reflections

2326 observed reflections [$I > 3\sigma(I)$]
 R_{int} = 0.009
 θ_{\max} = 25°
 h = 0 → 11
 k = -10 → 11
 l = -11 → 12
 3 standard reflections frequency: 60 min intensity decay: none

Refinement

Refinement on *F*
 R = 0.028
 wR = 0.030
 S = 0.403
 2326 reflections
 241 parameters
 H atoms refined isotropically
 $w = 1/[\sigma^2(F) + 0.0004F^2 + 1]$

$(\Delta/\sigma)_{\max}$ = 0.01
 $\Delta\rho_{\max}$ = 0.70 (5) e Å⁻³
 $\Delta\rho_{\min}$ = -0.14 (5) e Å⁻³
 Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

$$B_{\text{eq}} = (4/3)\sum_i \sum_j \beta_{ij} a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B_{eq}</i>
I	0.74887 (6)	0.64772 (5)	0.92321 (5)	7.49 (1)
Sn	0.61920 (4)	0.82667 (4)	0.72634 (4)	4.567 (8)
C1	0.3866 (6)	0.8640 (5)	0.7915 (5)	4.7 (1)
C2	0.2928 (7)	1.0029 (6)	0.7784 (6)	6.0 (2)
C3	0.1419 (7)	1.0236 (8)	0.8091 (7)	7.5 (2)
C4	0.0806 (7)	0.9109 (7)	0.8538 (6)	6.8 (2)
C5	0.1717 (7)	0.7735 (7)	0.8701 (7)	7.5 (2)
C6	0.3235 (7)	0.7501 (6)	0.8391 (7)	6.4 (2)
C7	0.6902 (5)	1.0191 (5)	0.7010 (5)	4.5 (1)
C8	0.7620 (6)	1.0636 (6)	0.5747 (6)	5.6 (1)
C9	0.8005 (6)	1.1935 (6)	0.5497 (7)	6.7 (2)
C10	0.7689 (7)	1.2792 (6)	0.6541 (8)	7.4 (2)
C11	0.6998 (7)	1.2377 (6)	0.7815 (7)	7.3 (2)
C12	0.6604 (7)	1.1075 (6)	0.8048 (6)	6.1 (2)
C13	0.6940 (5)	0.7114 (5)	0.5486 (5)	4.4 (1)
C14	0.8392 (6)	0.6741 (6)	0.4848 (6)	5.3 (1)
C15	0.8856 (7)	0.6011 (7)	0.3697 (6)	6.2 (2)
C16	0.7836 (7)	0.5660 (7)	0.3165 (6)	6.4 (2)
C17	0.6385 (7)	0.6020 (7)	0.3771 (6)	6.6 (2)
C18	0.5933 (6)	0.6745 (6)	0.4926 (6)	5.6 (1)

Table 2. Selected geometric parameters (Å, °)

I—Sn	2.7081 (6)	C7—C12	1.375 (8)
Sn—C1	2.120 (5)	C8—C9	1.376 (9)
Sn—C7	2.114 (5)	C9—C10	1.36 (1)
Sn—C13	2.125 (5)	C10—C11	1.37 (1)
C1—C2	1.381 (8)	C11—C12	1.38 (1)
C1—C6	1.378 (8)	C13—C14	1.365 (8)
C2—C3	1.37 (1)	C13—C18	1.375 (8)
C3—C4	1.35 (1)	C14—C15	1.368 (8)
C4—C5	1.36 (1)	C15—C16	1.36 (1)
C5—C6	1.38 (1)	C16—C17	1.35 (1)
C7—C8	1.381 (8)	C17—C18	1.369 (9)
I—Sn—C1	107.3 (1)	Sn—C7—C12	122.2 (5)
I—Sn—C7	106.9 (1)	C8—C7—C12	117.9 (6)
I—Sn—C13	106.2 (1)	C7—C8—C9	122.3 (7)
C1—Sn—C7	113.3 (2)	C8—C9—C10	118.5 (7)
C1—Sn—C13	110.5 (2)	C9—C10—C11	121.0 (7)
C7—Sn—C13	112.1 (2)	C10—C11—C12	119.8 (7)
Sn—C1—C2	120.7 (4)	C7—C12—C11	120.4 (7)
Sn—C1—C6	121.4 (4)	Sn—C13—C14	122.6 (4)
C2—C1—C6	117.8 (6)	Sn—C13—C18	119.5 (4)
C1—C2—C3	120.1 (7)	C14—C13—C18	117.8 (5)
C2—C3—C4	121.8 (7)	C13—C14—C15	122.0 (6)
C3—C4—C5	118.8 (7)	C14—C15—C16	118.9 (7)
C4—C5—C6	120.3 (7)	C15—C16—C17	120.5 (7)
C1—C6—C5	121.1 (7)	C16—C17—C18	120.1 (7)
Sn—C7—C8	119.8 (4)	C13—C18—C17	120.6 (6)

Direct phase determination gave the Sn and I atoms, and the other non-H atoms were located from the difference Fourier syntheses. Non-H atoms were refined anisotropically and H atoms were located and refined with $B = 5 \text{ \AA}^2$.

Data collection: CAD-4 VAX/PC Fortran System (Enraf-Nonius, 1988). Cell refinement: CAD-4 VAX/PC Fortran System. Data reduction: MolEN (Fair, 1990). Structure refinement: MolEN. Molecular graphics: ORTEPII (Johnson, 1976). Preparation of material for publication: MolEN.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, including H-atom geometry, have been deposited with the IUCr (Reference: TA1013). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Bauje, S. A., Bramwell, F. B., Charles, M., Cervantes-Lee, F. & Pannell, K. (1992). *Inorg. Chim. Acta*, **197**, 83–87.
- Enraf-Nonius (1988). CAD-4 VAX/PC Fortran System. *Operator's Guide to the Enraf-Nonius CAD-4 Diffractometer Hardware, its Software and the Operating System*. Enraf-Nonius, Delft, The Netherlands.
- Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis*. Enraf-Nonius, Delft, The Netherlands.
- Johnson, C. K. (1976). ORTEPII. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- Ng, S. W., Kumar Das, V. G., van Meurs, F., Schagen, J. D. & Straver, L. H. (1989). *Acta Cryst.* **C45**, 570–572.
- North, A. C. T., Phillips, D. C. & Mathews, F. W. (1968). *Acta Cryst.* **A24**, 351–358.
- Preut, H. & Huber, F. (1979). *Acta Cryst.* **B35**, 744–746.
- Simard, M. G. & Wharf, I. (1994). *Acta Cryst.* **C50**, 397–403.
- Tse, J. S., Lee, F. L. & Gabe, E. J. (1986). *Acta Cryst.* **C42**, 1876–1878.
- Tudela, D., Gutierrez-Puebla, E. & Monge, A. (1992). *J. Chem. Soc. Dalton Trans.* pp. 1069–1071.

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{N-(1-Adamantyl)[(pentafluoro-2-propenyl)thio]amino}(fluoro)bis[2,4,6-tris(trifluoromethyl)phenyl]tin at 153 K

STEFANIE FREITAG, REGINE HERBST-IRMER,
JENS T. AHLEMANN AND HERBERT W. ROESKY

*Institut für Anorganische Chemie, Universität
Göttingen, Tammannstrasse 4, 37077 Göttingen,
Germany*

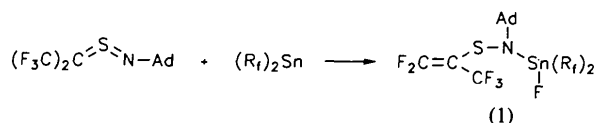
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Abstract

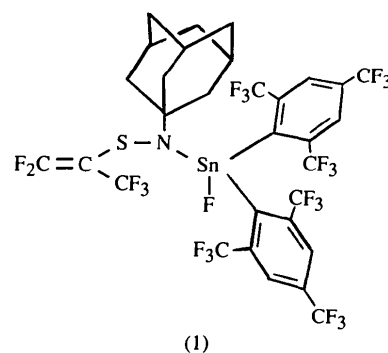
The synthesis and structure of the title compound (adamantane is tricyclo[3.3.1.1^{3,7}]decane), $[\text{SnF}(\text{C}_9\text{H}_2\text{F}_9)_2\{\text{C}_6\text{F}_3\text{S}(\text{C}_{10}\text{H}_{15}\text{N})\}]$, (1), are reported. (1) crystallizes as a monomer with short Sn...F interactions.

Comment

The title compound, (1), was synthesized by the reaction of hexafluoro-2-propanethione 1-adamantylimide with bis[2,4,6-tris(trifluoromethyl)phenyl]stannylene (Ahlemann, 1992):



$\text{R}_f = 2,4,6\text{-Tris(trifluoromethyl)phenyl}$
Ad = Adamantyl



The Sn atom has a distorted tetrahedral environment. The Sn1—N1 [2.037(5) Å] and Sn1—F6 [1.938(3) Å] bonds are typical single bonds (Blom & Haarland, 1985). As a result of the different oxidation state of Sn, the Sn1—C distances [2.179(6) and 2.187(5) Å] are about 0.10 Å shorter than in bis[2,4,6-tris(trifluoromethyl)phenyl]stannylene [2.278(5) and 2.284(5) Å; Grützmacher, Pritzkow & Edelmann,