

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

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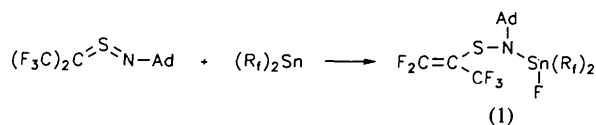
(Received 22 June 1994; accepted 24 October 1994)

Abstract

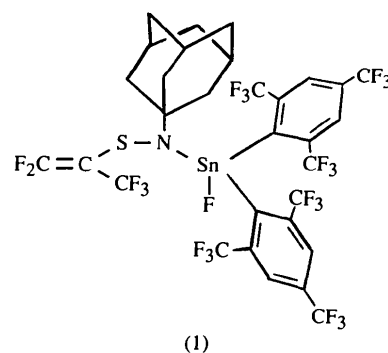
The synthesis and structure of the title compound (adamantane is tricyclo[3.3.1.1^{3,7}]decane), $[\text{SnF}(\text{C}_9\text{H}_2\text{F}_9)_2\{\text{C}_3\text{F}_5\text{S}(\text{C}_{10}\text{H}_{15}\text{N})\}]$, (1), are reported. (1) crystallizes as a monomer with short $\text{Sn} \cdots \text{F}$ 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,

1991]. In comparison with typical C—S single bonds (1.83 Å; Rademacher, 1987) and the C=S double bond in hexafluoro-2-propanethione 1-adamantylimide [1.646(1) Å; May, Roesky, Stalke, Pauer & Sheldrick, 1990], the C1—S1 distance in (1) [1.760(6) Å] is a short single bond. Bis[2,4,6-tris(trifluoromethyl)phenyl]stannylene crystallizes as a monomer stabilized by four intramolecular Sn···F contacts [2.807(4), 2.681(4), 2.663(4) and 2.833(4) Å]. Similar short Sn···F interactions were found in the title compound [Sn1···F8 2.942(3), Sn1···F13 2.814(3), Sn1···F17 2.713(3) and Sn1···F22 2.951(3) Å].

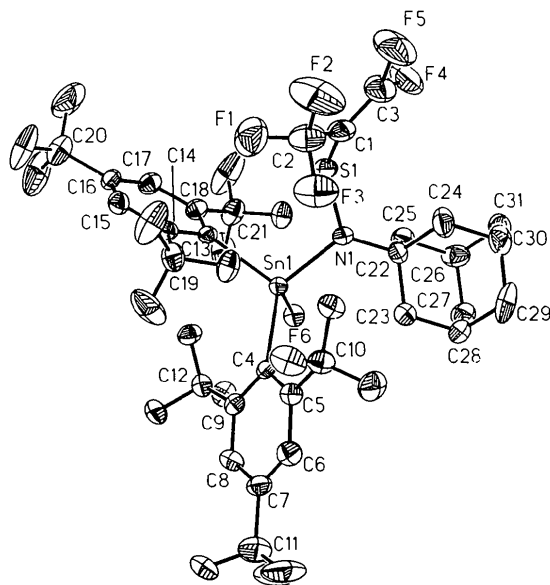


Fig. 1. Structure of (1) showing 50% probability displacement ellipsoids. The H atoms and some of the F-atom labels have been omitted for clarity.

Profile data from $\omega/2\theta$ scans
Absorption correction:
refined from ΔF
 $T_{\min} = 0.73$, $T_{\max} = 0.93$
5172 measured reflections
4632 independent reflections
3627 observed reflections
[$I > 2\sigma(I)$]

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.0422$
 $wR(F^2) = 0.1022$
 $S = 1.143$
4631 reflections
533 parameters
 $w = 1/[\sigma^2(F_o^2) + (0.0338P)^2 + 15.3065P]$
where $P = (F_o^2 + 2F_c^2)/3$

$h = -15 \rightarrow 15$
 $k = -10 \rightarrow 14$
 $l = -19 \rightarrow 21$
3 standard reflections
frequency: 90 min
intensity decay: none

$(\Delta/\sigma)_{\max} = 0.004$
 $\Delta\rho_{\max} = 1.684 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.543 \text{ e } \text{Å}^{-3}$
Extinction correction: none
Atomic scattering factors
from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	U_{eq}
S1	0.33721 (12)	0.31746 (12)	0.04144 (8)	0.0327 (4)
C1	0.4118 (5)	0.3223 (5)	-0.0168 (3)	0.038 (2)
C2	0.5197 (6)	0.3378 (6)	0.0114 (4)	0.055 (2)
F1	0.5410 (3)	0.4128 (4)	0.0589 (3)	0.0791 (15)
F2	0.5637 (4)	0.3580 (5)	-0.0387 (3)	0.102 (2)
F3	0.5649 (3)	0.2585 (4)	0.0469 (3)	0.0713 (14)
C3	0.3662 (6)	0.3241 (6)	-0.0865 (4)	0.055 (2)
F4	0.2699 (4)	0.3171 (4)	-0.1147 (2)	0.0717 (14)
F5	0.4062 (4)	0.3394 (5)	-0.1378 (2)	0.093 (2)
Sn1	0.40978 (3)	0.23338 (3)	0.19549 (2)	0.02346 (14)
F6	0.2985 (2)	0.1933 (2)	0.2282 (2)	0.0304 (8)
C4	0.5065 (4)	0.1360 (4)	0.2760 (3)	0.0231 (13)
C5	0.5691 (4)	0.0624 (4)	0.2628 (3)	0.0260 (14)
C6	0.6139 (4)	-0.0093 (5)	0.3142 (3)	0.033 (2)
C7	0.6002 (5)	-0.0057 (5)	0.3811 (3)	0.034 (2)
C8	0.5442 (4)	0.0688 (5)	0.3976 (3)	0.033 (2)
C9	0.4984 (4)	0.1391 (4)	0.3468 (3)	0.0256 (14)
C10	0.6006 (4)	0.0560 (5)	0.1961 (3)	0.033 (2)
F7	0.5983 (3)	-0.0368 (3)	0.1715 (2)	0.0507 (11)
F8	0.5484 (2)	0.1132 (3)	0.1425 (2)	0.0352 (9)
F9	0.6947 (3)	0.0877 (3)	0.2092 (2)	0.0464 (10)
C11	0.6499 (5)	-0.0809 (6)	0.4383 (4)	0.052 (2)
F10†	0.7038 (6)	-0.1470 (5)	0.4155 (3)	0.077 (2)
F11†	0.5835 (5)	-0.1369 (6)	0.4562 (4)	0.091 (3)
F12†	0.7041 (8)	-0.0405 (5)	0.4949 (4)	0.111 (4)
F10'†	0.623 (3)	-0.069 (3)	0.495 (2)	0.077 (2)
F11'†	0.745 (2)	-0.056 (3)	0.456 (2)	0.091 (3)
F12'†	0.646 (4)	-0.173 (2)	0.421 (2)	0.111 (4)
C12	0.4431 (4)	0.2184 (5)	0.3743 (3)	0.0318 (15)
F13	0.4302 (3)	0.3025 (2)	0.3356 (2)	0.0373 (9)
F14	0.3556 (3)	0.1898 (3)	0.3792 (2)	0.0387 (9)
F15	0.4942 (3)	0.2456 (3)	0.4415 (2)	0.0414 (9)
C13	0.4293 (4)	0.3947 (4)	0.2120 (3)	0.0229 (13)
C14	0.5246 (4)	0.4357 (4)	0.2317 (3)	0.0257 (14)
C15	0.5414 (4)	0.5355 (5)	0.2507 (3)	0.0311 (15)
C16	0.4637 (4)	0.5980 (4)	0.2493 (3)	0.0279 (14)
C17	0.3691 (5)	0.5608 (5)	0.2293 (3)	0.034 (2)
C18	0.3523 (4)	0.4608 (4)	0.2103 (3)	0.0290 (14)
C19	0.6143 (5)	0.3739 (5)	0.2356 (4)	0.037 (2)
F16	0.6794 (3)	0.4243 (3)	0.2115 (3)	0.0625 (13)
F17	0.5930 (2)	0.2914 (3)	0.1946 (2)	0.0381 (9)
F18	0.6587 (3)	0.3430 (4)	0.3007 (2)	0.0717 (15)
C20	0.4797 (6)	0.7069 (5)	0.2695 (4)	0.048 (2)
F19	0.5734 (3)	0.7326 (3)	0.2847 (3)	0.0740 (15)
F20	0.4490 (4)	0.7296 (4)	0.3252 (3)	0.085 (2)
F21	0.4325 (4)	0.7669 (3)	0.2169 (3)	0.089 (2)

C21	0.2461 (5)	0.4269 (5)	0.1914 (4)	0.041 (2)
F22	0.2263 (2)	0.3512 (3)	0.1459 (2)	0.0374 (9)
F23	0.2225 (3)	0.3960 (4)	0.2493 (2)	0.0721 (15)
F24	0.1854 (3)	0.4989 (3)	0.1640 (4)	0.098 (2)
N1	0.3632 (3)	0.2081 (3)	0.0878 (2)	0.0264 (12)
C22	0.2979 (4)	0.1201 (4)	0.0584 (3)	0.0293 (14)
C23	0.3286 (5)	0.0310 (5)	0.1093 (3)	0.037 (2)
C24	0.3076 (5)	0.0914 (5)	-0.0150 (3)	0.045 (2)
C25	0.1893 (4)	0.1446 (5)	0.0514 (4)	0.037 (2)
C26	0.1250 (5)	0.0537 (5)	0.0217 (4)	0.045 (2)
C27	0.1569 (5)	-0.0353 (5)	0.0714 (4)	0.045 (2)
C28	0.2638 (5)	-0.0603 (5)	0.0808 (4)	0.041 (2)
C29	0.2752 (6)	-0.0879 (6)	0.0063 (5)	0.065 (2)
C30	0.2400 (6)	-0.0004 (6)	-0.0456 (4)	0.052 (2)
C31	0.1345 (5)	0.0263 (6)	-0.0508 (4)	0.048 (2)

† Occupancy 0.85.

‡ Occupancy 0.15.

Table 2. Selected geometric parameters (Å, °)

Sn1—N1	1.698 (5)	Sn1—N1	2.037 (5)
Sn1—C1	1.760 (6)	Sn1—C13	2.179 (6)
C1—C3	1.324 (10)	Sn1—C4	2.187 (5)
C1—C2	1.483 (10)	N1—C22	1.501 (7)
Sn1—F6	1.938 (3)		
N1—S1—C1	107.2 (3)	F6—Sn1—C4	90.1 (2)
C3—C1—C2	121.6 (6)	N1—Sn1—C4	126.2 (2)
C3—C1—S1	117.0 (6)	C13—Sn1—C4	116.9 (2)
C2—C1—S1	120.8 (5)	C22—N1—S1	116.9 (4)
F6—Sn1—N1	103.8 (2)	C22—N1—Sn1	120.2 (3)
F6—Sn1—C13	107.7 (2)	S1—N1—Sn1	111.2 (3)
N1—Sn1—C13	107.7 (2)		

The *para*-trifluoromethyl group at the C4—C9 phenyl ring was found to be disordered. Two positions for each F atom were refined to occupancies of 0.85 and 0.15. Distance restraints for 1,2 and 1,3 distances were applied. The *U* values of F atoms opposite each other were constrained to be equal. H atoms were included in calculated positions and refined using a riding model. The *U* value of each H atom was fixed to 1.2*U*_{eq} of the corresponding C atom.

Data collection: *DIF4* (Stoe & Cie, 1988a). Cell refinement: *DIF4*. Data reduction: *REDU4* (Stoe & Cie, 1988b). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991). Software used to prepare material for publication: *SHELXL93*.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: NA1120). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Ahlemann, J. T. (1992). Diplomarbeit, Univ. of Göttingen, Germany.
 Blom, R. & Haarland, A. (1985). *J. Mol. Struct.* **128**, 21–27.
 Grützmacher, H., Pritzkow, H. & Edelman, F. T. (1991). *Organometallics*, **10**, 23–25.
 May, A., Roesky, H. W., Stalke, D., Pauer, F. & Sheldrick, G. M. (1990). *Chem. Ber.* **123**, 1475–1478.
 Rademacher, P. (1987). *Strukturen organischer Moleküle. Physikalische Organische Chemie*, Vol. 2, edited by M. Klessinger. New York: VCH.
 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. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. Univ. of Göttingen, Germany.
 Stoe & Cie (1988a). *DIF4. Diffractometer Control Program*. Version 6.2. Stoe & Cie, Darmstadt, Germany.
 Stoe & Cie (1988b). *REDU4. Data Reduction Program*. Version 6.2. Stoe & Cie, Darmstadt, Germany.

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Dibromodiethylbis(3*H*-imidazole-2-thione-*S*)tin(IV)

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Abstract

The crystal structure of the title compound, [SnBr₂(C₂H₅)₂(C₃H₄N₂S)₂], comprises discrete all-*trans* octahedral [SnBr₂Et₂(Himt)₂] units (Himt = imidazol-2-thione) with the Sn atom coordinated to two ethyl C atoms [Sn—C = 2.158 (7) Å], two Br atoms [Sn—Br = 2.759 (3) Å] and two Himt S atoms [Sn—S = 2.771 (2) Å]. The structural parameters suggest the presence of an intermolecular N—H...Br hydrogen bond.

Comment

Interest in the properties of Sn—S bonds is prompted by the desire to understand the biological behaviour of diorganotin(IV) compounds (Barbieri, 1992, and references therein). We have recently described the interaction of dimethyltin(IV) (García Martínez, Sánchez González, Casas, Sordo, Valle & Russo, 1993) and diphenyltin(IV) (García Martínez, Sánchez González, Casas, Sordo, Casellato, Graziani & Russo, 1993) derivatives with the ligand 1-methyl-2(3*H*)-imidazo-