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## Comparison of the Structures of Two Trimesityltin Iodide Solvates and Triphenyltin Iodide

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### Abstract

The structures of iodotris(2,4,6-trimethylphenyl)tin(IV) trichloro(<sup>2</sup>H)methane hemisolvate, [SnI(C<sub>9</sub>H<sub>11</sub>)<sub>3</sub>]<sub>2</sub>.CDCl<sub>3</sub> (I), iodotris(2,4,6-trimethylphenyl)-

tin(IV) toluene hemisolvate, [SnI(C<sub>9</sub>H<sub>11</sub>)<sub>3</sub>]<sub>2</sub>.C<sub>7</sub>H<sub>8</sub> (II), and iodotriphenyltin(IV), [SnI(C<sub>6</sub>H<sub>5</sub>)<sub>3</sub>] (III), have been determined by single-crystal X-ray diffraction. All three crystallize in the same space group (*P* $\bar{1}$ ) with Ar<sub>3</sub>SnI molecules forming chains through the lattice using overlapping aryl groups. In (III), these are closely packed, but in (I) and (II), *p*-CH<sub>3</sub> groups prevent this, leaving voids which are filled by solvent molecules. Comparison of (I) and (II) with (III) shows the usual distortions due to *o*-CH<sub>3</sub> groups, except for the fact that distal methyls are closer to the Sn—I axis than expected, with the Sn—I distance increased by 0.05 Å. In Ar<sub>3</sub>SnI, making the aryl group more sterically demanding has the same effect on the crystal structure as replacing iodine with a smaller halogen atom. Thus, (*p*-CH<sub>3</sub>OC<sub>6</sub>H<sub>4</sub>)<sub>3</sub>SnI (IV) crystallizes in the same space group as Ph<sub>3</sub>SnBr (*P*<sub>2</sub><sub>1</sub>/*c*).

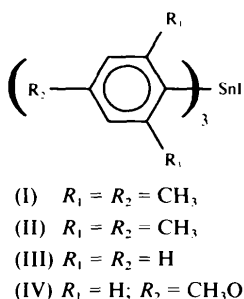
### Comment

Replacement of phenyl groups in organometallics by mesityl (mes = 2,4,6-trimethylphenyl) often results in structural or chemical changes attributed to the steric effects of the two *ortho*-methyl groups. As examples in tin chemistry, we note that while compounds of the type Ph<sub>3</sub>SnX, where X = OH (Glidewell & Liles, 1978), F (Tudela, Gutierrez-Puebla & Monge, 1992) or OAc (Molloy, Purcell, Quill & Newell, 1984), are polymeric with five-coordinate tin and planar C<sub>3</sub>Sn moieties, the corresponding mesityl compounds, with X = OH or F (Reuter & Puff, 1989), or OAc (Wharf & Simard, 1992), are monomeric with pyramidal C<sub>3</sub>SnX structures. Furthermore, while dehydrating Ph<sub>3</sub>SnOH produces (Ph<sub>3</sub>Sn)<sub>2</sub>O (Kushlefsky, Simmons & Ross, 1963), the mesityl analogue in refluxing ethanol loses mesitylene to form (mes<sub>2</sub>SnO)<sub>3</sub> (Weber, Pauls, Winter & Stegmann, 1982). These changes may well obscure the more subtle differences that might exist between phenyl- and mesityltin compounds with the same molecular structure.

In this paper, we compare two trimesityltin iodide solvates with the unsolvated triphenyl analogue. The former were prepared as part of an extension of earlier work on *para*-substituted triaryltin compounds (Wharf & Simard, 1991) to systems substituted at the *meta*- or *ortho*-positions. More recently, the crystal structure of Ph<sub>3</sub>SnI was determined, confirming the prediction from infrared data in the phenyl ring region that it was not isomorphous with the corresponding chloride and bromide structures (Wharf, 1993).

Trimesityltin iodide was prepared from the bromide (Lapkin & Sedel'nikova, 1960) and a small sample of (II) was recrystallized from toluene. Since the m.p. (479 K, literature 485 K) suggested incomplete conversion, an X-ray structure determination

was undertaken initially to test this. Subsequently, NMR data [CDCl<sub>3</sub> solution,  $\delta(^{119}\text{Sn})$ : -217.10 p.p.m., (CH<sub>3</sub>)<sub>4</sub>Sn] showed that the material was only the iodide, and crystals of (I) from the CDCl<sub>3</sub> solution were then examined because of the disorder that had been found in (II). Triphenyltin iodide (Wharf, Cuenca & Onyszchuk, 1984) was recrystallized from ethanol, giving (III), and the single-crystal FT-Raman spectrum (150w, 173vs, 216m, 235w, 272w) was almost the same as that measured previously for the bulk sample. Compound (IV), from cyclohexane, has been reported previously (Wharf, 1989); the crystal data are given in the *Experimental* for comparison with those of (I), (II) and (III).



The two iodotrimesityltin solvates (mes)<sub>3</sub>SnI· $\frac{1}{2}$ S [S = CDCl<sub>3</sub> (I) or C<sub>6</sub>H<sub>5</sub>CH<sub>3</sub> (II)], have very similar crystal structures (Fig. 1) with the same space group. In both cases, two chains of (mes)<sub>2</sub>Sn units are formed by overlapping phenyl rings parallel to the *b* axis while the third mesityl group on each Sn atom is directed towards an inversion centre. The *para*-methyl groups, however, hinder close-packing in this arrangement resulting in vacancies in the crystal lattice. In (I), these are filled by globular CDCl<sub>3</sub> molecules forming channels through the lattice, while in (II), planar toluenes (with the required disorder) slide in between mesityl groups bracketing the inversion centres in the unit cell. In both cases, the remaining space is then filled by neighbouring molecules closing up, leaving the solvates as tightly packed structures. In contrast, unsolvated Ph<sub>3</sub>SnI (III) is already tightly packed (Fig. 2).

For (I) and (II), the geometries around the tin (Table 2), including the disposition of the *o*-CH<sub>3</sub> groups, are almost identical, indicating that differences from that found for (III) are intra- rather than intermolecular in origin. Crystal packing requirements, however, may dictate the lack of trigonal symmetry that is found in these systems. The average geometries around tin for (I) and (II) when compared with that for (III) (Fig. 4) show differences similar to those predicted for (mes)<sub>3</sub>CH when compared with Ph<sub>3</sub>CH (Andose & Mislow, 1974). Thus,

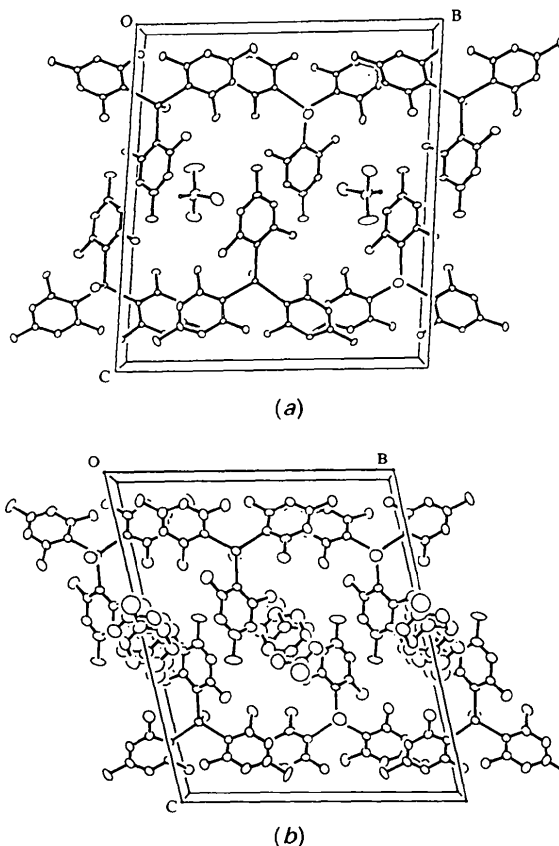


Fig. 1. Packing diagrams for (a) (I) and (b) (II).

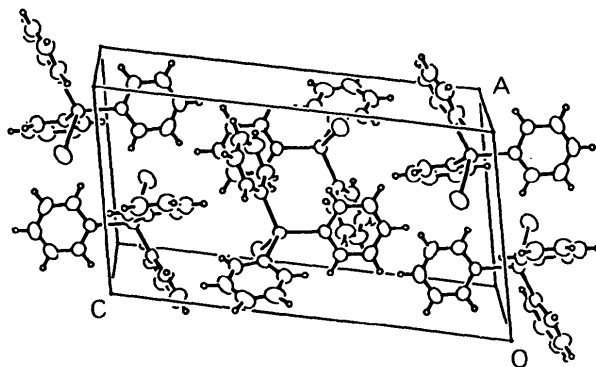


Fig. 2. Packing diagram for (III).

replacement of *ortho*-H atoms by methyl groups results in Sn—C and C—Sn—C increasing while *o*-CH<sub>3</sub> groups are bent back as predicted. There is also a significant lengthening of the Sn—I bond. The change in dihedral angles ( $\varphi$ ) (Table 3), *i.e.* a decrease, on average of  $\sim 4^\circ$ , on going from (III) to (I) or (II) is the opposite of that predicted for Ph<sub>3</sub>CH to (mes)<sub>3</sub>CH ( $\sim 4^\circ$  increase). In fact, these compounds have essentially identical  $\varphi$  values; Ph<sub>3</sub>CH,



## Triclinic

*P* $\bar{1}$   
 $a = 8.6431$  (13) Å  
 $b = 16.823$  (2) Å  
 $c = 19.970$  (2) Å  
 $\alpha = 77.738$  (11)°  
 $\beta = 84.234$  (11)°  
 $\gamma = 89.421$  (12)°  
 $V = 2822.9$  (6) Å<sup>3</sup>  
 $Z = 2$   
 $D_x = 1.528$  Mg m<sup>-3</sup>

## Data collection

Enraf-Nonius CAD-4  
 diffractometer  
 $\omega/2\theta$  scans [ $\Delta\omega = (1.00 + 0.35\tan\theta)^\circ$ ]  
 Absorption correction:  
 Gaussian by integration  
 from crystal shape (20 × 20 × 20)  
 $T_{\min} = 0.58$ ,  $T_{\max} = 0.79$   
 19 834 measured reflections  
 9923 independent reflections

## Refinement

Refinement on  $F$   
 $R = 0.068$   
 $wR = 0.079$   
 $S = 2.28$   
 5425 reflections  
 643 parameters  
 $w = 1/[\sigma^2(F) + 0.0001F^2]$   
 $(\Delta/\sigma)_{\max} = 0.37$

## Compound (III)

## Crystal data

[SnI(C<sub>6</sub>H<sub>5</sub>)<sub>3</sub>]  
 $M_r = 476.91$   
 Triclinic  
 $P\bar{1}$   
 $a = 9.8737$  (12) Å  
 $b = 9.9216$  (16) Å  
 $c = 18.799$  (3) Å  
 $\alpha = 83.928$  (14)°  
 $\beta = 78.372$  (12)°  
 $\gamma = 76.748$  (11)°  
 $V = 1752.3$  (5) Å<sup>3</sup>  
 $Z = 4$   
 $D_x = 1.808$  Mg m<sup>-3</sup>

## Data collection

Enraf-Nonius CAD-4  
 diffractometer  
 $\omega/2\theta$  scans [ $\Delta\omega = (0.80 + 0.14\tan\theta)^\circ$ ]

Cell parameters from 25  
 reflections  
 $\theta = 10.0$ – $11.0^\circ$   
 $\mu = 2.00$  mm<sup>-1</sup>  
 $T = 295$  K  
 Parallelepiped  
 $0.34$  ( $1\bar{1}\bar{1}, 111$ ) ×  $0.25$   
 $(11\bar{2}, 1\bar{1}\bar{2})$  ×  $0.14$   
 $(001, 00\bar{1})$  mm  
 White

5425 observed reflections  
 $[I \geq 3\sigma(I)]$   
 $R_{\text{int}} = 0.029$   
 $\theta_{\max} = 25^\circ$   
 $h = -10 \rightarrow 10$   
 $k = -19 \rightarrow 19$   
 $l = 0 \rightarrow 23$   
 7 standard reflections  
 frequency: 60 min  
 intensity variation:  $\pm 4.2\%$

$\Delta\rho_{\max} = 4.52$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -1.01$  e Å<sup>-3</sup>  
 Atomic scattering factors  
 from Cromer & Mann  
 (1968) for non-H atoms  
 and Stewart, Davidson  
 & Simpson (1965) for H  
 atoms

Absorption correction:  
 Gaussian by integration  
 from crystal shape (20 × 20 × 20)  
 $T_{\min} = 0.01$ ,  $T_{\max} = 0.20$   
 13 046 measured reflections  
 6625 independent reflections

## Refinement

Refinement on  $F$   
 $R = 0.037$   
 $wR = 0.039$   
 $S = 1.62$   
 4711 reflections  
 482 parameters  
 $w = 1/[\sigma^2(F) + 0.0001F^2]$   
 $(\Delta/\sigma)_{\max} = 0.15$   
 $\Delta\rho_{\max} = 0.73$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -1.06$  e Å<sup>-3</sup>

## Compound (IV)

## Crystal data

[SnI(C<sub>7</sub>H<sub>7</sub>O)<sub>3</sub>]  
 $M_r = 566.99$   
 Monoclinic  
 $P2_1/c$   
 $a = 9.805$  (6) Å  
 $b = 19.880$  (6) Å  
 $c = 13.353$  (3) Å  
 $\beta = 124.48$  (3)°  
 $V = 2145.6$  (15) Å<sup>3</sup>

$h = -11 \rightarrow 12$   
 $k = 0 \rightarrow 12$   
 $l = -22 \rightarrow 22$   
 5 standard reflections  
 frequency: 60 min  
 intensity variation:  $\pm 1.5\%$

Extinction correction:  
 Zachariasen (1967)  
 Extinction coefficient:  
 $0.629$  (13)

Atomic scattering factors  
 from Cromer & Mann  
 (1968) for non-H atoms  
 and Stewart, Davidson  
 & Simpson (1965) for H  
 atoms

$Z = 4$   
 $D_x = 1.755$  Mg m<sup>-3</sup>  
 Cu  $K\alpha$  radiation  
 $\lambda = 1.5418$  Å  
 Cell parameters from 25  
 reflections  
 $\theta = 12.0$ – $25.0^\circ$   
 $\mu = 21.26$  mm<sup>-1</sup>  
 $T = 295$  K  
 White

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å<sup>2</sup>)

Compound (I)	$B_{\text{eq}} = (8\pi^2/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$			$B_{\text{eq}}$
	$x$	$y$	$z$	
I(1)	0.42157 (6)	0.90377 (4)	0.76239 (3)	4.41 (3)
I(2)	0.29144 (6)	0.57751 (4)	0.26782 (3)	4.32 (3)
Sn(1)	0.74827 (5)	0.92008 (3)	0.76372 (3)	2.33 (2)
Sn(2)	0.60944 (5)	0.56964 (3)	0.24909 (3)	1.97 (2)
Cl(1)†	0.5668 (5)	0.7098 (2)	0.4891 (2)	10.5 (2)
Cl(2)†	0.2860 (4)	0.7893 (3)	0.4366 (2)	9.2 (2)
Cl(3)†	0.3871 (5)	0.7951 (4)	0.5813 (2)	13.1 (3)
C(10)†	0.4489 (12)	0.7867 (7)	0.5020 (6)	6.5 (6)
C(111)	0.7890 (8)	1.0366 (4)	0.8177 (3)	2.1 (3)
C(112)	0.7171 (7)	1.0540 (4)	0.8794 (4)	2.2 (3)
C(113)	0.7133 (8)	1.1319 (4)	0.9067 (4)	2.6 (3)
C(114)	0.7757 (8)	1.1946 (4)	0.8743 (4)	2.6 (3)
C(115)	0.8558 (9)	1.1758 (4)	0.8170 (4)	3.0 (3)
C(116)	0.8657 (8)	1.0987 (4)	0.7883 (4)	2.5 (3)
C(117)	0.6482 (9)	0.9908 (4)	0.9202 (4)	3.4 (3)
C(118)	0.7590 (10)	1.2793 (5)	0.9013 (5)	4.4 (4)
C(119)	0.9636 (10)	1.0856 (5)	0.7280 (4)	4.1 (4)
C(121)	0.7859 (8)	0.9165 (4)	0.6542 (4)	2.6 (3)
C(122)	0.6978 (9)	0.9654 (5)	0.6093 (4)	3.1 (3)
C(123)	0.7245 (9)	0.9639 (5)	0.5387 (4)	3.6 (4)
C(124)	0.8337 (9)	0.9143 (5)	0.5109 (4)	3.7 (4)
C(125)	0.9206 (9)	0.8685 (5)	0.5562 (4)	3.8 (4)
C(126)	0.9000 (8)	0.8673 (4)	0.6269 (4)	3.0 (3)
C(127)	0.5769 (10)	1.0220 (5)	0.6334 (4)	4.2 (4)
C(128)	0.8601 (11)	0.9142 (6)	0.4342 (5)	5.2 (5)
C(129)	1.0051 (10)	0.8156 (5)	0.6714 (5)	4.4 (4)

C(131)	0.8378 (8)	0.8197 (4)	0.8179 (4)	2.4 (3)	C(134)	0.421 (2)	0.1758 (11)	0.5123 (9)	5.1 (6)
C(132)	0.7765 (8)	0.7431 (4)	0.7933 (4)	2.8 (3)	C(135)	0.3377 (19)	0.2118 (10)	0.5601 (9)	4.7 (6)
C(133)	0.8298 (9)	0.6781 (4)	0.8281 (4)	2.9 (3)	C(136)	0.3548 (17)	0.1919 (9)	0.6305 (7)	3.5 (5)
C(134)	0.9413 (9)	0.6850 (4)	0.8869 (4)	3.0 (3)	C(137)	0.657 (2)	0.0258 (11)	0.6265 (9)	6.0 (7)
C(135)	1.0013 (8)	0.7611 (4)	0.9102 (4)	3.0 (3)	C(138)	0.397 (3)	0.2010 (13)	0.4356 (10)	7.4 (8)
C(136)	0.9529 (8)	0.8286 (4)	0.8761 (4)	2.5 (3)	C(139)	0.247 (2)	0.2322 (10)	0.6761 (9)	5.0 (6)
C(137)	0.6583 (9)	0.7273 (5)	0.7282 (4)	3.6 (3)	C(211)	0.0826 (16)	0.2946 (8)	0.1867 (8)	3.3 (5)
C(138)	0.9991 (10)	0.6133 (5)	0.9225 (5)	4.1 (4)	C(212)	0.1304 (16)	0.2146 (9)	0.2115 (8)	3.6 (5)
C(139)	1.0370 (9)	0.9062 (4)	0.9032 (5)	3.5 (3)	C(213)	0.0939 (18)	0.1533 (8)	0.1773 (8)	4.0 (5)
C(211)	0.6017 (7)	0.4607 (4)	0.1822 (3)	2.0 (3)	C(214)	0.0100 (18)	0.1689 (9)	0.1212 (9)	4.1 (6)
C(212)	0.4947 (8)	0.4529 (4)	0.1199 (3)	2.1 (3)	C(215)	-0.0392 (19)	0.2473 (10)	0.0985 (8)	4.5 (6)
C(213)	0.4732 (8)	0.3795 (4)	0.0825 (4)	2.4 (3)	C(216)	-0.0088 (17)	0.3085 (9)	0.1309 (8)	4.0 (6)
C(214)	0.5516 (8)	0.3133 (4)	0.1048 (4)	2.5 (3)	C(217)	0.216 (2)	0.1910 (10)	0.2758 (10)	5.6 (6)
C(215)	0.6621 (8)	0.3224 (4)	0.1630 (4)	2.5 (3)	C(218)	-0.029 (2)	0.1020 (10)	0.0872 (10)	6.0 (7)
C(216)	0.6904 (8)	0.3948 (4)	0.2020 (4)	2.2 (3)	C(219)	-0.080 (2)	0.3904 (10)	0.1033 (10)	5.8 (7)
C(217)	0.4033 (9)	0.5218 (4)	0.0910 (4)	3.0 (3)	C(221)	0.1257 (16)	0.5142 (9)	0.1789 (8)	3.6 (5)
C(218)	0.5121 (10)	0.2328 (4)	0.0654 (4)	3.5 (3)	C(222)	0.2256 (17)	0.5369 (9)	0.1190 (7)	3.5 (5)
C(219)	0.8233 (9)	0.3990 (5)	0.2628 (4)	3.1 (3)	C(223)	0.2423 (19)	0.6191 (10)	0.0879 (8)	4.3 (6)
C(221)	0.7243 (8)	0.5690 (4)	0.3559 (4)	2.3 (3)	C(224)	0.164 (2)	0.6789 (9)	0.1141 (8)	4.1 (6)
C(222)	0.6664 (9)	0.5164 (4)	0.4017 (4)	3.0 (3)	C(225)	0.0611 (19)	0.6538 (9)	0.1729 (8)	4.3 (6)
C(223)	0.7301 (9)	0.5209 (5)	0.4719 (4)	3.1 (3)	C(226)	0.0373 (18)	0.5738 (10)	0.2038 (8)	4.0 (6)
C(224)	0.8494 (9)	0.5760 (5)	0.4985 (4)	3.3 (3)	C(227)	0.314 (2)	0.4768 (10)	0.0835 (9)	5.4 (6)
C(225)	0.9092 (8)	0.6252 (4)	0.4524 (4)	3.3 (3)	C(228)	0.193 (2)	0.7662 (10)	0.0859 (10)	6.3 (7)
C(226)	0.8502 (8)	0.6230 (4)	0.3823 (4)	2.5 (3)	C(229)	-0.090 (2)	0.5532 (11)	0.2618 (10)	6.6 (7)
C(227)	0.5399 (11)	0.4526 (5)	0.3780 (4)	4.1 (4)	C(231)	0.0376 (17)	0.3664 (9)	0.3456 (7)	3.5 (5)
C(228)	0.9157 (11)	0.5815 (6)	0.5756 (4)	4.7 (4)	C(232)	0.0977 (19)	0.4055 (10)	0.3918 (9)	4.4 (6)
C(229)	0.9269 (9)	0.6783 (5)	0.3361 (4)	3.6 (4)	C(233)	0.044 (2)	0.3832 (11)	0.4614 (9)	5.2 (6)
C(231)	0.6666 (8)	0.6736 (4)	0.1963 (4)	2.3 (3)	C(234)	-0.071 (2)	0.3249 (12)	0.4863 (9)	5.4 (7)
C(232)	0.6164 (8)	0.7493 (4)	0.2189 (4)	2.8 (3)	C(235)	-0.1349 (19)	0.2886 (10)	0.4394 (10)	5.2 (6)
C(233)	0.6504 (9)	0.8136 (4)	0.1810 (5)	3.4 (3)	C(236)	-0.0853 (16)	0.3092 (9)	0.3690 (7)	3.6 (5)
C(234)	0.7310 (9)	0.8076 (4)	0.1236 (4)	3.4 (3)	C(237)	0.216 (2)	0.4725 (11)	0.3707 (10)	6.3 (7)
C(235)	0.7843 (8)	0.7335 (4)	0.1028 (4)	3.2 (3)	C(238)	-0.130 (3)	0.2997 (15)	0.5610 (11)	8.3 (8)
C(236)	0.7565 (8)	0.6666 (4)	0.1388 (4)	2.5 (3)	C(239)	-0.1755 (19)	0.2725 (11)	0.3218 (9)	5.3 (6)
C(237)	0.5328 (11)	0.7629 (5)	0.2828 (5)	4.3 (4)					
C(238)	0.7669 (11)	0.8792 (5)	0.0844 (5)	5.1 (4)					
C(239)	0.8315 (9)	0.5907 (4)	0.1162 (4)	3.3 (3)					
D(10)†	0.5115	0.8337	0.4985						

## Compound (II)

I(1)	0.83917 (13)	0.12396 (8)	0.74520 (7)	5.41 (5)
I(2)	0.45043 (14)	0.37811 (8)	0.24745 (7)	6.12 (6)
Sn(1)	0.51982 (12)	0.11110 (6)	0.75872 (5)	3.00 (4)
Sn(2)	0.13507 (12)	0.38974 (6)	0.23839 (5)	3.26 (4)
C(11)‡	0.950 (3)	0.0000 (17)	0.4618 (12)	6.4 (10)
C(12)‡	0.981 (3)	0.0781 (17)	0.4710 (12)	9.1 (12)
C(13)‡	1.055 (3)	0.0883 (17)	0.5276 (12)	7.6 (11)
C(14)‡	1.098 (3)	0.0204 (17)	0.5751 (12)	9.2 (11)
C(15)‡	1.068 (3)	-0.0576 (17)	0.5659 (12)	9.2 (11)
C(16)‡	0.994 (3)	-0.0678 (17)	0.5092 (12)	6.0 (10)
C(17)‡	0.890 (6)	-0.0109 (19)	0.3960 (15)	14.9 (2)
C(21)‡	0.559 (3)	0.4985 (16)	0.4624 (12)	5.6 (10)
C(22)‡	0.532 (3)	0.5779 (16)	0.4707 (12)	8.3 (11)
C(23)‡	0.439 (3)	0.5917 (16)	0.5283 (12)	8.8 (11)
C(24)‡	0.374 (3)	0.5261 (16)	0.5776 (12)	8.4 (11)
C(25)‡	0.401 (3)	0.4467 (16)	0.5692 (12)	9.0 (11)
C(26)‡	0.494 (3)	0.4330 (16)	0.5116 (12)	5.7 (10)
C(27)‡	0.648 (6)	0.4841 (18)	0.3973 (16)	15.6 (12)
C(111)	0.4371 (16)	0.2053 (8)	0.8121 (7)	3.0 (5)
C(112)	0.4938 (17)	0.2850 (9)	0.7900 (8)	3.8 (5)
C(113)	0.439 (2)	0.3440 (9)	0.8270 (10)	4.8 (6)
C(114)	0.329 (2)	0.3256 (10)	0.8829 (10)	5.0 (6)
C(115)	0.2700 (19)	0.2481 (11)	0.9008 (8)	4.6 (6)
C(116)	0.3173 (18)	0.1864 (9)	0.8675 (8)	3.9 (5)
C(117)	0.611 (2)	0.3116 (10)	0.7272 (9)	5.5 (6)
C(118)	0.270 (3)	0.3886 (12)	0.9214 (12)	7.9 (8)
C(119)	0.2341 (19)	0.1057 (10)	0.8887 (9)	4.9 (6)
C(121)	0.4933 (15)	-0.0110 (8)	0.8201 (7)	3.0 (5)
C(122)	0.5644 (17)	-0.0310 (9)	0.8821 (8)	3.9 (5)
C(123)	0.5653 (18)	-0.1124 (9)	0.9159 (8)	3.9 (5)
C(124)	0.501 (2)	-0.1731 (9)	0.8901 (9)	4.5 (6)
C(125)	0.4218 (19)	-0.1526 (9)	0.8332 (9)	4.3 (6)
C(126)	0.4169 (17)	-0.0737 (9)	0.7985 (8)	3.7 (5)
C(127)	0.6377 (19)	0.0300 (9)	0.9149 (8)	4.4 (6)
C(128)	0.519 (2)	-0.2617 (10)	0.9266 (10)	6.6 (7)
C(129)	0.318 (2)	-0.0553 (11)	0.7380 (9)	6.1 (7)
C(131)	0.4671 (16)	0.1336 (8)	0.6528 (7)	3.2 (5)
C(132)	0.5501 (18)	0.0952 (9)	0.6052 (8)	3.9 (5)
C(133)	0.525 (2)	0.1169 (11)	0.5358 (8)	4.9 (6)

## Compound (III)

I(1)	0.17373 (7)	0.48232 (5)	0.61574 (3)	7.06 (3)
I(2)	0.61274 (8)	0.16675 (7)	0.09909 (4)	8.55 (4)
Sn(1)	0.33466 (5)	0.24262 (4)	0.56223 (2)	4.38 (2)
Sn(2)	0.80102 (5)	0.33013 (4)	0.05519 (2)	4.49 (2)
C(111)	0.3570 (7)	0.2773 (6)	0.4472 (3)	4.3 (3)
C(112)	0.2399 (8)	0.3139 (8)	0.4131 (4)	5.3 (4)
C(113)	0.2586 (9)	0.3340 (9)	0.3377 (4)	6.1 (4)
C(114)	0.3927 (9)	0.3177 (9)	0.2977 (4)	6.2 (4)
C(115)	0.5089 (9)	0.2811 (8)	0.3291 (4)	5.9 (4)
C(116)	0.4924 (8)	0.2617 (7)	0.4046 (4)	5.0 (3)
C(121)	0.5301 (7)	0.2150 (7)	0.5988 (3)	4.4 (3)
C(122)	0.6066 (9)	0.3183 (7)	0.5846 (4)	5.9 (4)
C(123)	0.7356 (10)	0.2962 (9)	0.6067 (5)	7.5 (5)
C(124)	0.7883 (9)	0.1740 (9)	0.6410 (5)	7.0 (5)
C(125)	0.7141 (9)	0.0718 (8)	0.6560 (4)	6.3 (4)
C(126)	0.5861 (8)	0.0923 (7)	0.6337 (4)	5.2 (4)
C(131)	0.2240 (7)	0.0840 (6)	0.6048 (4)	4.4 (3)
C(132)	0.1940 (8)	0.0488 (8)	0.6786 (4)	5.5 (4)
C(133)	0.1232 (10)	-0.0549 (9)	0.7056 (4)	6.8 (4)
C(134)	0.0844 (9)	-0.1286 (8)	0.6589 (5)	7.3 (4)
C(135)	0.1119 (9)	-0.0990 (8)	0.5849 (5)	7.0 (5)
C(136)	0.1810 (8)	0.0081 (7)	0.5577 (4)	5.4 (4)
C(211)	0.8375 (7)	0.3394 (6)	-0.0600 (3)	4.2 (3)
C(212)	0.9776 (8)	0.3034 (7)	-0.0970 (4)	5.2 (4)
C(213)	1.0052 (9)	0.3084 (9)	-0.1730 (4)	6.7 (5)
C(214)	0.8984 (11)	0.3447 (10)	-0.2107 (4)	7.2 (5)
C(215)	0.7608 (10)	0.3788 (9)	-0.1748 (4)	6.9 (5)
C(216)	0.7296 (9)	0.3776 (8)	-0.0990 (4)	5.7 (4)
C(221)	0.7050 (7)	0.5240 (6)	0.0994 (3)	4.3 (3)
C(222)	0.6743 (9)	0.6430 (7)	0.0552 (4)	5.5 (4)
C(223)	0.6105 (10)	0.7683 (8)	0.0848 (5)	7.3 (5)
C(224)	0.5749 (10)	0.7749 (9)	0.1594 (5)	7.2 (5)
C(225)	0.6048 (10)	0.6584 (10)	0.2044 (4)	6.9 (5)
C(226)	0.6682 (9)	0.5320 (8)	0.1749 (4)	6.0 (4)
C(231)	0.9854 (8)	0.2299 (6)	0.0963 (3)	4.5 (3)
C(232)	1.0539 (9)	0.3040 (7)	0.1303 (4)	5.6 (4)
C(233)	1.1745 (10)	0.2400 (10)	0.1558 (5)	7.1 (5)
C(234)	1.2292 (10)	0.1025 (10)	0.1481 (5)	7.4 (5)
C(235)	1.1637 (10)	0.0274 (8)	0.1151 (5)	7.4 (5)
C(236)	1.0412 (10)	0.0907 (8)	0.0895 (4)	6.5 (4)

† Chloroform coordinates

‡ Toluene coordinates, occupancy of 0.5.

Table 2. Bond distances (Å) and angles (°) around tin

Compound (I)	x = 1	x = 2
Sn(x)—I(x)	2.753 (2)	2.745 (2)
Sn(x)—C(x11)	2.156 (7)	2.160 (6)
Sn(x)—C(x21)	2.158 (7)	2.163 (7)
Sn(x)—C(x31)	2.157 (7)	2.156 (7)
C(x11)—C(x12)	1.40 (1)	1.41 (1)
C(x11)—C(x16)	1.40 (1)	1.40 (1)
C(x21)—C(x22)	1.40 (1)	1.41 (1)
C(x21)—C(x26)	1.40 (1)	1.40 (1)
C(x31)—C(x32)	1.41 (1)	1.41 (1)
C(x31)—C(x36)	1.39 (1)	1.41 (1)
I(x)—Sn(x)—C(x11)	99.9 (2)	99.6 (2)
I(x)—Sn(x)—C(x21)	104.7 (2)	102.5 (2)
I(x)—Sn(x)—C(x31)	103.6 (2)	106.4 (2)
C(x11)—Sn(x)—C(x21)	113.6 (3)	118.2 (3)
C(x11)—Sn(x)—C(x31)	117.7 (3)	113.2 (2)
C(x21)—Sn(x)—C(x31)	114.6 (3)	114.4 (3)
Sn(x)—C(x11)—C(x12)	118.5 (5)	119.1 (5)
Sn(x)—C(x11)—C(x16)	121.9 (5)	121.8 (5)
Sn(x)—C(x21)—C(x22)	119.6 (5)	119.6 (5)
Sn(x)—C(x21)—C(x26)	121.5 (5)	121.7 (5)
Sn(x)—C(x31)—C(x32)	118.5 (5)	120.9 (5)
Sn(x)—C(x31)—C(x36)	122.0 (5)	120.1 (5)
C(x11)—C(x12)—C(x17)	123.2 (6)	123.0 (6)
C(x11)—C(x16)—C(x19)	123.1 (6)	123.3 (6)
C(x21)—C(x22)—C(x27)	123.2 (7)	123.1 (7)
C(x21)—C(x26)—C(x29)	122.9 (7)	122.4 (7)
C(x31)—C(x32)—C(x37)	123.1 (7)	122.3 (7)
C(x31)—C(x36)—C(x39)	124.1 (6)	122.9 (6)
Compound (II)		
Sn(x)—I(x)	2.752 (2)	2.752 (2)
Sn(x)—C(x11)	2.17 (1)	2.16 (1)
Sn(x)—C(x21)	2.16 (1)	2.18 (2)
Sn(x)—C(x31)	2.16 (1)	2.18 (1)
C(x11)—C(x12)	1.40 (2)	1.40 (2)
C(x11)—C(x16)	1.42 (2)	1.41 (2)
C(x21)—C(x22)	1.41 (2)	1.39 (2)
C(x21)—C(x26)	1.41 (2)	1.40 (2)
C(x31)—C(x32)	1.40 (2)	1.38 (2)
C(x31)—C(x36)	1.41 (2)	1.42 (2)
I(x)—Sn(x)—C(x11)	105.6 (4)	104.3 (4)
I(x)—Sn(x)—C(x21)	99.1 (4)	99.4 (4)
I(x)—Sn(x)—C(x31)	102.5 (4)	103.3 (4)
C(x11)—Sn(x)—C(x21)	114.5 (5)	116.4 (5)
C(x11)—Sn(x)—C(x31)	113.4 (5)	113.1 (5)
C(x21)—Sn(x)—C(x31)	118.7 (5)	117.3 (6)
Sn(x)—C(x11)—C(x12)	121 (1)	120 (1)
Sn(x)—C(x11)—C(x16)	119 (1)	122 (1)
Sn(x)—C(x21)—C(x22)	119 (1)	118 (1)
Sn(x)—C(x21)—C(x26)	123 (1)	122 (1)
Sn(x)—C(x31)—C(x32)	121 (1)	120 (1)
Sn(x)—C(x31)—C(x36)	119 (1)	120 (1)
C(x11)—C(x12)—C(x17)	123 (1)	122 (1)
C(x11)—C(x16)—C(x19)	124 (1)	123 (1)
C(x21)—C(x22)—C(x27)	124 (1)	124 (1)
C(x21)—C(x26)—C(x29)	122 (1)	122 (1)
C(x31)—C(x32)—C(x37)	122 (1)	123 (1)
C(x31)—C(x36)—C(x39)	126 (1)	124 (1)
Compound (III)		
Sn(x)—I(x)	2.7060 (8)	2.6916 (8)
Sn(x)—C(x11)	2.128 (6)	2.118 (6)
Sn(x)—C(x21)	2.127 (7)	2.116 (6)
Sn(x)—C(x31)	2.117 (7)	2.116 (7)
C(x11)—C(x12)	1.39 (1)	1.40 (1)
C(x11)—C(x16)	1.40 (1)	1.37 (1)
C(x21)—C(x22)	1.38 (1)	1.38 (1)
C(x21)—C(x26)	1.37 (1)	1.40 (1)
C(x31)—C(x32)	1.38 (1)	1.38 (1)
C(x31)—C(x36)	1.40 (1)	1.37 (1)
I(x)—Sn(x)—C(x11)	104.9 (2)	105.5 (2)
I(x)—Sn(x)—C(x21)	105.7 (2)	106.1 (2)
I(x)—Sn(x)—C(x31)	106.9 (2)	106.3 (2)
C(x11)—Sn(x)—C(x21)	112.6 (3)	113.5 (2)

C(x11)—Sn(x)—C(x31)	113.3 (3)	111.7 (3)
C(x21)—Sn(x)—C(x31)	112.6 (3)	113.0 (3)
Sn(x)—C(x11)—C(x12)	121.6 (5)	118.0 (5)
Sn(x)—C(x11)—C(x16)	119.4 (5)	122.6 (5)
Sn(x)—C(x21)—C(x22)	120.3 (5)	121.3 (5)
Sn(x)—C(x21)—C(x26)	121.0 (5)	119.6 (5)
Sn(x)—C(x31)—C(x32)	122.3 (5)	120.4 (5)
Sn(x)—C(x31)—C(x36)	120.2 (5)	121.4 (5)
C(x11)—C(x12)—H(x12)	119 (4)	119 (4)
C(x11)—C(x16)—H(x16)	119 (4)	120 (4)
C(x21)—C(x22)—H(x22)	118 (5)	120 (4)
C(x21)—C(x26)—H(x26)	118 (4)	118 (4)
C(x31)—C(x32)—H(x32)	118 (4)	119 (4)
C(x31)—C(x36)—H(x36)	121 (4)	119 (4)

Table 3. Dihedral angles (°)

Inclination of the plane of ring (xX) defined by the ring C atoms C(xX1) to C(xX6) from the plane given by I(x), Sn(x), C(xX1).

Compound (I)	X = 1	X = 2	X = 3	Average
x = 1	52.4 (2)	49.4 (2)	53.8 (2)	
x = 2	57.2 (2)	50.7 (2)	45.8 (2)	51.6 (2)
Compound (II)				
x = 1	47.0 (2)	56.3 (2)	49.3 (2)	
x = 2	50.2 (2)	55.2 (2)	48.5 (2)	51.1 (2)
Compound (III)				
x = 1	57.4 (2)	53.2 (2)	61.6 (2)	
x = 2	53.6 (2)	62.2 (2)	46.6 (2)	55.8 (2)

For compound (I), the structure was solved by the heavy-atom method and difference Fourier syntheses using *NRCVAX* (Gabe, Le Page, Charland, Lee & White, 1989). The refinement, as for compounds (II) and (III), was by full-matrix least squares with all non-H atoms anisotropic and all H atoms isotropic. The methyl H atoms of (I) were located from at least one peak obtained from a difference Fourier map; the positions of the other H atoms were calculated. Final H-atom coordinates were introduced at ideal positions (C—H = 0.95 Å) with  $U_{\text{iso}}$  refined initially and fixed in the final cycles [ $U_{\text{iso}} = 0.04$  (phenyl),  $U_{\text{iso}} = 0.07$  (methyl) and  $U_{\text{iso}} = 0.10 \text{ \AA}^2$  (deuterium of CDCl<sub>3</sub>)]. Anomalous-dispersion terms (Cromer & Liberman, 1970) were included for I, Sn and Cl. The secondary-extinction coefficient was not refined. The final Fourier map showed five peaks of  $2.28\text{--}0.93 \text{ e \AA}^{-3}$  at  $0.26\text{--}1.74 \text{ \AA}$  from Cl and four peaks of  $2.17\text{--}0.80 \text{ e \AA}^{-3}$  at  $0.71\text{--}1.94 \text{ \AA}$  from Sn or I. The background was  $\leq 0.70 \text{ e \AA}^{-3}$ .

For compound (II), the structure was solved by the heavy-atom method and difference Fourier syntheses using *NRCVAX* and final refinement was carried out using *SHELX76* (Sheldrick, 1976). The highly disordered toluene solvent molecules were found at symmetry centers ( $\frac{1}{2}, \frac{1}{2}, \frac{1}{2}$ ) and  $(0, 0, \frac{1}{2})$ . The ideal toluene model, imposed to help convergence, was retained in the final cycles (C=C = 1.395, C—C = 1.51 Å). The methyl H atoms were located from at least one peak obtained from a difference Fourier map; the positions of the others were calculated. Final H-atom coordinates were introduced at ideal positions (C—H = 0.95 Å) with  $U_{\text{iso}}$  refined initially and fixed in the final cycles [ $U_{\text{iso}} = 0.07$  (phenyl),  $U_{\text{iso}} = 0.085$  (methyl),  $U_{\text{iso}} = 0.10$  (toluene) and  $U_{\text{iso}} = 0.15 \text{ \AA}^2$  (methyl of toluene)]. Anomalous-dispersion terms were included for all atoms. The secondary-extinction coefficient was not refined. The final Fourier map showed ten peaks of  $4.52\text{--}0.98 \text{ e \AA}^{-3}$  at  $0.53\text{--}1.89 \text{ \AA}$  from Sn or I. The background was  $\leq 0.90 \text{ e \AA}^{-3}$ .

For compound (III), the structure was solved by direct methods using *SHELXS86* (Sheldrick, 1985) and difference Fourier

syntheses using *NRCVAX*. All H atoms were calculated initially and refined in the final cycles. Anomalous-dispersion terms (Cromer & Liberman, 1970) were included for I, Sn and Cl. The final Fourier map showed seven peaks of  $0.73\text{--}0.45\text{ e \AA}^{-3}$  at  $0.41\text{--}1.69\text{ \AA}$  from I and four peaks of  $0.60\text{--}0.44\text{ e \AA}^{-3}$  at  $1.32\text{--}1.49\text{ \AA}$  from Sn. The background was  $\leq 0.44\text{ e \AA}^{-3}$ .

Stereodiagrams for compounds (1), (2) and (3) were produced using *ORTEP* (Johnson, 1965).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry, least-squares-planes data, intermolecular contacts and H-atom geometry for (II), and stereoviews of the three unit cells, *ORTEP* views of the solvate molecules and a view of the numbering scheme for molecule (1) of (II) have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71594 (179 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: BR1044]

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## {Hexakis[2,4,6-tris(trifluoromethyl)phenyl]-cyclotristannoxane}<sub>2</sub> †

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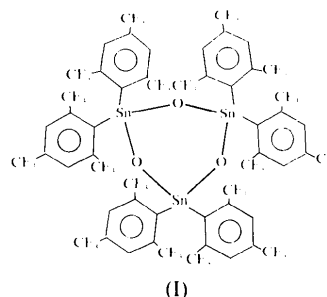
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## Abstract

The structure of the title compound,  $[\text{Sn}_3\text{O}_3(\text{C}_6\text{H}_2\text{F}_9)_6]$ , is reported. There are two molecules of hexakis[2,4,6-tris(trifluoromethyl)phenyl]cyclotristannoxane in the asymmetric unit; 20 of the 36 trifluoromethyl groups are disordered.

## Comment

The title compound (I) is a derivative of the monomer bis[2,4,6-tris(trifluoromethyl)phenyl]stannylene (Grützmacher, Pritzkow & Edelmann, 1991) and is stabilized by



(I)

† Alternative name: *cyclo-tri-μ-oxo-tris{bis[2,4,6-tris(trifluoromethyl)phenyl]tin}*.