Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

Bis[1,3-bis(diphenylphosphino)propane-κ^2P,P']-silver(I) hemi[cyclopentyldiphenylbis(trifluoroacetato-κO)stannate(IV)] hemi[triphenylbis(trifluoroacetato-κO)stannate(IV)]

Yin Yin Teo, Kong Mun Lo and Seik Weng Ng*

Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia

Correspondence e-mail: seikweng@um.edu.my

Received 8 September 2004
Accepted 16 September 2004
Online 25 September 2004

In the crystal structure of the title co-crystal, $[Ag(C_{27}H_{26}P_2)_2][Sn(C_6H_5)_3(C_2F_3O_4)_2]_{0.5}[Sn(C_6H_5)_2(C_5H_9)(C_2F_3O_4)_2]_{0.5}$, the Ag atom shows tetrahedral coordination, whereas the Sn atom shows trigonal–bipyramidal coordination. There are no significant interactions between the cations and anions.

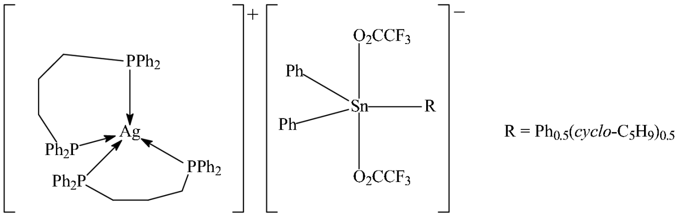
Key indicators

Single-crystal X-ray study
T = 113 K
Mean σ(C–C) = 0.006 Å
Disorder in main residue
R factor = 0.047
wR factor = 0.133
Data-to-parameter ratio = 14.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

Several reports have detailed mixed triorganotin compounds, the 'mixed' groups being a chlorophenyl and two phenyl groups (Lo *et al.*, 1997), chlorophenyl and two tolyl groups (Lo & Ng, 2004*b*), as well as a cyclopentyl and two phenyl groups (Lo *et al.*, 1999; Lo & Ng, 2004*a*). Interest in the solid-state structures of such mixed compounds arises from their generally biological activities compared with those of the symmetrical compounds (Kumar Das *et al.*, 1989).



(I)

As triphenyltin trifluoroacetate is a Lewis acid, it can be converted to the triphenylbis(trifluoroacetato)stannate anion; the anion has been isolated as the bis[1,2-bis(diphenylphosphino)ethane]silver salt (Chee *et al.*, 2003). The report is now extended to the bis[1,2-bis(diphenylphosphino)propane]silver derivative of the mixed cyclopentyldiphenyltin system; however, the silver stannate that was isolated is an intimate 1/1 co-crystal of the cyclopentyldiphenylbis(trifluoroacetato)stannate and triphenylbis(trifluoroacetato)stannate salts (Figs. 1, 2, 3 and 4).

The Ag atom in the cation shows tetrahedral coordination; the Ag—P distances are marginally shorter than those of the bis[bis(diphenylphosphino)ethane)]silver salt (Chee *et al.*, 2003) owing to the greater flexibility in the ligand, which has three methylene linkages. For the anion, strict comparison of the bond dimensions is difficult as the anion is severely disordered; nevertheless, the Sn atom is unambiguously five-coordinate in a *trans*-trigonal–bipyramidal environment.

Experimental

Cyclopentyldiphenyltin hydroxide was synthesized as described previously (Lo *et al.*, 1999) from the cleavage of cyclopentyltriphenyltin by iodine followed by the hydrolysis of the iodide by sodium hydroxide; the mixed cycloalkyl/aryl compound (0.18 g,

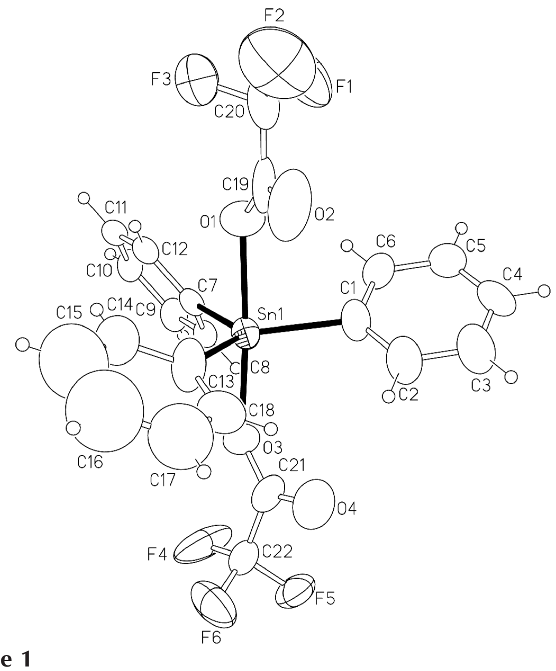


Figure 1
*ORTEP*II (Johnson, 1976) plot of the triphenylbis(trifluoroacetato)-stannate ion, with displacement ellipsoids drawn at 50% probability level. H atoms are drawn as spheres of arbitrary radii.

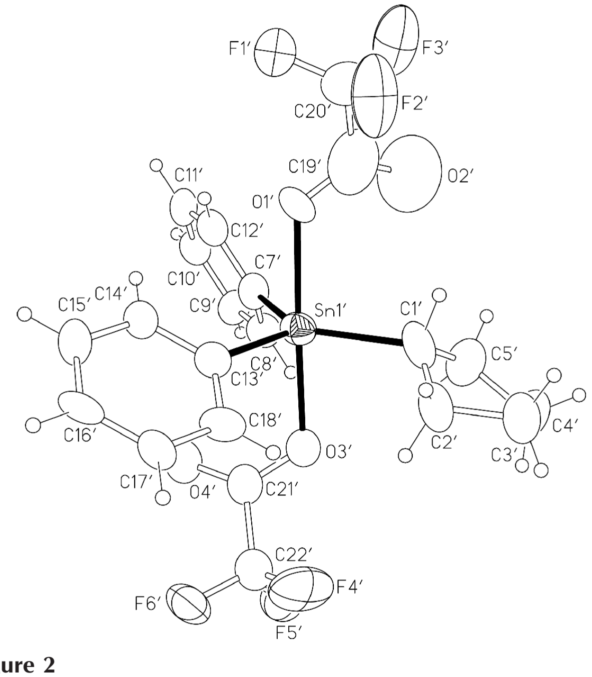


Figure 2
*ORTEP*II (Johnson, 1976) plot of the cyclopentyldiphenylbis(trifluoroacetato)stannate ion, with displacement ellipsoids drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radii.

0.5 mmol) and trifluoroacetic acid (0.06 g, 0.5 mmol) were heated in a small volume of ethanol. 1,3-Bis(diphenylphosphino)propane (0.41 g, 1 mmol) and silver trifluoroacetate (0.11 g, 0.5 mmol) were dissolved in a small volume of ethanol. The two solutions were mixed and heated for several minutes. The solution was filtered; evaporation of the solvent gave a product that was then recrystallized from ethanol. The compound was expected to be the simple salt, with the bis(trifluoroacetato)cyclopentyldiphenylstannate ion as the counter-ion.

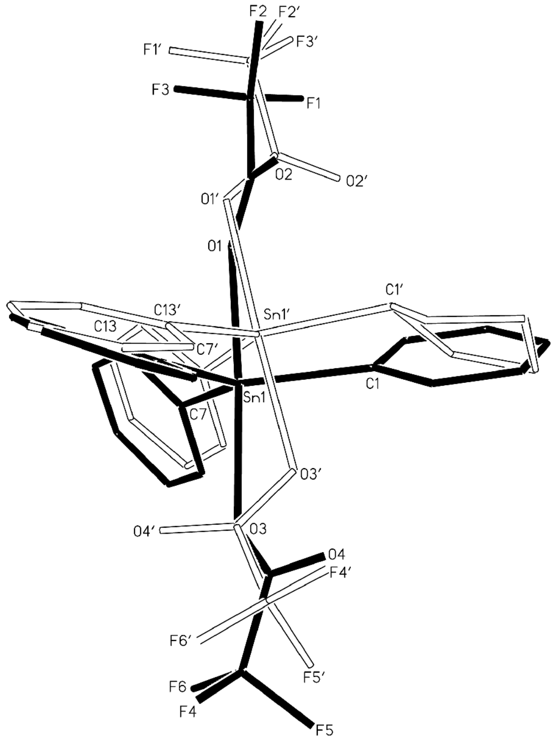


Figure 3
Scheme illustrating the disorder in the anion.

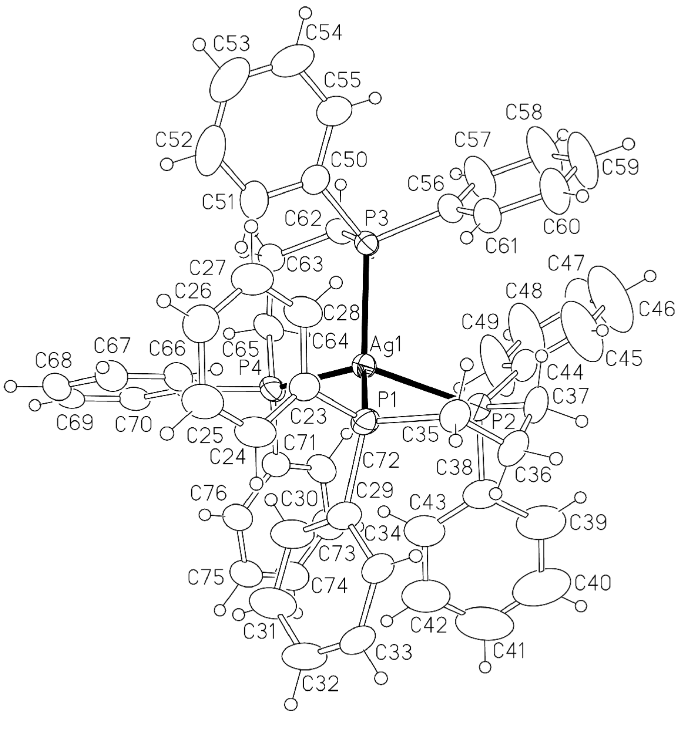


Figure 4
*ORTEP*II (Johnson, 1976) plot of the bis[1,3-bis(diphenylphosphino)-propane]silver cation, with displacement ellipsoids drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radii.

However, the refinement showed that the anion is a 50:50 mixture of cyclopentyldiphenylbis(trifluoroacetato)stannate and triphenylbis-(trifluoroacetato)stannate ions. The presence of the triphenyltin entity is possibly attributed to the presence of triphenyltin hydroxide

Acta Cryst. (2004). E**60**, m1478–m1480 Teo *et al.* • $[Ag(C_{27}H_{26}P_2)_2][SnPh_{2.5}(C_5H_9)_{0.5}(C_2F_3O_4)_2]$ **m1479**

that is formed through the iodine cleavage of the cycloalkyl–tin bond of the cyclopentyltriphenyltin reagent.

Crystal data

$C_{27}H_{26}AgP_2^+\cdot 0.5C_{22}H_{15}F_6O_4Sn^-\cdot 0.5C_{21}H_{19}F_6O_4Sn^-$
$M_r = 1504.75$
Triclinic, $P\overline{1}$
$a = 10.6019$ (3) Å
$b = 18.0331$ (5) Å
$c = 18.3529$ (5) Å
$\alpha = 91.372$ (1)°
$\beta = 93.985$ (1)°
$\gamma = 105.418$ (1)°
$V = 3371.06$ (16) Å^3
$Z = 2$
$D_x = 1.482$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 14392 reflections
$\theta = 2.3$–26.4°
$\mu = 0.82$ mm^{-1}
$T = 113$ (2) K
Irregular block, colorless
0.60 × 0.51 × 0.42 mm

Data collection

Siemens *P*4/CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan *SADABS* (Bruker, 2001)
$T_{\min} = 0.616$, $T_{\max} = 0.710$
26881 measured reflections
13501 independent reflections
10515 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.028$
$\theta_{\max} = 26.4°$
$h = -13 \rightarrow 13$
$k = -22 \rightarrow 22$
$l = -22 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.133$
$S = 1.02$
13501 reflections
961 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0642P)^2 + 6.1836P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 1.04$ e Å^{-3}
$\Delta\rho_{\min} = -0.95$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Sn1—C1	2.139 (4)	Sn1′—C13′	2.114 (4)
Sn1—C7	2.149 (5)	Sn1′—O1′	2.254 (8)
Sn1—C13	2.110 (7)	Sn1′—O3′	2.244 (6)
Sn1—O1	2.252 (8)	Ag1—P1	2.468 (1)
Sn1—O3	2.236 (6)	Ag1—P2	2.499 (1)
Sn1′—C1′	2.160 (7)	Ag1—P3	2.479 (1)
Sn1′—C7′	2.140 (5)	Ag1—P4	2.470 (1)
C1—Sn1—C7	115.0 (2)	C1′—Sn1′—O3′	84.5 (3)
C1—Sn1—C13	124.2 (4)	C7′—Sn1′—C13′	114.7 (3)
C1—Sn1—O1	85.8 (3)	C7′—Sn1′—O1′	88.5 (3)
C1—Sn1—O3	97.4 (3)	C7′—Sn1′—O3′	93.0 (3)
C7—Sn1—C13	120.4 (4)	C13′—Sn1′—O1′	90.2 (3)
C7—Sn1—O1	86.4 (3)	C13′—Sn1′—O3′	90.4 (3)
C7—Sn1—O3	86.7 (3)	O3′—Sn1′—O1′	178.0 (3)
C13—Sn1—O1	91.0 (4)	P1—Ag1—P2	92.50 (4)
C13—Sn1—O3	92.3 (4)	P1—Ag1—P3	123.21 (4)
O3—Sn1—O1	173.1 (3)	P1—Ag1—P4	119.25 (3)
C1′—Sn1′—C7′	127.0 (3)	P2—Ag1—P3	106.11 (3)
C1′—Sn1′—C13′	118.2 (3)	P2—Ag1—P4	120.12 (4)
C1′—Sn1′—O1′	93.5 (3)	P3—Ag1—P4	96.84 (3)

The triorganobis(trifluoroacetato)stannate anion was intially refined as cyclopentyldiphenylbis(trifluoroacetato)stannate; however, as the refinement was not satisfactory, it was refined as disordered, but the refinement was still not satisfactory. The refinement was only meaningful when the anion was refined as an $[Sn(C_6H_5)_2(C_5H_9)(O_2CCF_3)_2]$ anion that is disordered with respect to an $[Sn(C_6H_5)_3(O_2CCF_3)_2]$ anion. As the disorder refined to almost 50:50, the occupancies were fixed as 0.5 each. A number of restraints were applied to model the disorder. The disorder also affected the cation, and the phenyl rings of the phosphine ligand were refined as rigid hexagons with 1.39 Å bonds. In the cyclopentyl ring, the C—C distances were restrained to 1.50 (1) Å and the 1,3-related distances to 2.45 (2) Å. For the carboxyl fragments, the C—O distances were restrained to 1.25 (1) Å; the C—C distance was restrained to 1.54 (1) Å. The C—F distances were restrained to 1.33 (1) Å; the F···F distance was restrained to 2.17 (1) Å and the C···F distance to 2.35 (2) Å. Additionally, the Sn—C and Sn—O distances for the pairs of unprimed atoms were set to be within 0.01 Å of those for the pairs of primed atoms. The four-atom C—CO_2 fragments were restrained to be nearly planar. The vibrations of the atoms other than Sn, Ag and P were restrained to be approximately isotropic for the F, O and C atoms. H atoms were placed at calculated postions in the riding-model approximation [aromatic C—H = 0.95 Å and aliphatic C—H = 0.99 Å, and $U_{\rm iso}(H) = 1.2U_{\rm eq}(C)$]. The final difference Fourier map had a peak at 1 Å from atom Sn1 and a hole at 1 Å from Sn1′

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

We thank Dr Jan Wikaira of the University of Canterbury for the diffraction measurements, and the University of Malaya (F0190/2004A and IRPA 09-02-03-1021) for generously supporting this work.

References

Bruker (2001). *SADABS, SAINT* and *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.

Chee, C. F., Lo, K. M. & Ng, S. W. (2003). *Acta Cryst.* E**59**, m273–m275.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Kumar Das, V. G., Kuthubutheen, A. J., Balabaskaran, S. & Ng, S. W. (1989). *Main Group Met. Chem.* **12**, 389–424.

Lo, K. M., Kumar Das, V. G. & Ng, S. W. (1999). *Acta Cryst.* C**55**, 889–894.

Lo, K. M. & Ng, S. W. (2004*a*). *Acta Cryst.* E**60**, m715–m716.

Lo, K. M. & Ng, S. W. (2004*b*). *Acta Cryst.* E**60**, m717–m719.

Lo, K. M., Ng, S. W. & Kumar Das, V. G. (1997). *Acta Cryst.* C**53**, 545–546.

Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.

m1480 Teo *et al.* • $[Ag(C_{27}H_{26}P_2)_2][SnPh_{2.5}(C_5H_9)_{0.5}(C_2F_3O_4)_2]$ *Acta Cryst.* (2004). E**60**, m1478–m1480