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Bis[1,2-bis(diphenylphosphino)ethane-κ^2P,P']silver(I) bis(chlorodifluoroacetato-κO)(cyclohexyl)diphenylstannate(IV)

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Key indicators

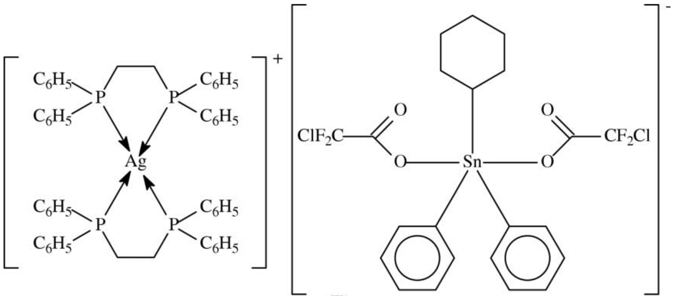
Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.008 Å
Disorder in main residue
R factor = 0.049
wR factor = 0.168
Data-to-parameter ratio = 12.6

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

In the title salt, $[Ag(Ph_2CH_2CH_2Ph_2)_2]^+[c\text{-}HexPh_2Sn(ClF_2CCO_2)_2]^-$ or $[Ag(C_{26}H_{24}P_2)_2][Sn(C_6H_5)_2(C_6H_{11})(C_2ClF_2O_2)_2]$, the Ag^I atom shows a tetrahedral coordination, while the Sn^{IV} atom is in a *trans*-trigonal–bipyramidal coordination.

Comment

The bis(carboxylato)triorganostannate(IV) anion has been isolated as its phosphine-silver(I) salts, as exemplified by, for example, bis[1,2-bis(diphenylphosphino)ethane]silver(I) bis(trifluoroacetato)triphenylstannate(IV), whose crystal structure consists of discrete cations and anions (Chee *et al.*, 2003). The present study involves a triorganostannate having mixed organic groups; the study unsuccessfully attempted the synthesis of a mixed triorganostannate having mixed carboxylate groups. The salt (I) has a tetrahedral $[(Ph_2CH_2CH_2Ph_2)_2Ag]^+$ cation (Fig. 1) and a *trans*-C_3SnO_2 trigonal-bipyramidal $[c\text{-}HexPh_2Sn(ClF_2CCO_2)_2]^-$ anion (Fig. 2). There are no strong interactions between the cation and anion; probably because of this, the two species are severely disordered.



(I)

Experimental

Cyclohexyldiphenyltin(IV) hydroxide (0.18 g, 0.48 mmol) was dissolved in a 1:1 dichloromethane–methanol mixture (50 ml). Chlorodifluoroacetic acid (0.05 ml, 0.5 mmol) and trifluoroacetic acid (0.04 ml, 0.5 mmol) were added. The mixture was heated until the hydroxide dissolved completely. Another solution containing 1,3-bis(diphenylphosphino)ethane (0.4 g, 1.0 mmol) and silver trifluoroacetate (0.11 g, 0.5 mmol) was prepared; this was also heated until the reagents dissolved completely. The two solutions were mixed and and allowed to evaporate at room temperature to obtain single crystals (yield 80%; m.p. 409–411 K). Analysis: found (calculated) C 59.10 (59.12), H 4.58 (4.63)%.

Crystal data

$[Ag(C_{26}H_{24}P_2)_2][Sn(C_6H_5)_2(C_6H_{11})(C_2ClF_2O_2)_2]$
$M_r = 1519.63$
Triclinic, $P\overline{1}$
$a = 11.7453$ (2) Å
$b = 14.7704$ (2) Å
$c = 21.1302$ (3) Å
$\alpha = 97.582$ (1)°
$\beta = 94.641$ (1)°
$\gamma = 105.550$ (1)°
$V = 3474.62$ (9) Å^3
$Z = 2$
Mo $K\alpha$ radiation
$\mu = 0.87$ mm^{-1}
$T = 173$ (2) K
$0.49 \times 0.32 \times 0.16$ mm

Data collection

Bruker APEXII area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\rm min} = 0.735$, $T_{\rm max} = 0.874$
54063 measured reflections
15831 independent reflections
12918 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.028$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.168$
$S = 1.30$
15831 reflections
1252 parameters
1021 restraints
H-atom parameters constrained
$\Delta\rho_{\rm max} = 2.61$ e Å^{-3}
$\Delta\rho_{\rm min} = -1.35$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Sn1—C13	2.144 (10)	Ag1—P3	2.4682 (10)
Sn1—C1	2.161 (4)	Ag1—P2	2.4721 (10)
Sn1—O3	2.176 (9)	Ag1—P1	2.4803 (11)
Sn1—C7	2.186 (8)	Ag1—P4	2.4910 (11)
Sn1—O1	2.270 (6)		
P3—Ag1—P2	123.91 (4)	P3—Ag1—P4	85.16 (4)
P3—Ag1—P1	120.82 (4)	P2—Ag1—P4	127.78 (4)
P2—Ag1—P1	84.65 (3)	P1—Ag1—P4	119.08 (4)

All atoms in both cation and anion are disordered except the Ag, P and methylene C atoms. As the disorder refined to nearly 50:50, the occupancies were fixed at 0.5. All phenyl rings were refined as rigid hexagons (C—C = 1.39 Å). The 1,2-related and 1,3-related distances in the cyclohexyl ring of the anion were restrained to 1.54 (1) and 2.52 (2) Å, respectively. The C—O, C—C, C—Cl, C—F, O···O, O···C, F···F and F···Cl distances in the chlorodifluoroacetate groups were restrained to 1.25 (1), 1.50 (1), 1.75 (1), 1.35 (1), 2.17 (2), 2.39 (2), 2.20 (2) and 2.54 (2) Å, respectively. The four-atom C–C(O)–O units were each restrained to planarity within 0.01 Å. The displacement parameters of each set of one Cl and two F atoms were set to be equal, and further splitting in the trihaloacetato groups rendered the refinement unstable. The pair of Sn—C/Sn′—C′ and Sn—O/Sn′—O′ distances were restrained to within 0.01 Å of each other. Similarly, distances for each pair of P—C and P—C′ bonds were restrained to within 0.02 Å of each other. C-bound H atoms were placed at calculated positions (C–H = 0.95–1.00 Å), and were included in the refinement in the riding-model approximation with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$. The vibrations of the halogen, O and C atoms were all restrained to be nearly isotropic. In the final difference Fourier map, the largest peak is 1.2 Å from C19 and the deepest hole is 0.7 Å from Ag1.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2007).

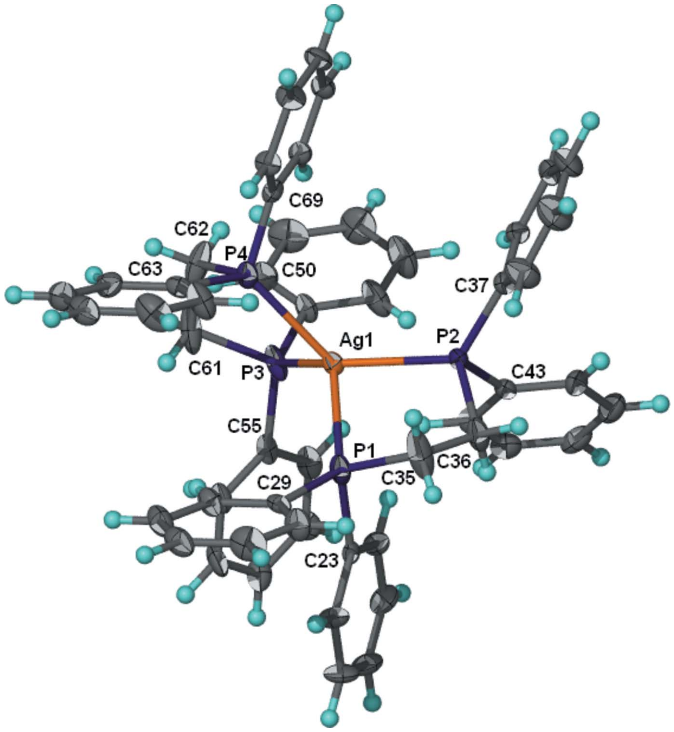


Figure 1
The structure of the cation of (I). Displacement ellipsoids are drawn at the 70% probability level. Only one of the disorder components is shown. H atoms are shown as spheres of arbitrary radius.

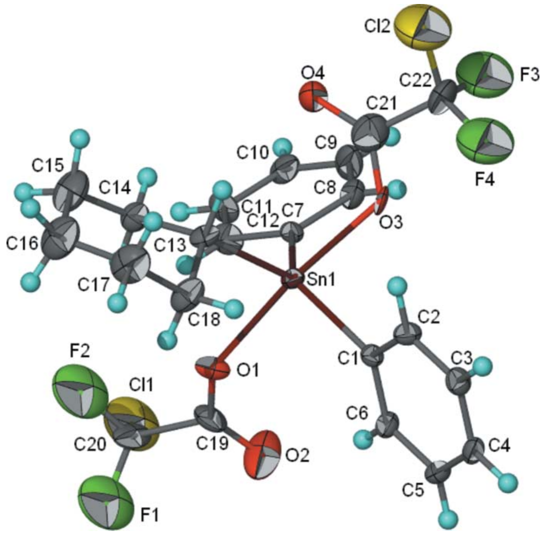


Figure 2
The structure of the anion of (I). Displacement ellipsoids are drawn at the 70% probability level. Only one of the disorder components is shown. H atoms are shown as spheres of arbitrary radius.

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Acta Cryst. (2007). E**63**, m1365–m1367 Teo *et al.* • $[Ag(C_{26}H_{24}P_2)_2][Sn(C_6H_5)_2(C_6H_{11})(C_2ClF_2O_2)_2]$ **m1367**