

# Tris[ $\mu$ -bis(diphenylphosphino)methane- $\kappa^2P:P'$ ]-di- $\mu_3$ -sulfanido-trisilver(I) hexafluoroantimonate dichloromethane disolvate

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## Key indicators

Single-crystal X-ray study

$T = 293\text{ K}$

Mean  $\sigma(\text{C}-\text{C}) = 0.019\text{ \AA}$

$R$  factor = 0.080

$wR$  factor = 0.210

Data-to-parameter ratio = 17.5

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

The  $\mu_3$ -SH dicapped trisilver compound  $[\text{Ag}_3(\mu\text{-dppm})_3(\mu_3\text{-SH})_2]\text{SbF}_6 \cdot 2\text{CH}_2\text{Cl}_2$  [dppm is bis(diphenylphosphino)methane,  $\text{C}_{25}\text{H}_{22}\text{P}_2$ ], formed by the reaction between  $[\text{Ag}_2(\mu\text{-dppm})_2(\text{MeCN})_2](\text{SbF}_6)_2$  and  $[\text{Pd}(\text{phen})(\text{tdt})]$  (phen is *o*-phenanthroline and tdt is toluene-3,4-dithiolate) through the disruption of C–S bonds, exhibits a trigonal bipyramid core, with two  $\mu_3$ -SH ligands at the apices. The Ag atoms are located in tetrahedral environments with  $\text{P}_2\text{S}_2$  donors.

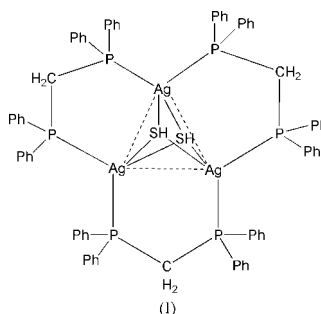
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## Comment

There is considerable interest in designing novel metal complexes with mixed phosphorus- and sulfur-containing ligands, owing to their unique optical and opto-electronic properties (Yam *et al.*, 2000; Tang *et al.*, 2002). It is well known that  $\text{S}^{2-}$  and  $\text{SH}^-$  as versatile coordinating groups could exhibit various bonding modes, to produce metal cluster complexes with diverse structural topologies (Kitagawa *et al.*, 1990; Su *et al.*, 1998). Here we report the crystal structure of a trisilver complex,  $[\text{Ag}_3(\mu\text{-dppm})_3(\mu_3\text{-SH})_2]\text{SbF}_6$  (I), which resulted from the self-assembly between the metal components  $[\text{Pd}(\text{phen})(\text{tdt})]$  (phen is *o*-phenanthroline and tdt is toluene-3,4-dithiolate) and  $[\text{Ag}_2(\mu_2\text{-dppm})_2(\text{MeCN})_2](\text{SbF}_6)_2$  through the disruption of C–S bonds of tdt.



A perspective drawing of the complex cation, with the atomic numbering scheme, is depicted in Fig. 1 and selected bonding parameters are presented in Table 1. The complex consists of the  $[\text{Ag}_3(\mu\text{-dppm})_3(\mu_3\text{-SH})_2]^+$  cation, an  $\text{SbF}_6^-$  anion and dichloromethane solvent molecules. The  $\mu_3$ -SH dicapped trisilver complex exhibits a trigonal bipyramidal core, with two  $\mu_3$ -SH ligands in the apices and the three Ag atoms in equatorial sites. The Ag atoms are located in distorted tetrahedral environments with a  $\text{P}_2\text{S}_2$  donor set. The Ag–Ag distances are in the range 3.337 (1)–3.477 (1) Å, and are shorter than the sum of van der Waals radii for two Ag atoms, indicating a weak metal bonding contact. The Ag–S distances are in the range 2.661 (3)–2.825 (3) Å and the Ag–P bond distances are in the range 2.465 (2)–2.491 (3) Å, close to

those found in other similar complexes of silver (Albano *et al.*, 1999; Meyer *et al.*, 1989; Lang *et al.*, 1993; Henkel *et al.*, 1988).

## Experimental

The title compound was isolated from the reaction between  $[\text{Ag}_2(\mu_2\text{-dppm})_2(\text{MeCN})_2](\text{SbF}_6)_2$  (Diez *et al.*, 1987) and  $[\text{Pd}(\text{phen})(\text{tdt})]$  (Scott & Richard, 1996) in an equimolar ratio in dichloromethane under anaerobic condition for 12 h. Well shaped pale yellow crystals, suitable for X-ray diffraction study, were grown by slow diffusion of diethyl ether into a dichloromethane solution at room temperature.

### Crystal data

$[\text{Ag}_3(\text{C}_{25}\text{H}_{22}\text{P}_2)_3(\text{HS})_2]\text{SbF}_6 \cdot 2\text{CH}_2\text{Cl}_2$	$D_x = 1.597 \text{ Mg m}^{-3}$
$M_r = 1948.45$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 8192 reflections
$a = 16.0813 (1) \text{ \AA}$	$\theta = 1.3\text{--}25.0^\circ$
$b = 18.0049 (2) \text{ \AA}$	$\mu = 1.40 \text{ mm}^{-1}$
$c = 28.1089 (3) \text{ \AA}$	$T = 293 (2) \text{ K}$
$\beta = 95.4170 (10)^\circ$	Prism, pale yellow
$V = 8102.36 (13) \text{ \AA}^3$	$0.72 \times 0.60 \times 0.46 \text{ mm}$
$Z = 4$	

### Data collection

Siemens SMART CCD diffractometer	14 088 independent reflections
$\omega$ scans	10 084 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$R_{\text{int}} = 0.047$
$T_{\text{min}} = 0.354$ , $T_{\text{max}} = 0.525$	$\theta_{\text{max}} = 25.0^\circ$
25 151 measured reflections	$h = -10 \rightarrow 19$
	$k = -13 \rightarrow 21$
	$l = -33 \rightarrow 32$

### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0537P)^2 + 77.3298P]$
$R[F^2 > 2\sigma(F^2)] = 0.080$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.210$	$(\Delta/\sigma)_{\text{max}} = 0.006$
$S = 1.21$	$\Delta\rho_{\text{max}} = 1.50 \text{ e \AA}^{-3}$
14 088 reflections	$\Delta\rho_{\text{min}} = -1.23 \text{ e \AA}^{-3}$
807 parameters	
H-atom parameters constrained	

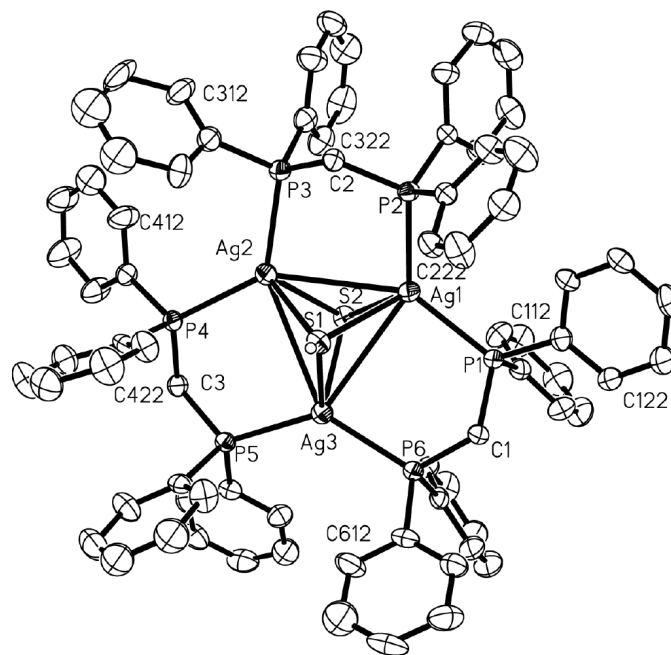
**Table 1**

Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ).

Ag1–P2	2.465 (2)	Ag2–S2	2.661 (3)
Ag1–P1	2.465 (2)	Ag2–S1	2.825 (3)
Ag1–S2	2.734 (3)	Ag3–P5	2.483 (3)
Ag1–S1	2.760 (3)	Ag3–P6	2.491 (3)
Ag1–Ag2	3.3369 (11)	Ag3–S1	2.686 (3)
Ag2–P4	2.468 (2)	Ag3–S2	2.771 (3)
Ag2–P3	2.469 (3)		
P2–Ag1–P1	126.27 (8)	P4–Ag2–P3	125.91 (9)
P2–Ag1–S1	96.47 (8)	P3–Ag2–S2	116.19 (9)
P2–Ag1–Ag2	86.89 (6)	P5–Ag3–S2	103.27 (9)
P1–Ag1–Ag2	146.48 (6)	Ag3–S1–Ag1	79.33 (7)
S2–Ag1–Ag2	50.81 (6)	Ag3–S1–Ag2	77.18 (7)
S1–Ag1–Ag2	54.21 (6)	Ag1–S1–Ag2	73.38 (7)

All H atoms were included in calculated positions, with C–H distances of 0.93 ( $sp^2$ ) and 0.97  $\text{\AA}$  ( $sp^3$ ), and H–S distances of 1.20  $\text{\AA}$ . They were included in the refinement in riding motion approximation with  $U_{\text{iso}} = 1.2U_{\text{eq}}$  ( $1.5U_{\text{eq}}$  for  $sp^3$ ) of the carrier atom.

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1994); data reduction: SAINT and XPREP (Siemens,



**Figure 1**

A view of the cation of (I), with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

1994); program(s) used to solve structure: SHELXTL (Siemens, 1994); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

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