

Bis[μ -bis(diphenylphosphino)amine- $\kappa P:P'$]-bis[acetonitrilesilver(I)] tris(*cis*-1,2-dicyanoethylene-1,2-dithiolate)molybdate(IV)

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Key indicators

Single-crystal X-ray study

$T = 293\text{ K}$

Mean $\sigma(\text{C}-\text{C}) = 0.014\text{ \AA}$

R factor = 0.063

w R factor = 0.135

Data-to-parameter ratio = 15.6

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

The reaction between the metal diphosphine $[\text{Ag}_2(\text{dppa})_2](\text{BF}_4)_2$ [dppa is bis(diphenylphosphino)amine, $\text{C}_{24}\text{H}_{21}\text{N}_2\text{P}_2$] and the metal thiolate $[\text{Bu}_4\text{N}]_2[\text{Mo}(\text{mnt})_3]$ (mnt is *cis*-1,2-dicyanoethylene-1,2-dithiolate, $\text{C}_4\text{N}_2\text{S}_2$) gave the title compound, $[\text{Ag}_2(\text{dppa})_2(\text{MeCN})_2][\text{Mo}(\text{mnt})_3]$. The $\text{Ag}\cdots\text{Ag}$ distance of 2.9501 (13) \AA is shorter than the sum of van der Waals radii for two Ag atoms, indicating a weak metal–metal contact in the complex. The cation lies on an inversion centre and the anion lies on a twofold axis.

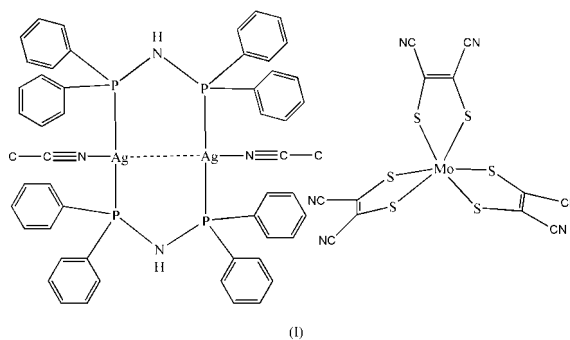
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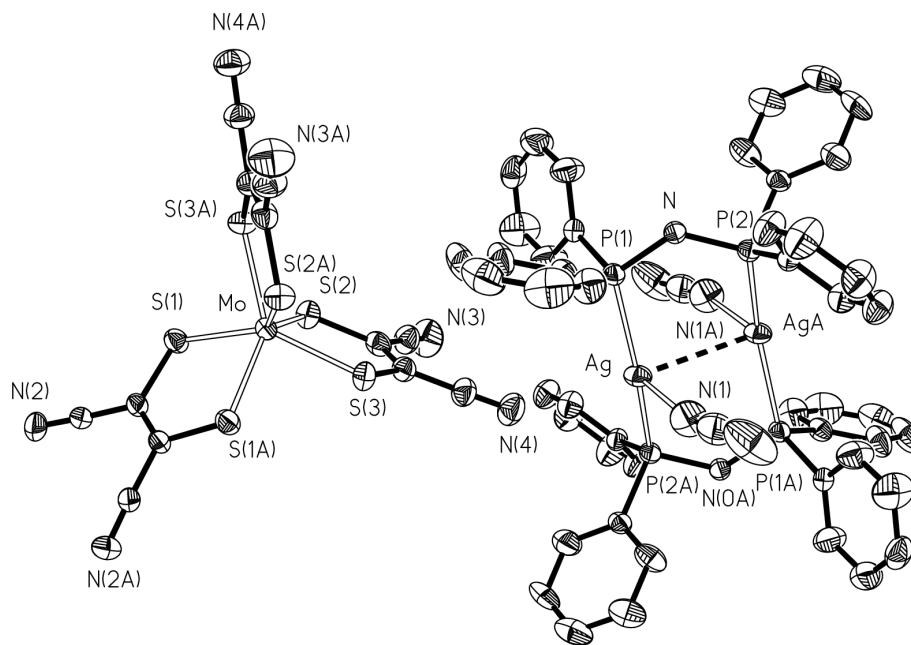
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Comment

Interest in the metal complexes of diphosphine compounds has grown rapidly in recent years (Yam *et al.*, 1998). We are interested in the design of photoluminescent heterometallic cluster complexes by self-assembly between two metal components, one with potential bridging donors and the other with substitutable or vacant coordination sites (Xu *et al.*, 2002). Thus, the reaction between $[\text{Bu}_4\text{N}]_2[\text{Mo}(\text{mnt})_3]$ (mnt is *cis*-1,2-dicyanoethylene-1,2-dithiolate), with potential bridging sulfur donors, and $[\text{Ag}_2(\text{dppa})_2]^{2+}$ [dppa is bis(diphenylphosphino)amine], with vacant coordination sites, was expected to afford an Ag–Mo heteronuclear species. However, the only isolated product of the reaction between $[\text{Bu}_4\text{N}]_2[\text{Mo}(\text{mnt})_3]$ and $[\text{Ag}_2(\text{dppa})_2](\text{BF}_4)_2$ was the title compound $[\text{Ag}_2(\text{dppa})_2(\text{MeCN})_2][\text{Mo}(\text{mnt})_3]$, (I), in which



$[\text{Mo}(\text{mnt})_3]^{2-}$ acts only as an anion. A perspective drawing of the complex with the atomic numbering scheme is depicted in Fig. 1 and selected bonding parameters are presented in Table 1. The Ag^{I} atoms are located in a distorted triangular-planar environment of a P_2N chromophore and the $\text{Ag}\cdots\text{Ag}$ distance [2.9501 (13) \AA] is shorter than the sum of the van der Waals radii of two Ag atoms, indicating a weak metal–metal contact; the two Ag atoms are bridged by P–N–P groups of two dppa molecules. The Mo atom is located in an octahedral environment of six S atoms, and the Mo–S distances [2.382 (2)–2.385 (2) \AA] are similar to those observed in $[\text{Ph}_4\text{As}]_2[\text{Mo}(\text{mnt})_3]$ (Brown & Stiefel, 1973).


Figure 1

A view of the title complex with the atomic numbering scheme; symmetry-related atoms have a suffix *A*. Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity.

Experimental

The starting materials $[\text{Bu}_4\text{N}]_2[\text{Mo}(\text{mnt})_3]$ (Stiefel *et al.*, 1970) and $[\text{Ag}_2(\text{dppa})_2](\text{BF}_4)_2$ (Sekabange *et al.*, 2002) were prepared according to the reported procedures. The reaction between equimolar quantities of $[\text{Ag}_2(\text{dppa})_2](\text{BF}_4)_2$ and $[\text{Bu}_4\text{N}]_2[\text{Mo}(\text{mnt})_3]$ was carried out in dichloromethane under anaerobic conditions for 1 d, after which the solvent was removed *in vacuo* and the residue was extracted with acetonitrile. Well-shaped dark-green crystals suitable for X-ray diffraction analysis were grown by slow diffusion of diethyl ether into the acetonitrile solution.

Crystal data

$[\text{Ag}_2(\text{C}_{24}\text{H}_{21}\text{N}_2\text{P}_2)_2(\text{C}_2\text{H}_3\text{N})_2]\cdot$
 $[\text{Mo}(\text{C}_4\text{N}_2\text{S}_2)_3]$
 $M_r = 1585.1$
 Orthorhombic, *Pbcn*
 $a = 17.181(3) \text{ \AA}$
 $b = 15.313(3) \text{ \AA}$
 $c = 26.386(5) \text{ \AA}$
 $V = 6942(2) \text{ \AA}^3$
 $Z = 4$

$D_x = 1.517 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 Cell parameters from 5955
 reflections
 $\theta = 1.5\text{--}25.1^\circ$
 $\mu = 1.06 \text{ mm}^{-1}$
 $T = 293(2) \text{ K}$
 Prism, dark green
 $0.36 \times 0.34 \times 0.28 \text{ mm}$

Data collection

Siemens SMART CCD
 diffractometer
 ω scans
 Absorption correction: multi-scan
 (*SADABS*; Sheldrick, 1996)
 $T_{\text{min}} = 0.729$, $T_{\text{max}} = 1.000$
 19 737 measured reflections

6143 independent reflections
 3749 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.080$
 $\theta_{\text{max}} = 25.1^\circ$
 $h = -13 \rightarrow 20$
 $k = -18 \rightarrow 17$
 $l = -31 \rightarrow 21$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.063$
 $wR(F^2) = 0.135$
 $S = 1.17$
 6143 reflections
 393 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0205P)^2 + 30.6018P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.40 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.55 \text{ e \AA}^{-3}$

Table 1

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

Mo—S2	2.382 (2)	Ag...Ag ⁱ	2.9501 (13)
Mo—S1	2.384 (2)	P1—N	1.677 (6)
Mo—S3	2.385 (2)	P2—N	1.688 (6)
Ag—P1	2.434 (2)	P2—Ag ⁱ	2.430 (2)
Ag—N1	2.457 (9)	N1—C1	1.111 (13)
S2 ⁱⁱ —Mo—S2	141.21 (11)	P1—Ag—N1	99.9 (2)
S2 ⁱⁱ —Mo—S1	130.07 (8)	P2 ⁱ —Ag...Ag ⁱ	88.65 (5)
S2—Mo—S1	81.88 (7)	P1—Ag...Ag ⁱ	88.52 (5)
S2—Mo—S1 ⁱⁱ	130.07 (8)	N1—Ag...Ag ⁱ	84.5 (2)
S1—Mo—S1 ⁱⁱ	81.70 (10)	N—P1—Ag	114.0 (2)
S2 ⁱⁱ —Mo—S3	81.93 (8)	C21—P1—Ag	115.5 (3)
S2—Mo—S3	81.54 (7)	C11—P1—Ag	112.9 (3)
S1—Mo—S3	141.35 (8)	N—P2—Ag ⁱ	114.0 (2)
S1 ⁱⁱ —Mo—S3	82.77 (7)	C41—P2—Ag ⁱ	112.2 (2)
S2 ⁱⁱ —Mo—S3 ⁱⁱ	81.54 (7)	C31—P2—Ag ⁱ	116.7 (3)
S3—Mo—S3 ⁱⁱ	128.69 (11)	P1—N—P2	125.9 (3)
P2 ⁱ —Ag—P1	156.48 (7)	C1—N1—Ag	168.3 (10)
P2 ⁱ —Ag—N1	103.0 (2)		

Symmetry codes: (i) $1 - x, 1 - y, 1 - z$; (ii) $1 - x, \frac{1}{2} - z$.

The positions of the H atoms were generated geometrically (C—H fixed at 0.96 Å); these atoms were assigned isotropic displacement parameters and allowed to ride on their respective parent C atoms in the refinement.

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