

Bis[μ_2 -bis(diphenylphosphino)methane]-bis(μ_2 -ethane-1,2-dithiolato)- μ_4 -sulfido- μ_2 -sulfido-disulfidodimolybdenum(V)-disilver(I) dimethylformamide trisolvate

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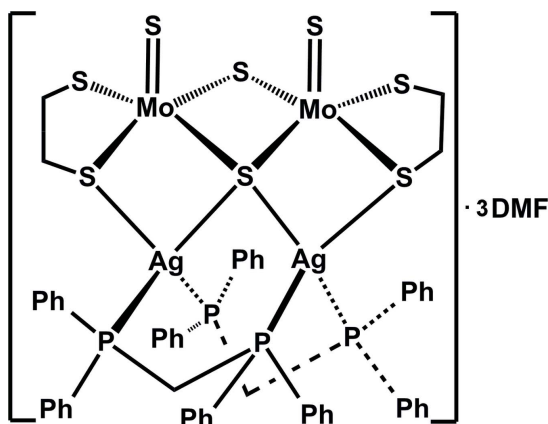
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 Key indicators: single-crystal X-ray study; $T = 223$ K; mean $\sigma(\text{C}-\text{C}) = 0.015$ Å; R factor = 0.085; wR factor = 0.188; data-to-parameter ratio = 16.0.

Treatment of $[\text{Et}_4\text{N}]_2[(\text{edt})_2\text{Mo}_2\text{S}_2(\mu\text{-S})_2]$ (edt = ethanedithiolate) with two equivalents of $\text{Ag}(\text{CH}_3\text{CN})_4\text{ClO}_4$ in the presence of bis(diphenylphosphino)methane (dppm) ligand gives rise to the title tetranuclear cluster, $[\text{Ag}_2\text{Mo}_2(\text{C}_2\text{H}_4\text{S}_2)_2\text{S}_4(\text{C}_{25}\text{H}_{22}\text{P}_2)_2] \cdot 3\text{C}_3\text{H}_7\text{NO}$. The complex molecule and one of the dimethylformamide (DMF) solvent molecules occupy special positions on a mirror plane. The molecular structure of the complex may be visualized as being built of $[\text{Mo}_2\text{S}_2(\mu\text{-S})_2(\text{edt})_2]^{2-}$ dianions and $[\text{Ag}_2(\text{dppm})_2]^{2+}$ dications connected by two $\text{Ag}-\mu\text{-S}_{\text{edt}}$ and two $\text{Ag}-\mu_4\text{-S}$ bonds.

Related literature

For general background to the chemistry of sulfido-bridged dinuclear clusters consisting of a $[\text{M}_2\text{S}_4]$ core ($M = \text{Mo}, \text{W}$) and various transition metals, see: Kuwata & Hidai (2001); Curtis *et al.* (1997); Halbert *et al.* (1985); Kawaguchi *et al.* (1997); Brunner *et al.* (1985). For the synthesis and structure of the starting material, see: Pan *et al.* (1984). For related structures, see: Zhu *et al.* (1990); Lin *et al.* (1997); Wei *et al.* (2008).



Experimental

Crystal data

| | |
|---|-----------------------------------|
| $[\text{Ag}_2\text{Mo}_2(\text{C}_2\text{H}_4\text{S}_2)_2\text{S}_4(\text{C}_{25}\text{H}_{22}\text{P}_2)_2] \cdot 3\text{C}_3\text{H}_7\text{NO}$ | $V = 7076$ (3) Å ³ |
| $M_r = 1708.3$ | $Z = 4$ |
| Orthorhombic, <i>Pnma</i> | Mo $K\alpha$ radiation |
| $a = 26.022$ (5) Å | $\mu = 1.26$ mm ⁻¹ |
| $b = 21.375$ (4) Å | $T = 223$ K |
| $c = 12.721$ (3) Å | $0.35 \times 0.20 \times 0.07$ mm |

Data collection

| | |
|--|--|
| Rigaku Mercury diffractometer | 57121 measured reflections |
| Absorption correction: multi-scan (<i>REQAB</i> ; Jacobson, 1998) | 6401 independent reflections |
| $T_{\text{min}} = 0.746$, $T_{\text{max}} = 0.915$ | 5931 reflections with $I > 2\sigma(I)$ |
| | $R_{\text{int}} = 0.090$ |

Refinement

| | |
|---------------------------------|---|
| $R[F^2 > 2\sigma(F^2)] = 0.085$ | 401 parameters |
| $wR(F^2) = 0.188$ | H-atom parameters constrained |
| $S = 1.35$ | $\Delta\rho_{\text{max}} = 0.95$ e Å ⁻³ |
| 6401 reflections | $\Delta\rho_{\text{min}} = -0.58$ e Å ⁻³ |

Data collection: *CrystalClear* (Rigaku/MSC, 2001); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: YA2110).

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Bis[μ_2 -bis(diphenylphosphino)methane]bis(μ_2 -ethane-1,2-dithiolato)- μ_4 -sulfido- μ_2 -sulfido-disulfidodimolybdenum(V)disilver(I) dimethylformamide trisolvate

X.-C. Wang and X.-L. You

Comment

In the past decades, the chemistry of the sulfido-bridged dinuclear clusters consisting of $[M_2S_4]$ core ($M = \text{Mo}, \text{W}$) and various transition metals has attracted much attention. For example, precursors $[(\text{dtc})_2\text{Mo}_2\text{S}_2(\mu\text{-S})_2]$ ($\text{dtc} = \text{S}_2\text{CNEt}_2$) (Kuwata & Hidai, 2001) and $[\text{Cp}^x_2\text{Mo}_2\text{S}_2(\mu\text{-S})_2]$ ($\text{Cp}^x = \text{pentamethyl-, pentaethyl- or pentabutyl-cyclopentadienyl}$) (Curtis *et al.*, 1997; Halbert *et al.*, 1985; Kawaguchi *et al.*, 1997; Brunner *et al.*, 1985) were shown to react with transition metals to form both incomplete cubane-like $[\text{Mo}_2\text{M}'\text{S}_4]$ and complete cubane-like $[\text{Mo}_2\text{M}'_2\text{S}_4]$ clusters. The type of cluster formed is dependent upon the ability of the terminal S_t and the bridging $\mu\text{-S}_\text{b}$ groups in $[\text{Mo}_2\text{S}_4]$ core to bind further metal centers. Recently, another precursor $[\text{Et}_4\text{N}]_2[(\text{edt})_2\text{Mo}_2\text{S}_2(\mu\text{-S})_2]$ (**1**), which has a chelating edt at each Mo site of the $[\text{Mo}_2\text{S}_4]$ core, has been introduced; its terminal S_t , the doubly bridging $\mu\text{-S}_\text{b}$, and chelating S_{edt} are capable of binding Cu(I) centers (Zhu *et al.*, 1990; Lin *et al.*, 1997; Wei *et al.*, 2008). However, until now, only quite limited data have been reported involving precursor **1** bound to Ag(I) complexes. In this paper we describe the result of our efforts to generate a Mo/Ag/S cluster $[\text{Mo}_2\text{S}_2(\mu\text{-S})_2(\text{edt})_2\text{Ag}_2(\text{dppm})_2] \cdot 3\text{DMF}$ (**2.3DMF**) by reaction of **1** with two equivalents of $\text{Ag}(\text{CH}_3\text{CN})_4\text{ClO}_4$ in the presence of dppm ligand.

The asymmetric unit of **2.3DMF** contains half of the $[\text{Mo}_2\text{S}_2(\mu\text{-S})_2(\text{edt})_2\text{Ag}_2(\text{dppm})_2]$ molecule, and one and a half DMF molecules (Fig. 1). The complex may be considered as having a $[\text{Mo}_2\text{S}_2(\mu\text{-S})_2(\text{edt})_2]^{2-}$ anionic unit bound to a $[\text{Ag}_2(\text{dppm})_2]^{2+}$ cation *via* two $\text{Ag}-\mu\text{-S}_{\text{edt}}$ and two $\text{Ag}-\mu_4\text{-S}_\text{b}$ bonds. A crystallographic mirror plane runs through S3, S5, C27 and C28 atoms. Each Mo center has a distorted square pyramidal environment, consisting of one terminal S_t , one S_{edt} , one $\mu\text{-S}_{\text{edt}}$, and two $\mu\text{-S}$ atoms. Each Ag center has a distorted tetrahedral coordination made up of one $\mu\text{-S}_{\text{edt}}$, one $\mu_4\text{-S}$ and two P atoms from two dppm ligands. The $\text{Ag1}-\text{S5}$ bond [2.924 (3) Å], involving the $\mu_4\text{-S}$ atom, is much longer than $\text{Ag1}-\text{S4}$ with the S_{edt} atom [2.588 (2) Å]. The eight-membered $[\text{Ag}-\text{P}-\text{C}-\text{P}-\text{Ag}-\text{P}-\text{C}-\text{P}]$ ring in the $[\text{Ag}_2(\text{dppm})_2]^{2+}$ dication adopts a boat conformation. The $\text{Mo}\cdots\text{Mo}$ distance [2.8772 (14) Å] is longer than that in the precursor **1** [2.863 (3) Å] (Pan *et al.*, 1984). The $\text{Mo1}-\mu\text{-S4}$ bond length is elongated by 0.05 Å relative to that of $\text{Mo1}-\text{S2}$ as the S4 atom is involved in coordination to the Ag1 atom. The $\text{Mo1}-\text{S5}$ bond [2.344 (2) Å] is slightly longer than $\text{Mo1}-\text{S3}$ [2.322 (2) Å] due to the μ_4 -character of the S5 atom.

Experimental

To a solution of **1** (76 mg, 0.1 mmol) in 10 ml of CH_2Cl_2 was added dropwise a solution of $\text{Ag}(\text{CH}_3\text{CN})_4\text{ClO}_4$ (29 mg, 0.2 mmol) in 20 ml of MeCN. A bulk of deep red precipitate was formed within s. The red slurry was stirred for 10 minutes, and a solution of dppm (79 mg, 0.2 mmol) in CH_2Cl_2 (10 ml) was added. The resulting mixture was stirred for 30 min, forming a homogenous red solution. Addition of MeOH to this solution yielded a red microcrystalline solid, which was collected

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by filtration, washed with MeCN and Et₂O, and dried *in vacuo*. Recrystallization of the solid in DMF/*i*-PrOH afforded red crystals of **2.3DMF** two days later. Yield: 63 mg (50% based on Mo).

Refinement

Even though packing analysis shows solvent accessible voids, our attempts to locate additional solvent proved unsuccessful, and none of the geometrically placed atoms, centered around the void, could be reasonably refined. Because of the quick loss of crystallinity upon removal from the mother liquor, the structure has a limited accuracy with high *R*-factors and goodness of fit; optimization of weighting scheme results in high value of the second coefficient. Some of the phenyl atoms show intense thermal motion, however attempts to introduce disorder of the phenyl ring did not produce noticeable improvement of the accuracy of the model.

All H atoms were placed geometrically (C—H 0.93 Å for aromatic and formate, 0.96 Å and 0.97 Å for methyl and methylene groups respectively) and included in the refinement in the riding motion approximation with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}$ of the parent atom [$1.5U_{\text{eq}}$ for methyl groups].

Figures

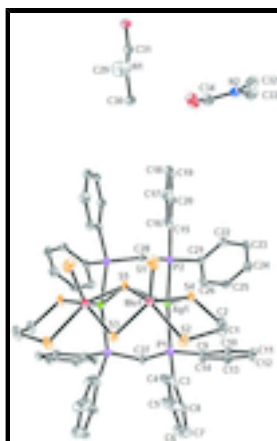


Fig. 1. Molecular structure of **2.3DMF** with thermal ellipsoids, drawn at 30% probability level; hydrogen atoms are omitted for clarity. The unlabeled atoms are derived from the reference atoms by means of the $(x, 1/2 - y, z)$ symmetry transformation.

Bis[μ_2 -bis(diphenylphosphino)methane]bis(μ_2 -ethane-1,2-dithiolato)- μ_4 -sulfido- μ_2 -sulfido-disulfidodimolybdenum(V)disilver(I) dimethylformamide trisolvate

Crystal data

[Ag₂Mo₂(C₂H₄S₂)₂S₄(C₂₅H₂₂P₂)₂] \cdot 3C₃H₇NO

$M_r = 1708.3$

Orthorhombic, *Pnma*

Hall symbol: -P 2ac 2n

$a = 26.022$ (5) Å

$b = 21.375$ (4) Å

$c = 12.721$ (3) Å

$V = 7076$ (3) Å³

$Z = 4$

$F_{000} = 3448$

$D_x = 1.604$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 7734 reflections

$\theta = 2.0$ – 25.0°

$\mu = 1.26$ mm⁻¹

$T = 223$ K

Platelet, red

$0.35 \times 0.20 \times 0.07$ mm

Data collection

| | |
|---|--|
| Rigaku Mercury diffractometer | 6401 independent reflections |
| Radiation source: fine-focus sealed tube | 5931 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{\text{int}} = 0.090$ |
| $T = 223$ K | $\theta_{\text{max}} = 25.0^\circ$ |
| ω scans | $\theta_{\text{min}} = 3.4^\circ$ |
| Absorption correction: Multi-scan (REQAB; Jacobson, 1998) | $h = -30 \rightarrow 30$ |
| $T_{\text{min}} = 0.746$, $T_{\text{max}} = 0.915$ | $k = -25 \rightarrow 25$ |
| 57121 measured reflections | $l = -15 \rightarrow 15$ |

Refinement

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|--|--|
| Refinement on F^2 | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.085$ | H-atom parameters constrained |
| $wR(F^2) = 0.188$ | $w = 1/[\sigma^2(F_o^2) + (0.0747P)^2 + 33.869P]$ |
| $S = 1.35$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| 6401 reflections | $(\Delta/\sigma)_{\text{max}} < 0.001$ |
| 401 parameters | $\Delta\rho_{\text{max}} = 0.95 \text{ e } \text{\AA}^{-3}$ |
| Primary atom site location: structure-invariant direct methods | $\Delta\rho_{\text{min}} = -0.58 \text{ e } \text{\AA}^{-3}$ |
| | Extinction correction: none |

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ | Occ. (<1) |
|-----|--------------|--------------|--------------|----------------------------------|-----------|
| Ag1 | 0.00904 (2) | 0.17618 (3) | 0.71271 (5) | 0.0236 (2) | |
| Mo1 | 0.12522 (3) | 0.18270 (3) | 0.92175 (5) | 0.0211 (2) | |
| P1 | -0.07622 (8) | 0.17918 (10) | 0.79382 (16) | 0.0218 (5) | |
| S1 | 0.20195 (11) | 0.16455 (14) | 0.8910 (3) | 0.0520 (7) | |
| O1 | 0.3659 (4) | 0.2500 | -0.0188 (7) | 0.038 (2) | |

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| | | | | |
|------|-------------|------------|-------------|-------------|
| N1 | 0.3070 (4) | 0.2500 | 0.1140 (8) | 0.025 (2) |
| C1 | 0.0888 (5) | 0.0364 (4) | 0.9861 (8) | 0.046 (3) |
| H9A | 0.1210 | 0.0199 | 0.9597 | 0.055* |
| H9B | 0.0743 | 0.0058 | 1.0341 | 0.055* |
| C2 | 0.0528 (4) | 0.0471 (4) | 0.8971 (7) | 0.038 (2) |
| H16A | 0.0192 | 0.0582 | 0.9243 | 0.045* |
| H16B | 0.0493 | 0.0089 | 0.8565 | 0.045* |
| C3 | -0.0801 (4) | 0.1653 (4) | 0.9351 (7) | 0.028 (2) |
| C4 | -0.0361 (5) | 0.1676 (6) | 0.9945 (8) | 0.056 (3) |
| H8A | -0.0049 | 0.1781 | 0.9634 | 0.067* |
| C5 | -0.0384 (7) | 0.1543 (8) | 1.1014 (9) | 0.086 (5) |
| H1A | -0.0086 | 0.1561 | 1.1416 | 0.103* |
| C6 | -0.0835 (8) | 0.1388 (6) | 1.1481 (10) | 0.083 (5) |
| H2A | -0.0845 | 0.1296 | 1.2195 | 0.099* |
| C7 | -0.1276 (7) | 0.1368 (6) | 1.0894 (10) | 0.071 (5) |
| H6A | -0.1588 | 0.1270 | 1.1210 | 0.085* |
| C8 | -0.1255 (5) | 0.1492 (5) | 0.9838 (8) | 0.044 (3) |
| H5A | -0.1554 | 0.1468 | 0.9441 | 0.053* |
| C9 | -0.1156 (3) | 0.1152 (4) | 0.7415 (6) | 0.0232 (18) |
| C10 | -0.0974 (4) | 0.0548 (4) | 0.7605 (7) | 0.0274 (19) |
| H22A | -0.0673 | 0.0489 | 0.7988 | 0.033* |
| C11 | -0.1241 (4) | 0.0037 (4) | 0.7223 (7) | 0.034 (2) |
| H29A | -0.1122 | -0.0364 | 0.7365 | 0.041* |
| C12 | -0.1677 (4) | 0.0114 (4) | 0.6640 (8) | 0.038 (2) |
| H17A | -0.1854 | -0.0231 | 0.6376 | 0.046* |
| C13 | -0.1850 (4) | 0.0714 (5) | 0.6449 (8) | 0.040 (2) |
| H13A | -0.2148 | 0.0771 | 0.6054 | 0.049* |
| C14 | -0.1595 (3) | 0.1233 (4) | 0.6829 (7) | 0.031 (2) |
| H20A | -0.1718 | 0.1633 | 0.6690 | 0.037* |
| C15 | 0.0903 (3) | 0.1697 (3) | 0.4725 (6) | 0.0202 (17) |
| C16 | 0.1304 (3) | 0.1645 (4) | 0.5441 (7) | 0.0269 (19) |
| H26A | 0.1235 | 0.1638 | 0.6158 | 0.032* |
| C17 | 0.1805 (4) | 0.1605 (5) | 0.5084 (8) | 0.040 (2) |
| H7A | 0.2071 | 0.1577 | 0.5569 | 0.048* |
| C18 | 0.1916 (4) | 0.1606 (4) | 0.4042 (8) | 0.035 (2) |
| H14A | 0.2255 | 0.1576 | 0.3817 | 0.042* |
| C19 | 0.1521 (3) | 0.1651 (4) | 0.3318 (7) | 0.031 (2) |
| H30A | 0.1596 | 0.1646 | 0.2603 | 0.037* |
| C20 | 0.1019 (4) | 0.1704 (4) | 0.3646 (7) | 0.0277 (19) |
| H31A | 0.0756 | 0.1745 | 0.3154 | 0.033* |
| C21 | -0.0123 (3) | 0.1164 (4) | 0.4566 (6) | 0.0224 (17) |
| C22 | 0.0119 (3) | 0.0654 (4) | 0.4086 (7) | 0.029 (2) |
| H25A | 0.0475 | 0.0638 | 0.4033 | 0.035* |
| C23 | -0.0184 (4) | 0.0170 (4) | 0.3688 (7) | 0.034 (2) |
| H19A | -0.0027 | -0.0173 | 0.3373 | 0.041* |
| C24 | -0.0713 (4) | 0.0194 (4) | 0.3758 (7) | 0.036 (2) |
| H18A | -0.0912 | -0.0130 | 0.3488 | 0.043* |
| C25 | -0.0947 (4) | 0.0702 (4) | 0.4231 (7) | 0.034 (2) |
| H15A | -0.1304 | 0.0721 | 0.4270 | 0.041* |

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|------|--------------|--------------|--------------|-------------|------|
| C26 | -0.0657 (3) | 0.1177 (4) | 0.4643 (7) | 0.0282 (19) | |
| H27A | -0.0818 | 0.1511 | 0.4977 | 0.034* | |
| C27 | -0.1134 (5) | 0.2500 | 0.7688 (9) | 0.024 (3) | |
| H33A | -0.1248 | 0.2500 | 0.6963 | 0.028* | |
| H33B | -0.1436 | 0.2500 | 0.8134 | 0.028* | |
| C28 | 0.0006 (4) | 0.2500 | 0.4579 (8) | 0.018 (2) | |
| H34A | 0.0108 | 0.2500 | 0.3845 | 0.021* | |
| H34B | -0.0366 | 0.2500 | 0.4605 | 0.021* | |
| C29 | 0.3438 (7) | 0.2500 | 0.1950 (14) | 0.081 (7) | |
| H3A | 0.3744 | 0.2296 | 0.1711 | 0.121* | 0.50 |
| H3B | 0.3303 | 0.2281 | 0.2549 | 0.121* | 0.50 |
| H3C | 0.3516 | 0.2923 | 0.2144 | 0.121* | 0.50 |
| C30 | 0.2532 (5) | 0.2500 | 0.1436 (13) | 0.042 (3) | |
| H30B | 0.2436 | 0.2909 | 0.1677 | 0.063* | 0.50 |
| H30C | 0.2477 | 0.2202 | 0.1989 | 0.063* | 0.50 |
| H30D | 0.2326 | 0.2389 | 0.0839 | 0.063* | 0.50 |
| C31 | 0.3212 (5) | 0.2500 | 0.0161 (11) | 0.033 (3) | |
| H12 | 0.2950 | 0.2500 | -0.0337 | 0.040* | |
| C32 | 0.2952 (5) | -0.0235 (6) | 0.1897 (11) | 0.067 (4) | |
| H4A | 0.3174 | -0.0415 | 0.1376 | 0.100* | |
| H4B | 0.2776 | -0.0563 | 0.2268 | 0.100* | |
| H4C | 0.3152 | 0.0006 | 0.2384 | 0.100* | |
| C33 | 0.2240 (5) | -0.0106 (7) | 0.0615 (11) | 0.070 (4) | |
| H10A | 0.2012 | -0.0397 | 0.0950 | 0.105* | |
| H10B | 0.2438 | -0.0320 | 0.0090 | 0.105* | |
| H10C | 0.2043 | 0.0220 | 0.0288 | 0.105* | |
| C34 | 0.2541 (4) | 0.0767 (5) | 0.1667 (10) | 0.051 (3) | |
| H11 | 0.2296 | 0.1006 | 0.1316 | 0.061* | |
| P2 | 0.02494 (8) | 0.17823 (9) | 0.52142 (16) | 0.0187 (4) | |
| S2 | 0.10053 (10) | 0.11032 (11) | 1.05640 (18) | 0.0370 (6) | |
| N2 | 0.2582 (3) | 0.0164 (4) | 0.1394 (7) | 0.042 (2) | |
| O2 | 0.2797 (3) | 0.1029 (4) | 0.2337 (7) | 0.069 (3) | |
| S3 | 0.11821 (12) | 0.2500 | 1.0644 (2) | 0.0269 (7) | |
| S4 | 0.07638 (8) | 0.10971 (9) | 0.81247 (16) | 0.0246 (5) | |
| S5 | 0.09599 (12) | 0.2500 | 0.7891 (2) | 0.0243 (6) | |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|-------------|--------------|--------------|
| Ag1 | 0.0258 (3) | 0.0255 (3) | 0.0194 (3) | 0.0015 (3) | 0.0011 (3) | 0.0018 (3) |
| Mo1 | 0.0231 (4) | 0.0186 (4) | 0.0217 (4) | 0.0017 (3) | -0.0047 (3) | 0.0009 (3) |
| P1 | 0.0248 (11) | 0.0197 (10) | 0.0210 (11) | 0.0013 (8) | 0.0007 (9) | 0.0021 (9) |
| S1 | 0.0415 (15) | 0.0467 (16) | 0.0678 (19) | 0.0071 (12) | -0.0064 (14) | -0.0074 (14) |
| O1 | 0.038 (6) | 0.040 (5) | 0.035 (5) | 0.000 | 0.010 (4) | 0.000 |
| N1 | 0.022 (5) | 0.027 (5) | 0.026 (6) | 0.000 | 0.004 (4) | 0.000 |
| C1 | 0.074 (8) | 0.027 (5) | 0.036 (6) | -0.007 (5) | -0.017 (5) | 0.010 (4) |
| C2 | 0.053 (6) | 0.023 (5) | 0.037 (5) | -0.002 (4) | -0.015 (5) | 0.008 (4) |
| C3 | 0.040 (5) | 0.020 (4) | 0.024 (5) | 0.008 (4) | 0.003 (4) | 0.002 (4) |

supplementary materials

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|-----|-------------|-------------|-------------|--------------|--------------|-------------|
| C4 | 0.057 (7) | 0.084 (9) | 0.026 (5) | 0.029 (6) | -0.001 (5) | -0.006 (6) |
| C5 | 0.111 (12) | 0.130 (13) | 0.018 (6) | 0.066 (11) | -0.010 (7) | -0.005 (7) |
| C6 | 0.159 (16) | 0.064 (9) | 0.025 (6) | 0.053 (10) | 0.024 (9) | 0.017 (6) |
| C7 | 0.131 (14) | 0.043 (7) | 0.039 (7) | -0.022 (8) | 0.047 (8) | -0.006 (6) |
| C8 | 0.068 (7) | 0.037 (6) | 0.028 (5) | -0.015 (5) | 0.018 (5) | -0.005 (4) |
| C9 | 0.025 (5) | 0.029 (4) | 0.015 (4) | 0.000 (4) | 0.002 (3) | 0.000 (3) |
| C10 | 0.037 (5) | 0.020 (4) | 0.025 (5) | -0.002 (4) | 0.002 (4) | 0.000 (4) |
| C11 | 0.046 (6) | 0.023 (4) | 0.032 (5) | 0.001 (4) | 0.009 (5) | 0.004 (4) |
| C12 | 0.055 (6) | 0.018 (5) | 0.042 (6) | -0.009 (4) | -0.002 (5) | -0.002 (4) |
| C13 | 0.035 (5) | 0.035 (5) | 0.052 (6) | -0.006 (4) | -0.008 (5) | -0.003 (5) |
| C14 | 0.034 (5) | 0.021 (4) | 0.037 (5) | 0.002 (4) | -0.006 (4) | 0.006 (4) |
| C15 | 0.022 (4) | 0.014 (4) | 0.025 (4) | 0.000 (3) | -0.004 (3) | -0.002 (3) |
| C16 | 0.036 (5) | 0.024 (4) | 0.021 (4) | 0.002 (4) | -0.006 (4) | -0.004 (3) |
| C17 | 0.033 (5) | 0.044 (6) | 0.044 (6) | 0.003 (4) | -0.009 (5) | 0.001 (5) |
| C18 | 0.025 (5) | 0.029 (5) | 0.050 (6) | 0.000 (4) | 0.009 (4) | 0.000 (4) |
| C19 | 0.033 (5) | 0.030 (5) | 0.030 (5) | -0.005 (4) | 0.007 (4) | 0.002 (4) |
| C20 | 0.035 (5) | 0.028 (5) | 0.020 (4) | 0.000 (4) | 0.000 (4) | 0.000 (4) |
| C21 | 0.029 (4) | 0.019 (4) | 0.019 (4) | 0.001 (3) | -0.005 (4) | 0.001 (3) |
| C22 | 0.028 (5) | 0.027 (4) | 0.032 (5) | -0.001 (4) | 0.004 (4) | 0.000 (4) |
| C23 | 0.046 (6) | 0.023 (5) | 0.032 (5) | 0.000 (4) | 0.000 (4) | -0.009 (4) |
| C24 | 0.040 (5) | 0.034 (5) | 0.034 (5) | -0.011 (4) | -0.006 (4) | -0.010 (4) |
| C25 | 0.028 (5) | 0.042 (5) | 0.033 (5) | -0.010 (4) | 0.007 (4) | -0.003 (4) |
| C26 | 0.025 (5) | 0.033 (5) | 0.026 (5) | 0.002 (4) | 0.002 (4) | -0.007 (4) |
| C27 | 0.032 (7) | 0.015 (5) | 0.023 (6) | 0.000 | -0.004 (5) | 0.000 |
| C28 | 0.020 (6) | 0.020 (5) | 0.013 (5) | 0.000 | -0.001 (5) | 0.000 |
| C29 | 0.058 (12) | 0.14 (2) | 0.044 (10) | 0.000 | -0.026 (9) | 0.000 |
| C30 | 0.022 (7) | 0.041 (8) | 0.062 (10) | 0.000 | 0.008 (7) | 0.000 |
| C31 | 0.029 (7) | 0.032 (7) | 0.039 (8) | 0.000 | -0.003 (6) | 0.000 |
| C32 | 0.048 (7) | 0.057 (8) | 0.095 (10) | -0.003 (6) | 0.008 (7) | 0.018 (7) |
| C33 | 0.048 (7) | 0.080 (9) | 0.082 (9) | -0.010 (7) | 0.003 (7) | -0.023 (8) |
| C34 | 0.042 (6) | 0.052 (7) | 0.060 (7) | 0.007 (5) | 0.005 (6) | 0.003 (6) |
| P2 | 0.0201 (10) | 0.0189 (10) | 0.0171 (10) | 0.0009 (8) | 0.0003 (8) | -0.0010 (8) |
| S2 | 0.0540 (15) | 0.0328 (12) | 0.0243 (12) | -0.0104 (11) | -0.0123 (11) | 0.0080 (10) |
| N2 | 0.030 (4) | 0.041 (5) | 0.056 (6) | -0.007 (4) | 0.008 (4) | -0.005 (4) |
| O2 | 0.061 (6) | 0.064 (6) | 0.080 (6) | 0.000 (4) | -0.012 (5) | -0.032 (5) |
| S3 | 0.0349 (17) | 0.0261 (15) | 0.0197 (15) | 0.000 | -0.0050 (13) | 0.000 |
| S4 | 0.0325 (11) | 0.0182 (10) | 0.0229 (11) | 0.0005 (8) | -0.0081 (9) | 0.0003 (8) |
| S5 | 0.0351 (17) | 0.0176 (14) | 0.0203 (15) | 0.000 | -0.0031 (13) | 0.000 |

Geometric parameters (Å, °)

| | | | |
|----------------------|-------------|----------|------------|
| Ag1—P1 | 2.448 (2) | C16—C17 | 1.384 (13) |
| Ag1—P2 | 2.469 (2) | C16—H26A | 0.9300 |
| Ag1—S4 | 2.588 (2) | C17—C18 | 1.356 (14) |
| Ag1—S5 | 2.924 (3) | C17—H7A | 0.9300 |
| Ag1—Ag1 ⁱ | 3.1558 (14) | C18—C19 | 1.383 (13) |
| Mo1—S1 | 2.071 (3) | C18—H14A | 0.9300 |
| Mo1—S3 | 2.322 (2) | C19—C20 | 1.377 (13) |
| Mo1—S5 | 2.344 (2) | C19—H30A | 0.9300 |

| | | | |
|----------------------|-------------|---------------------|------------|
| Mo1—S2 | 2.396 (2) | C20—H31A | 0.9300 |
| Mo1—S4 | 2.446 (2) | C21—C26 | 1.392 (12) |
| Mo1—Mo1 ⁱ | 2.8772 (14) | C21—C22 | 1.400 (12) |
| P1—C27 | 1.824 (7) | C21—P2 | 1.834 (8) |
| P1—C3 | 1.824 (9) | C22—C23 | 1.396 (12) |
| P1—C9 | 1.835 (8) | C22—H25A | 0.9300 |
| O1—C31 | 1.245 (15) | C23—C24 | 1.382 (13) |
| N1—C31 | 1.300 (16) | C23—H19A | 0.9300 |
| N1—C29 | 1.406 (18) | C24—C25 | 1.382 (13) |
| N1—C30 | 1.450 (16) | C24—H18A | 0.9300 |
| C1—C2 | 1.487 (13) | C25—C26 | 1.370 (12) |
| C1—S2 | 1.842 (10) | C25—H15A | 0.9300 |
| C1—H9A | 0.9700 | C26—H27A | 0.9300 |
| C1—H9B | 0.9700 | C27—P1 ⁱ | 1.824 (7) |
| C2—S4 | 1.824 (9) | C27—H33A | 0.9700 |
| C2—H16A | 0.9700 | C27—H33B | 0.9700 |
| C2—H16B | 0.9700 | C28—P2 ⁱ | 1.846 (6) |
| C3—C4 | 1.372 (15) | C28—P2 | 1.846 (6) |
| C3—C8 | 1.378 (13) | C28—H34A | 0.9700 |
| C4—C5 | 1.392 (16) | C28—H34B | 0.9700 |
| C4—H8A | 0.9300 | C29—H3A | 0.9600 |
| C5—C6 | 1.36 (2) | C29—H3B | 0.9600 |
| C5—H1A | 0.9300 | C29—H3C | 0.9600 |
| C6—C7 | 1.37 (2) | C30—H30B | 0.9600 |
| C6—H2A | 0.9300 | C30—H30C | 0.9600 |
| C7—C8 | 1.370 (16) | C30—H30D | 0.9600 |
| C7—H6A | 0.9300 | C31—H12 | 0.9300 |
| C8—H5A | 0.9300 | C32—N2 | 1.437 (15) |
| C9—C14 | 1.373 (12) | C32—H4A | 0.9600 |
| C9—C10 | 1.396 (11) | C32—H4B | 0.9600 |
| C10—C11 | 1.382 (12) | C32—H4C | 0.9600 |
| C10—H22A | 0.9300 | C33—N2 | 1.452 (14) |
| C11—C12 | 1.365 (14) | C33—H10A | 0.9600 |
| C11—H29A | 0.9300 | C33—H10B | 0.9600 |
| C12—C13 | 1.382 (13) | C33—H10C | 0.9600 |
| C12—H17A | 0.9300 | C34—O2 | 1.218 (13) |
| C13—C14 | 1.381 (13) | C34—N2 | 1.339 (14) |
| C13—H13A | 0.9300 | C34—H11 | 0.9300 |
| C14—H20A | 0.9300 | S3—Mo1 ⁱ | 2.322 (2) |
| C15—C16 | 1.389 (11) | S5—Mo1 ⁱ | 2.344 (2) |
| C15—C20 | 1.405 (12) | S5—Ag1 ⁱ | 2.924 (3) |
| C15—P2 | 1.820 (8) | | |
| P1—Ag1—P2 | 124.54 (7) | C16—C17—H7A | 119.3 |
| P1—Ag1—S4 | 114.92 (7) | C17—C18—C19 | 119.5 (9) |
| P2—Ag1—S4 | 112.31 (7) | C17—C18—H14A | 120.2 |
| P1—Ag1—S5 | 123.16 (8) | C19—C18—H14A | 120.2 |
| P2—Ag1—S5 | 100.85 (8) | C20—C19—C18 | 120.6 (9) |

supplementary materials

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|-------------------------|-------------|---------------------------|-----------|
| S4—Ag1—S5 | 67.02 (6) | C20—C19—H30A | 119.7 |
| P1—Ag1—Ag1 ⁱ | 88.50 (5) | C18—C19—H30A | 119.7 |
| P2—Ag1—Ag1 ⁱ | 88.98 (5) | C19—C20—C15 | 119.9 (8) |
| S4—Ag1—Ag1 ⁱ | 123.29 (5) | C19—C20—H31A | 120.1 |
| S5—Ag1—Ag1 ⁱ | 57.35 (4) | C15—C20—H31A | 120.1 |
| S1—Mo1—S3 | 109.83 (12) | C26—C21—C22 | 119.7 (8) |
| S1—Mo1—S5 | 106.97 (12) | C26—C21—P2 | 118.8 (6) |
| S3—Mo1—S5 | 99.01 (8) | C22—C21—P2 | 121.2 (7) |
| S1—Mo1—S2 | 105.81 (11) | C23—C22—C21 | 118.8 (8) |
| S3—Mo1—S2 | 79.66 (8) | C23—C22—H25A | 120.6 |
| S5—Mo1—S2 | 145.47 (11) | C21—C22—H25A | 120.6 |
| S1—Mo1—S4 | 105.92 (10) | C24—C23—C22 | 120.8 (8) |
| S3—Mo1—S4 | 142.96 (10) | C24—C23—H19A | 119.6 |
| S5—Mo1—S4 | 79.26 (7) | C22—C23—H19A | 119.6 |
| S2—Mo1—S4 | 81.67 (8) | C23—C24—C25 | 119.7 (8) |
| S1—Mo1—Mo1 ⁱ | 100.80 (8) | C23—C24—H18A | 120.1 |
| S3—Mo1—Mo1 ⁱ | 51.72 (5) | C25—C24—H18A | 120.1 |
| S5—Mo1—Mo1 ⁱ | 52.15 (5) | C26—C25—C24 | 120.4 (9) |
| S2—Mo1—Mo1 ⁱ | 130.22 (6) | C26—C25—H15A | 119.8 |
| S4—Mo1—Mo1 ⁱ | 129.64 (5) | C24—C25—H15A | 119.8 |
| C27—P1—C3 | 106.1 (5) | C25—C26—C21 | 120.5 (8) |
| C27—P1—C9 | 105.0 (4) | C25—C26—H27A | 119.7 |
| C3—P1—C9 | 101.8 (4) | C21—C26—H27A | 119.7 |
| C27—P1—Ag1 | 115.4 (4) | P1 ⁱ —C27—P1 | 112.2 (6) |
| C3—P1—Ag1 | 117.4 (3) | P1 ⁱ —C27—H33A | 109.2 |
| C9—P1—Ag1 | 109.5 (3) | P1—C27—H33A | 109.2 |
| C31—N1—C29 | 120.6 (13) | P1 ⁱ —C27—H33B | 109.2 |
| C31—N1—C30 | 121.6 (11) | P1—C27—H33B | 109.2 |
| C29—N1—C30 | 117.8 (13) | H33A—C27—H33B | 107.9 |
| C2—C1—S2 | 110.0 (7) | P2 ⁱ —C28—P2 | 112.4 (6) |
| C2—C1—H9A | 109.7 | P2 ⁱ —C28—H34A | 109.1 |
| S2—C1—H9A | 109.7 | P2—C28—H34A | 109.1 |
| C2—C1—H9B | 109.7 | P2 ⁱ —C28—H34B | 109.1 |
| S2—C1—H9B | 109.7 | P2—C28—H34B | 109.1 |
| H9A—C1—H9B | 108.2 | H34A—C28—H34B | 107.9 |
| C1—C2—S4 | 110.5 (7) | N1—C29—H3A | 109.5 |
| C1—C2—H16A | 109.5 | N1—C29—H3B | 109.5 |
| S4—C2—H16A | 109.5 | H3A—C29—H3B | 109.5 |
| C1—C2—H16B | 109.5 | N1—C29—H3C | 109.5 |
| S4—C2—H16B | 109.5 | H3A—C29—H3C | 109.5 |
| H16A—C2—H16B | 108.1 | H3B—C29—H3C | 109.5 |
| C4—C3—C8 | 118.5 (9) | N1—C30—H30B | 109.5 |
| C4—C3—P1 | 119.4 (8) | N1—C30—H30C | 109.5 |
| C8—C3—P1 | 122.1 (8) | H30B—C30—H30C | 109.5 |
| C3—C4—C5 | 119.6 (13) | N1—C30—H30D | 109.5 |
| C3—C4—H8A | 120.2 | H30B—C30—H30D | 109.5 |

| | | | |
|--------------|------------|---------------------------------------|-------------|
| C5—C4—H8A | 120.2 | H30C—C30—H30D | 109.5 |
| C6—C5—C4 | 121.0 (14) | O1—C31—N1 | 127.4 (13) |
| C6—C5—H1A | 119.5 | O1—C31—H12 | 116.3 |
| C4—C5—H1A | 119.5 | N1—C31—H12 | 116.3 |
| C5—C6—C7 | 119.6 (11) | N2—C32—H4A | 109.5 |
| C5—C6—H2A | 120.2 | N2—C32—H4B | 109.5 |
| C7—C6—H2A | 120.2 | H4A—C32—H4B | 109.5 |
| C8—C7—C6 | 119.7 (13) | N2—C32—H4C | 109.5 |
| C8—C7—H6A | 120.2 | H4A—C32—H4C | 109.5 |
| C6—C7—H6A | 120.2 | H4B—C32—H4C | 109.5 |
| C7—C8—C3 | 121.5 (12) | N2—C33—H10A | 109.5 |
| C7—C8—H5A | 119.2 | N2—C33—H10B | 109.5 |
| C3—C8—H5A | 119.2 | H10A—C33—H10B | 109.5 |
| C14—C9—C10 | 119.6 (8) | N2—C33—H10C | 109.5 |
| C14—C9—P1 | 124.5 (7) | H10A—C33—H10C | 109.5 |
| C10—C9—P1 | 115.9 (6) | H10B—C33—H10C | 109.5 |
| C11—C10—C9 | 119.9 (8) | O2—C34—N2 | 125.5 (11) |
| C11—C10—H22A | 120.1 | O2—C34—H11 | 117.3 |
| C9—C10—H22A | 120.1 | N2—C34—H11 | 117.3 |
| C12—C11—C10 | 121.0 (8) | C15—P2—C21 | 105.5 (4) |
| C12—C11—H29A | 119.5 | C15—P2—C28 | 104.7 (4) |
| C10—C11—H29A | 119.5 | C21—P2—C28 | 102.7 (4) |
| C11—C12—C13 | 118.5 (9) | C15—P2—Ag1 | 119.5 (3) |
| C11—C12—H17A | 120.7 | C21—P2—Ag1 | 110.0 (3) |
| C13—C12—H17A | 120.7 | C28—P2—Ag1 | 112.9 (3) |
| C14—C13—C12 | 121.8 (9) | C1—S2—Mo1 | 104.6 (3) |
| C14—C13—H13A | 119.1 | C34—N2—C32 | 120.7 (10) |
| C12—C13—H13A | 119.1 | C34—N2—C33 | 120.7 (10) |
| C9—C14—C13 | 119.3 (8) | C32—N2—C33 | 118.6 (10) |
| C9—C14—H20A | 120.4 | Mo1—S3—Mo1 ⁱ | 76.55 (10) |
| C13—C14—H20A | 120.4 | C2—S4—Mo1 | 107.9 (3) |
| C16—C15—C20 | 118.7 (8) | C2—S4—Ag1 | 117.7 (3) |
| C16—C15—P2 | 119.0 (6) | Mo1—S4—Ag1 | 106.28 (8) |
| C20—C15—P2 | 122.2 (6) | Mo1 ⁱ —S5—Mo1 | 75.71 (9) |
| C17—C16—C15 | 119.8 (8) | Mo1 ⁱ —S5—Ag1 | 145.22 (13) |
| C17—C16—H26A | 120.1 | Mo1—S5—Ag1 | 99.16 (6) |
| C15—C16—H26A | 120.1 | Mo1 ⁱ —S5—Ag1 ⁱ | 99.16 (6) |
| C18—C17—C16 | 121.4 (9) | Mo1—S5—Ag1 ⁱ | 145.22 (13) |
| C18—C17—H7A | 119.3 | Ag1—S5—Ag1 ⁱ | 65.31 (7) |

Symmetry codes: (i) $x, -y+1/2, z$.

