Silver(I) Complexes with $[(C_5H_5)_2MoH_2]$ and $[(C_5H_5)_2WH_2]$ Ligands

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Abstract: The reaction of $[Cp_2MH_2]$ ($Cp = C_5H_5$; M = Mo, W) with AgX ($X = BF_4, PF_6$) in a 2:1 molar ratio yields $[Ag(Cp_2MH_2)_2]X$ (**1**). X-ray crystal structure analyses show nearly linear coordination at the silver ion. The silver halide complexes with $[Cp_2MoH_2]$ exhibit a diversity of structures. For instance, in $[Ag(Cp_2MoH_2)_2Cl]$ (**2**) the coordination of the silver ion by two $[Cp_2MoH_2]$ ligands and the chloride ion is trigonal planar. The hydride ligands are located by using the difference Fourier method. The clusters $[Ag_3(Cp_2MoH_2)_3X_3]$ (X = Br, I) (**3**) are formed in the reaction of $[Ag(Cp_2MoH_2)_2]BF_4$ with $[N(n\text{Bu})_4]X$. X-ray crystal structure analyses reveal that these trinuclear clusters contain two different halide bridges and a single short Ag–Ag distance. The complex $[Ag_3(Cp_2MoH_2)_4Br_2]PF_6$ (**4**) with two different bromide bridges has an unusual trinuclear structure in which two silver ions are each bound to one $[Cp_2MoH_2]$ ligand, and one silver ion is bound to two $[Cp_2MoH_2]$ ligands.

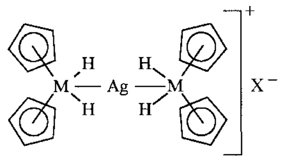
Keywords: clusters · molybdenum · silver · structure elucidation · tungsten

Introduction

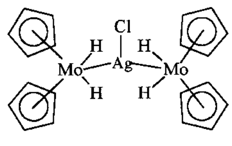
The complexes $[Cp_2MoH_2]$[1] and $[Cp_2WH_2]$[1] each contain three low-lying molecular orbitals[2] between the two bent cyclopentadienyl rings. The two outer orbitals are used to bind the σ-bonded hydride ligands, the orbital in the middle contains a lone pair of electrons. In dilute acids, $[Cp_2MoH_2]$ and $[Cp_2WH_2]$ behave as bases,[1] and the resulting cations $[Cp_2MoH_3]^+$ and $[Cp_2WH_3]^+$ are soluble in water. Protonation and deprotonation are reversible. Lewis acids such as BF_3,[3] $AlMe_3$[1] and $GaMe_3$[4] add to the lone pairs of electrons in $[Cp_2MoH_2]$ and $[Cp_2WH_2]$. In the reaction with $[M(CO)_5(thf)]$ (M = Cr, Mo, W) they form the complexes $[\{Cp_2MoH_2\}\{M(CO)_5\}]$ and $[\{Cp_2WH_2\}\{M(CO)_5\}]$.[5] There are relatively few transition metal complexes known that contain the ligands $[Cp_2MoH_2]$ and $[Cp_2WH_2]$.[6] In 1992 the reaction of $[Cp_2WH_2]$ with $AgBF_4$ was reported to give $[Ag(Cp_2WH_2)_2]BF_4$.[7] Here we describe the structure of the complexes $[Ag(Cp_2MH_2)_2]X$ (M = W, $X = BF_4$; M = Mo, $X = PF_6$) and the coordination chemistry of $[Cp_2MoH_2]$ with silver(I) halides.

Results and Discussion

Yellow solutions of $[Cp_2MoH_2]$ or $[Cp_2WH_2]$ in acetone or methanol react rapidly (as evident from the colour change) with $AgBF_4$ or $AgPF_6$ (molar ratio 2:1) to give the air-sensitive complexes $[Ag(Cp_2MH_2)_2]X$ (M = Mo, W; $X = BF_4$, PF_6) of type **1**. In the ^1H NMR spectra of the complexes **1**, a triplet appears for the Cp ligands and a multiplet for the hydride ligands.



1 M=Mo, W; X=BF4, PF6



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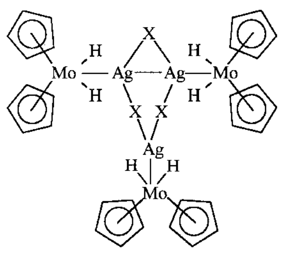
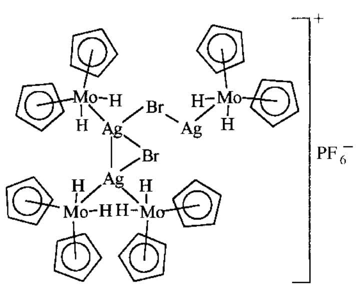


3 X=Br, I



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Crystals of the complexes $[Ag(Cp_2WH_2)_2]BF_4 \cdot H_2O$ and $[Ag(Cp_2MoH_2)_2]PF_6 \cdot H_2O$ suitable for X-ray structure analysis were obtained from a mixture of CH_2Cl_2, THF and toluene. Figure 1 shows the nearly linear arrangement of the two $[Cp_2WH_2]$ units at the Ag ion in $[Ag(Cp_2WH_2)_2]^+$.

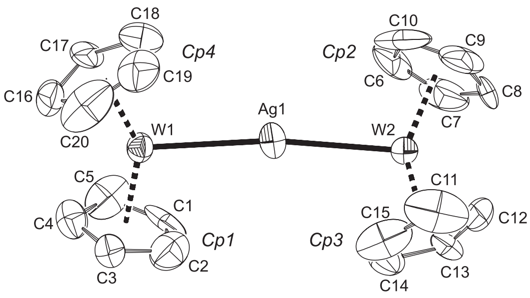

Figure 1. Molecular structure of the cation of $[Ag(Cp_2WH_2)_2]BF_4 \cdot H_2O$ in the crystal (ORTEP plot). Selected bond lengths [Å] and angle [°]: W1–Ag1 2.813(2), W2–Ag1 2.796(2) W1-Ag1-W2 170.8(1).

In $[Ag(Cp_2WH_2)_2]BF_4$ the W1-Ag1-W2 angle is 170.8°, and in $[Ag(Cp_2MoH_2)_2]PF_6$ the Mo1-Ag1-Mo2 angle is 175.2°. Assuming linearity for the M-Ag-M unit, the dihedral angle between the normals to the Cp planes should be 90° in an ideally staggered conformation of the $[Cp_2MH_2]$ ligands and 0° in an ideally eclipsed conformation. However, the difference between the dihedral angles ring centroid-M-Ag-M is 49.6° for Cp1 and Cp3 and 49.1° for Cp2 and Cp4 in $[Ag(Cp_2WH_2)_2]BF_4$. There are two independent molecules in the crystal of $[Ag(Cp_2MoH_2)_2]PF_6$, and in both cases the angle between Cp1 and Cp3 and between Cp2 and Cp4 is 57.7°. Thus, in both complexes the ligands $[Cp_2MH_2]$ adopt an orientation intermediate between a staggered and an eclipsed conformation.

The position of the hydride ligands could not be localised in the two structures; however, they are expected to lie in planes perpendicular to the metal–Cp centroid axes, and surround the silver ion as a rhombic disphenoid. As the angles between the M–H bonds and the M–Ag axes are extremly small (1/2 H-Mo-H angle in $[Cp_2MoH_2] = 37.4°$[8]), the four hydride ligands should be directed towards the silver ion.

The reaction of $[Ag(Cp_2MoH_2)_2]BF_4$ with tetrabutylammonium chloride in acetone gave the complex $[Ag(Cp_2MoH_2)_2Cl]$ (type **2**), which was crystallised from dichloromethane/toluene. Figure 2 shows that the silver ion is coordinated to two $[Cp_2MoH_2]$ ligands and one chloride ion in a trigonal planar fashion (sum of bond angles 359.9°). The Mo1-Ag1-Mo2 angle is 136.2°. The Cp rings at the two Mo centers adopt a twisted conformation similar to that in the Ag complexes **1**. Cp1 and Cp3 form an angle of 32.4°, Cp2 and Cp4 an angle of 33.6°. The dihedral angles ring centroid-Mo-Ag-Cl differ considerably; Cp2-Mo1-Ag1-Cl1 is 8.5°, whereas Cp4-Mo2-Ag1-Cl1 is 49.8°.

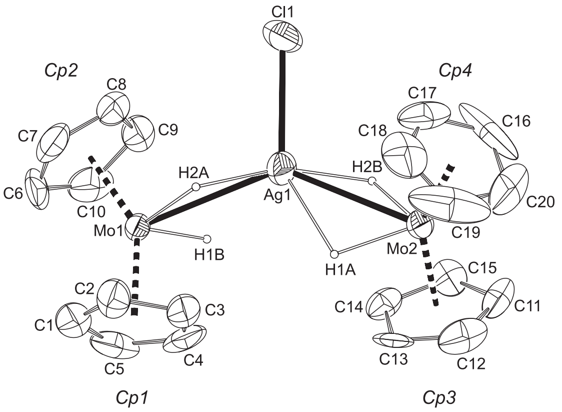

Figure 2. Molecular structure of $[Ag(Cp_2MoH_2)_2Cl]$ in the crystal (ORTEP plot). Selected bond lengths [Å] and angles [°]: Mo1–Ag1 3.007(2), Mo2–Ag1 2.966(2), Ag1–Cl1 2.583(3) Mo1-Ag1-Mo2 136.2(1), Mo1-Ag1-Cl1 109.6(1), Mo2-Ag1-Cl1 114.1(1).

The position of the hydride ligands H1A, H1B, H2A and H2B was determined by the difference Fourier method. The two planes formed by Mo, the two hydrides, H1 and H2, and Ag are tilted (dihedral angle H1B-Mo1-Ag1-H2A 20.9°, H1A-Mo2-Ag1-H2B 35.4°). The angle H1B-Mo1-H2A is 81.8° and the H1A-Mo2-H2A angle is 73.8°. The Mo–H distances lie between 1.54 and 1.80 Å, and the shortest Ag–H distance is 1.76 Å and the longest is 2.31 Å.

Solid AgI reacted with $[Cp_2MoH_2]$ in dichloromethane to give a yellow compound containing $[Cp_2MoH_2]$, Ag and I in the ratio 1:1:1. The complex is hardly soluble in dichloromethane or chloroform, but it can be purified by chromatography. The complex crystallised from dichloromethane/toluene in the form of yellow bipyramids. Figure 3 shows the crystal structure of the cluster $[Ag_3(Cp_2MoH_2)_3I_3]$ (type **3**).

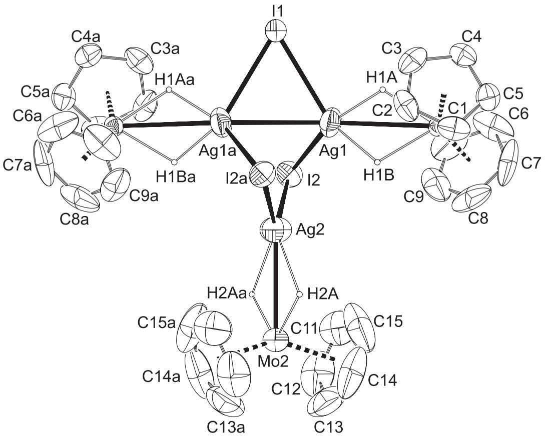

Figure 3. Molecular structure of $[Ag_3(Cp_2MoH_2)_3I_3]$ in the crystal (ORTEP plot). Selected bond lengths [Å] and angles [°]: Mo1–Ag1 2.930(1), Mo2–Ag2 2.944(2), Ag1–Ag1a 2.940(2), Ag1–I1 2.857(1), Ag2–I2 2.834(1); Mo1-Ag1-I1 121.2(1), I1-Ag1-I2 104.9(1), Mo1-Ag1-Ag1a 161.9(1), I2-Ag2-I2a 116.8(1), Ag1-I1-Ag1a 61.9(1).

The hydrides H1A, H1B and H2A were localised by the difference Fourier method. The C_2 axis of the molecule passes through the atoms I1, Ag2 and Mo2. The coordination of Ag2 by $[Cp_2MoH_2]$ and two I atoms is trigonal planar (sum of bond angles 360°). The planes I1-Ag1-Ag2 and Mo2-Ag2-I2 are arranged perpendicular to each other. The Ag1–Ag1a distance of 2.94 Å is very short.[9] Mo1, Ag1, Ag1a and Mo1a are arranged nearly in a straight line (the Mo1-Ag1-

Ag1a and Mo1a-Ag1a-Ag1 bond angles are both 161.9°). The two iodide bridges Ag1-I2-Ag2 and Ag1a-I2a-Ag2 lie above and below the plane of the three Ag atoms, respectively; the Ag1-I1-Ag1a bridge also lies in this plane. The distances of the silver atoms to the iodide bridge atoms are between 2.83 and 2.86 Å; the Ag1 – I2a and Ag1a – I2 distances of 3.41 Å are much larger. The rhomb formed by Mo2-H2A-Ag2-H2Aa is planar (dihedral angle 0°). In contrast, the dihedral angle Mo1-H1B-Ag1-H1A is 19.8°. The Mo – H bond lengths range from 1.78 to 1.89 Å, and the H-Mo-H angles lie between 65.0° and 96.1°. The Ag – H distances differ considerably; the shortest Ag – H distance is 1.70 Å and the longest is 2.19 Å.

The reaction of [Cp_2MoH_2] with $AgPF_6$ and tetrabutylammonium bromide in dichloromethane afforded two kinds of crystals: [$Ag_3(Cp_2MoH_2)_3Br_3$] (type **3**), whose structure is analogous to that of [$Ag_3(Cp_2MoH_2)_3I_3$], and the complex [$Ag_3(Cp_2MoH_2)_4Br_2$]PF_6 (type **4**), which represents a new class of a trinuclear structure with two different bromide bridges (Figure 4). In the structure of **4** two of the silver atoms are each coordinated to one [Cp_2MoH_2] ligand, whilst the third silver atom is coordinated to two [Cp_2MoH_2] ligands. The Ag1 – Br1 and Ag2 – Br2 distances of 3.31 and 3.51 Å, respectively, are much larger than in the Ag – Br bridges (2.69 – 3.05 Å). The position of the hydride ligands could not be localised. The Ag1 – Ag3 distance (3.11 Å) is longer than the Ag1 – Ag1a distance (3.02 Å) in [$Ag_3(Cp_2MoH_2)_3Br_3$].

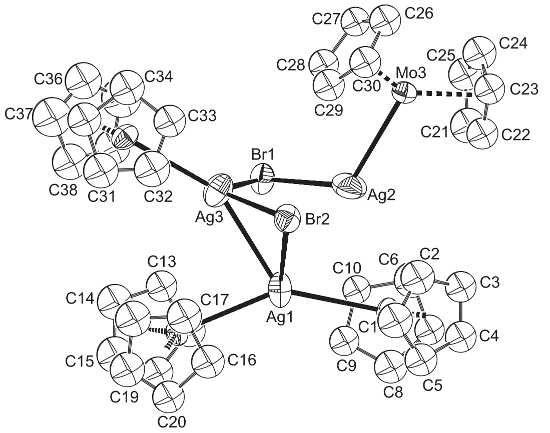

Figure 4. Molecular structure of [$Ag_3(Cp_2MoH_2)_4Br_2$]PF_6 in the crystal (ORTEP plot). Selected bond lengths [Å] and angles [°]: Mo1 – Ag1 2.962(4), Mo2 – Ag1 2.939(4), Mo3 – Ag2 2.931(4), Mo4 – Ag3 2.927(4), Ag1 – Ag3 3.114(4), Ag1 – Br2 3.048(5), Ag2 – Br1 2.760(4), Ag3 – Br2 2.722(4); Ag1-Br2-Ag3 65.0(1), Mo1-Ag1-Mo2 142.7(1), Mo2-Ag1-Ag2 145.4(1), Mo1-Ag1-Ag3 134.1(1).

Experimental Section

All reactions were carried out under an atmosphere of dry nitrogen with standard Schlenk techniques. $AgBF_4$ was purchased from Strem Chemical Co. and stored under argon. The synthesis of [Cp_2MH_2] M = Mo, W was described in reference [1]. Field desorption mass spectra were obtained from solutions of the compounds in dichloromethane on a Finnigan MAT 95 spectrometer. ^1H NMR spectra were recorded on a Bruker ARX 400 instrument. Elemental analyses were performed by the microanalytical laboratory of the Universität Regensburg. All X-ray structure analyses were recorded on a Syntex R3 diffractometer with $Mo_{K\alpha}$ radiation at $T = 296$ K. The structures were solved by direct methods using the SHELXTL Plus Program SHELXTL Plus (release 4.2/800), PC version. Further details of the crystal structure investigations may be obtained from the Fachinformationszentrum Karlsruhe, D-76344 Eggenstein-Leopoldshafen (Germany) on quoting the depository CSD number given in each case.

Reaction of [Cp_2MH_2] (M = Mo, W) with AgX (X = BF_4, PF_6): Typical procedure: To a solution of [Cp_2MoH_2] (245 mg, 1.07 mmol) in acetone (15 mL) was added $AgPF_6$ (136 mg, 0.54 mmol) and the mixture was stirred for 1 h. The solvent was removed under reduced pressure and the yellow residue was washed with ether (nearly quantitative yield). Crystals suitable for X-ray structure analysis were obtained from a mixture of CH_2Cl_2, THF and toluene.

[$Ag(Cp_2MoH_2)_2$]PF_6: M.p. 145 °C (decomp); ^1H NMR (400 MHz, [D_6]acetone): $\delta = 5.11$ (t, ^3J(H,H) = 0.8 Hz, 10 H Cp), −9.63 (m, ^3J(H,H) = 0.8 Hz, 2 H; MoH); FD-MS: *m/z* (%): 567 (10) [M^+], 230 (100) [$Cp_2MoH_2^+$]; elemental analysis for $C_{20}H_{24}AgF_6Mo_2P$ (709.1): calcd: C 33.88, H 3.41; found: C 33.71, H 3.39.

X-ray structure analysis of [$Ag(Cp_2MoH_2)_2$]$PF_6 \cdot H_2O$: Yellow needles, crystal dimensions 0.10 × 0.15 × 0.35 mm³, rhombic, space group P_{ccn} (D_{2h}^{10}) (no. 56), $a = 20.65(1)$, $b = 21.823(7)$, $c = 10.488(4)$ Å, $V = 4726(3)$ Å³, $Z = 4$ (two independent molecules), $\rho_{calcd} = 2.03$ g cm^{-3}, empirical absorption correction (4 reflections $7.0 < 2\theta < 32.0°$), ω scans, transmission factors 0.91 – 1.00, $\mu = 1.99$ mm^{-1}, $F(000) = 2784$; 4753 independent, 1337 observed reflections ($I > 2.5\sigma(I)$); parameters 162; $R = 0.102$, $R_w = 0.076$, residual electron density 1.17/ − 1.52 e Å^{-3}. CSD-406822.

[$Ag(Cp_2WH_2)_2$]PF_6: M.p. 153 °C (decomp); ^1H NMR (250 MHz, [D_6]acetone): $\delta = 5.03$ (t, ^3J(H,H) = 0.8 Hz, 10 H; Cp), −12.19 (m, ^3J(H,H) = 0.8 Hz, ^1J(W,H) = 73.0 Hz, 2 H; WH); FD-MS: *m/z* (%): 741 (10) [M^+], 316 (100) [$Cp_2WH_2^+$]; elemental analysis for $C_{20}H_{24}AgF_6PW_2 \cdot H_2O$ (903.0): calcd: C 26.60, H 2.90; found: C 26.65, H 2.83.

X-ray structure analysis of [$Ag(Cp_2WH_2)_2$]$BF_4 \cdot H_2O$: Yellow plates, irregular shape, crystal dimensions 0.05 × 0.25 × 0.45 mm³, monoclinic, space group $P2_1/c$ (C_{2h}^5) (no. 14), $a = 10.281(5)$, $b = 21.247(8)$, $c = 10.388(5)$ Å, $\beta = 99.34(4)°$, $V = 2239(2)$ Å³, $Z = 4$, $\rho_{calcd} = 2.49$ g cm^{-3}, empirical absorption correction (7 reflections $4.5 < 2\theta < 32.5°$), ω scans, transmission factors 0.24 – 1.00, $\mu = 11.16$ mm^{-1}, $F(000) = 1544$; 4432 independent, 2638 observed reflections ($I > 2.5\sigma(I)$); parameters 228; $R = 0.063$, $R_w = 0.052$, residual electron density 1.46/ − 1.46 e Å^{-3}. CSD-406823.

Reaction of [$Ag(Cp_2MoH_2)_2$]BF_4 with [$N(n Bu)_4$]X (X = Cl, Br, I): Typical procedure: To a solution of [$Ag(Cp_2MoH_2)_2$]BF_4 (57.8 mg, 0.09 mmol) in acetone (4 mL) was added [$N(n Bu)_4$]X (X = Cl, Br, I) (0.09 mmol) and the mixture was stirred for 18 h. The solvent was removed and the yellow residue was washed with ether (nearly quantitative yield).

[$Ag(Cp_2MoH_2)_2Cl$]: M.p. 85 °C (decomp); ^1H NMR (400 MHz, $CDCl_3$): $\delta = 4.90$ (t, ^3J(H,H) = 0.7 Hz, 10 H; Cp), −9.85 (bs, 2 H; MoH).

X-ray structure analysis of [$Ag(Cp_2MoH_2)_2Cl$]: Yellow prisms, crystal dimensions 0.05 × 0.15 × 0.30 mm³, rhombic, space group $P_{ca}2_1$ (C_{2v}^5) (no. 29), $a = 15.472(4)$, $b = 11.274(3)$, $c = 11.236(3)$ Å, $V = 1960(1)$ Å³, $Z = 4$, $\rho_{calcd} = 2.03$ g cm^{-3}, empirical absorption correction (7 reflections $8.0 < 2\theta < 37.0°$), ω scans, transmission factors 0.77 – 1.00, $\mu = 2.38$ mm^{-1}, $F(000) = 1168$; 5065 independent, 3148 observed reflections ($I > 2.5\sigma(I)$); parameters 218; H1A, H2A, H1B, H2B were found by difference Fourier synthesis and fixed. $R = 0.057$, $R_w = 0.044$, residual electron density 2.07/ − 1.90 e Å^{-3}. CSD-406817.

[$Ag_3(Cp_2MoH_2)_3I_3$]: M.p. 100 °C (decomp); ^1H NMR (400 MHz, CD_2Cl_2, 80 scans due to low solubility): $\delta = 4.85$ (t, ^3J(H,H) = 0.7 Hz, 10 H; Cp), −9.56 (m, ^3J(H,H) = 0.7 Hz, 2 H; MoH); elemental analysis for $C_{30}H_{36}Ag_3I_3Mo_3$ (1388.7): calcd: C 25.95, H 2.61; found: C 25.63, H 2.71.

X-ray structure analysis of [$Ag_3(Cp_2MoH_2)_3I_3$]: Yellow bipyramids, crystal dimensions 0.15 × 0.25 × 0.55 mm³, monoclinic, space group $C2/c$ (C_{2h}^6) (no. 15), $a = 11.559(4)$, $b = 18.236(6)$, $c = 16.591(5)$ Å, $\beta = 95.84(2)°$, $V = 3479(2)$ Å³, $Z = 4$, $\rho_{calcd} = 2.65$ g cm^{-3}, empirical absorption correction (7 reflections $8.0 < 2\theta < 46.0°$), ω scans, transmission factors 0.74 – 1.00, $\mu = 5.39$ mm^{-1}, $F(000) = 2568$; 5088 independent, 3122 observed reflections ($I > 2.5\sigma(I)$); parameters 179; H1A, H2A, H1B were found by difference Fourier synthesis and fixed. $R = 0.046$, $R_w = 0.038$, residual electron density 0.90/− 0.95 e Å^{-3}. CSD-406821.

[$Ag_3(Cp_2MoH_2)_3Br_3$]: M.p. 90 °C (decomp); ^{1}H NMR (400 MHz, $CDCl_3$): $\delta = 4.83$ (t, ^3J(H,H) = 0.7 Hz, 10H Cp), -9.41 (m, ^3J(H,H) = 0.7 Hz, 2H, MoH).

X-ray structure analysis of [$Ag_3(Cp_2MoH_2)_3Br_3$]: Yellow bipyramids, crystal dimensions $0.08 \times 0.15 \times 0.25$ mm^3, monoclinic, space group $C2/c\,(C^6_{2h})$ (no. 15), $a = 11.538(7)$, $b = 18.02(1)$, $c = 16.160(6)$ Å, $\beta = 96.15(4)^\circ$, $V = 3340(3)$ Å^3, $Z = 4$, $\rho_{calcd} = 2.48$ g cm^{-3}, empirical absorption correction (6 reflections $9.0 < 2\theta < 36.0^\circ$), ω scans, transmission factors 0.76–1.00, $\mu = 6.42$ mm^{-1}, $F(000) = 2352$; 2954 independent, 1363 observed reflections ($I > 2.5\sigma(I)$); parameters 176; H1A, H2A, H1B were found by difference Fourier synthesis and fixed. $R = 0.069$, $R_w = 0.051$, residual electron density 1.24/ – 1.20 e Å^{-3}. CSD-406819.

X-ray structure analysis of [$Ag_3(Cp_2MoH_2)_4Br_2$]PF_6: Yellow plates, crystal dimensions $0.05 \times 0.20 \times 0.30$ mm^3, monoclinic, space group $P2_1/c\,(C^5_{2h})$ (no. 14), $a = 11.724(9)$, $b = 14.70(1)$, $c = 25.94(2)$ Å, $\beta = 96.06(6)^\circ$, $V = 4447(6)$ Å^3, $Z = 4$, $\rho_{calcd} = 2.29$ g cm^{-3}, empirical absorption correction (6 reflections $8.5 < 2\theta < 34.0^\circ$), ω scans, transmission factors 0.67–1.00, $\mu = 4.27$ mm^{-1}, $F(000) = 2912$; 7391 independent, 3245 observed reflections ($I > 2.5\sigma(I)$); parameters 305; $R = 0.093$, $R_w = 0.070$, residual electron density 1.47/– 1.38 e Å^{-3}. CSD-406818.

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