

Phosphorus Hybrid Polymers

The Diphosphorus Complex $[\text{Cp}_2\text{Mo}_2(\text{CO})_4(\eta^2\text{-P}_2)]$ as a Building Block for the Synthesis of Mixed-Hybrid Coordination Polymers

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Dedicated to Professor Masaaki Yoshifuji on the occasion of his 75th birthday

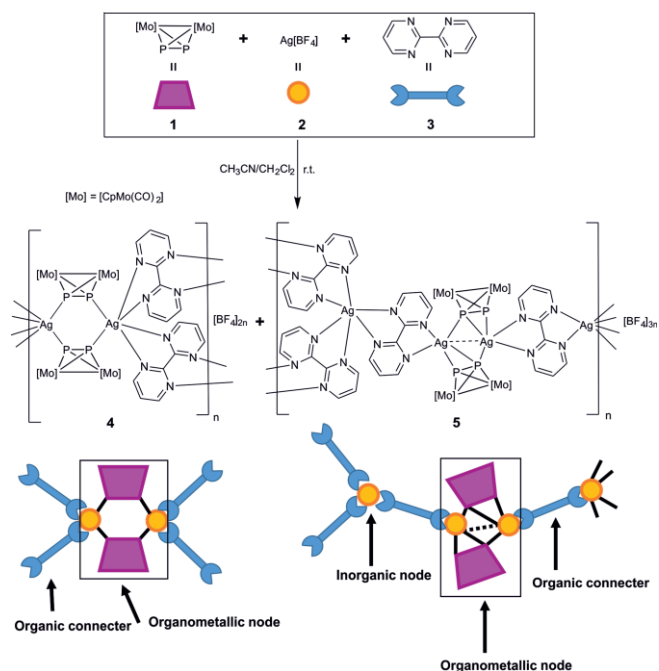
Abstract: The three-component reaction of the tetrahedral diphosphorus complex $[\text{Cp}_2\text{Mo}_2(\text{CO})_4(\eta^2\text{-P}_2)]$ (**1**), with $\text{Ag}[\text{BF}_4]$ (**2**) in the presence of 2,2'-bipyrimidine (**3**) leads to the formation of the two novel two-dimensional networks **4** and **5**. Compound **4** is a new two-dimensional organometallic-organic hybrid polymer, while derivative **5** represents a unique two-di-

mensional organometallic-inorganic-organic hybrid polymer. These results show the possibility of synthesizing a new class of coordination polymers, which could not be obtained from two-component reactions with organic molecules in addition of metal ions.

Introduction

Rapid development has been achieved in the construction of discrete and extended molecular architectures by self-assembly processes.^[1] Such derivatives can be classified as “inorganic-organic” supramolecular complexes as they are usually obtained from two-component reactions involving the coordination of organic ligands bearing N, O, or S donor atoms to Lewis-acidic inorganic metal cations.^[1h,2] Due to the lack of P donating connectors and the usual use of simple transition-metal cations, our group established an alternative approach by utilising organometallic polyphosphorus (P_n) ligand complexes with flexible coordination modes as connectors between metal ions. By using this novel method, it was possible to synthesize one- and two-dimensional coordination polymers,^[3] vast fullerene-like supramolecular spherical aggregates,^[4] and organometallic nanosized capsules.^[5] More recently, this concept was extended to three-component reactions by using additional bipyridine-based organic linkers in combination with P_n ligand complexes and metal ions. This novel approach succeeded in the synthesis of novel “organometallic-organic” hybrid polymers [where P_n -based organometallic fragments (nodes) are linked by organic spacers], which could not be obtained from classical two-

component reactions. For example, the tetrahedrane complex $[\text{Cp}_2\text{Mo}_2(\text{CO})_4(\eta^2\text{-P}_2)]$ ($\text{Cp} = \text{C}_5\text{H}_5$) (**1**) was treated with Ag^+ or Cu^+ salts in the presence of dipyrindylethene to yield unprecedented one-, two-, and three-dimensional “organometallic-organic” coordination polymers.^[6] These results motivated us to expand our research field raising the question as to whether it is possible to utilize some other N donor ligands in our reactions. One of those ligands is 2,2'-bipyrimidine “bpym”,^[7a] which is a spe-



Scheme 1. Reaction of **1** with $\text{Ag}[\text{BF}_4]$ (**2**) and bpym (**3**), synthesis of two-dimensional hybrid polymers **4** and **5**, and schematic illustration of nodes and connectors.

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cial molecule, because it can act both as a di- or tetradentate ligand. The supramolecular chemistry of this molecule has been extensively studied, and its coordination to transition metals afforded a rich library of zero- and multi-dimensional complexes.^[8] Now, however, we have shown that the reaction of the P₂ ligand complex **1** with Ag[BF₄] (**2**) in the presence of 2,2'-bipyrimidine "bpym" (**3**) allows access to the two polymeric networks **4** and **5**, with the latter representing a unique "organometallic-inorganic-organic" hybrid network. A variety of heterometallic metallacycles were synthesized by Jin et al. by linking a simple organometallic moiety and a metal cation with organic linkers.^[7b,7c] However, to the best of our knowledge, no extended hybrid networks with "mixed" complexed organometallic and inorganic nodes linked by organic spacers have been reported yet (Scheme 1).

Results and Discussion

In a first step, attention was focused on the reaction of the P₂ ligand complex **1** with the silver salt **2** and the bpym linker **3** in a 1:1:1 stoichiometric ratio in a mixture of CH₂Cl₂/CH₃CN at room temperature. This reaction afforded, after crystallization, two compounds **4** and **5** as orange crystals suitable for X-ray diffraction studies. The structures obtained from X-ray diffraction analysis performed on single crystals of **4** and **5** revealed that compound **4** is a novel two-dimensional organometallic-organic hybrid polymer with Ag₂(1)₂ organometallic nodes connected to each other by bpym linker molecules (Scheme 1, Figure 1). Furthermore, derivative **5** outlines a new class of two-dimensional coordination polymers composed of inorganic Ag-containing and organometallic Ag₂(1)₂-comprising nodes that are connected to each other by organic bpym linkers (Scheme 1, Figure 2).

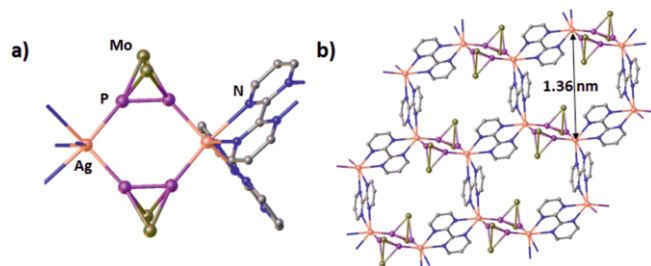


Figure 1. (a) Structure of the dicationic repeating unit of **4**. (b) Section of the 2D coordination polymer **4**. Cp and CO ligands, hydrogen atoms and counterions are omitted for clarity.

Within the organometallic nodes of **4**, the two P₂ ligands are coordinated to the silver ions by a frequently observed bridging η¹:η¹ coordination mode. In addition, four bpym molecules are also coordinated to those silver ions, each by two N atoms. As a consequence, each silver ion in **4** possesses a distorted octahedral coordination sphere consisting of two P atoms and four N atoms (Figure 1). The P–P bond in derivative **4** [2.0925(11) Å] is slightly elongated compared to that of the noncoordinated ligand **1** [2.079(6) Å]. The Ag–P bonds [2.5795(8)–2.6040(8) Å] are slightly elongated compared to those of similar hybrid polymers previously reported.^[6a] The

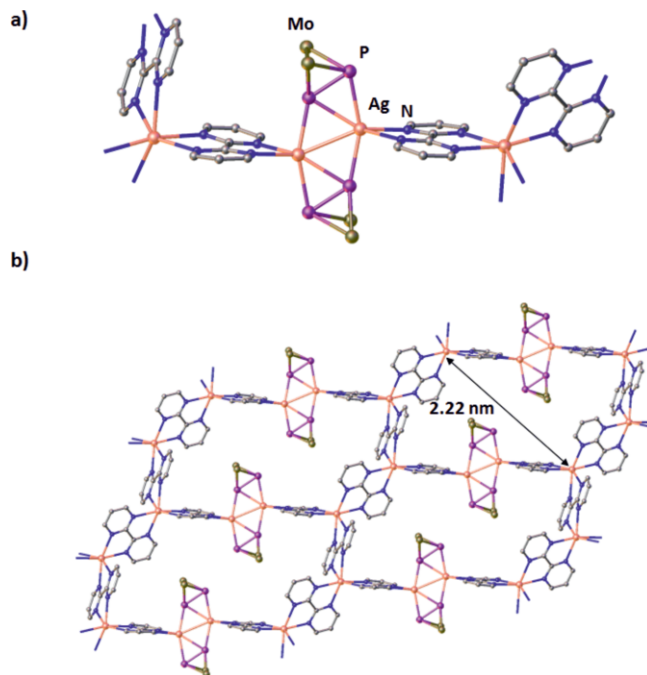


Figure 2. (a) Structure of the tetracationic repeating unit of **5**. (b) Section of the 2D coordination polymer **5**. Cp and CO ligands, hydrogen atoms and counterions are omitted for clarity.

Ag...Ag distance is long (5.480 Å) revealing no argentophilic interactions.^[9] The Ag₂P₄ six-membered motifs in **4** are nearly planar (folding angle 0.04°). The organometallic nodes in **4** form the vertices of a 2D network with cavities having "circular" meshes of a maximum diameter of 1.36 nm (Figure 1b).

In derivative **5**, each repeating unit is composed of one Ag₂(1)₂ organometallic fragment (organometallic node) and two Ag^I ions (inorganic nodes) connected to each other by bpym spacers (Figure 2a). Each organometallic node in **5** consists of two silver atoms to which two P₂ ligands are coordinated in a very rare bridging η¹:η² coordination mode (Figure 2b).^[10] In addition, two bpym molecules are also coordinated to the silver atoms, each by two of its N atoms. As a consequence, each silver ion within those organometallic nodes is coordinated to three P atoms, two N atoms and one Ag atom. The P–P bond in **5** [2.151(2) Å] is slightly elongated compared to that of the noncoordinated ligand **1** [2.079(6) Å] and ligand **1** in compound **4**. The Ag–P bond lengths [2.555(2)–2.630(2) Å] vary within a wider range compared to those of complex **4**. The Ag...Ag distance is short [2.939(1) Å] suggesting argentophilic interaction.^[9] The Ag₂P₄ six-membered motifs in the organometallic nodes of **5** are less planar than in **4** (folding angle 6.60°). Each organometallic node is connected to two 1D ribbon-like inorganic-organic fragments (Figure 2b). Those 1D fragments are composed each of silver ions (nodes) linked by organic bpym connectors. Each of those silver ions possesses a distorted octahedral coordination sphere with six coordinated N atoms of three bpym molecules. Two of those bpym molecules are a part of the 1D fragments, while the third one links the 1D fragment to one Ag₂(1)₂ organometallic node. In complex **5**, the inorganic Ag nodes within the 1D fragments form

the vertices of a 2D network with cavities having a “hexagonal shape” of a maximum diameter of 2.22 nm. It is worth noting that in the solid state packing of both **4** and **5** no solvent molecules are observed. In addition, all BF_4^- counteranions are located outside the self-assembled structures.

Regardless of several attempts, the new products **4** and **5** could not be separated from each other because of similar colour and solubility. Both compounds are well soluble in donor solvents like CH_3CN , but insoluble in other common organic solvents like CH_2Cl_2 , THF and *n*-pentane. Their room-temperature ^{31}P NMR spectrum in CD_3CN shows a broad signal centered at $\delta = -79.1$ ppm, which is upfield shifted compared to that of the free P_2 ligand complex **1** ($\delta = -43.2$ ppm).^[11] Their room-temperature ^1H and ^{13}C NMR spectra present simple sets of signals revealing all expected signals corresponding to the protons and carbon atoms of ligand **1** and the bpym linker **3**. The ^{19}F NMR spectrum features two signals centered at $\delta \approx -150.5$ and -150.6 ppm, corresponding to the BF_4^- counteranions (see the Supporting Information). In the mass spectrum, only fragments of $\text{Ag}_2(\mathbf{1})_2$ together with a bpym linker are found, revealing like the NMR data the depolymerisation of the aggregates in donor solvents.

Conclusions

The presented results contribute to a new direction in the area of metallo-supramolecular chemistry in which the synthesis of coordination polymers composed by “organometallic and inorganic” nodes linked together by organic linkers is accessible. Those polymers are obtained by simply mixing $\text{Ag}(\text{BF}_4)$, bpym linker and a P_2 ligand complex under ambient reaction conditions. The described synthesis opens the door to a novel class of coordination compounds for which compound **5** has been found as a first example. Here, the combination of “organometallic and inorganic” nodes (both containing transition metal coordination centers) linked by organic molecules was found for the first time resulting in a 2D “organometallic-inorganic-organic” hybrid network. It further shows the advantage of our approach using three-component reactions over the classical two-component reactions approach with metal ions and organic linkers. Taking into consideration that supramolecular aggregation reactions are usually sensitive to reaction conditions,^[12] current studies involve similar three-component reactions with careful optimization of the reaction conditions for the selective synthesis of hybrid “organometallic-inorganic-organic” polymers.

Experimental Section

General: All experiments were performed under dry argon using standard Schlenk techniques. The compounds 2,2'-bipyrimidine (**3**) and silver tetrafluoroborate (**2**) were purchased from Alfa Aesar and Sigma-Aldrich, respectively, and used as received without further purification. The ligand complex $[\text{CpMo}_2(\text{CO})_4(\eta^2\text{-P}_2)]$ (**1**) was synthesized according to a literature procedure.^[13] Solvents were freshly distilled under argon from CaH_2 (CH_2Cl_2 , CH_3CN) and from Na/K alloy (*n*-pentane). IR spectra were recorded with a Varian FTS-800 spectrometer. ^1H , ^{13}C , ^{31}P and ^{19}F NMR spectra were recorded

with a Bruker Avance 300 spectrometer. ^1H and ^{13}C NMR chemical shifts are reported in parts per million (ppm) relative to Me_4Si as external standard. ^{31}P NMR chemical shifts are expressed in ppm relative to external 85 % H_3PO_4 and were decoupled from the proton. ^{19}F NMR chemical shifts are reported relative to CFCl_3 . For the ESI mass spectra a Finnigan Thermoquest TSQ 7000 mass spectrometer was used. Elemental analyses were performed by the micro-analytical laboratory of the University of Regensburg.

Synthesis and NMR Characterization of Complexes 4 and 5: A solution of equimolar amounts of $\text{Ag}[\text{BF}_4]$ (**2**; 0.040 g, 0.20 mmol), $[\text{Cp}_2\text{Mo}_2(\text{CO})_4(\eta^2\text{-P}_2)]$ (**1**; 0.100 g, 0.20 mmol) and bpym (**3**; 0.032 g, 0.20 mmol) in a 1:1 mixture of CH_2Cl_2 (10 mL)/ CH_3CN (10 mL) was stirred at room temperature for 3 h. The reaction mixture was filtered using a Teflon capillary to remove any suspended particles, and the obtained solution was layered with the same amount of *n*-pentane and kept at room temperature. After 1 week, orange crystals of **4** and **5** had formed. The crystals were isolated together by decantation of the overlaying solution, washed two times with *n*-pentane (2 mL), and subsequently dried at reduced pressure. Overall yield 0.118 g, 69 % for **4** + **5**. ^1H NMR (300 MHz, CD_3CN , 25 °C): $\delta = 5.36$ (s, 5 H, C_5H_5), 7.70 (t, $^3J_{\text{HH}} = 4.9$ Hz, 2 H, H_{bpym}), 9.04 (d, $^3J_{\text{HH}} = 4.9$ Hz, 4 H, 4 H, H_{bpym}) ppm. $^{13}\text{C}\{^1\text{H}\}$ NMR (75.47 MHz, CD_3CN , 25 °C): $\delta = 88.1$ (s, C_5H_5), 124.1 (s, CH_{bpym}), 159.7 (s, CH_{bpym}), 160.7 (s, C_{bpym}), 224.6 (C_{CO}) ppm. $^{31}\text{P}\{^1\text{H}\}$ NMR (121.49 MHz, CD_3CN , 25 °C): $\delta = -79.1$ (s, $\omega_{1/2} = 13.9$ Hz) ppm. $^{19}\text{F}\{^1\text{H}\}$ NMR (282.40 MHz, CD_3CN , 25 °C): $\delta = -150.6$ (s, $^{11}\text{BF}_4$), -150.5 (s, $^{10}\text{BF}_4$) ppm. ESI-MS (CH_3CN): *m/z* (%) = 1452.7 (1) $[\text{Ag}_2\{\text{Cp}_2\text{Mo}_2(\text{CO})_4\text{P}_2\}_2(\text{C}_8\text{H}_6\text{N}_4)(\text{BF}_4)]^+$, 1100.6 (4) $[\text{Ag}\{\text{Cp}_2\text{Mo}_2(\text{CO})_4\text{P}_2\}]^+$, 760.9 (45) $[\text{Ag}\{\text{Cp}_2\text{Mo}_2(\text{CO})_4\text{P}_2\}(\text{C}_8\text{H}_6\text{N}_4)]^+$, 732.8 (12) $[\text{Ag}\{\text{Cp}_2\text{Mo}_2(\text{CO})_4\text{P}_2\}(\text{C}_8\text{H}_6\text{N}_4)\text{-CO}]^+$, 704.8 (20) $[\text{Ag}\{\text{Cp}_2\text{Mo}_2(\text{CO})_4\text{P}_2\}(\text{C}_8\text{H}_6\text{N}_4) - 2 \text{CO}]^+$, 676.8 (6) $[\text{Ag}\{\text{Cp}_2\text{Mo}_2(\text{CO})_4\text{P}_2\}(\text{C}_8\text{H}_6\text{N}_4) - 3 \text{CO}]^+$, 643.7 (10) $[\text{Ag}\{\text{Cp}_2\text{Mo}_2(\text{CO})_4\text{P}_2\}(\text{CH}_3\text{CN})]^+$, 422.9 (5) $[\text{Ag}(\text{C}_8\text{H}_6\text{N}_4)_2]^+$, 305.9 (100) $[\text{Ag}(\text{C}_8\text{H}_6\text{N}_4)(\text{CH}_3\text{CN})]^+$, 264.9 (21) $[\text{Ag}(\text{C}_8\text{H}_6\text{N})]^+$.

Single-Crystal X-ray Crystallography: All diffraction experiments were performed at 123 K. The data sets were collected with a Rigaku (formerly: Agilent Technologies or Oxford Diffraction) SuperNova diffractometer using Cu-K_α radiation. All crystal preparations were performed under mineral oil. The structure solution and refinement were done with ShelX.^[14] The H atoms were calculated geometrically, and a riding model was used during the refinement process. Graphical material was created with Olex2.^[15] CCDC 1483826 (for **4**), and 1483827 (for **5**) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre.

Supporting Information (see footnote on the first page of this article): Crystallographic data (Table S1).

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Keywords: Self-assembly · Phosphorus · Hybrid polymers · 2,2'-Bipyrimidine · Molybdenum

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