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Luminescent Frameworks

Cluster Linker Approach: Preparation of a Luminescent Porous Framework with NbO Topology by Linking Silver Ions with Gold(I) Clusters**

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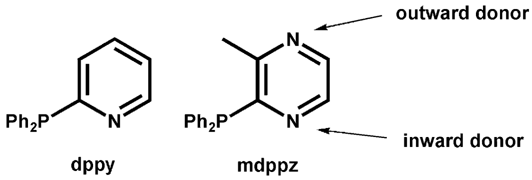
Abstract: *A cluster-based luminescent porous metal–organic framework has been constructed through a "cluster linker" approach. The luminescent gold(I) cluster, prefunctionalized with pyrazinyl groups, was used as a cluster linker, similar to an organic linker, to connect silver ions in order to form a 3D framework. 1D channels with 1.1 nm diameter were observed in the framework. The cluster with its intrinsic luminescence was incorporated into a porous framework to give a luminescent bifunctional NbO net. This MOF shows solvatochromic behavior, and the interactions between solvent molecules and silver ions inside the channels account for the changes in absorption and emission spectra.*

Metal–organic frameworks (MOFs) have been extensively studied as porous materials for applications in gas storage, separation, catalysis, drug delivery, and others. Generally, MOFs are prepared through the assembly of metal ions and organic linkers. An interesting alternative is the use of metal clusters as nodes in the construction of MOFs, which leads to the formation of cluster-based architectures.[1–4] There are several advantages to employ clusters in the assembly of MOF materials. First, highly connected frameworks may be created, because a metal cluster can provide more coordination sites than a single ion. Second, cluster-based MOFs normally possess large channels in comparison to those prepared from simple ion nodes.[5,6] Finally, the incorporation of cluster units may introduce intrinsic properties from the cluster into the framework.

Most cluster-based MOFs have been prepared through one-pot reactions, that is, the cluster nodes are formed in situ. Only a few stepwise preparations of cluster-based MOFs have been reported.[7] A stepwise assembly method involves the use of a preformed building unit containing labile coordinating species, such as a solvent molecule or an anion, which are amenable for a subsequent ligand replacement between the building unit and the organic linker. However, in the case that a cluster unit does not have any labile coordinating species, an inverted approach is needed to preorganize functionality in the cluster for further assembly. Our strategy involved the use of a prefunctionalized cluster as a linker, instead of an organic ligand, to connect metal ions in order to generate higher dimensional nets. We have successfully applied this "cluster linker" approach in the construction of a luminescent porous MOF.

Luminescent porous materials have potential applications because of their bifunctional nature.[8–15] In order to combine both luminescence and porosity in one compound, we used luminescent clusters bearing coordinating donors to link metal ions in order to form porous MOFs.

The cluster $[(C)(Au\text{-dppy})_6Ag_2](BF_4)_4$ (dppy = 2-pyridyldiphenylphosphine)[16] is highly emissive with a long life time at room temperature. In order to introduce these features into MOFs, we needed to install functionality into the cluster through organic modification for subsequent binding to metal ions. By using (2-(3-methylpyrazinyl)-diphenylphosphine) (mdppz) in place of dppy, we prepared cluster linker $[(C)(Au\text{-mdppz})_6](BF_4)_2$ (**1**), which bears six outward N donors that are available for coordinating to metal ions in order to generate high-dimensional cluster-based polymers or frameworks (see Scheme 1 and Figure 1). Herein, we report the synthesis and structures of cluster linker **1** and its application in the preparation of a luminescent cluster-based MOF with NbO topology, $[(C)(Au\text{-mdppz})_6Ag_6](BF_4)_8$ (**2**). The photophysical properties and sensing behavior responsive to common solvents are also described.



Scheme 1. Structures of dppy and mdppz.

The synthesis and characterization of ligand mdppz and cluster **1** are detailed in the Experimental Section and the Supporting Information.[16,17] The structure of **1** was determined by X-ray single-crystal diffraction (Figure 1 a). The structural core of **1** is an octahedral Au_6 unit with a hypercoordinated carbon atom in the center. Each gold atom is coordinated by one phosphine ligand mdppz. This configuration is similar to those previously reported by Schmidbaur et al.[18,19] Cluster **1** was very stable in air and kept intact in solution. The strong peak at m/z 1431.7 in the ESI-MS

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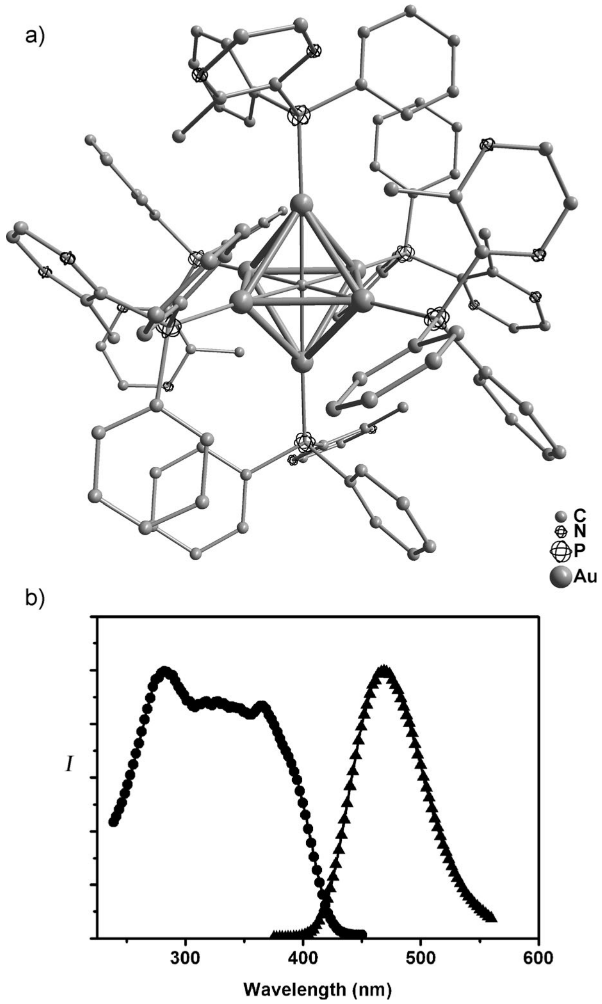

Figure 1. a) Molecular structure of the cluster linker $[(C)(Au\text{-mdppz})_6](BF_4)_2$ (**1**) bearing six outward N donors. b) Excitation (●) and emission (▲) spectra of **1** in the solid state; λ_{max}(●) = 287.8 nm, λ_{max}(▲) = 468.4 nm.

corresponds to the molecular ion $[(C)(Au\text{-mdppz})_6]^{2+}$ (Figure S5 in the Supporting Information). Cluster **1** was intensely emissive in the solid state. Under excitation at a wavelength of 365 nm, the blue emission of **1** could be detected by the naked eye, even in daylight (Figure 2g). The maximum emission wavelengths of previously reported hexanuclear gold(I) clusters are in the range of 495–550 nm,[16–19] whereas for **1** it was blue-shifted to 468 nm. This result suggests that the heterocycles of mdppz are involved in the emissive state.

Compound **2** was assembled by connecting silver(I) ions with cluster linker **1**. Six equivalents of $AgBF_4$ in methanol were slowly added to a solution of **1** in dichloromethane under stirring. The colorless solution turned yellow immediately. After filtration, slow evaporation of the filtrate at room temperature in the dark gave yellowish green crystals of **2** in about 62% yield.

Structure determination showed the 3D framework of **2**.[20] The basic building unit is shown in Figure 2a and b. Cluster **1** connects silver(I) ions with the outward N donors of its pyrazinyl groups to form an extending structure. There are three kinds of crystallographically distinct silver(I) ions in **2**, Ag1, Ag2, and Ag3. Ag1 is coordinated to inward N donors of mdppz to form intracluster binding; Ag2 is bound to an N donor of the pyrazinyl group, but is terminally ligated by BF_4^- and H_2O. Ag3 is coordinated to two N donors from two neighboring units **1** to generate an extending structure.

The 3D crystal structure of **2** is shown in Figure 2c. The structure contains two interpenetrated frameworks based on the 6^48^2 topology (Figure 2d).[21–23] Although interpenetration occurs in this structure, it features 1D channels along the *c* direction. The diameter of each hexagonal channel is about 1.1 nm. The topology of the framework is of NbO type, and the interpenetration style is shown in Figure 2e. Cluster **1** has six outward N donors for extending connections, but actually only functions as a four-connected linker because of the terminal blocking of the silver ions of type Ag2 by H_2O and BF_4^-.

Compound **2** is brightly emissive in the solid state. It emits yellowish green light with λ_{max} at 539 nm under UV irradiation (Figure 2g). The emission lifetimes of **1** and **2** were 2.99 ± 0.22 μs and 4.67 ± 0.28 μs, respectively. The quantum yields (QY) in the solid state were 1.5% and 25.6% for **1** and **2**, respectively. The large Stokes shift and the lifetime in microseconds suggest that **2** is a phosphorescent porous material with channels at nanometer scale. Silver atoms coordinated to the inward N donors (Ag1) and outward N donors (Ag2 and Ag3) contribute to the improvement of the QY, as well as the red shift of the solid state emission from **1** to **2**.

To assign the different roles to Ag1, Ag2, and Ag3, we synthesized $[(C)(Au\text{-mdppz})_6Ag_2](BF_4)_4$ (**3**) by adding exactly two equivalents of $AgBF_4$ to a solution of **1** in CH_2Cl_2/MeOH. The structure of **3** is similar to the building units in **2** (Figure 3a). Six inward N donors in **3** were ligated, but all outward N donors were nonligated. The yellow emission of crystalline **3** under UV irradiation is shown in Figure 2g, and the excitation and emission spectra of **3** in the solid state are shown in Figure 3b. QY and lifetime of the yellow-emitting **3** were 3.2% and 6.54 ± 0.43 μs, respectively. The emission of **3** is distinctly red-shifted in comparison to that of **1**, that is, the ligation of inward N donors to silver atoms and the formation of gold–silver interactions resulted in a decrease of the emission energy. Thus, the nature of the emission for **3** can be tentatively assigned to a cluster-based metal-centered (MC) excited state,[16] mixed with a metal-to-ligand charge-transfer (MLCT) state.

On the basis of the above data, we suggest that the ligation of Ag1 to the inward N donors in **2** mainly contributed to the red shift of the solid-state emission, while the large enhancement in QY mainly resulted from the rigidity generated through the formation of the 3D structure through coordination of Ag2 and Ag3. Furthermore, the coordination of Ag2 and Ag3 in **2** perturbed the excited state by introducing an MLCT process (from Ag2/Ag3 to the pyrazinyl group).

Compound **2** shows solvatochromic behavior. Freshly prepared yellowish green crystals of **2** gradually turned to orange after about a week, and its luminescence also changed from yellowish green to orange. This process can be accelerated by heating **2** or exposing it to a vacuum. The reverse

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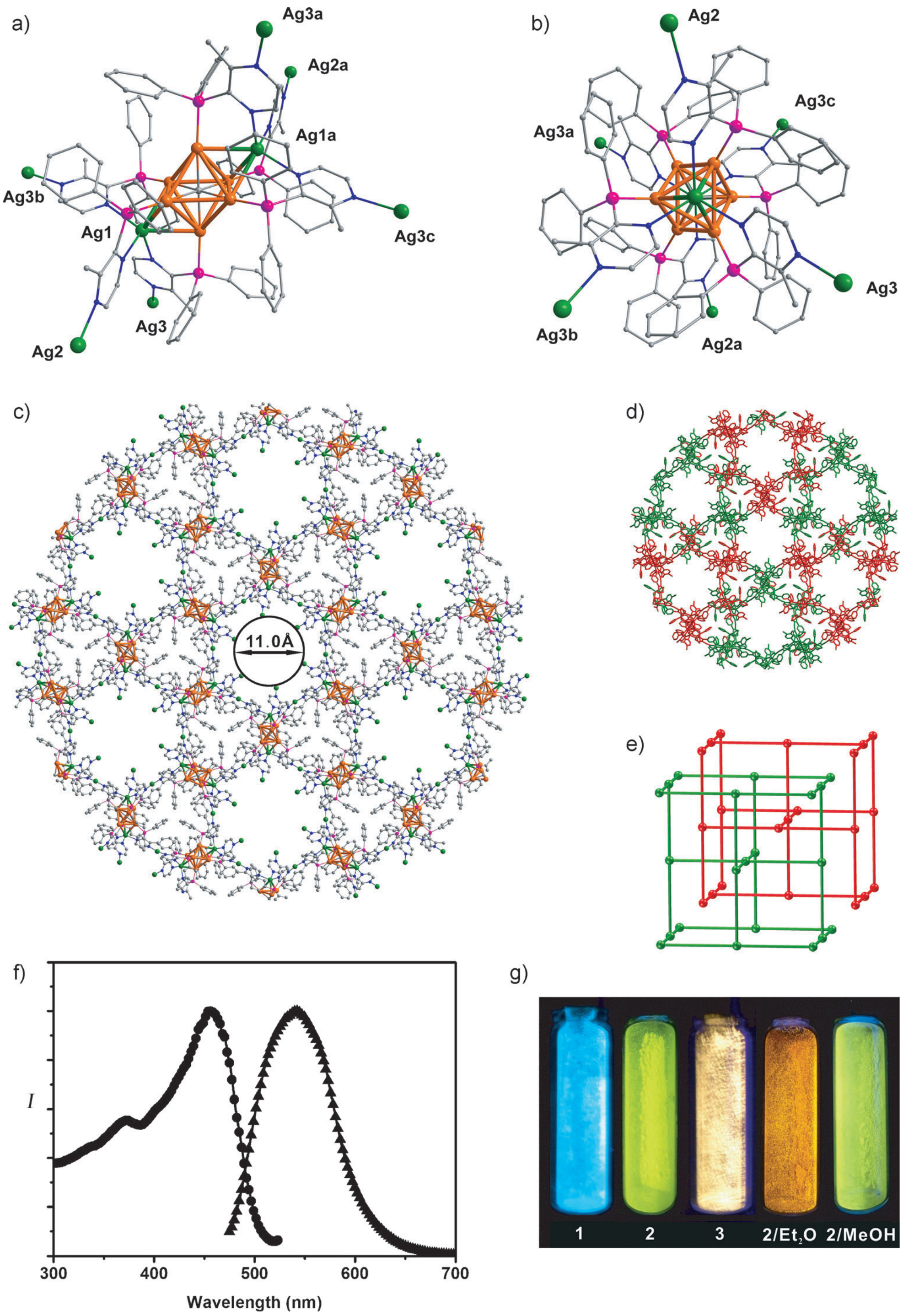

Figure 2. a) Structure of the building unit of **2**. b) Four extending sites: Ag3, Ag3a, Ag3b, Ag3c. Note: Ag2 and Ag2a are not involved in the structural extension. Symmetry code: a: $1/3-x$, $4/3-y$, $2/3-z$; b: $1+x+y$, $1+x$, $-z$; c: $-2/3-x+y$, $2/3-x$, $2/3+z$. c) Perspective view of **2** along the c direction; Au = orange, Ag = green, P = purple, N = blue, C = gray. d) Two interpenetrated nets shown in different colors; anions and solvents omitted for clarity. e) Schematic representation of NbO topology in **2**. f) Excitation (●) and emission (▲) spectra of **2** in the solid state; λ_{max}(●) = 459.4 nm, λ_{max}(▲) = 538.6 nm. g) From left to right: photos of **1**, **2**, **3**, **2** soaked with Et_2O, and **2** soaked with Et_2O and then MeOH under excitation at a wavelength of 365 nm.

process was realized by soaking orange-colored **2** in methanol or water. The emission of **2** was red-shifted after it was soaked in diethyl ether (or hexane) at ambient conditions. Reverse luminescence was observed when **2** was in contact with methanol (or water). The emission spectra and photos of this process are shown in Figure 4a and Figure 2g, respectively. These data indicate that the response is reversible.

PXRD measurements indicate that the crystal structure of **2** is robust during the solvent-exchange process (Figure 4b). The freshly prepared sample was soaked in Et_2O and then in MeOH. The PXRD patterns stayed almost the same during this process. The interactions between solvent molecules and silver ions of type Ag2 inside the channels may account for the changes in absorption and emission spectra of **2**.

The application of heat or a vacuum resulted in the loss of H_2O molecules coordinated to Ag2, thus leading to the corresponding changes in color and luminescence. Soaking in ethyl ether also resulted in a loss of H_2O molecules, which had a similar effect on the color and luminescence as heat or a vacuum. When methanol or water were added, the coordination of methanol or water molecules was restored on Ag2, which resulted in the recovery of **2** and a corresponding change in emission.

In conclusion, a luminescent gold(I) cluster **1** was synthesized and used as a cluster linker for the assembly of a porous 3D framework. The luminescence of the cluster linker was introduced into the MOF, thus giving the luminescent porous material **2**. Compound **2** exhibited an interpenetrated NbO topology and hexagonal channels with a size of about 1.1 nm along the c axis. The channels were available for solvent molecule exchanges along with corresponding luminescence responses.

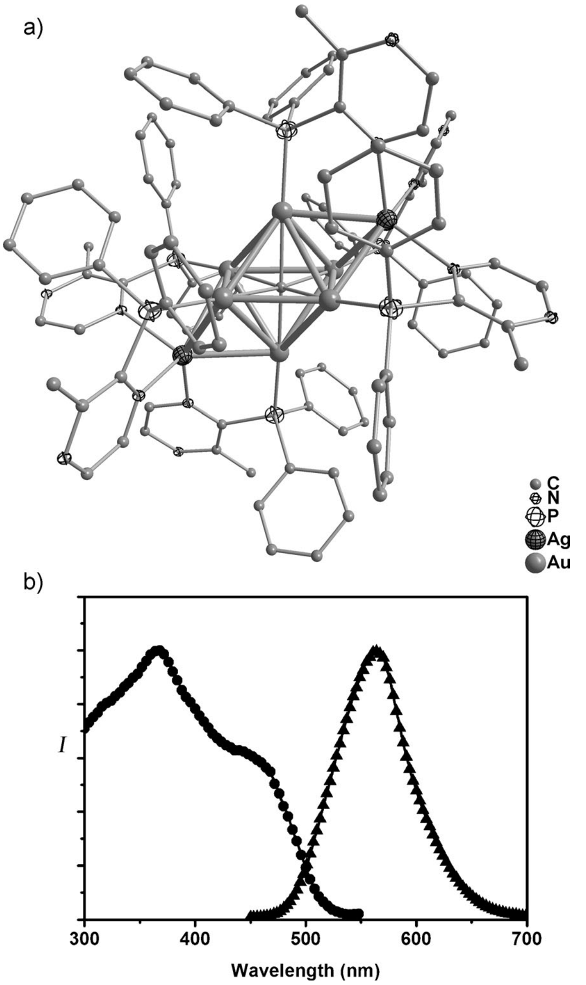

Figure 3. a) Molecular structure of the cationic part of $[(C)(Au\text{-}mdppz)_6Ag_2](BF_4)_4$ (**3**). b) Excitation (●) and emission (▲) spectra of **3** in the solid state; $\lambda_{max}(●) = 366.8$ nm, $\lambda_{max}(▲) = 563.2$ nm.

It should be noted that the silver ions with open sites in **2** are available for further postsynthetic modifications. This cluster linker approach should be useful for the preparation of various bifunctional MOF materials.

Experimental Section

2-(3-Methylpyrazinyl)-diphenylphosphine (mdppz): This compound was prepared in a synthetic procedure similar to a reported method.[17] *n*BuLi in hexane (1.6 M, 18.0 mL) was added dropwise to a solution of Ph_2PH (4.5 mL, 25.3 mmol) in THF (20 mL) at 0 °C. The resulting mixture was stirred for 10 min and added dropwise to a solution of 2-chloro-3-methylpyrazine (2.7 mL, 26.0 mmol) in THF (30 mL) at −78 °C. The mixture was kept at −78 °C and stirred for 3 h, then it was allowed to warm slowly to room temperature. The solvents were removed under vacuum, and water (50 mL) was added. The aqueous phase was extracted with CH_2Cl_2 (3 × 20 mL) and the organic phase was dried with anhydrous Na_2SO_4 overnight. Most of the CH_2Cl_2 was removed under vacuum, and hexane (100 mL) was added to deposit a light yellow solid, which upon recrystallization from CH_2Cl_2/hexane afforded a colorless product. Yield: 5.07 g, 70.1 %. ^{1}H NMR (500.2 MHz, $CDCl_3$): $\delta = 8.43$ (d, 1 H, pz), 8.34 (d, 1 H, pz), 7.32–7.41 (m, 10 H, Ph), 2.59 ppm (s, 3 H, CH_3). ^{31}P NMR (202.5 MHz, $CDCl_3$): $\delta = -7.52$ ppm (s).

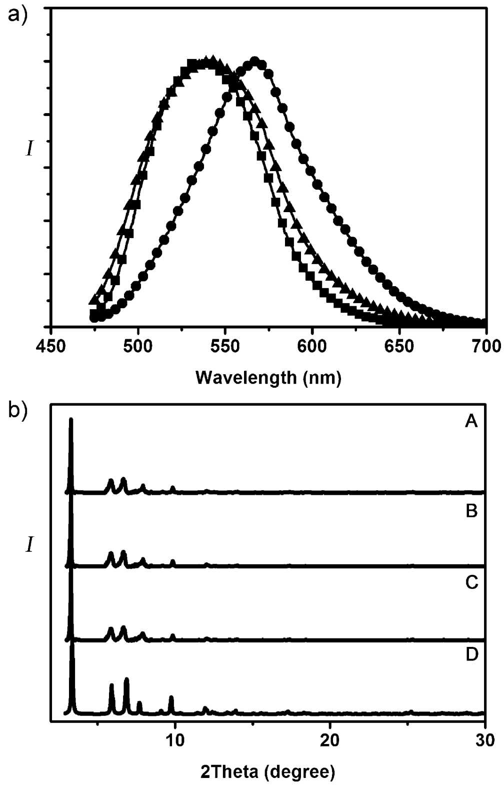

Figure 4. a) Emission spectra of freshly prepared **2** (■; $\lambda_{max} = 538.8$ nm), **2** soaked with Et_2O (●; $\lambda_{max} = 567.0$ nm), and **2** soaked with Et_2O and then MeOH (▲; $\lambda_{max} = 540.0$ nm) at $\lambda_{ex} = 459.0$ nm. b) PXRD of freshly prepared **2** (C), **2** soaked with Et_2O (B), **2** soaked with Et_2O and then MeOH (A), and the simulated pattern (D).

$[(C)(Au\text{-}mdppz)_6](BF_4)_2$ (**1**): This compound was prepared in a synthetic procedure similar to the reported method.[16] Me_2SAuCl (295.0 mg, 1.0 mmol) was added to a solution of mdppz (278.0 mg, 1.0 mmol) in dichloromethane. The solution was stirred for 10 min and the solvents were then evaporated. Ag_2O (464.0 mg, 2.0 mmol), $NaBF_4$ (550.0 mg, 5.0 mmol), and acetone (100 mL) were then added. After 3 h stirring, the solution was evaporated to dryness and the residual was extracted with dichloromethane (3 × 20 mL). A brown solid was obtained after the solvents were removed again. The brown solid was added to a Schlenk tube with N_2 atmosphere, and dichloromethane (5 mL), Et_3N (0.05 mL, 0.40 mmol), and a 2 M solution of Me_3SiCHN_2 in hexane (0.16 mL, 0.32 mmol) were added. The resulting mixture was stirred for 3 h. After filtration, a layer of Et_2O (5 mL) was then allowed to diffuse into the filtrate, which gave the product as colorless block-like crystals after several days. Yield: 243.0 mg (48.0 %, based on gold). Anal. calcd for $C_{103}H_{90}N_{12}B_2F_8P_6Au_6 \cdot 2\,CH_2Cl_2 \cdot C_4H_{10}O$: C: 39.90, N: 5.12, H: 3.19. Found: C: 40.02, N: 4.89, H: 3.14. IR (KBr): $\tilde{\nu} = 1084$ cm^{-1} (br, B-F). ^{1}H NMR (500.1 MHz, CD_2Cl_2): δ = 8.36 (d, 12 H, pz), 7.36–7.26 (m, 36 H, Ph), 6.88–6.82 (t, 24 H, Ph), 1.48 ppm (s, 18 H, CH_3). ^{31}P NMR (202.5 MHz, CD_2Cl_2): δ = 27.51 ppm (s). Lifetime: 2.99 ± 0.22 μs in the solid state. Quantum yield: 1.5 %.

$[(C)(Au\text{-}mdppz)_6]Ag_6(BF_4)_8$ (**2**): Complex **1** (30.4 mg, 0.01 mmol) was dissolved in dichloromethane (5 mL). $AgBF_4$ (12.0 mg, 0.06 mmol) in methanol (3 mL) was added dropwise under stirring to this solution. The mixture was kept in the dark and the volatiles were allowed to evaporate slowly at room temperature. After a few days, yellowish green crystals were obtained. Yield: 26.0 mg (61.8 %, based on **1**). Anal. calcd for $C_{103}H_{90}N_{12}B_8F_{32}P_6Ag_6Au_6 \cdot 2\,H_2O \cdot 2\,CH_3OH$: C: 29.29, N: 3.90, H: 2.39. Found: C: 29.50, N: 4.05, H:

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2.56. IR (KBr): $\tilde{\nu}$ =1099 cm^{-1} (br, B-F). Lifetime: 4.67 ± 0.28 μs in the solid state. Quantum yield: 25.6 %.

[(C)(Au-mdppz)$_6$]Ag$_2$(BF$_4$)$_4$ (**3**): Complex **1** (30.4 mg, 0.01 mmol) was dissolved in dichloromethane (3 mL) and methanol (1 mL). AgBF$_4$ (4.0 mg, 0.02 mmol) was added to the solution under stirring. The mixture was then filtered and the filtrate was layered with Et$_2$O. Yellow crystals were obtained after two days. Yield: 16.7 mg (48.8 %, based on **1**). Anal. calcd for $C_{103}H_{90}N_{12}B_4F_{16}P_6Ag_2Au_6$: C: 36.10, N: 4.91, H: 2.65. Found: C: 36.00, N: 4.85, H: 2.56. IR (KBr): $\tilde{\nu}$ =1084 cm^{-1} (br, B-F). Lifetime: 6.54 ± 0.43 μs in the solid state. Quantum yield (solid state): 3.2 %.

Further experimental details and spectroscopic characterizations are given in the Supporting Information.

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[20] Crystal data for **1**: $C_{103}H_{94}B_2N_{12}F_8P_6Au_6 \cdot 2CH_2Cl_2$, $a = 14.3329(6)$, $b = 14.7905(6)$, $c = 15.0691(5)$ Å, $\alpha = 118.838(4)$, $\beta = 100.608(3)$, $\gamma = 93.512(3)°$, $V = 2708.43(18)$ Å^3, triclinic space group $P\bar{1}$, $Z = 1$, $T = 173.0(1)$ K, 23 945 reflections measured, 9522 unique ($R_{int} = 0.0326$), final $R1 = 0.0355$, $wR2 = 0.0804$ for 8271 observed reflections [$I > 2\sigma(I)$]. Crystal data for **2**: $C_{103}H_{90}B_8N_{12}F_{32}P_6Ag_6Au_6 \cdot 8H_2O$, $a = 51.1116(9)$, $b = 51.1116(9)$, $c = 15.3938(4)$ Å, $\gamma = 120°$, $V = 34826.9(13)$ Å^3, hexagonal space group $R\bar{3}$, $Z = 9$, $T = 173.0(1)$ K, 76630 reflections measured, 21 291 unique ($R_{int} = 0.1075$), final $R1 = 0.0929$, $wR2 = 0.1795$ for 13 502 observed reflections [$I > 2\sigma(I)$]. Crystal data for **3**: $C_{103}H_{90}B_4N_{12}F_{16}P_6Ag_2Au_6 \cdot H_2O \cdot 3\,CH_2Cl_2 \cdot CH_3OH$, $a = 23.4729(4)$, $b = 23.3466(5)$, $c = 22.2668(4)$ Å, $\beta = 94.785(2)°$, $V = 12160.0(4)$ Å^3, monoclinic space group $P2_1/c$, $Z = 4$, $T = 173.0(5)$ K, 49 135 reflections measured, 21 360 unique ($R_{int} = 0.0461$), final $R1 = 0.0403$, $wR2 = 0.1006$ for 17 639 observed reflections [$I > 2\sigma(I)$]. CCDC 1011481 (**1**), 1011482 (**2**), and 1011483 (**3**) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

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