

Supporting Information

Synthesis, Photophysical and Theoretical Studies of Luminescent Silver(I)–Copper(I) Alkynyl-Diphosphine Complexes

Igor O. Koshevoy,^{*, †} Antti J. Karttunen,[†] Yi-Chih Lin,[‡] Chao-Chen Lin,[‡] Pi-Tai Chou,^{*, ‡} Sergey P. Tunik,[#] Matti Haukka[†] and Tapani A. Pakkanen^{*, †}

Department of Chemistry, University of Joensuu, FI-80101, Joensuu, Finland;

Department of Chemistry, National Taiwan University, Taipei 106, Taiwan;

Department of Chemistry, St.-Petersburg State University, Universitetskii pr. 26, 198504, St.-Petersburg, Russia

E-mail: igor.koshevoy@joensuu.fi; tapani.pakkanen@joensuu.fi; chop@ntu.edu.tw

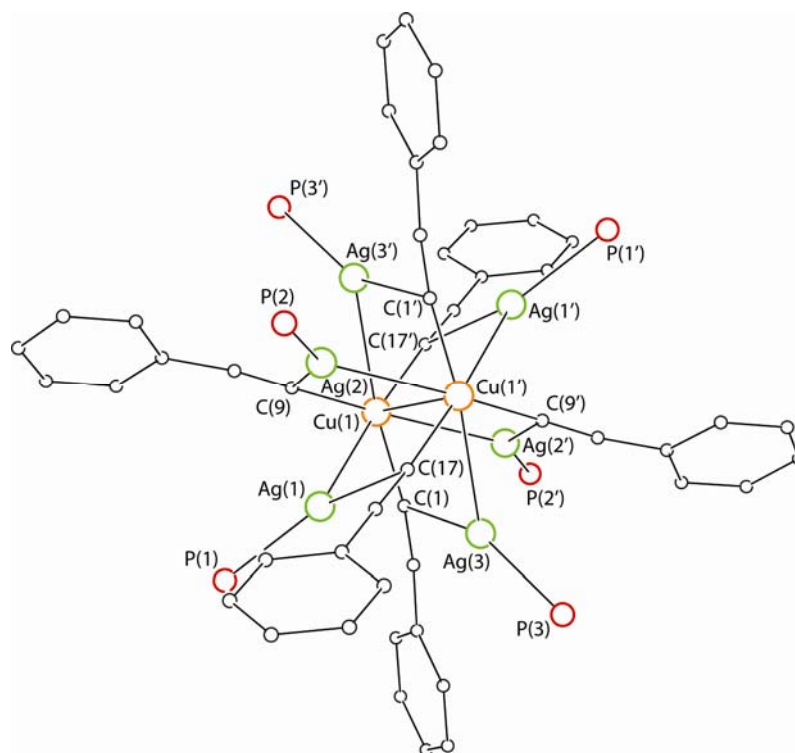


Figure S1. ORTEP view of the central heterometallic cluster of **1**. Selected interatomic distances (Å) are: Ag(1)–C(1) 2.520(5); Ag(1)–C(17) 2.425(5); Ag(1)–C(9) 2.514(5); Ag(2)–C(1') 2.527(5); Ag(2)–C(9) 2.436(5); Ag(2)–C(17) 2.602(5); Ag(3)–C(1) 2.431(5); Ag(3)–C(9') 2.529(5); Ag(3)–C(17) 2.503(5).

X-ray Structure Determinations. The weakly diffracting crystal of **3** was immersed in cryo-oil, mounted in a Nylon loop, and measured at a temperature of 100 K. The X-ray diffraction data was collected on a Nonius KappaCCD diffractometer using Mo K α radiation ($\lambda = 0.71073 \text{ \AA}$). The *Denzo-Scalepack*¹ package was used for cell refinements and data reductions. The structure was solved by direct methods using the *SHELXS-97*² program with the *WinGX*³ graphical user interface. A semi-empirical absorption correction (*SADABS*)⁴ was applied to data. Structural refinement was carried out using *SHELXL-97*.² Two of the PF₆ anions in the asymmetric unit were disordered over two sites with occupancy ratios 0.51/0.49 and 0.50/0.50. Furthermore, the P-F and F...F distances in one of the PF₆ anions were set to be equal. All three NMe₂ -groups in the asymmetric unit were also disordered and all C---N distances were constrained to be similar, but otherwise no disorder model was used in the final refinement. One of the CH₂Cl₂ solvent molecules was disordered over two sites with equal occupancies. Additional disorder was found in some phenyl rings. However, no disorder model was used for these groups in the final refinement. A series of displacement restraints were applied to the disordered moieties so that their U_{ij} components approximate to isotropic behavior. One CH₂Cl₂ and one CH₃CN solvent molecules were heavily disordered and partially lost. The final structural model was refined without these solvent molecules. The contribution of the missing solvent molecules to the calculated structure factors was taken into account using SQUEEZE routine implemented in PLATON⁵ program. The missing solvent molecules were taken into account in the cell content. Hydrogen atoms were positioned geometrically and constrained to ride on their parent atoms, with C-H = 0.95-0.99 Å and U_{iso} = 1.2-1.5 U_{eq}(parent atom).

1. Otwinowski, Z.; Minor, W. Processing of X-ray Diffraction Data Collected in Oscillation Mode, Academic Press, New York, pp. 307-326, 1997. In *Methods in Enzymology, Volume 276, Macromolecular Crystallography, Part A*, Carter, C. W., Sweet, J., Eds.; Academic Press: New York, USA, 1997; pp 307-326.
2. Sheldrick, G. M. *Acta Crystallogr. Sect. A* **2008**, A64, 112-122.
3. Farrugia, L. J. *J. Appl. Crystallogr.* **1999**, 32, 837.
4. Sheldrick, G. M. *SADABS-2008/1 - Bruker AXS area detector scaling and absorption correction*, Bruker AXS: Madison, Wisconsin, USA, 2008
5. Spek, A. L. *A Multipurpose Crystallographic Tool*, Utrecht University: Utrecht, The Netherlands, 2003

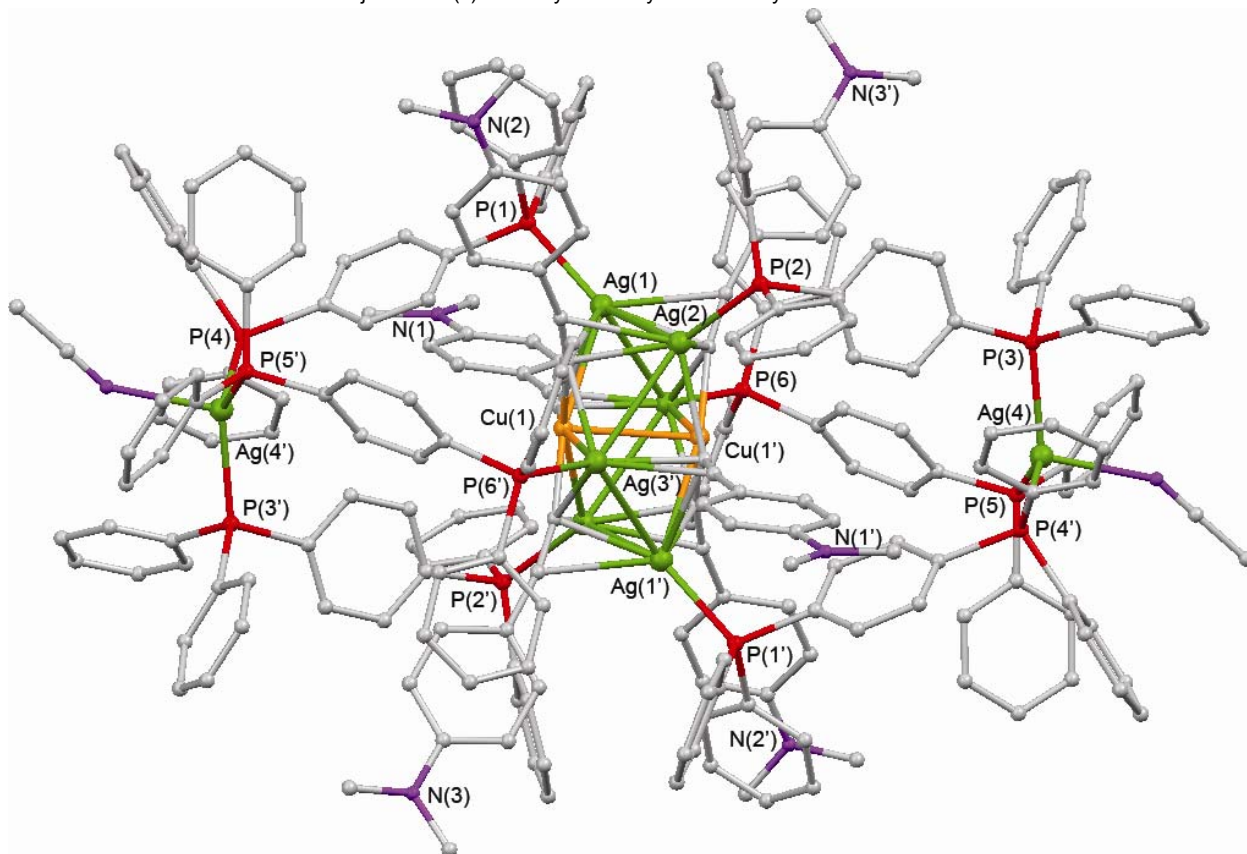


Figure S2. Molecular structure of the cluster **3**. Selected intermetallic distances (Å) are: Ag(1)-Cu(1) 2.7998(9); Ag(1)-Ag(3) 3.1011(7); Ag(1)-Ag(2) 3.1949(8); Ag(2)-Cu(1') 2.7888(8); Ag(2)-Ag(3') 3.1412(7); Ag(3)-Cu(1') 2.7847(9); Cu(1)-Cu(1') 2.8748(15). Symmetry transformations used to generate equivalent atoms: ' -x,-y,-z.

Table S1. Crystal data and structure refinement for **3**.

	3	
Identification code	3	
Empirical formula	C ₂₅₇ H ₂₃₃ Ag ₈ Cl ₆ Cu ₂ F ₂₄ N ₁₃ P ₁₆	
Formula weight	5657.82	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P $\bar{1}$	
Unit cell dimensions	a = 17.4900(2) Å	$\alpha = 81.9800(10)^\circ$.
	b = 18.5715(3) Å	$\beta = 87.162(9)^\circ$.
	c = 20.4761(4) Å	$\gamma = 78.3190(10)^\circ$.
Volume	6447.96(18) Å ³	
Z	1	
Density (calculated)	1.457 Mg/m ³	
Absorption coefficient	0.987 mm ⁻¹	
F(000)	2858	
Crystal size	0.25 x 0.17 x 0.13 mm ³	
Theta range for data collection	2.43 to 25.15°.	
Index ranges	-20 ≤ h ≤ 20, -22 ≤ k ≤ 22, -24 ≤ l ≤ 24	
Reflections collected	116760	
Independent reflections	23031 [R(int) = 0.0467]	
Completeness to theta = 25.15°	99.7 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.8857 and 0.7932	

Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	23031 / 265 / 1461
Goodness-of-fit on F ²	1.062
Final R indices [I > 2σ(I)]	R1 = 0.0687, wR2 = 0.1765
R indices (all data)	R1 = 0.0817, wR2 = 0.1839
Largest diff. peak and hole	2.883 and -1.873 e.Å ⁻³

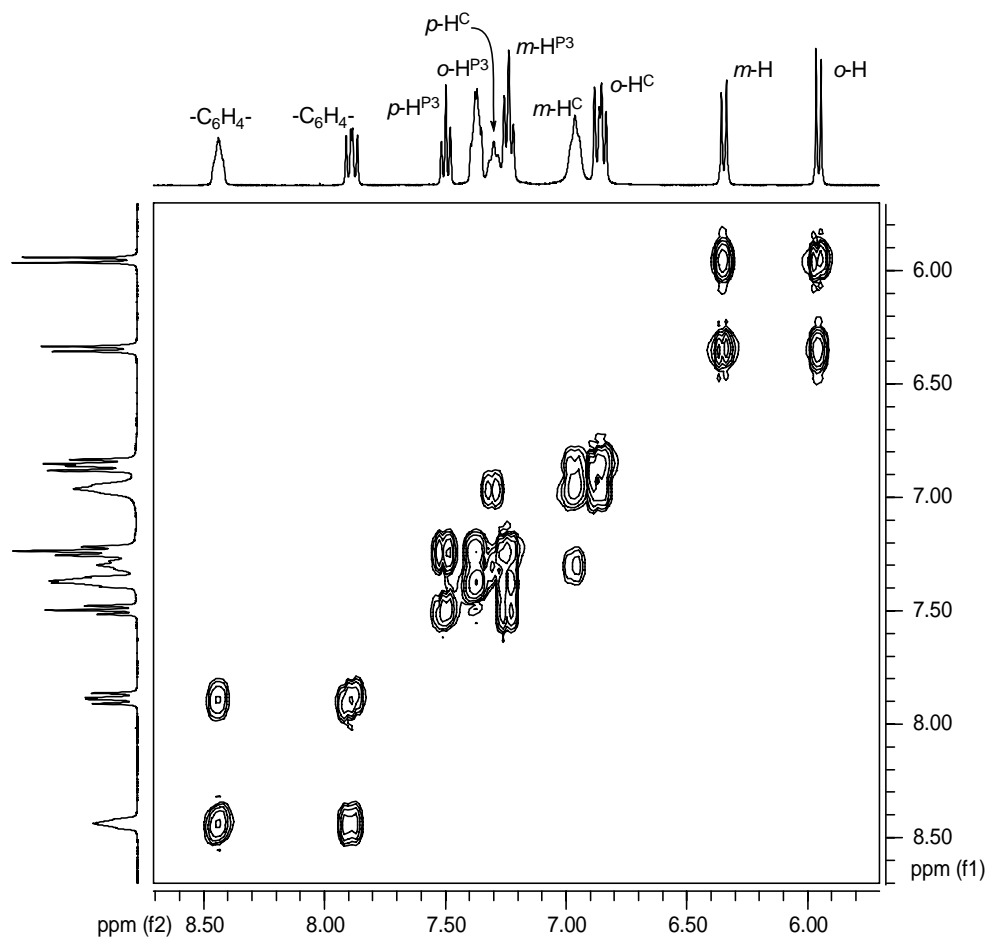


Figure S3. The ¹H COSY spectrum of **2** (aromatic region), 400 MHz, acetone-d₆, 298 K. H – phenyl protons of the C₂Ph ligands; H^{P3} – phenyl protons of the dppb (PPh₂C₆H₄PPh₂) ligands, adjacent to the {AgP₃} fragment; H^C – phenyl protons of the dppb ligands, adjacent to the {Ag₆Cu₂} fragment.

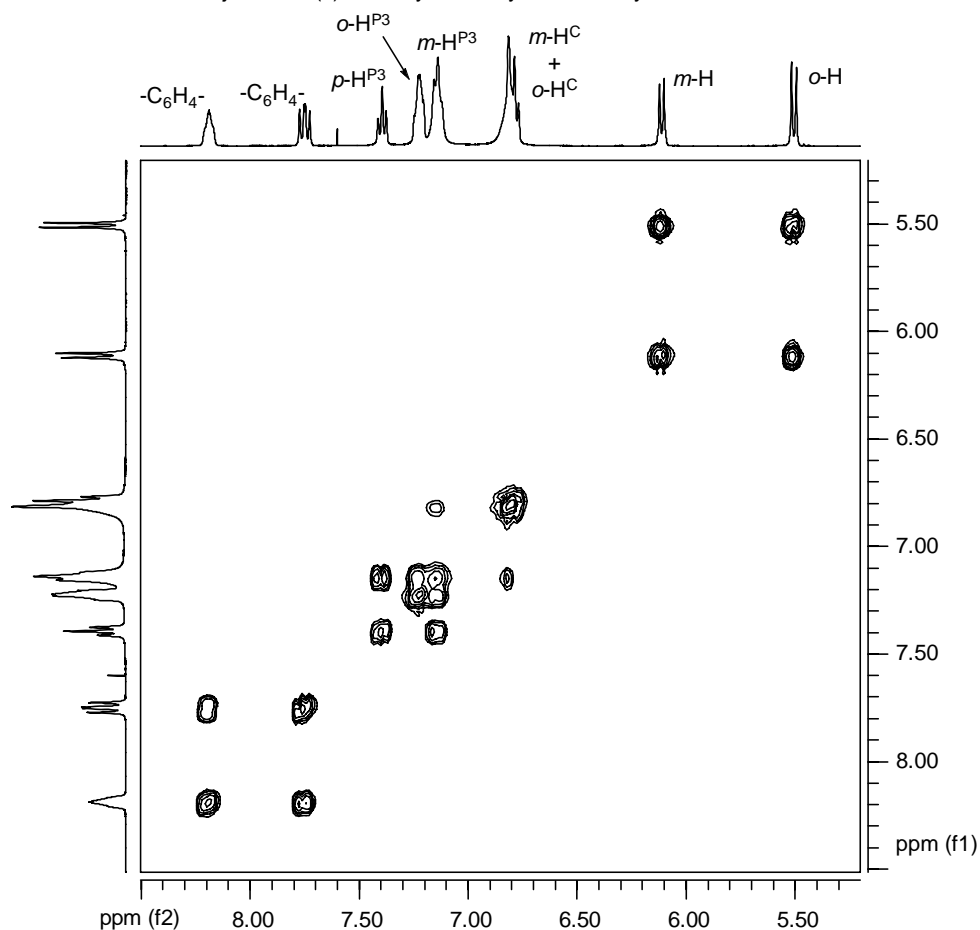


Figure S4. The ¹H COSY spectrum of **3** (aromatic region), 400 MHz, acetone-d₆, 298 K. H – phenyl protons of the C₂Ph ligands; H^{P3} – phenyl protons of the dppb ligands, adjacent to the {AgP₃} fragment; H^C – phenyl protons of the dppb ligands, adjacent to the {Ag₆Cu₂} fragment.

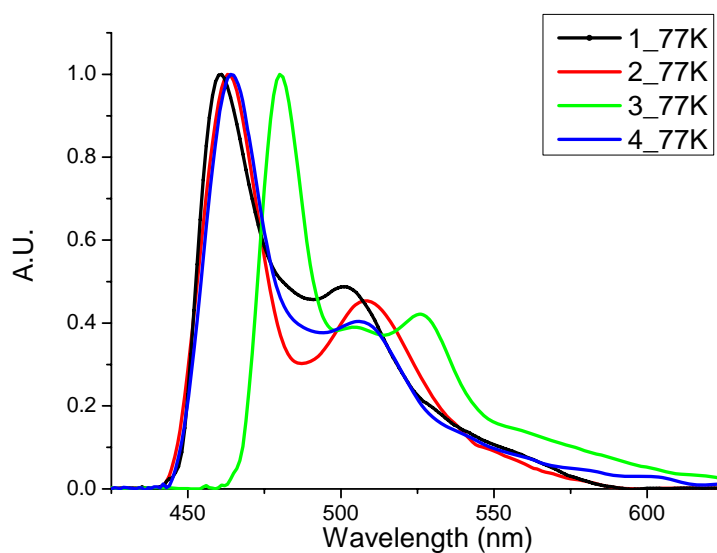
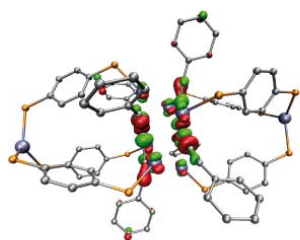
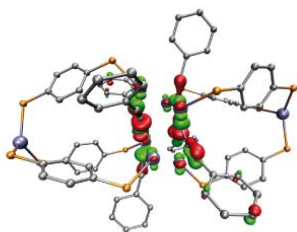


Figure S5. The emission spectra of complexes **1-4** in acetonitrile at 77 K.

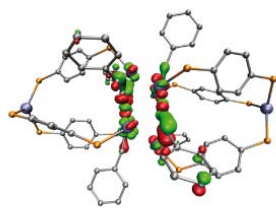
Computational Data



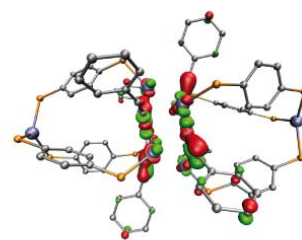
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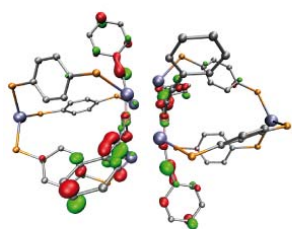
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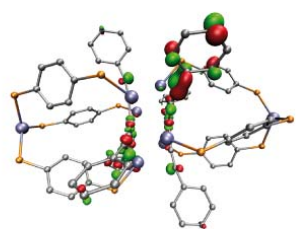
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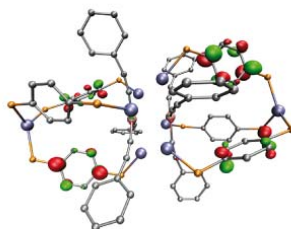
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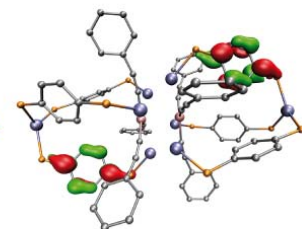
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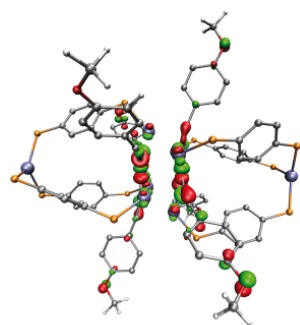
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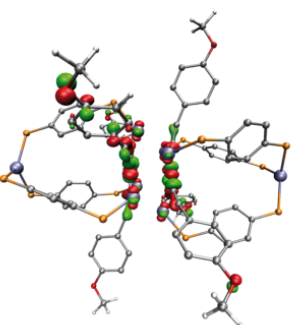
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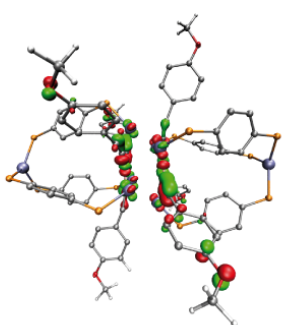
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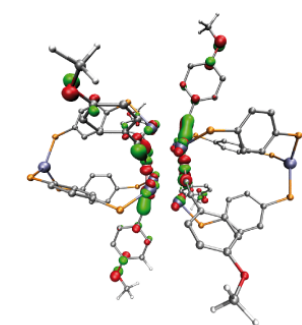
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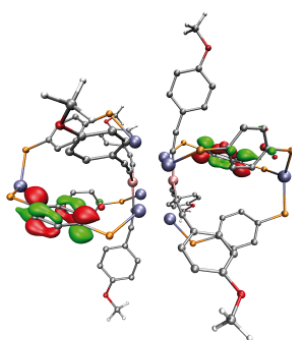
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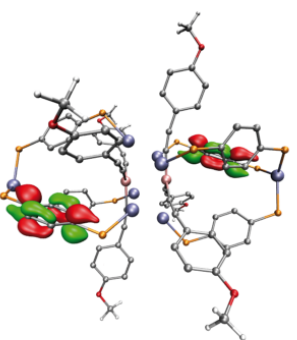
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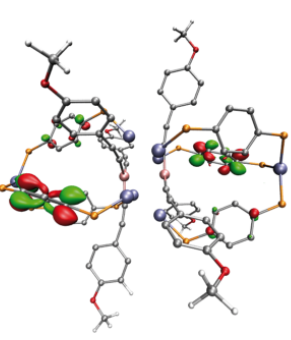
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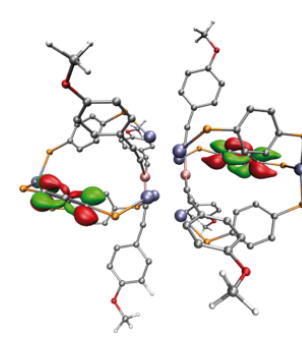
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(2) LUMO + 2



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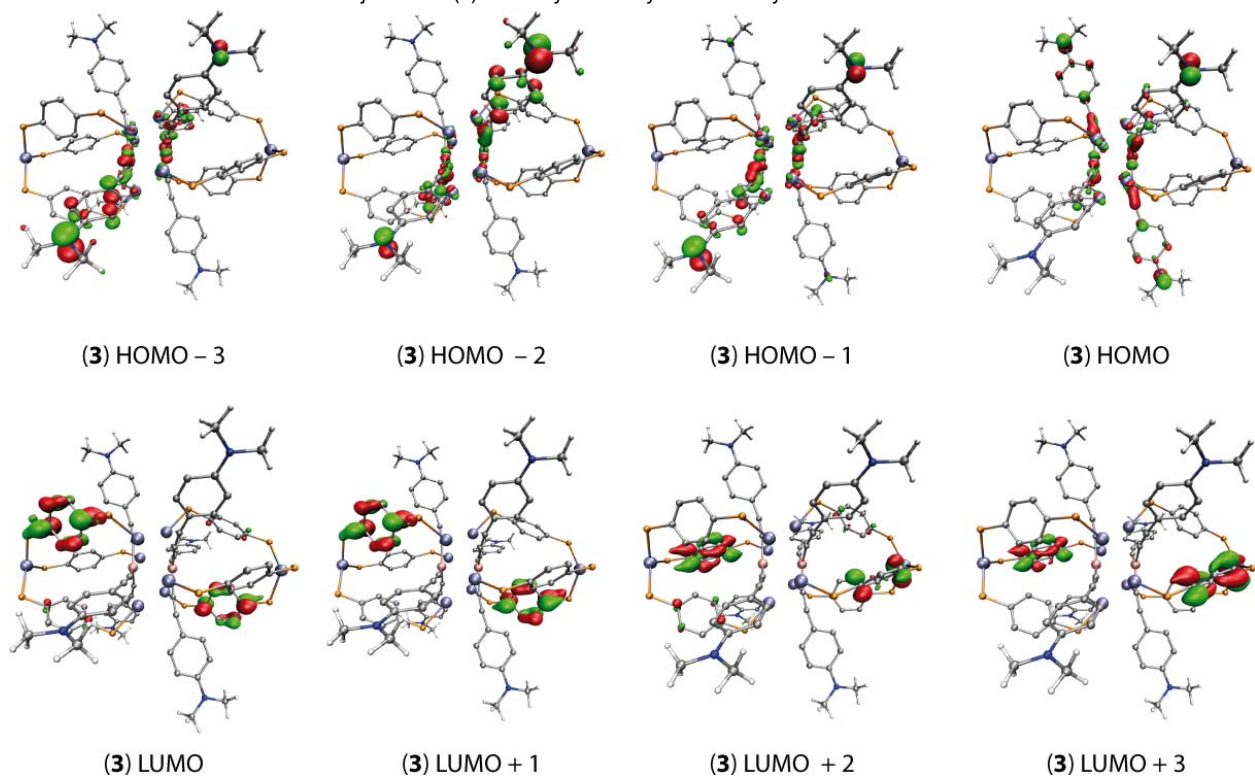


Figure S6. Frontier molecular orbital isodensity plots for the complexes **1–3** (isodensity value 0.04 a.u.). Phenyl rings of the diphosphine ligands, acetonitrile ligands, hydrogen atoms, and most bonds within the central metal cluster have been omitted for clarity.

Optimized Cartesian coordinates of the studied systems in atomic units (BP86 level of theory)

$[\text{Ag}_8\text{Cu}_2(\text{C}_2\text{Ph})_6(\text{PPh}_2\text{C}_6\text{H}_4\text{PPh}_2)_6]^{4+}$ (**1**)

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14.76461647938457	5.74010154329322	-21.24975722224434	c
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$[\text{Ag}_8\text{Cu}_2(\text{C}_2\text{C}_6\text{H}_4\text{OMe})_6(\text{PPh}_2\text{C}_6\text{H}_4\text{PPh}_2)_6]^{4+}$ (2)

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-2.30018084000805	-9.21045938808436	3.20192320675321	p
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-3.47832951714730	-3.67780480720327	-8.78565796311399	p
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6.41984770664152	-1.83961438646816	-16.00271764991599	p
-6.70052695747133	-6.10656652480516	14.35950574476839	p
0.71873262726322	5.32965047167866	-0.96483845378447	ag
-4.89069396341186	2.32191009142327	-0.65297538176637	ag
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-9.09855127527199	-1.93228556792462	14.37736935489070	ag
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10.49568352961109	-10.36132329215865	12.17484816647430	c
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10.87315806484649	-11.56939921049718	13.84241874119831	h
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16.13329898277112	7.70547623333040	6.04487383295321	h
17.93364357801741	10.04313706514307	4.30998395275052	h
14.71273138617257	10.73227328008855	5.24513632822055	h

[Ag₈Cu₂(C₂C₆H₄NMe₂)₆(PPh₂C₆H₄PPh₂)₆]⁴⁺ (3)

-0.68393455340306	-5.34087866095055	0.98996476670922	ag
4.88584699040289	-2.33210566744029	0.64877850894963	ag
-2.51389564530056	-2.30961287119027	-4.30387738412413	ag
9.11465068256650	1.87451075700906	-14.35979277728182	ag

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-1.40058286369109	-0.31045167397849	2.23301802934775	cu
-2.24638566825400	-9.16464018986576	3.24312827660926	p
9.20443327539280	-3.75920168580463	-0.51327186598011	p
-3.42217300178581	-3.69786030849171	-8.69356639317345	p
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-6.62463725782972	-6.01012536797865	14.39351611339870	p
0.71869406152995	5.33169157135635	-0.96850207997878	ag
-4.85586086535510	2.32414164344493	-0.62498639754639	ag
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-9.08424375655586	-1.87314615079946	14.37782070570539	ag
1.43171593415601	0.29959685006995	-2.21191095676128	cu
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