

Synthesis, structure and spectroscopic properties of two new cyanido-bridged trinuclear 9-atom molecular  $\text{Ag}-\text{N}\equiv\text{C}-\text{Cu}-\text{N}\equiv\text{C}-\text{Au}-\text{C}\equiv\text{N}$  assembly of formula  $[\text{AgCuAu}(\text{CN})_3(\text{PPh}_3)_5](\text{H}_2\text{O})_2$  and a dinuclear gold–copper one-dimensional coordination polymer of formula  $[\text{AuCu}(\text{CN})_2(\text{PPh}_3)_2](\text{H}_2\text{O})_2$

Mohammed Jaafar<sup>a</sup>, Andrej Pevec<sup>b</sup>, Sebastiaan Akerboom<sup>c</sup>, Ali Alsalmeh<sup>a</sup>, Khalid Al-Farhan<sup>a</sup>, Mohamed Ghazzali<sup>a,\*</sup>, Jan Reedijk<sup>a,c,\*</sup>

<sup>a</sup> Department of Chemistry, College of Science, King Saud University, P.O. Box 2455, Riyadh 11451, Kingdom of Saudi Arabia

<sup>b</sup> Faculty of Chemistry and Chemical Technology, University of Ljubljana, Aškerčeva 5, P.O. Box 537, 1000 Ljubljana, Slovenia

<sup>c</sup> Leiden Institute of Chemistry, Leiden University, P.O. Box 9502, 2300 RA Leiden, The Netherlands

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## ABSTRACT

Two new cyanido-bridged coinage metal compounds are presented. A novel hetero trinuclear Ag–Cu–Au compound of formula  $[\text{AgCuAu}(\text{CN})_3(\text{PPh}_3)_5](\text{H}_2\text{O})_2$  **1** is described with bridging cyanide ligands between the metals, and terminal phosphane ligands for Cu(I) and Ag(I). Silver is tetrahedrally coordinated by 3 phosphane ligands and a bridging cyanido ligand, copper is coordinated tetrahedrally by 2 phosphanes, and 2 bridging cyanide ligands, whereas gold has a linear coordination by 2 cyanido-κC ligands. Under slightly different synthetic conditions, an alternating chain compound  $[\text{AuCu}(\text{CN})_2(\text{PPh}_3)_2](\text{H}_2\text{O})_2$  **2** was found, isostructural with the related Ag–Cu compound. In **2**, Cu(I) is tetrahedrally coordinated by 2 phosphane ligands and 2N atoms of the bridging cyanido ligand to the Au(I). The qualitative solid-state luminescence emission intensity of **1** is reported and compared with that of the related bimetallic Au–Cu, Ag–Cu, Ag–Ag and Ag–Au cyanido-bridged chain compounds. The luminescence of **2** was found somewhat stronger than that of **1**. The electronic molecular orbitals were calculated by DFT, where a strong contribution of Cu(I) orbitals in HOMO/LUMO character of the two complexes is observed.

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## 1. Introduction

Hetero-nuclear metal compounds are nowadays quite common, several of these display interesting properties, like in catalysis and advanced materials [1]. Within this class of compounds, those with hetero-nuclear monovalent group 11 metals (Cu, Ag, Au) received quite some attention as therapeutics [2,3] and also as photo-reactive agents [4]. Consequently, luminescent d<sup>10</sup> Ag(I) or Au(I) compounds have been actively investigated in recent years, with a focus on their emission properties in relation to the electronic structure of the compounds [4–9].

In our recent work we have reported [10,11] on chain-type bimetallic cyanido-bridged compounds of formulae  $[\text{M}(\text{CN})_2$

$(\text{PPh}_3)_2\text{Ag}]_n$  (M is Ag, Cu or Au). These compounds are easily prepared by room temperature reactions of triphenylphosphane with the cyanide salts of silver(I), copper(I) and gold(I). These compounds belong to a class of one-dimensional cyanido-bridged chain coordination polymers, where the tetrahedral Ag<sup>I</sup> ions are coordinated by 2 triphenylphosphane ligands and 2N-bound cyanides, in a  $\text{AgN}_2\text{P}_2$  chromophore. The linear Ag(I), Cu(I) and Au(I) centers are in a two-coordinate geometry with C-bound cyanides. This series was shown to display interesting luminescence properties [10].

In an attempt to improve the luminescence of these compounds we tried to expand the series Ag–M (M = Ag, Cu, Au) to Cu–M (M = Au, Cu). The first product (abbreviated Cu–Au) is isostructural with the known Ag–Au product, but now with Ag replaced by Cu. The structure and qualitative luminescence of this compound are described. By using the 3 metals together, where both Cu and Au are added simultaneously to the Ag phosphane units, the chain

\* Corresponding authors at: Leiden Institute of Chemistry, Leiden University, P.O. Box 9502, 2300 RA Leiden, The Netherlands.

E-mail address: [reedijk@chem.leidenuniv.nl](mailto:reedijk@chem.leidenuniv.nl) (J. Reedijk).

propagation is halted and surprisingly an unprecedented trinuclear molecular structure of formula  $[\text{AgCuAu}(\text{CN})_3(\text{PPh}_3)_5](\text{H}_2\text{O})_2$  is found. In this case, Cu binds to phosphane ligands, while the linear Au(I) has, in addition to the bridging cyanide, only a terminal C-bound cyanide. Both the trinuclear AgCuAu and dinuclear AuCu compounds, abbreviated as **1** and **2** were found stable in day light. Both X-ray single crystal derived-structures are presented below, together with their solid state luminescence properties and DFT theoretical calculations.

## 2. Experimental

### 2.1. Starting materials

The starting chemicals silver(I) cyanide, copper(I) cyanide, gold(I) cyanide and tri(phenyl phosphane) were commercially available (BDH-Analar grade). Hexane and dichloro-methane were kept over molecular sieves.

### 2.2. Analytical and physical methods

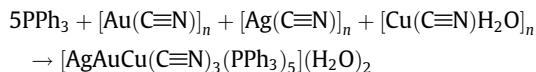
The elemental analyses were performed by using a Perkin Elmer Series II-2400 analyzer, whereas metal analyses were performed using ICP equipment, after dissolving the compound in *aqua regia*.

The FT-IR spectra were recorded on a Thermo Scientific Nicolet iS10. The excitation and emission spectra were recorded at room temperature using a Shimadzu RF-5301PC spectrofluorophotometer equipped with a solid-state sample holder. The excitation spectra were recorded by constantly monitoring the emission spectrum at the wavelength of most intense luminescence, while scanning the excitation wavelength from 220 to 400 nm. The excitation spectrum has been corrected for the response of the detector using an 8 mg/mL solution of rhodamine-B in ethylene glycol as a standard.

### 2.3. Synthesis of $[\text{AgAuCu}(\text{C}\equiv\text{N})_3(\text{PPh}_3)_5](\text{H}_2\text{O})_2$ **1**

Gold(I) cyanide,  $[\text{Au}(\text{C}\equiv\text{N})]_n$  (0.223 g, 1 mmol) was added to a clear solution of tri(phenyl)phosphane,  $\text{PPh}_3$  (1.310 g, 5 mmol) in dichloromethane (40 mL). Immediately a clear solution was obtained, and subsequently silver(I) cyanide  $[\text{AgC}\equiv\text{N}]_n$  (0.134 g, 1 mmol) with copper(I) cyanide  $[\text{Cu}(\text{CN})\text{H}_2\text{O}]_n$  (0.108 g, 1 mmol) were simultaneously added into this solution. A white crystalline solid was gradually precipitating, filtered and recrystallized from a 10/20 mixture dichloromethane/hexane (30 mL) and finally collected as crystalline material, from which a suitable crystal analyzed according to the formula  $[\text{AgAuCu}(\text{C}\equiv\text{N})_3(\text{PPh}_3)_5](\text{H}_2\text{O})_2$  (yield 1.751 g, 97%), m.p. 198 °C. Elemental Analysis for  $\text{C}_{93}\text{H}_{79}\text{AgAuCuO}_2\text{N}_3\text{P}_5$ : Found (calc.): %C: 62.64 (62.26), %H: 4.43 (4.40), %N: 2.52 (2.34), %Cu: 4.1 (3.54), %Ag: 5.8 (6.01), %Au: 10.8 (10.98).

The reaction that has taken place appears to be:



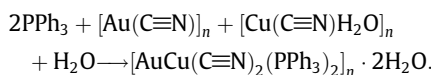
Part of the re-crystallized precipitate, when dried at 100 °C for 3 h gave a white powder, apparently without the two molecules water and with formula  $\text{C}_{93}\text{H}_{75}\text{AgAuCuN}_3\text{P}_5$ , m.p. 190 °C: Found (calc.): %C: 63.93 (63.53), %H: 4.12 (4.27), %N: 2.52 (2.39). FT-IR:  $\nu$  max  $\text{cm}^{-1}$  (KBr disk) 1093 (P–C), 2168(w), 2145(m) and 2118(s)  $\text{cm}^{-1}$  (C≡N).

### 2.4. Synthesis of $[\text{AuCu}(\text{C}\equiv\text{N})_2(\text{PPh}_3)_2]_n(\text{H}_2\text{O})_{2n}$ **2**

Under conditions with a lower Ag ion concentration in the solution, a different product has been isolated, analyzed as

$\text{C}_{38}\text{H}_{34}\text{AuCuN}_2\text{O}_2\text{P}_2$ . It turned out that even without the presence of Ag, this product could be prepared. Gold(I) cyanide,  $[\text{Au}(\text{C}\equiv\text{N})]_n$  (0.223 g, 1 mmol) was added to a clear solution of tri(phenyl)phosphane,  $\text{PPh}_3$  (0.524 g, 2 mmol) in dichloromethane (45 mL). During stirring a clear solution was obtained, and subsequently copper cyanide  $[\text{Cu}(\text{CN})\text{H}_2\text{O}]_n$  (0.108 g, 1 mmol), was added and dissolved within 10 min. After about 45 min a white crystalline solid started to precipitate. The solid was filtered off and washed with dichloromethane. After recrystallization from a 50/50 mixture dichloromethane/hexane, from which a suitable crystal turned out to have the formula  $[\text{AuCu}(\text{C}\equiv\text{N})_2(\text{PPh}_3)_2](\text{H}_2\text{O})_2$  (yield 0.770 g; 88%), m.p. 132 °C. FT-IR:  $\nu$  max  $\text{cm}^{-1}$  (KBr disk): 1093(s); 2161(s), 2125(m)  $\text{cm}^{-1}$  (C≡N). Elemental analysis: Found (calc. for  $[\text{AuCu}(\text{C}\equiv\text{N})_2(\text{PPh}_3)_2](\text{H}_2\text{O})$ ): %C: 53.09 (52.27), %H: 3.52 (3.90), %N: 3.16 (3.21).

The reaction that has taken place appears to be:



Elemental analysis after drying *in vacuo*: Found (calc. for  $[\text{AuCu}(\text{C}\equiv\text{N})_2(\text{PPh}_3)_2]$ ): %C: 54.93 (54.5), %H: 3.32 (3.59), %N: 3.39 (3.35).

### 2.5. X-ray single crystal diffraction

Suitable colorless single-crystals of compounds **1** and **2** were selected and glued on a glass thread. Diffraction data were collected at room temperature with an Agilent SuperNova dual source using an Atlas detector and equipped with mirror-monochromated Mo K $\alpha$  radiation ( $\lambda = 0.71073$  Å). The data were processed by using CrysAlis PRO [12]. The structure was solved by direct methods and refined by full-matrix least-square procedure based on  $F^2$  using SHELX package [13]. All hydrogen atoms bound to carbon were included in the model at geometrically calculated positions and refined using a riding model. The other hydrogen atoms (O–H) were located in the difference Fourier map and refined with the help of distance restraints. Crystal and diffraction collection data are summarized in Table 1.

### 2.6. Theoretical calculations

The *ab initio* DFT molecular electronic calculations were performed using the ADF package [14] on ground state geometry truncated from X-ray single crystal structure z-matrices, without the two water molecules. In **2**, the  $\text{d}^{10}$   $[\text{Au}(\text{PPh}_3)_2\text{Cu}(\text{CN})]$  cationic fragment with tetrahedral Cu<sup>I</sup> and linear Au<sup>I</sup> coordination environments are sustained by two molecules of HCN, while the geometrical positions of the two hydrogen atoms were optimized. Single-point calculations were performed on geometry optimized molecules with exchange correlation energy calculated without geometrical constraints, employing the hybrid exchange functional B3LYP, augmented with the scalar zeroth-order regular approximation (ZORA) triple- $\zeta$ -plus two polarization function (TZ2P) basis set, as implemented. The A-DIIS restricted SCF relativistic calculations were performed, employing a Slater Type Orbital (STO) triple- $\zeta$ -plus one polarization function (TZP) for metal ions.

## 3. Results and discussion

### 3.1. Structural description

The molecular structure of the trinuclear compound **1** is presented in Fig. 1left, together with the two water lattice molecules; these lattice molecules fill up a cavity in the lattice, and only are mutually H bonded. They can be removed upon drying. Relevant

**Table 1**  
Crystal data and structure refinement details for **1** and **2**.

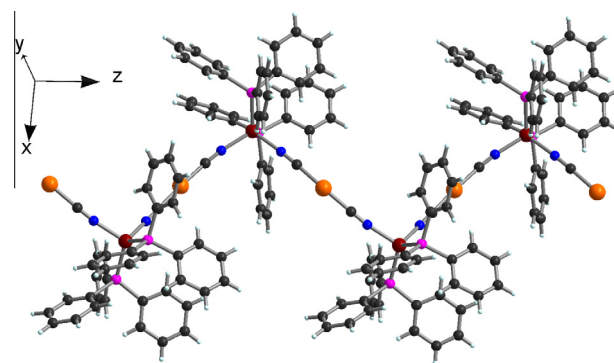
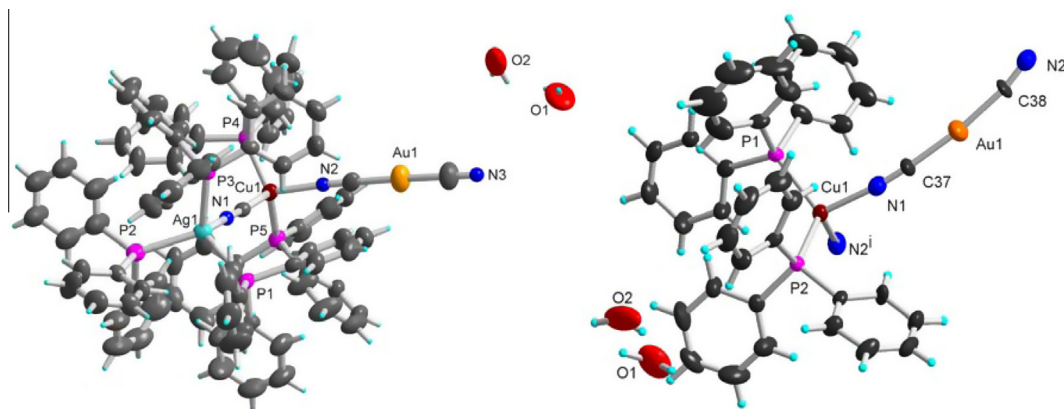
	1	2
Formula	C <sub>93</sub> H <sub>79</sub> AgAuCuN <sub>3</sub> O <sub>2</sub> P <sub>5</sub>	C <sub>38</sub> H <sub>34</sub> AuCuN <sub>2</sub> O <sub>2</sub> P <sub>2</sub>
Formula weight (g mol <sup>-1</sup> )	1793.82	873.12
Crystal system	triclinic	orthorhombic
Space group	<i>P</i> $\bar{1}$	<i>Pna</i> 2 <sub>1</sub>
<i>a</i> (Å)	14.3572(2)	25.6835(3)
<i>b</i> (Å)	16.6540(3)	9.91100(10)
<i>c</i> (Å)	18.8183(4)	14.9309(2)
$\alpha$ (°)	80.310(2)	90
$\beta$ (°)	73.5610(10)	90
$\gamma$ (°)	85.953(2)	90
<i>V</i> (Å <sup>3</sup> )	4252.79(13)	3800.65(8)
<i>Z</i>	2	4
<i>D</i> <sub>calc</sub> (g cm <sup>-3</sup> )	1.401	1.526
No. of collected reflections	36236	34107
No. of independent reflections	17361	8695
<i>R</i> <sub>int</sub>	0.0515	0.0334
No. of reflections observed	9664	7811
No. parameters	897	417
<i>R</i> [ <i>I</i> > 2σ( <i>I</i> )] <sup>a</sup>	0.0742	0.0507
<i>wR</i> <sub>2</sub> (all data) <sup>b</sup>	0.2334	0.1532
Goodness-of-fit, <i>S</i> <sup>c</sup>	1.022	1.129
Maximum/minimum residual electron density (e Å <sup>-3</sup> )	+1.73/−1.84	+1.65/−1.89

<sup>a</sup>  $R = \sum ||F_o| - |F_c|| / \sum |F_o|$ .<sup>b</sup>  $wR_2 = \{ \sum [w(F_o^2 - F_c^2)^2] / \sum [w(F_o^2)^2] \}^{1/2}$ .<sup>c</sup>  $S = \{ \sum [(F_o^2 - F_c^2)^2] / (n/p) \}^{1/2}$  where *n* is the number of reflections and *p* is the total number of reflections.**Table 2**Selection of important metal–ligand bond, O...O contact and M...M contacts distances (Å) and angles (degrees) in the compounds [AgAuCu(C≡N)<sub>3</sub>(PPh<sub>3</sub>)<sub>5</sub>](H<sub>2</sub>O)<sub>2</sub> (**1**) and [AuCu(C≡N)(PPh<sub>3</sub>)<sub>2</sub>](H<sub>2</sub>O)<sub>2</sub> (**2**).

	1	2
Au–C	1.988(12) and 1.989(10)	1.940(7) and 1.942(6)
Cu–N	2.050(8)	2.033(7)
Cu–C/N	1.977(8)	
Cu–P	2.292(2) and 2.283(2)	2.2735(16) and 2.2882(17)
Ag–N/C	2.262(8)	
Ag–P	2.568(2); 2.516(2); 2.574(3)	
O...O (H bond)	2.816(17)	2.865(15)
Ag...Cu	5.343(1)	
Cu...Au	5.149(1)	5.0813(8) and 5.0672(9)
Ag...Au	7.952(1)	
Angles (degrees)		
C–Au–C	178.1(4)	173.5(3)
N–Cu–C/N	107.2(3)	
P–Cu–P	120.26(8)	118.46(6)
P–Cu–N	100.5(2); 109.3(2)	109.4(2); 109.8(2); 105.2(2); 109.1(2)
P–Cu–C	108.6(2); 110.1(2)	
P–Ag–P	114.81(7); 114.36(8); 111.88(7)	
P–Ag–N/C	102.9(2); 101.85(19); 109.8(2)	
Ag...Cu...Au	98.54(2)	

M–L bond distances are given in Table 2. Assignment of the metal ions was clear, and no intermolecular M–M interactions are observed.

The coordination around silver is trigonal, 4-coordinated with 3 phosphane ligands and one cyanide bridging to Cu(I). Regarding this bridging cyanide between Cu and Ag, it is not *a priori* possible to ascertain the position of N and C between Ag and Cu from a crystallographic point of view. A survey of CSD [15] entries, release November 2013, showed that there are 36 structures with the fragment Ag–C≡N–Cu(II) and no structure with Ag–N≡C–Cu(II). In fact only one structure with Ag–N≡C–Cu(I) [10] is known from a related compound and one other case for an octanuclear ring cluster with alternating Ag/Cu [16]. We carefully also checked the other possibility, and when tried to refine the structure with Ag–C≡N–Cu the anisotropic displacement parameters (ADPs) of C and N appeared with a bit less realistic than in the other case.

**Fig. 2.** Presentation of the one dimensional zigzag-like chain compound of **2**.**Fig. 1.** Atomic displacement parameters (50% probability) with numbering scheme for compound **1** (left) and **2** (right). Hydrogen atoms are presented as spheres of arbitrary radii.

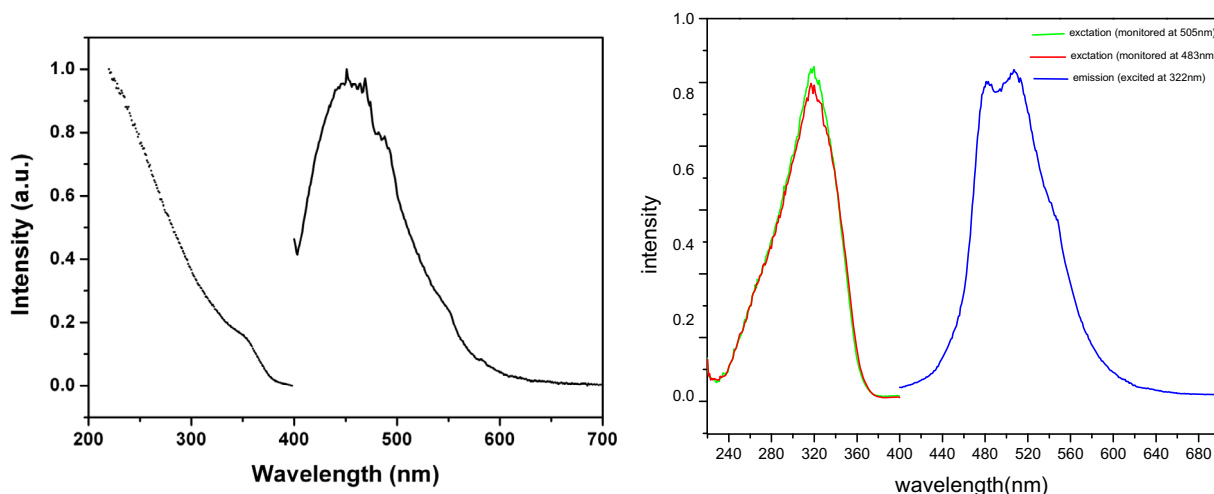


Fig. 3. Room temperature excitation (left) and emission (right) spectra for compound **1** (left) and compound **2** (right). Intensities are given in arbitrary units.

Given the fact that Ag is a softer cation than Cu one could prefer the structure with  $\text{Ag}-\text{C}\equiv\text{N}-\text{Cu}$ , despite its slightly poorer refinement. The Cu(I) is further coordinated by an N-bound cyanide (bridging to gold), and 2 phosphane ligands, in a tetrahedral chromophore. The Au(I) is linearly coordinated by 2 cyanide- $\kappa\text{C}$  ligands, one of them bridging between gold and copper.

The structure of the chain-type Au–Cu compound **2** is of a known type for the combinations Ag–Au and Ag–Cu. This missing link with the tetrahedral coordination for Cu(I) and the linear  $[\text{Au}(\text{CN})_2]$  units appears as normal regarding bond lengths and angles. The structure is projected in Fig. 1right, together with the used atomic numbering.

It is worth mentioning here the effect of the Cu(I) ions on the crystallographic symmetry of the one dimensional bimetallic series  $[\text{MM}'(\text{CN})_2(\text{PPh}_3)_2]_n$ ; where M and M' are coinage metals [10,11]. Compared to Ag–Au and Ag–Ag crystallizing in a centrosymmetrical monoclinic symmetry, the Cu–Au and Cu–Ag analogs were found to be crystallizing in the same chiral orthorhombic system, despite the lattice water molecules. In **2**, the molecular chain propagation angle is  $96.42(1)^\circ$ , compared to  $96.16(2)^\circ$  in the analog Cu–Ag [8], Fig. 2 depicts the asymmetrical unit of the molecular chain in **2**.

The packing of both compounds is uneventful, with no short contacts and no stacking between the aromatic rings of the phosphane ligand. The water molecules appear to fill up gaps in the lattice, and are arranged as dimers with H bond links ( $\text{O}\cdots\text{O} = 2.82\text{--}2.87 \text{ \AA}$ )

### 3.2. Luminescence studies

The room temperature excitation and emission spectra for compounds **1** and **2** are depicted in Fig. 3left and right. The excitation spectrum features a broad band in the region from 220 to 375 nm, showing a steady decline in intensity on going from 220

to 375 nm. A small shoulder around 347 nm is the only other feature in the spectrum. It is to be noted that the spikes on the emission peak seem to appear when weakly luminescent compounds are measured, and are a characteristic of the used spectrometer.

It is clear anyway that the intensity of the emission is very weak for this sample. Dehydration of the sample *in vacuo* did not result in major changes of the luminescence spectrum (data not shown). For compound **2** the luminescence appears as somewhat stronger, and especially in the excitation band a different pattern is seen. Qualitative comparison with the earlier reported compounds of formula  $[\text{MM}'(\text{CN})_2(\text{PPh}_3)_2](\text{H}_2\text{O})_2$  (M, M' = Cu, Ag, Cu) shows weaker luminescence in the present case [10,11]. Detailed studies of luminescence spectra and reflectance spectra are left for future studies where the  $-\text{CN}$  groups are also varied to  $-\text{C}\equiv\text{CR}$ .

### 3.3. DFT calculations

The single-point geometry-optimized calculations revealed the HOMO characters of **1** are containing the  $d_{yz}^2$  orbitals on the  $\text{CuN}_2\text{P}_2$  chromophore with  $\sigma(\text{Cu}-\text{P})$  bonding,  $\pi$ -orbital contribution from phenyl rings as well as  $\pi(\text{C}\equiv\text{N})$ -bonding. The cyanide  $\pi^*$ -orbital mixes with the  $\text{M}^{+1} p_z^*$ -orbital to form the HOMO's{−1}, see Fig. 4 for frontier orbitals depiction. The LUMO's and LUMO's{−1} in **1** are localized mainly on the phenyl rings of the tri-phenylphosphane ligands. There is no contribution of the Au or the Ag in the frontier molecular orbitals of **1**. The HOMO/LUMO gaps of ca. 0.11 in **1**, compared to ca. 0.07 in **2** and ca. 0.06 a.u. in the Cu–Ag compound [10] were found in accordance with the observed luminescence emission intensities. The orbital distribution in **2** was almost identical to its related compounds [10,11] with HOMO/LUMOs mixing is strongly favoring the MMLCT ( $d-p^*$ ) transition.

## 4. Concluding remarks

The structure of (**1**) presented and discussed above is a unique heteronuclear molecular array with the presence of an atomic  $\text{N}\equiv\text{C}-\text{Au}-\text{C}\equiv\text{N}-\text{Cu}-\text{C}\equiv\text{N}-\text{Ag}$  sequence. Evidence is presented for cyanide bridging between Cu(I) and Ag(I) with C coordinating at Cu. The solid state luminescence intensity, present prominently in the related dinuclear cyanido-bridged Ag–Au, Ag–Cu, and Ag–Ag compounds, appears significantly reduced in the current trinuclear compound. The presence of the water molecules in the lattice is not responsible for this, as dehydration *in vacuo* did hardly change the luminescence spectrum and intensity. The structure of **2** is a missing member in a family of dinuclear-based

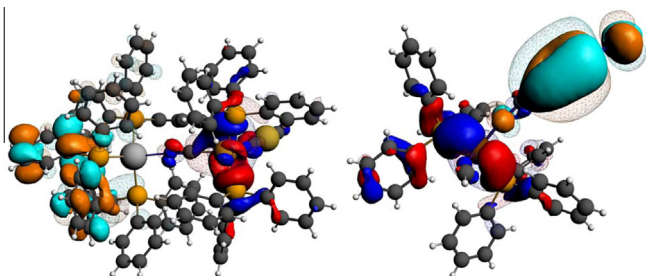


Fig. 4. Molecular orbitals surface diagrams of **1** (left) and **2** (right). HOMO/LUMO are in solid, HOMO{−1}/LUMO{+1} are in mesh.

coinage metal-chain compounds. Its luminescence is also quite weak, showing a different excitation band.

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### Appendix A. Supplementary material

CCDC 1006352–1006353 contains 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](http://www.ccdc.cam.ac.uk/data_request/cif). Supplementary data associated with this article can be found, in the online version, at <http://dx.doi.org/10.1016/j.ica.2014.08.017>.

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