

Supporting Information

Intensely, Oxygen Independent Phosphorescent Gold(I)-Silver(I) Complex: “Trapping” an Au₈Ag₁₀ Oligomer by Two Gold-Alkynyl-Diphosphine Molecules

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Experimental

General comments

Au(tht)Cl (tht = tetrahydrothiophene),^[1] [AuC₂Ph]_n,^[2] [AgC₂Ph]_n,^[3] [Au₂(PPh₂(C₆H₄)₂PPh₂)₂][PF₆]₂,^[4] 4,4''-PPh₂(C₆H₄)₃PPh₂^[5] were synthesized according to published procedures. Other reagents and solvents were used as received. Solution ¹H, ¹³C and ³¹P NMR spectra were recorded on Bruker Avance 400 and Bruker DPX 300 spectrometers. Mass spectra were determined on a Bruker APEX-Qe ESI FT-ICR instrument, in the ESI⁺ mode. Microanalyses were carried out in the analytical laboratory of the University of Joensuu.

[Au₂(PPh₂(C₆H₄)₃PPh₂)₂][PF₆]₂ (3)

Au(tht)Cl (80 mg, 0.250 mmol) was dissolved in CH₂Cl₂ (10 cm³) and solid PPh₂(C₆H₄)₃PPh₂ (150 mg, 0.251 mmol) was added. The resulting colourless solution was stirred for 15 min and then treated dropwise with the solution of AgPF₆ (64 mg, 0.253 mmol) in acetone (5 cm³). White flaky precipitate of AgCl gradually formed. The suspension was stirred in the absence of light for 30 min. Solids were separated by centrifugation and colourless solution was passed through Silica gel (0.5×2 cm, 70-230 mesh, Merck Kieselgel 60) diluted with heptane (5 cm³) and vacuum dried. White powder was washed with diethyl ether (3×5 cm³), dried, then suspended in acetone (4 cm³) and precipitated by addition of heptane (10 cm³). Resulting solid was washed again with ether (3×5 cm³) and vacuum dried to give analytically pure sample (198 mg, 84%).

ES MS (m/z): $[\text{Au}_2(\text{PPh}_2(\text{C}_6\text{H}_4)_3\text{PPh}_2)_2]^{2+}$ 795 (calcd 795). $^{31}\text{P}\{^1\text{H}\}$ NMR (CD_2Cl_2 ; δ): 43.98, 44.11 (rel. intensity 1/1.6), -144.8 (sept, PF_6). ^1H NMR (CD_2Cl_2 ; two sets of signals (rel. intensity 1/1.6) in the aromatic region corresponding to phenylene spacers and phenyl substituents at phosphorus (**NB 1**). Anal. Calc. for $\text{C}_{84}\text{H}_{64}\text{Au}_2\text{F}_{12}\text{P}_6$: C, 53.63; H, 3.43. Found: C, 53.44; H, 3.45.

NB 1. The spectrum displays two groups of resonances with relative intensities 1/1.6, similar to the spectroscopic pattern observed in the ^{31}P NMR spectrum. These groups of signals evidently correspond to the isomeric forms of the molecule, which very probably differ in conformation of the digold-diphosphine fragment adopting either “chair” or “boat” forms shown below. Relative intensities of these groups of resonances match well the corresponding number of protons. Rigidity of the phosphorus atom tetrahedral environment prevents fast isomerization of the molecule. The dimeric structure of **3** is supported by its ES mass spectrum reported by us earlier.^[5]

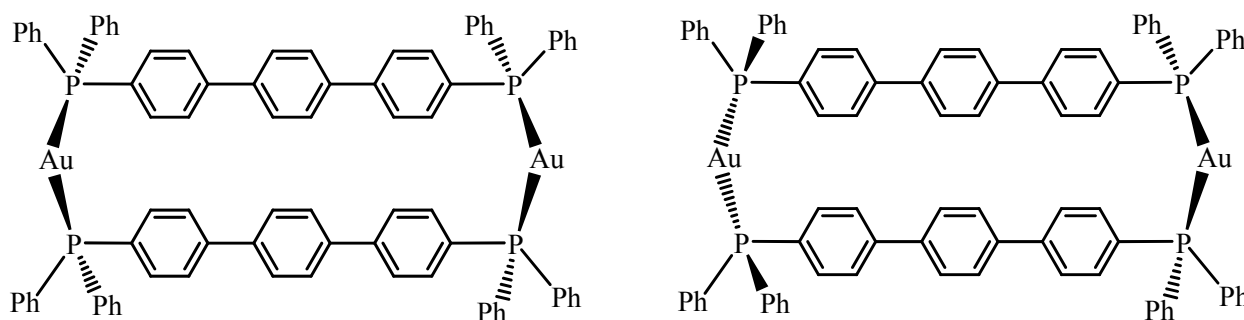


Chart S1. Proposed isomers of the dication **3**.

$[\{\text{Au}_8\text{Ag}_{10}(\text{C}_2\text{Ph})_{16}\}\{(\text{PhC}_2\text{Au})_2\text{PPh}_2(\text{C}_6\text{H}_4)_3\text{PPh}_2\}_2][\text{PF}_6]_2$ (1**)**

$[\text{AuC}_2\text{Ph}]_n$ (100 mg, 0.336 mmol) and $[\text{AgC}_2\text{Ph}]_n$ (70 mg, 0.335 mmol) were suspended in CH_2Cl_2 (10 cm^3) and solid **1** (94 mg, 0.05 mmol) was added. The yellow suspension was stirred in the absence of light for 2h. The resulting bright canary-yellow solution containing small amount of unidentified precipitate was filtered and evaporated. The residue was dissolved in acetone (3 cm^3) and some bright yellow microcrystalline solid formed. Slow addition of hexane (3 cm^3) increased amount of precipitate that was collected by centrifugation, washed with acetone-hexane (2:1 v/v) mixture (2×5 cm^3), diethyl ether (5 cm^3) and vacuum dried. Recrystallization by gas-phase diffusion of pentane into the CH_2Cl_2 /acetone solution of **1** at -15 °C gave yellow blocks (143 mg, 61%). Single crystals suitable for X-ray diffraction study were grown under the same conditions in the presence of small amount of methanol. **1** can be handled in solution at room temperature for short periods of time (3-4 h) as is unstable towards formation of unidentified insoluble material and **2**, especially in concentrated (> 30 mg/ml) solutions. ES

MS (*m/z*): $[\text{Au}_{12}\text{Ag}_{10}(\text{C}_2\text{Ph})_{20}(\text{PPh}_2(\text{C}_6\text{H}_4)_3\text{PPh}_2)_2]^{2+}$ 3331 (calcd 3331). $^{31}\text{P}\{^1\text{H}\}$ NMR (CD_2Cl_2 , δ): 38.5 (s, 4P), -143 (sept, 2P, PF_6). ^1H NMR (CD_2Cl_2 , δ): **diphosphine**: 8.23 (s, $-\text{C}_6\text{H}_4-$, 8H), 8.17 (dd, meta-H, $-\text{C}_6\text{H}_4-\text{P}$, 8H, $J(\text{H}-\text{H})$ 8.3 Hz, $J(\text{P}-\text{H})$ 2.2 Hz), 7.54 (dd, ortho-H, $-\text{C}_6\text{H}_4-\text{P}$, 8H, $J(\text{H}-\text{H})$ 8.5 Hz, $J(\text{P}-\text{H})$ 12.0 Hz), **Ph rings 1**: 7.65 (dd, ortho-H, 8H, $J(\text{P}-\text{H})$ 13 Hz, $J(\text{H}-\text{H})$ 7.5 Hz), 7.28 (t, para-H, 4H, $J(\text{H}-\text{H})$ 7.7 Hz), 6.93 (ddd, meta-H, 8H, $J(\text{P}-\text{H})$ 2.1 Hz, $J(\text{H}-\text{H})$ 7.7, 7.5 Hz); **Ph rings 2**: 7.69 (t, para-H, 4H, $J(\text{H}-\text{H})$ 7.7 Hz), 7.51 (dd, ortho-H, 8H, $J(\text{P}-\text{H})$ 13 Hz, $J(\text{H}-\text{H})$ ca. 7.7 Hz), 7.395 (ddd, meta-H, 8H, $J(\text{P}-\text{H})$ 2.0 Hz, $J(\text{H}-\text{H})$ ca. 7.7 Hz); **$\{\text{C}_2\text{Ph}\}$ moieties** (five groups of $\text{PhC}_2\text{AuC}_2\text{Ph}$ rods, for assignment details see text below (**NB 2**)) (**I**): 6.65 (dd, meta-H, 4H, $J(\text{H}-\text{H})$ 7.5, 7.8 Hz), 6.62 (t, para-H, 4H, $J(\text{H}-\text{H})$ 7.8 Hz), 6.20 (d, ortho-H, 8H, $J(\text{H}-\text{H})$ 7.5 Hz); (**II**): 6.72 (t, para-H, 4H, $J(\text{H}-\text{H})$ 7.5 Hz), 6.65 (d, ortho-H, 8H, $J(\text{H}-\text{H})$ 7.5 Hz), 6.25 (dd, meta-H, 8H, $J(\text{H}-\text{H})$ 7.5, 7.5 Hz); (**III**): 6.84 (d, ortho-H, 8H, $J(\text{H}-\text{H})$ 7.6 Hz), 6.81 (t, para-H, 4H, $J(\text{H}-\text{H})$ 8.2 Hz), 6.33 (dd, meta-H, 8H, $J(\text{H}-\text{H})$ ca. 7.6, 8.2 Hz); (**IV**): 7.00 (dd, meta-H, 8H, $J(\text{H}-\text{H})$ 7.4, 8.0 Hz), 6.77 (d, ortho-H, 8H, $J(\text{H}-\text{H})$ 7.4 Hz), ca. 6.61 (t, para-H, 4H, $J(\text{H}-\text{H})$ 8.0 Hz); (**V**): 7.67 (d, ortho-H, 8H, $J(\text{H}-\text{H})$ ca. 8 Hz), 7.17 (tt, para-H, 4H, $J(\text{H}-\text{H})$ 7.5, 1.8 Hz), 7.07 (dd, meta-H, 8H, $J(\text{H}-\text{H})$ 7.5, 8 Hz). Anal. Calc. for $\text{C}_{244}\text{H}_{164}\text{Ag}_{10}\text{Au}_{12}\text{F}_{12}\text{P}_6$: C, 42.16; H, 2.38. Found: C, 42.35; H, 2.32.

$[\{\text{Au}_6\text{Ag}_8(\text{C}_2\text{Ph})_{12}\}\{(\text{PhC}_2\text{Au})_2\text{PPh}_2(\text{C}_6\text{H}_4)_2\text{PPh}_2\}_2][\text{PF}_6]_2$ (4**)**

$[\text{AuC}_2\text{Ph}]_n$ (50 mg, 0.168 mmol) and $[\text{AgC}_2\text{Ph}]_n$ (35 mg, 0.167 mmol) were suspended in CH_2Cl_2 (10 cm^3) and solid $[\text{Au}_2(\text{PPh}_2(\text{C}_6\text{H}_4)_2\text{PPh}_2)_2][\text{PF}_6]_2$ (54 mg, 0.031 mmol) was added. The yellow suspension was stirred at this temperature for 15 minutes followed by stirring at room temperature in the absence of light for additional 1h. The resulting bright yellow-greenish solution containing small amount of unidentified precipitate was filtered and evaporated. The residue was dissolved in acetone (1 cm^3) and small amount of bright yellow microcrystalline solid formed. Slow addition of hexane (2 cm^3) increased amount of precipitate that was collected by centrifugation, washed with acetone-hexane (1:1 v/v) mixture (3 $\times$ 5 cm^3), diethyl ether (5 cm^3) and vacuum dried. Repetitious recrystallization by gas-phase diffusion of pentane into the CH_2Cl_2 /acetone solution of **4** at -15 $^\circ\text{C}$ gave yellow-greenish crystalline solid (28 mg, 24%). **4** was found to be unstable in solution and solid state at temperature above 0 $^\circ\text{C}$. ES MS (*m/z*): $[\text{Au}_{12}\text{Ag}_{10}(\text{C}_2\text{Ph})_{20}(\text{PPh}_2(\text{C}_6\text{H}_4)_3\text{PPh}_2)_2]^{2+}$ 2748 (calcd 2748). $^{31}\text{P}\{^1\text{H}\}$ NMR (CD_2Cl_2 , δ): 39.0 (s, 4P), -143 (sept, 2P, PF_6). ^1H NMR (CD_2Cl_2 , δ): **diphosphine**: 8.21 (dd, meta-H, ($-\text{C}_6\text{H}_4-\text{P}$), 8H, $J(\text{H}-\text{H})$ 8.6 Hz, $J(\text{P}-\text{H})$ 2.2 Hz), 7.61 (dd, ortho-H, ($-\text{C}_6\text{H}_4-\text{P}$), 8H, $J(\text{H}-\text{H})$ 8.6 Hz, $J(\text{P}-\text{H})$ 11.9 Hz), **Ph rings I**: 7.70 (dd, ortho-H, 8H, $J(\text{P}-\text{H})$ 13 Hz, $J(\text{H}-\text{H})$ 7.7 Hz), 7.29 (t, para-H, 4H, $J(\text{H}-\text{H})$ 7.6 Hz), 6.95 (ddd, meta-H, 8H, $J(\text{P}-\text{H})$ 2.2 Hz, $J(\text{H}-\text{H})$ 7.7, 7.6 Hz); **Ph rings II**: 7.54 (t, para-H, 4H, $J(\text{H}-\text{H})$ 7.7 Hz), 7.72 (dd, ortho-H, 8H, $J(\text{P}-\text{H})$ 12.5 Hz, $J(\text{H}-\text{H})$ ca. 7.7 Hz), 7.43 (ddd, meta-H, 8H, $J(\text{P}-\text{H})$ 2.2 Hz, $J(\text{H}-\text{H})$ ca. 7.6 Hz); **$\{\text{C}_2\text{Ph}\}$ moieties** (four groups of

PhC₂AuC₂Ph rods, for assignment see text below (**NB 2**)) (**I**): 6.65 (dd, meta-H, 8H, $J(\text{H-H})$ 7.5, 7.8 Hz), 7.02 (t, para-H, 4H, $J(\text{H-H})$ 7.5 Hz), 6.23 (d, ortho-H, 8H, $J(\text{H-H})$ 7.8 Hz); (**II**): 6.86 (t, para-H, 4H, $J(\text{H-H})$ 7.8 Hz), 6.76(d, ortho-H, 8H, $J(\text{H-H})$ 7.9 Hz), 6.34 (dd, meta-H, 8H, $J(\text{H-H})$ 7.8, 7.9Hz); (**III**): 6.87 (d, ortho-H, 8H, $J(\text{H-H})$ 8.0 Hz), 7.04 (t, para-H, 4H, $J(\text{H-H})$ 7.5 Hz), 6.59 (dd, meta-H, 8H, $J(\text{H-H})$ ca 7.5, 8.0 Hz); (**IV**): 7.12 (dd, meta-H, 8H, $J(\text{H-H})$ 7.5, 8.2 Hz), 7.68 (d, ortho-H, 8H, $J(\text{H-H})$ 8.2 Hz), 7.22 (t, para-H, 4H, $J(\text{H-H})$ 7.5 Hz). Anal. Calc. for C₂₀₀H₁₃₆Ag₈Au₁₀F₁₂P₆: C, 41.52; H, 2.37. Found: C, 41.72; H, 2.30.

NB 2. The idealized symmetry group of **1** is D₂ that contains three C₂ axes through the center of the molecule, two of which lies in the plane formed by the phosphorus atoms and the other is perpendicular to it. These symmetry operations make equivalent the phosphorus atoms of the diphosphine ligands, eight protons of the central phenylene spacers, eight ortho- and eight meta-protons of the P-C₆H₄ moieties but differentiate the phenyl substituents at phosphorus into two groups, four rings in each of them. In turn, twenty phenyl rings of the alkynyl fragments are divided into five groups, one of which consists of the terminal phenyl-alkynyl moieties bound to the phosphino-gold centers (Au1, Au12, Au6, Au7. Four other groups belong to the phenyl-alkynyl moieties from the central {Au(C₂Ph)₂}₈ core. Assignment of the proton spectra of **1** and **4** is based on the ¹H-¹H COSY experiments of these complexes (Figs. S2 and S4), where the signals are clearly related to each other inside the groups of phenyl (“ortho-meta-para” resonances) and phenylene protons. The C₂Ph moieties coordinated to the gold ions do not display additional couplings, in contrast to the protons of the P-Ph₂ and P-C₆H₄ fragments, which belong to the diphosphine ligands. The long range ³¹P-¹H couplings observed in the latter case allow for differentiation of the phenyl rings signals in these spectra. For example, in the spectrum of **1** the unique singlet (8H) at 8.23 ppm is evidently generated by the protons of the central phenylene fragment. Two dd resonances (8H) at 7.54 and 8.17 ppm are related to each other in COSY spectrum ($J(\text{H-H})$ 8.5 Hz) and display characteristic three- and four-bond coupling to phosphorus (12.0 and 2.2 Hz). This allows for their assignment to ortho- and meta-protons of the phenylene spacers directly bound to the phosphorus atoms. In a similar manner two sets of resonances (“ortho-meta-para” protons related to each other in the COSY spectrum) should be assigned to phenyl rings of the diphosphine ligands because of typical ^{3,4}J(P-H) coupling mentioned above. In the case of **1** the exact values of coupling constants were rectified after application of resolution enhancement procedure. In a similar manner the resonances corresponding to the diphosphine ligands were assigned in the spectrum of **4**. It is worth noting that the disposition of the signals as well as the values of coupling constants in **4** are essentially similar to those found in the proton spectrum of **1**. In accord with the symmetry of **1** and **4** the “-

C₂Ph'' moieties form five and four groups, respectively, each of which contains four phenyl rings. Relative intensities of the corresponding signals (8 para-, 8 meta- and 4 ortho-protons in all these groups of signals) are in excellent agreement with the structural patterns revealed for these complexes in solid state, but it is impossible to assign the signals of alkynyl-phenyl rings in detail because of their structural similarity.

X-ray Structure Determinations

The crystal of **1** was immersed in cryo-oil, mounted in a Nylon loop, and measured at a temperature of 90 K. The X-ray diffraction data were collected by means of a Nonius KappaCCD diffractometer using Mo K α radiation ($\lambda = 0.71073$ Å). The *Denzo-Scalepack*^[6] program package was used for cell refinements and data reductions. The structure was solved by direct methods using the *SHELXS-97*^[7] with the *WinGX*^[8] graphical user interface. A semi-empirical absorption correction (*SADABS*)^[9] was applied to all data. Structural refinements were carried out using *SHELXL-97*.^[10]

Crystal contained methanol, acetone and dichloromethane as solvent of crystallization. Some of the solvent molecules were disordered and partially lost, which lowered the quality of data. One of the CH₂Cl₂ (C257, Cl5, Cl6) and MeOH (C256, O6) molecules were disordered over the same site and were refined with equal occupancies of 0.5. Partially lost MeOH molecule C255-O4 was refined with occupancy of 0.5. The heavy atoms of these disordered solvent molecules were restrained so that their U_{ij} components approximate to isotropic behavior. Furthermore, the anisotropic displacement parameters of oxygen O4 and carbon C255 were set to be equal. Similarly, O3 and C254 in another MeOH molecule were set to be equal.

One of the PF₆ counter anions was also disordered over two (P6 and P7) sites with equal occupancies. The P-F and F-F distances were restrained and were not allowed to refine freely. All hydrogen atoms were positioned geometrically and were also constrained to ride on their parent atoms, with C-H = 0.95-0.99 Å, U_{iso} = 1.2-1.5 U_{eq}(parent atom), O-H = 0.98 Å and U_{iso} = 1.5 U_{eq}(parent atom).

The highest peak is located 0.11 Å from atom C255 and the deepest hole is located 0.36 Å from atom O5.

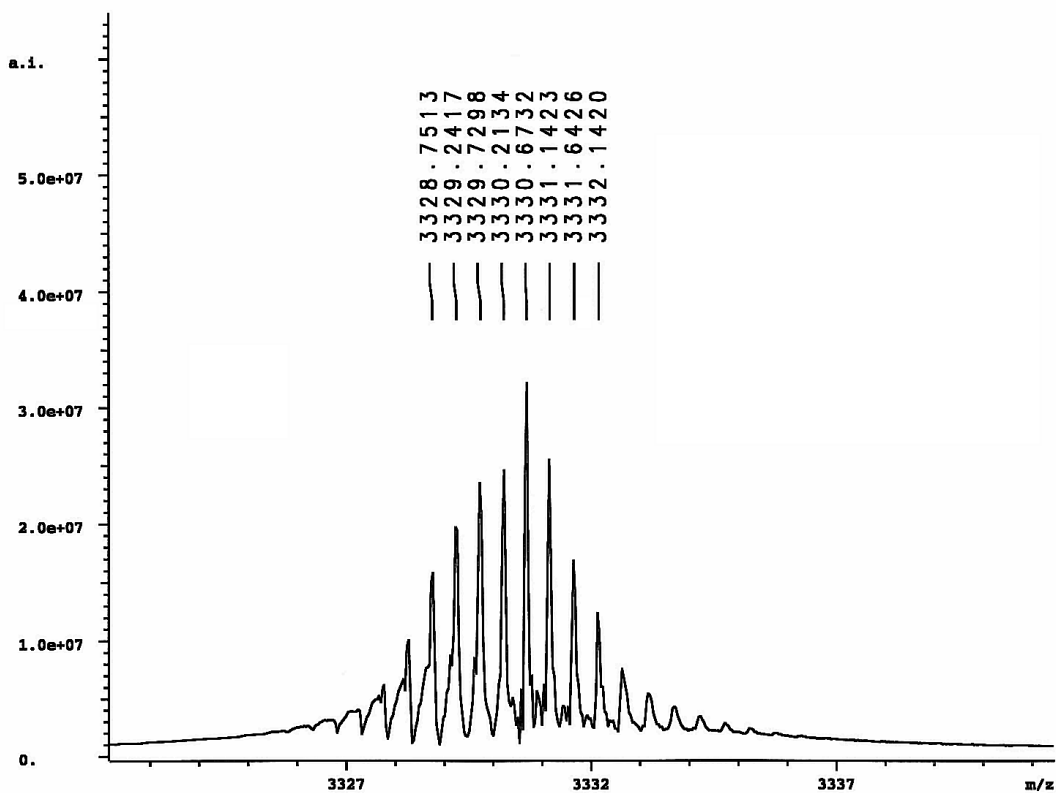


Figure S1. ESI-MS spectrum of the $[\text{Au}_{12}\text{Ag}_{10}(\text{C}_2\text{Ph})_{20}(\text{PPh}_2(\text{C}_6\text{H}_4)_3\text{PPh}_2)_2]^{2+}$ dication (**1**).

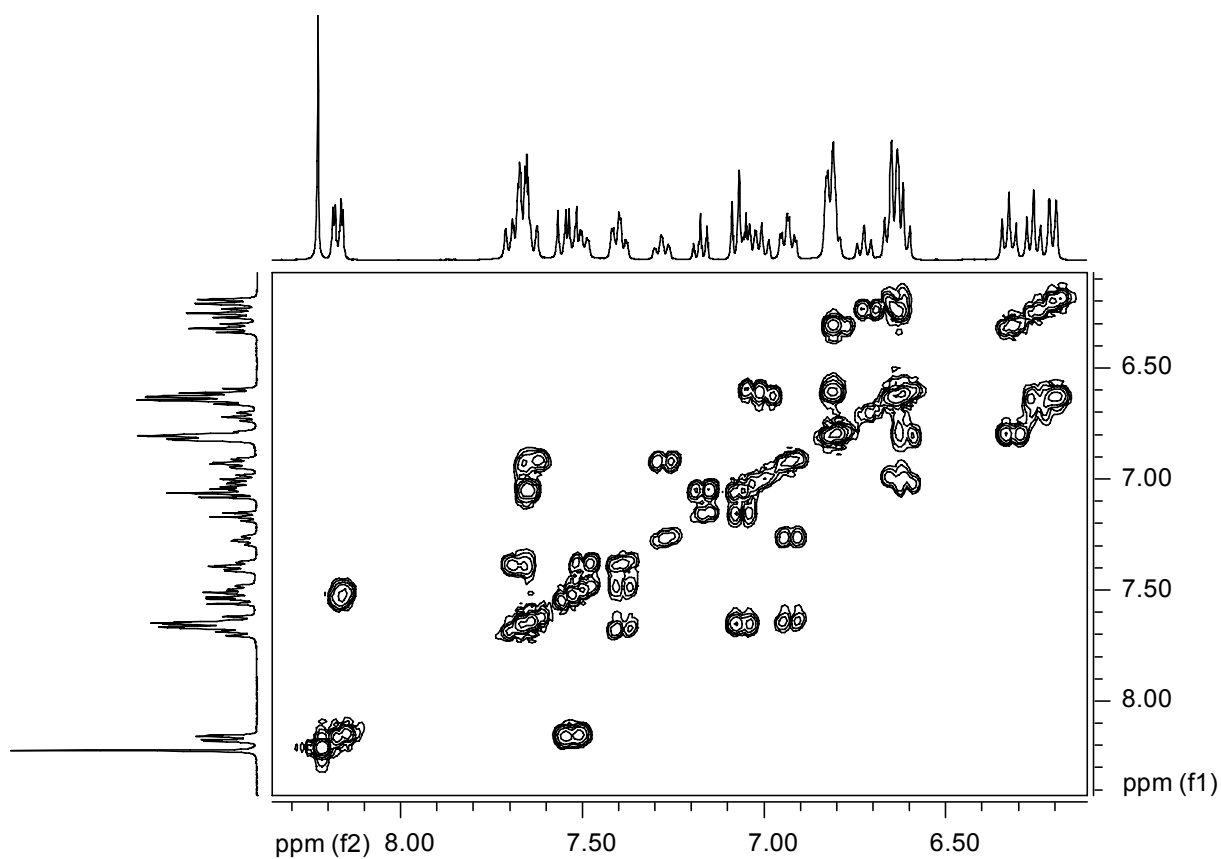


Figure S2. ^1H - ^1H COSY spectrum of **1** (400 MHz, CD_2Cl_2 , 298 K).

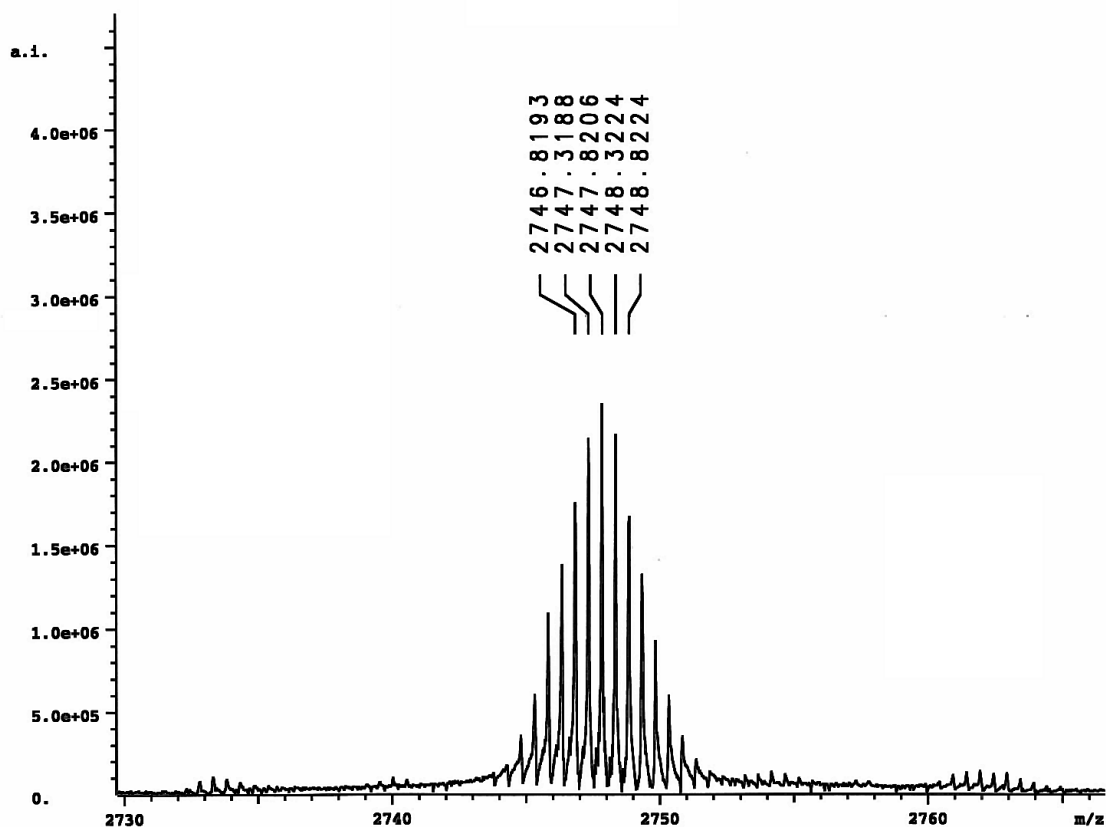


Figure S3. ESI-MS spectrum of the $[\text{Au}_{10}\text{Ag}_8(\text{C}_2\text{Ph})_{16}(\text{PPh}_2(\text{C}_6\text{H}_4)_2\text{PPh}_2)_2]^{2+}$ dication (**4**).

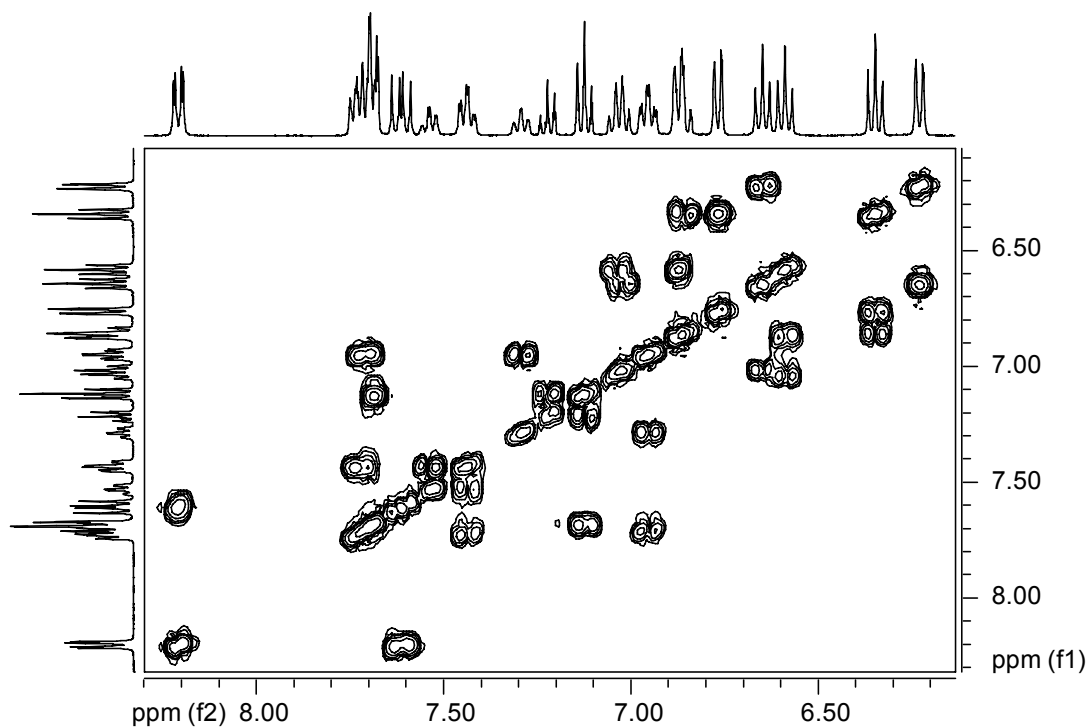


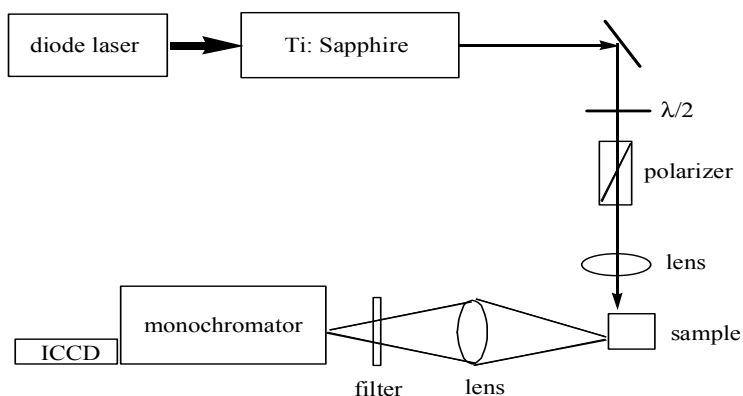
Figure S4. ^1H - ^1H COSY spectrum of **4** (400 MHz, CD_2Cl_2 , 298 K).

Photophysical details

Steady-state absorption and emission spectra were recorded on a Hitachi (U-3310) spectrophotometer and an Edinburgh (FS920) fluorometer, respectively. Both wavelength-

dependent excitation and emission response of the fluorometer have been calibrated. To determine the photoluminescence quantum yield in solution, samples were degassed by three freeze-pump-thaw cycles. Coumarin 480 in methanol, with quantum yield of ~ 0.87 , served as the standard for measuring the quantum yield. The error of the quantum yield measurement was in the range of $< 2\%$ (two replica). Lifetime studies were performed with an Edinburgh FL 900 photon-counting system using a hydrogen-filled lamp as the excitation source. Data were analyzed using the nonlinear least squares procedure in combination with an iterative convolution method. The emission decays were analyzed by the sum of exponential functions, which allows partial removal of the instrument time broadening and consequently renders a temporal resolution of ~ 300 ps.

The measurement of two-photon induced emission excitation spectra and two-photon absorption cross-section was described in our previous report.^[11] In brief, the setup for the two photon excitation spectra and two photon excitation cross-section measurement is depicted in Scheme S1 and is similar to that used by Xu et al.^[12] A femtosecond mode-locked Ti:sapphire laser (Spectra Physics) generates 120 fs pulses at repetition rate of 80 MHz with an average power of 1.4 W. The laser beam was focused on a sample cell (1 cm) by a lens with the focal length 6 cm. To minimize the effects of re-absorption, the excitation beam was focused as close as possible to the wall of the quartz cell, which faced the slit of the imaging spectrograph. Two-photon emission fluorescence was detected in a direction perpendicular to the pump beam. The emission was focused by a lens with the focal length 8 cm into an optical spectrum analyzer. Our optical spectrum analyzer consists of a CCD with detector control (PI-MAX camera, Princeton Instruments Inc.) in conjunction with a monochromator (SP2300i, Acton Research Corporation) was used as a recorder. The concentration of compound **1** for the comparative two-photon emission measurements is consistent with the quantum yield determination to be 5.03×10^{-5} M in dichloromethane. The error of two-photon absorption measurement was in the range of $< 10\%$ (ten replica).



Scheme S1. Experimental setup for two-photon absorption cross-section measurement.

The open aperture Z-scan experiments were conducted by using the essentially the same experimental setup and procedure as previously described.^[13] In this study, a mode-locked Ti:sapphire laser (Tsunami, Spectra Physics) produced single Gaussian pulse which was coupled to a regenerative amplifier that generated a ~200 fs, 1 mJ pulse (780-820 nm, 1 kHz, Spitfire Pro, Spectra Physics). The pulse energies, after suitable attenuation, were reduced to 0.95 μ J. After passing through an $f = 30$ cm lens, the laser beam was focused and passed through a 2.00 mm cell filled with the sample solution, compound **1** in dichloromethane (4.13×10^{-3} M), and the beam radius at the focal position is 3.82×10^{-3} cm. When the sample cell changed its position along the beam direction (z-axis), the transmitted laser beam from the sample cell was detected by a photodiode (PD-10, Ophir). Accordingly, in theory, the two-photon absorption induced decrease of transmittance, $T(z)$, can be expressed as equations (1) and (2) and two-photon absorption coefficient (β) can be obtained from experimental data by fitting Z-scan curves to relationship (1) and (2).

$$T(z) = \sum_{n=0}^{\infty} \frac{(-q)^n}{(n+1)^{3/2}} \quad (1)$$

$$q = \frac{\beta I_0 L}{1 + \frac{z^2}{z_0^2}} \quad (2)$$

where n is an integer number from 0 to ∞ and has been truncated at $n = 1000$, L is the sample length, I_0 is the input intensity, z is the sample position with respect to the focal plane, and z_0 is the diffraction length of the incident beam (Rayleigh range).^[13] After obtaining the two-photon absorption coefficient (β), two-photon absorption cross-section (σ_2) can be deduced by using equation (3)

$$\beta = \frac{\sigma_2 N_A d \times 10^{-3}}{h \nu} \quad (3)$$

where N_A is the Avogadro constant, d is the concentration, h is the Planck constant, and ν is the frequency of the incident beam. As for the open aperture Z-scan experiments, we have measured 2PA cross-sections of a well known 2PA dye, coumarin 480, to ensure that the 2PA cross-section values were not over-estimated. The resulting value of 165.1 GM is consistent with the reported data of 168.2 GM, which is obtained via correcting the two-photon induced fluorescence value of 146.2 GM^[14] by the emission quantum yield of 0.87 in methanol, within 5% uncertainty (five replicas, shown in Figure S5).

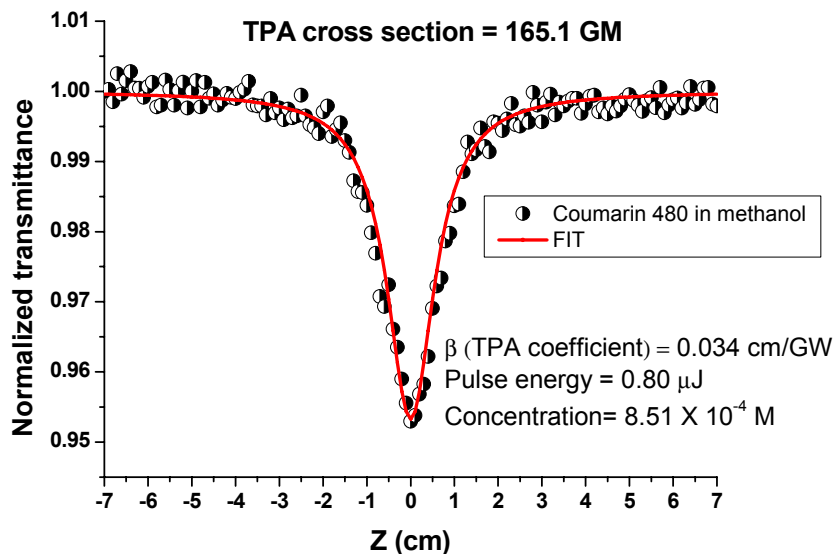


Figure S5. Z-scan experimental data of Coumarin 480 in methanol (circles) in a 2-mm cell. Solid lines are the best fit for the data points by equations (1) and (2).

Photophysical results

Figure S6 depicts the steady state absorption and luminescence spectra of the titled compounds in dichloromethane and the relevant photophysical data are presented in Table 1. The higher-energy absorption bands at ~ 260 nm for compound **1** and **2** are ascribed to the intraligand $\pi \rightarrow \pi^*$ ($C \equiv CAr$) transitions of the alkynyls moiety.^[4, 5, 15, 16] As for the lower lying transitions, the bands centered at ~ 320 nm can reasonably be assigned to an electronic transition from the σ (Au-P) orbital to an empty π^* ($C \equiv CPh$ or phosphine) antibonding orbital located at the bridging phenyl group.^[15, 16] The 390 nm absorption band for the compound **2** is mainly due to the Ag- π -alkynyl fragment metal- and/or cluster-centered transitions bands.^[4, 15] These assignments are consistent with previous reports on the relevant Au(I)–Cu(I) complexes, while the shift of the absorption bands ranging from 300 nm to 400 nm is attributed to the different electronic property between Ag(I) and Cu(I). The lowest transition for compound **1** featured with a shoulder and extended to ~ 450 nm is likely to exhibit, in part, metal (d of Au and Ag) $\rightarrow$ alkynes (π^*) charge transfer character. However, due to the complicated nature for both HOMO and LUMO (see computational section), the transition cannot be described as a pure MLCT character. For compound **2**, the lowest transition at 450–500 nm is plausibly ascribed to the metal-perturbed interligand ($\pi \rightarrow \pi^*$), mixed with some metal cluster-centered sp transitions. Further support of these viewpoints will be elaborated in the computational section.

In our study, the value of the two-photon cross section was measured by two-photon induced fluorescence technique and the open-aperture Z-scan method. As depicted in the insert of Figure S7, the up-converted fluorescence for compound **1** is linearly dependent on the square of the incident power (slope=2.03), firmly supporting the occurrence of two-photon absorption. Figure S7 and Figure S8 show the results of the wavelength dependence for the two-photon excitation spectra and the Z-scan curve, respectively.

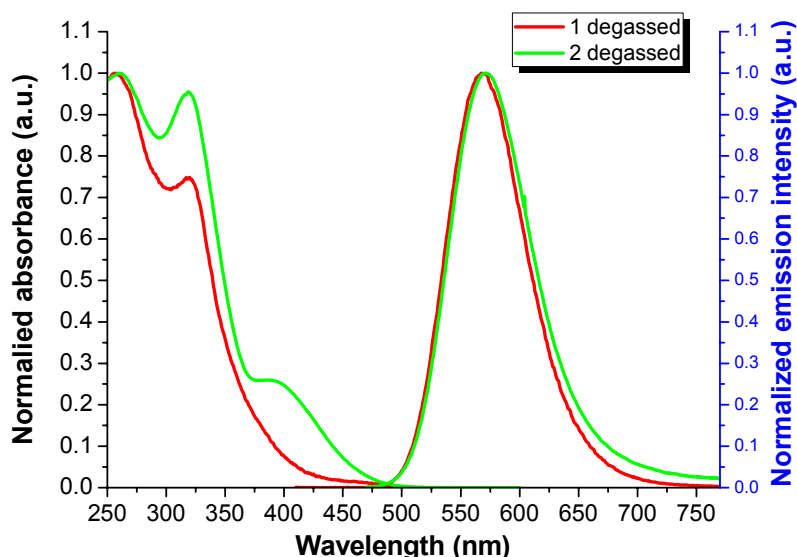


Figure S6. UV/VIS absorption and normalized emission spectra of **1** and **2** in deaerated CH_2Cl_2 at room temperature, $\lambda_{\text{excit}} = 450 \text{ nm}$.

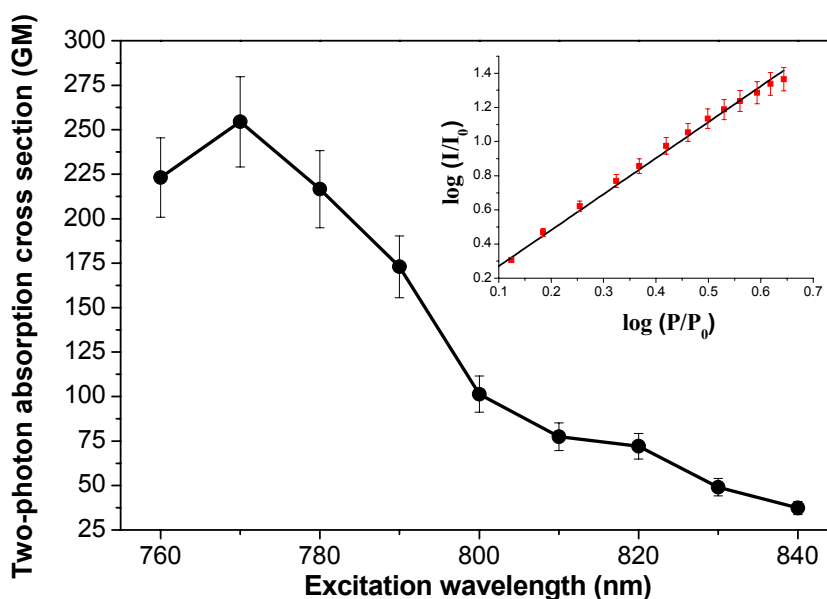


Figure S7. The two-photon excitation spectra (760–840 nm) of compound **1** (●) in dichloromethane. Insert: The log-log plot of normalized excitation power (P/P_0) versus normalized two-photon emission intensity (I/I_0).

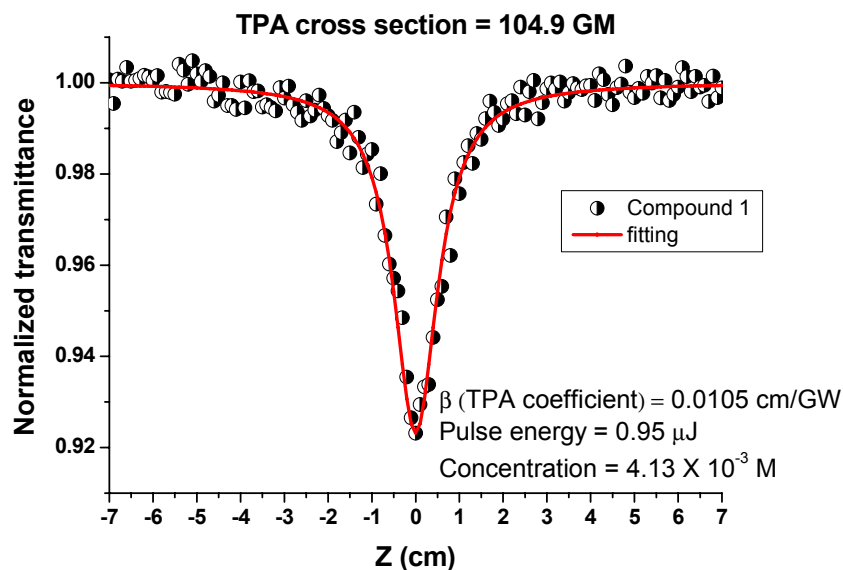


Figure S8. Z-scan experimental data of compound **1** in dichloromethane (circles) in a 2-mm cell. Solid lines are the best fit for the data points by equations (1) and (2).

Computational details

The studied Au(I)–Ag(I) complexes were optimized without any symmetry constraints using the BP86 density functional method.^[17] Because the weak Au–Ag interactions between the central and external fragments of the linear complexes **1** and **4** are not properly described by DFT methods, all metal atoms were fixed at the positions determined by X-ray diffraction analysis in the structural optimizations of these two complexes. The silver and gold atoms were both described with a triple-valence-zeta quality basis set with polarization functions (def2-TZVP),^[18] employing 28-electron and 60-electron relativistic effective core potentials for Ag and Au, respectively.^[19] A split-valence basis set with polarization functions on non-hydrogen atoms was used for all the other atoms (def2-SV(P)).^[20] The multipole-accelerated resolution-of-the-identity technique was used to speed up the calculations.^[21] All the electronic structure calculations were carried out with TURBOMOLE version 5.10.^[22]

Computational results

The electronic properties of the supramolecular Au(I)–Ag(I) complexes were investigated with quantum chemical methods to obtain insights into their photophysical behavior and structural characteristics. The frontier orbital characteristics of the linear complex **1** and triangular complex **2** are illustrated in Figure S9. In the case of **1**, the main contributions to HOMO come from sd(Au) and d(Ag) orbitals within the heterometallic core, while the LUMO is mainly composed of sp(Ag) and alkynyl ligand $\pi^*(C\equiv CPh)$ orbitals. Hence, the frontier orbitals of **1** are located within the heterometallic core of the complex, which is protected by bulky

ligands (Figure S10). The frontier orbital characteristics of **1** suggest the observed phosphorescence to originate from metal-centered $[sp(\text{Ag}) \rightarrow sd(\text{Au}), d(\text{Ag})]$ triplet emission. The protected nature of the central chromophore of **1** is likely to be a significant contributing factor to the observed high efficiency of the phosphorescence, as the bulky ligands hinder O_2 quenching of the excited triplet state. In the case of the triangular complex **2**, the HOMOs are located in the corners of the heterometallic core and are composed primarily of alkynyl ligand $\pi(\text{C}\equiv\text{CPh})$ orbitals, with small contributions from metal d orbitals. The LUMO of **2** is composed of central fragment $sp(\text{Ag})$, $sp(\text{Au})$, and ligand $\pi^*(\text{C}\equiv\text{CPh})$ orbitals. In comparison to **1**, the frontier orbitals of **2** are less protected.

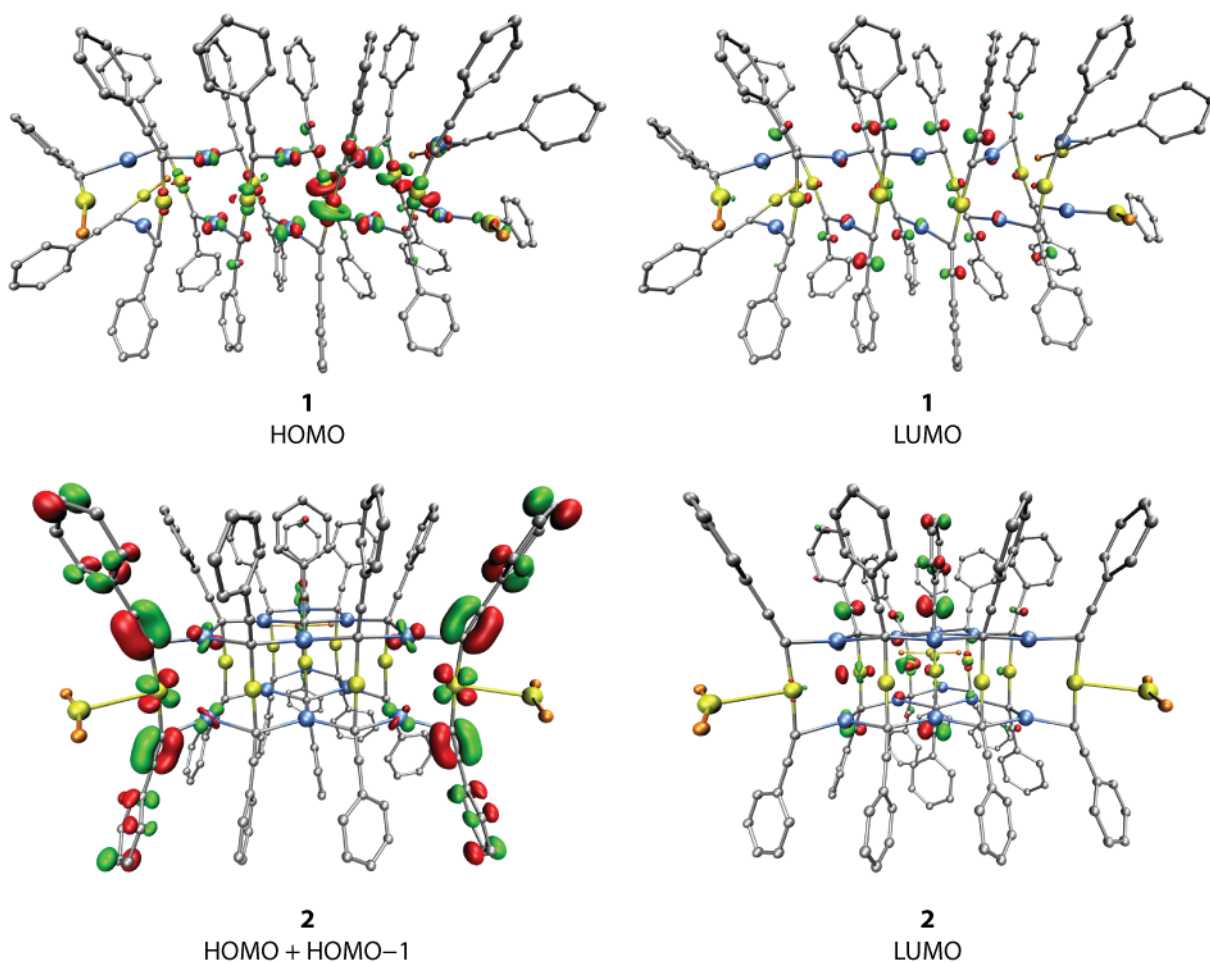


Figure S9. Frontier molecular orbital isodensity plots for the Au(I)-Ag(I) complexes **1** and **2** (isodensity value 0.04). Hydrogen atoms and diphosphine-based phenyl rings omitted for clarity.

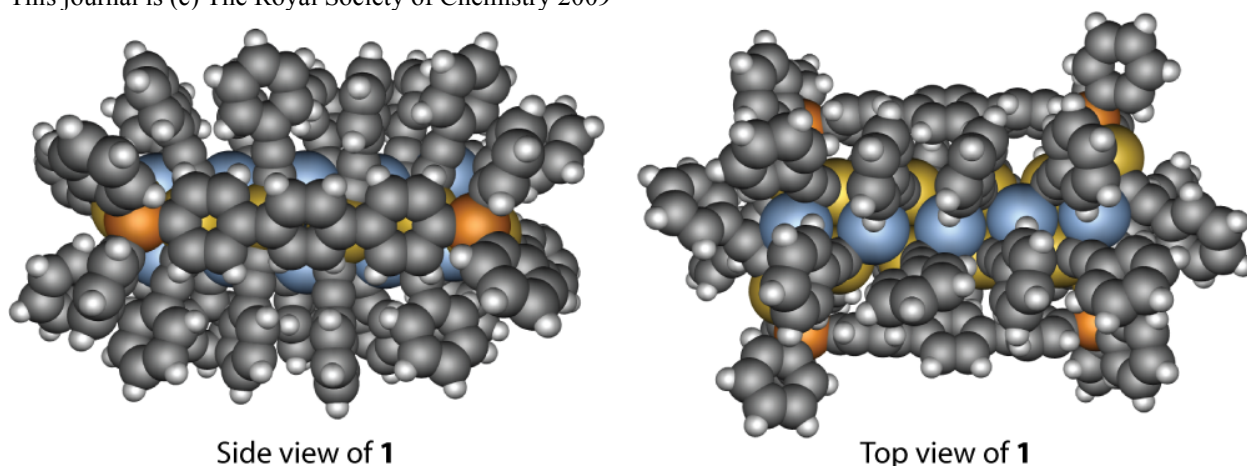


Figure S10. Two space-filling models of the Au(I)-Ag(I) complex **1** at the optimized geometry. The heterometallic core is protected by bulky ligands.

The structural and electronic characteristics of the smaller linear Au(I)-Ag(I) complex **4** are very similar to **1**, the frontier orbitals being located within the heterometallic core protected by bulky ligands. However, the experimental evidence suggests the complex **4** to be unstable towards decomposition into unidentified insoluble material and a triangular $[\{\text{Au}_6\text{Ag}_6(\text{C}_2\text{Ph})_{12}\}\text{Au}_3(\text{PPh}_2(\text{C}_6\text{H}_4)_2\text{PPh}_2)_3]^{3+}$ complex structurally related to triangular complex **2**. Although detailed quantum chemical studies on the relative thermodynamic stabilities of the supramolecular Au(I)-Ag(I) complexes are not currently feasible due to their large size, the calculated HOMO-LUMO gaps of the species provide some insight into their relative kinetic stabilities. The linear complex **4** possesses a HOMO-LUMO gap of 1.78 eV, which is smaller than the gap of 1.95 eV calculated for the triangular complex $[\{\text{Au}_6\text{Ag}_6(\text{C}_2\text{Ph})_{12}\}\text{Au}_3(\text{PPh}_2(\text{C}_6\text{H}_4)_2\text{PPh}_2)_3]^{3+}$. For comparison, the larger linear complex **1** also possesses a gap of 1.78 eV, while the gap of the larger triangular complex **2** is only 1.37 eV. Hence, from the point of view of kinetic stability, the experimentally observed stability trends of **1** and **4** with respect to their triangular counterparts are understandable. Thermodynamic studies are expected to shed further light on the self-assembly processes of the supramolecular Au(I)-Ag(I) complexes.

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Additional computational data

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

$[\{\text{Au}_8\text{Ag}_{10}(\text{C}_2\text{Ph})_{16}\}\{(\text{PhC}_2\text{Au})_2\text{PPh}_2(\text{C}_6\text{H}_4)_3\text{PPh}_2\}_2]^{2+}$ (1)

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21.12752204337807	32.05014215846771	5.88158526750952	au
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33.74012471364500	28.03730758850356	0.64420781959067	au
37.24141027579692	26.64552390082820	-3.90209658748246	au
42.54001486761350	30.04003990649179	6.78903201132717	au
36.86970104108443	31.53783725999007	5.33658809775314	au
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46.37321521432319	32.88310454476623	18.73682809828946	h
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27.45039099412205	35.00126106439359	15.91030404338456	h
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31.63027796123034	33.57429700552967	14.49105801077111	h
25.80230225188314	39.84839487409047	16.62752047013658	c
24.62306203180868	41.92387413665423	15.41625550702817	c
25.62633485901781	42.92687035650060	13.89212379496015	h
22.16386298361344	42.66769195476499	16.05671725043387	c
21.29232510618725	44.26438797263086	15.04228156206586	h
20.80758545201659	41.39327960166521	17.97359675784434	c
21.96336491126802	39.32276543511954	19.19223624500588	c
20.95300674369566	38.29737574284857	20.69580927684182	h
24.41353970850284	38.56331302957291	18.51655823146465	c
25.28890702387487	36.96227757563845	19.52022631305345	h
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15.12825490262707	38.95947813264031	22.01039040202128	c
14.21739682146142	38.23522078190020	20.28125178953297	h
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13.23445601510372	36.26682696741823	24.45962942193621	h
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15.27857238812406	37.92816364568595	28.41582185971061	h
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21.81461177011903	45.84625534736840	20.31106079411618	h
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21.91148619794498	50.44556293571461	21.33506767154465	h
17.92049541798096	51.03046595933920	20.50693094918044	c
17.97109081705735	53.06370353783625	20.95865796541065	h
15.65191929622217	49.89852857164276	19.71276204094110	c
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13.12769268129262	31.87745493080378	20.23256978602591	c
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[{Au₁₀Ag₁₂(C₂Ph)₂₀}Au₃(PPh₂(C₆H₄)₃PPh₂)₃]⁵⁺ (2)

-0.79583619108621	-16.77762497299080	8.69233815157566	au
-0.16453841117458	15.73793446543545	10.40820807049853	au
1.05530698559708	0.57903317023802	-18.60418975839398	au
1.01106968620564	0.31127155439319	-13.17253764017862	au
0.69002334378737	-3.35471260314048	-6.17492882985509	au
0.67055651558642	3.47376659684432	-6.37502520856717	au
0.30324328836904	-7.39432933856261	0.72144412070188	au
0.32277249093565	0.05476791746924	0.36716376346900	au
0.59177861701095	7.50261899808204	0.20974289396185	au
-0.04679450825243	-3.85460246794014	6.73745886364367	au
-0.40250650402834	-11.76137650410939	6.56639435047260	au
-0.26142494945115	4.28503718762213	6.32564782342585	au
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3.74164971868121	8.10646929809167	4.95425479288262	ag
-3.43271331304524	8.58133952692121	4.10406242406870	ag
3.38515029716418	0.14078937280071	5.32234578689576	ag
-3.45720336642924	0.12511807532800	4.92413832673370	ag
3.43858895020789	-8.09812882840686	5.45126508962883	ag
-3.67761505338846	-7.80655697621842	4.86285212398700	ag
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3.93009194974537	-4.12302453066469	-1.84990455421571	ag
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-2.98591238815999	4.02580538621196	-2.15169653226787	ag
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-2.73756623417734	-0.08046585546433	-9.33243081170726	ag
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$$[\{\text{Au}_6\text{Ag}_8(\text{C}_2\text{Ph})_{12}\}\{(\text{PhC}_2\text{Au})_2\text{PPh}_2(\text{C}_6\text{H}_4)_2\text{PPh}_2\}_2]^{2+} \text{ (4)}$$

19.68471573578794	31.07995648942152	7.52867102742511	au
13.53819079005428	33.59234808855849	9.15496979404209	au
26.02172512920345	28.70702671805161	5.54219035874306	au
22.46299187648082	34.41248946052197	12.59257156763372	au
16.19798106894505	36.82680424582275	14.26611350083845	au
28.60328072422489	32.02406393051128	10.66826290618116	au
13.19482745529445	41.03333579879672	16.75412762601614	au
33.94100265797614	30.23959504215848	12.40246506528458	au
29.73144754234055	27.32488063636016	1.17786662936598	au
7.84085387223702	32.16522651227550	9.44088543823120	au
18.50401451642670	36.66013035410114	9.24133030256451	ag
17.65382640410357	31.31239286903329	12.91968325308735	ag
23.68016482044781	28.96062803629470	10.88463660915309	ag
24.42830760653155	34.17154931092440	7.18322899333543	ag
12.24580672487957	38.92156625232394	10.79147308464203	ag
11.36141464635674	33.58875760789782	14.24872801553416	ag
30.39058420257296	32.19735186555517	5.16991420603153	ag
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26.70371355054551	24.98292950940294	-1.06225625777268	p
34.26748560410357	33.35607909442429	15.55459680638044	p
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16.08665660439281	41.81523145406337	20.02610370631408	p
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27.08090735458390	34.70198180482073	18.29606505058542	c
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25.91656965301040	43.31497665382983	11.32785077456378	h
27.505567720340568	47.237777719413891	9.22199925351635	h
28.08273789379636	47.34545936190533	4.50691402070754	h
27.06041468551650	43.49247206135230	1.92186439845460	h
25.45747701549670	39.57989117492482	4.01593701259756	h
34.01976577100504	44.89088462889371	7.91902437898000	h
37.08362153672309	44.31372300339887	4.33492055571030	h
31.37194594530997	41.26348035469002	9.38881721859142	h
34.86056018726506	36.43066719806006	3.73040543962163	h
37.48245455595527	40.06368210983637	2.24501791847340	h
38.83209074873762	38.90682170064560	9.47280707161309	h
35.84103293281400	35.46256265618887	10.68252989568874	h
37.64014097974058	36.70926535170464	18.62258766858108	h
40.63129104560169	40.13879483456548	17.39932755116022	h
41.24770877927202	41.25458627409311	12.82796920165977	h
41.75598726727933	34.55496583209387	-0.17162854387777	h
43.98598048482712	34.28830920319226	4.00870551478608	h
41.81558606001039	32.27905060981526	7.72235427213623	h
37.43416468398063	30.53795063269247	7.26098459232461	h
37.36541673036010	32.83812841958404	-0.63220277296402	h
37.05778706369733	21.21611917373692	10.96116117708443	h
35.92292149124756	25.11187846045209	3.81222091996141	h
38.15008126472516	21.58936576692379	1.54781829139457	h
39.83822505636319	17.88517365545242	3.98802267453565	h
39.28736448919097	17.70339611352987	8.69643385805149	h
22.86612585331147	25.13792221478169	-5.27416538011529	h
30.93367319468464	26.44390717582373	-4.27722261819290	h
31.37815780661911	27.17544837888049	-8.92679748802921	h
27.57573903305655	26.89504926451832	-11.75602822441149	h
23.32625270144568	25.87862978282419	-9.90665987996975	h
24.13846258566263	20.59278491242939	-3.53535354059811	h
25.06480885430341	15.97618323585472	-3.50540971501183	h
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30.39603440210972	21.87609667911397	1.66259506544075	h

33.50081005115475	34.53954822697840	21.06313088779265	h
35.38830728898519	32.69154821655593	24.96959502876731	h
38.43473757938987	29.06584918133325	24.66784766545299	h
39.58209121944871	27.29148067525972	20.41590713518407	h
37.68724587240959	29.12176828256673	16.49081439778289	h
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29.90123268469163	22.96860825036535	21.46543224336933	h
27.23355826769059	19.17388549836189	20.45281365595662	h
24.54771689808016	19.15803404024488	16.52988578152110	h
24.51977334909508	22.90305179272204	13.65486654300894	h
26.00602924031324	18.90367652166999	5.13024421735612	h
30.22894898346794	21.56248543394347	11.66067992244148	h
31.25114613411314	17.09698732514295	12.81290956536085	h
29.64149390383119	13.51157443887595	10.14413227959071	h
22.21631623169458	21.48256244456793	7.70157709753015	h
21.75436923604876	16.90745522527703	8.84271025588804	h
18.81258317824139	15.61218457544172	12.33770071784105	h
16.35965482445505	18.92615253832730	14.70426895292024	h
16.82917298204607	23.48217994725136	13.57369657275657	h
20.35491269147777	25.26952958692757	17.16232547460756	h
20.33355171674582	22.60451543204375	21.06320235180141	h
21.06226361515728	24.58406416285620	25.31610705826105	h
21.78196322899934	29.26647697286657	25.64557045888111	h
21.72732556807249	31.95238631863876	21.74254877988402	h
30.58482438405833	34.18239427013287	-0.33054401420213	h
31.56051819698523	36.97064586561143	-4.02985626065841	h
28.15839830346982	39.67712495479709	-5.93729819927722	h
23.76737663766693	39.57507499105506	-4.11965325740417	h
22.78327594687021	36.79230788973121	-0.44475984149372	h
17.65475455661468	35.37271909407004	-0.65344793541912	h
19.12540229395682	41.28596599229583	4.86492420662672	h
18.56133284310958	44.55396125319399	1.50946993182002	h
17.59108298526332	43.25964503595902	-2.95775897193938	h
17.12680563217233	38.65114062927810	-4.02516477551852	h
23.54459964817358	29.48777099841054	-1.61211871075121	h
19.24478909518849	30.60649383545161	-0.20604891488996	h
18.26335123182007	23.05408265534035	2.87003340123161	h
22.59326255212935	21.93461619029280	1.51096260414891	h
16.02571831387840	25.40899580693478	5.80332952144141	h
11.84332491272974	26.68418272843846	7.39005735244149	h
10.74607099903038	31.40917301632005	0.74598264345677	h
14.93180209814419	30.08524621405203	-0.86390106416783	h
32.43335292195162	38.74531805226316	15.21827856969853	h
28.37412714470774	40.76157572347234	16.41445050769961	h
25.56563413111231	33.52619941815199	19.10169180910099	h
29.59969488712306	31.50982505260766	17.92262358722040	h
23.69038406788388	36.48453165324876	22.06674807041912	h
19.43685585851925	38.24886455051604	22.96801046260483	h
19.78540600627602	42.57501650879039	15.97693749215433	h
23.99647389328205	40.76541470858711	15.06987606628119	h
9.99231347392907	24.97708860282267	11.20006325392126	h
14.89262573223898	27.31614473487421	17.38287223907009	h
13.40157125397296	23.64271788680777	19.94528923625314	h
10.21312566894806	20.60895125489633	18.14575217115453	h
8.52179525365354	21.29764969478540	13.76144894037785	h
27.01276888746496	14.43938441759622	6.29849383317059	h

[{Au₆Ag₆(C₂Ph)₁₂}Au₃(PPh₂(C₆H₄)₂PPh₂)₃]³⁺ (structurally analogous to 2, based on a shorter diphosphine with biphenyl spacer)

14.33313012495524	-4.05899504901084	22.01385593281064	au
16.09879532837544	19.02115754703586	14.33049461734636	au
14.69075613837531	13.89600488230647	13.19006105942788	au
13.70922194154551	-0.18763763421882	18.24828756564606	au
11.94402704203522	1.47637152737577	11.50049593451669	au
12.24696425525507	8.08352174300693	8.54130636136536	au
13.96046012769708	7.15498927962795	15.51682398989583	au
9.01061889931814	1.52747416420017	-1.12494614334051	au
10.37962582115421	2.89768232888381	3.95354824471791	au
17.10361229179705	9.36367557549242	11.39479582953675	ag
8.00283714398703	4.35475388196895	8.64594865097404	ag
10.26213332167522	10.70769798589010	13.15033407088679	ag
16.72049117889962	2.20707549265461	14.40281378704930	ag
9.91863842111938	3.30634082900810	16.44741889481817	ag
14.90741948459616	3.38862740338749	6.92235396734371	ag
13.28625572330274	-7.56829569270751	19.37661034242932	p
17.35969027552814	18.31641776914393	18.60578813463687	p
15.48797053679968	-1.12257847996392	25.24920813715600	p

15.05184221730648	20.52509398384804	10.19928909709186	p
9.43796517379357	5.64008744316287	-2.93265196853830	p
8.37656764396957	-2.82557271675034	-0.08993745247409	p
23.00775761421275	-3.45500973747345	29.18434189285800	c
19.24071542468353	13.71879140355290	20.75724884730578	c
17.92662750688351	3.00369832760714	22.86814832355429	c
14.72415383954199	13.87230327656150	19.97130446635367	c
18.98295066823950	11.24373126757816	21.68767201923179	c
22.26482334899960	-0.90353332505013	29.19412750961661	c
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13.95996104849666	4.05178812794342	24.91913928576966	c
15.81173071780218	2.23185897446573	24.30667756792201	c
14.47877866515331	11.41068990837840	20.92427305732989	c
14.22020122519356	6.56754085876960	24.11049965976472	c
20.69105600373855	19.23482434222371	18.99979033861894	c
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18.52356823643656	-1.97102598363338	26.71390874304450	c
22.44310628006190	18.50706128918727	17.11807128014555	c
21.51915764122555	-5.26525353629889	27.93315159749334	c
16.60378565018621	10.02404049435721	21.78193782398840	c
18.19122762404064	5.52390623591729	22.09774537119306	c
25.00331569722887	19.16124760249047	17.36308890313811	c
25.82661116230191	20.56152246884839	19.46706901474416	c
24.08620437626166	21.30083785238275	21.33247150944041	c
16.33912710705330	7.36596410696563	22.68557585497081	c
15.96798101702969	-9.78012488210267	19.26513629916789	c
10.60216931559079	-9.36649358721249	20.65798963961591	c
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10.19669840416488	-9.37507891044592	23.29731313984598	c
20.17221850708198	-13.04165912605847	19.06749809056864	c
7.07517832483302	-12.29187023922974	20.13584431199730	c
20.52073624736136	-10.41345492010605	18.87769236320274	c
15.18746346230891	19.57933990324567	23.35818181417991	c
13.85725073960327	-7.91190894315199	14.00430540445757	c
9.02293707250667	-10.82956758406973	19.07738760257951	c
14.59394400474192	22.64175290259442	19.95662979240502	c
9.64757004411846	-4.86727619078103	12.98727266270701	c
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10.56013508281942	-1.53797281577691	27.17706156898013	c
13.83074737169185	-0.86142587946696	30.36266578031343	c
13.12961475269635	-1.17388362028997	27.80659002574750	c
13.17399484065973	-7.36441049606791	11.49614884253983	c
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15.54370780184264	20.29666831132438	20.81042209583687	c
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13.32559625157183	24.25845421901453	21.63594596559627	c
21.52311052424382	20.64143853911097	21.10851582400255	c
8.53494615485851	24.19738153657942	12.40150851153025	c
12.33551924192882	24.93917473117540	8.75129411143156	c
15.11666130852969	14.63785057327301	5.02694772548791	c
12.55484927642267	14.32027769810128	4.31601924980181	c
12.45796075245251	22.83617606846549	10.38983452297603	c
10.55269991708087	22.48075714021556	12.22678879466820	c
13.32650945153132	11.61831560750023	0.42710755225411	c
14.03968355152284	18.13716342525644	7.85074649013772	c
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10.32020291303537	26.65568610242605	8.94669626486394	c
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2.18259979652501	-3.07431262037466	23.19498396289832	c
15.94200861034053	4.11747442792077	-6.93981960753977	c
4.31345017730275	3.62240989849271	4.48638734698055	c
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26.02685357633475	-3.48984907981701	19.78473995402246	c
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