

Supporting Information

Synthesis, Characterization, Photophysical and Theoretical Studies of the Supramolecular Gold(I)-Silver(I) Alkynyl-Phosphine Complexes

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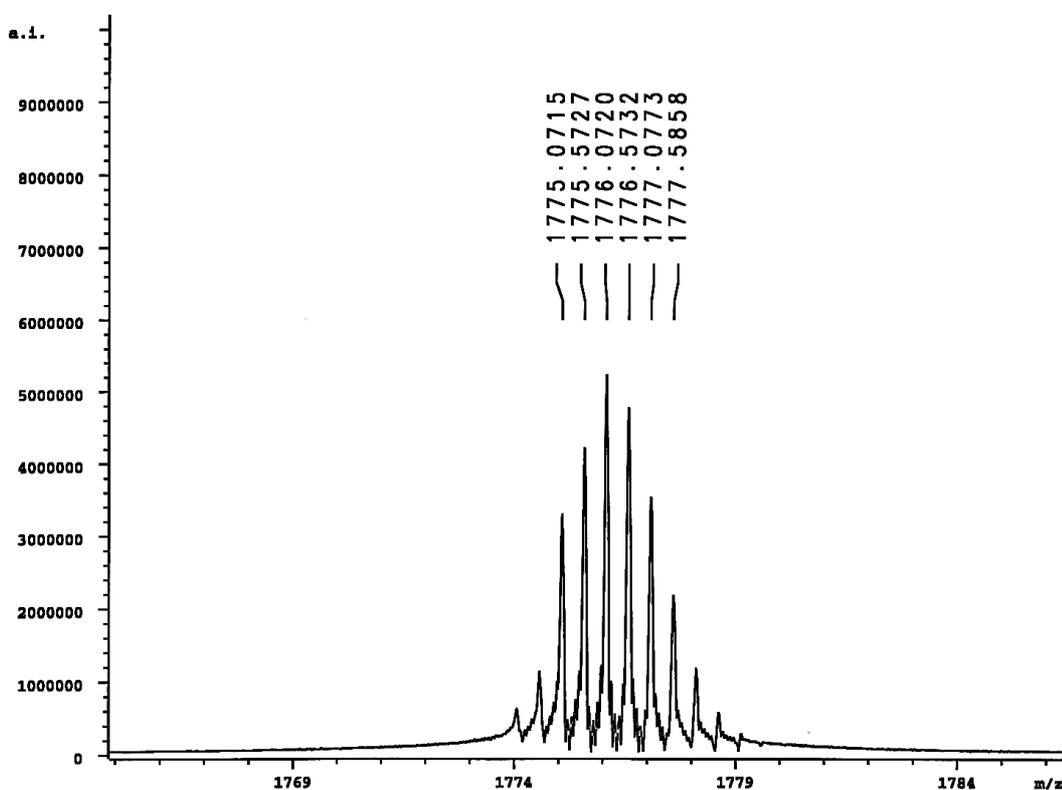


Figure S1. ESI-MS spectrum of the $[\text{Au}_6\text{Ag}_3(\text{C}_2\text{Ph})_7(\text{PPh}_2\text{C}_6\text{H}_4\text{PPh}_2)_3]^{2+}$ dication (**1-2PF₆**).

X-ray Crystal Structure Determinations

The crystal was immersed in cryo-oil, mounted in a Nylon loop and measured at a temperature of 120 K. The X-ray diffraction data was collected by means of a Nonius KappaCCD diffractometer using Mo K α radiation ($\lambda=0.71073$ Å). The Denzo-Scalepack program package was used for cell refinements and data reduction. The structures were solved by direct methods using the *SHELXS-97*¹ with the WinGX² graphical user interface. An empirical

absorption correction was to the data (*SADABS*).³ Structural refinements were carried out using *SHELXL-97*.⁴ Because of the low data quality this structure has been submitted only as supplementary material. Due to the low data quality U_{eq} values of some non-solvent atoms remained high. All phenyl rings have been refined as rigid phenyl groups with C-C 1.39 Å and C-C-C angles of 120°. Furthermore, all carbon atoms were restrained so that their U_{ij} components approximate to isotropic behavior. Carbons C63-C66, C109-C114, and C147-C153 were further restrained to have equal displacement parameters. Only one of the solvent molecules (toluene) was located from the difference Fourier map and refined. The rest of the residual electron density related to solvent of crystallization could not be unambiguously assigned and it was treated as a diffuse contribution using the *SQUEEZE* procedure as implemented in *PLATON*.⁵ All hydrogen atoms were positioned geometrically and constrained to ride on their parent atoms, with C-H = 0.95-0.98 Å, and $U_{iso} = 1.2-1.5 U_{eq}(\text{parent atom})$. The highest peak is located 1.07 Å from atom Au₄ and the deepest hole is located 0.67 Å from atom Au₄. The crystallographic details are summarized in Table S1.

1. Sheldrick, G. M. *SHELXS97, Program for Crystal Structure Determination*, University of Gottingen, 1997.
2. Farrugia, L. J. *J. Appl. Cryst* **1999**, *32*, 837-838.
3. Sheldrick, G. M. *SADABS - Bruker Nonius scaling and absorption correction*, 2.10; Bruker Analytical X-ray Systems, Bruker Axs Inc.: Madison, Wisconsin, USA, 2003.
4. Sheldrick, G. M. *SHELXL97, Program for Crystal Structure Refinement*, University of Gottingen: 1997.
5. Spek, A. L. *J. Appl. Cryst.* 2003, *36*, 7-13.

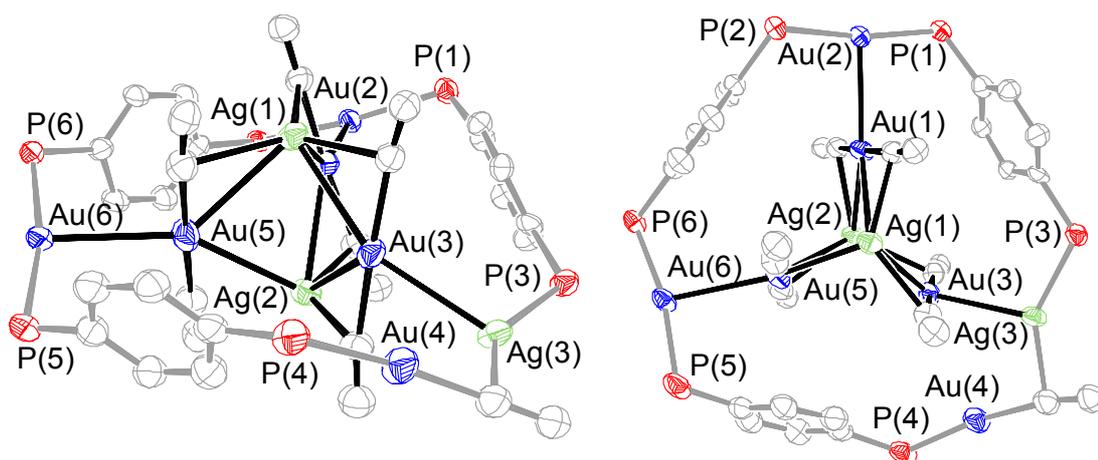


Figure S2. Two ORTEP projections of the dication **1**. Phenyl rings and hydrogen atoms omitted for clarity. Selected interatomic distances (Å) are Au(1)-Au(2) = 2.9588(8), Au(3)-Ag(3) = 2.8850(15), Au(4)-Ag(3) = 3.1876(13), Au(5)-Au(6) = 2.9594(12), Au(1)-Ag(1) = 2.9403(13),

Au(1)-Ag(2) = 2.9421(15), Au(3)-Ag(1) = 2.9148(15), Au(3)-Ag(2) = 2.9402(15), Au(5)-Ag(1) = 3.0099(16), Au(5)-Ag(2) = 2.9490(15).

Table S1. Crystal data and structure refinement for **1**.

Identification code	1	
Empirical formula	C ₁₅₃ H ₁₁₅ Ag ₃ Au ₆ F ₁₂ P ₈	
Formula weight	3934.62	
Temperature	120(2) K	
Wavelength	0.71069 Å	
Crystal system	Triclinic	
Space group	P $\bar{1}$	
Unit cell dimensions	a = 16.7570(12) Å b = 16.871(4) Å c = 27.920(7) Å	α = 101.104(14)° β = 100.071(8)° γ = 106.638(9)°
Volume	7196(2) Å ³	
Z	2	
Density (calculated)	1.816 Mg/m ³	
Absorption coefficient	6.643 mm ⁻¹	
F(000)	3752	
Crystal size	0.33 x 0.16 x 0.08 mm ³	
Theta range for data collection	2.48 to 25.04°	
Index ranges	-19 ≤ h ≤ 19, -20 ≤ k ≤ 20, -33 ≤ l ≤ 33	
Reflections collected	79074	
Independent reflections	24184 [R(int) = 0.0689]	
Completeness to theta = 25.04°	95.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.6322 and 0.2171	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	24184 / 921 / 1316	
Goodness-of-fit on F ²	1.115	
Final R indices [I > 2σ(I)]	R1 = 0.0786, wR2 = 0.2122	
R indices (all data)	R1 = 0.1296, wR2 = 0.2339	
Largest diff. peak and hole	4.144 and -3.130 e.Å ⁻³	

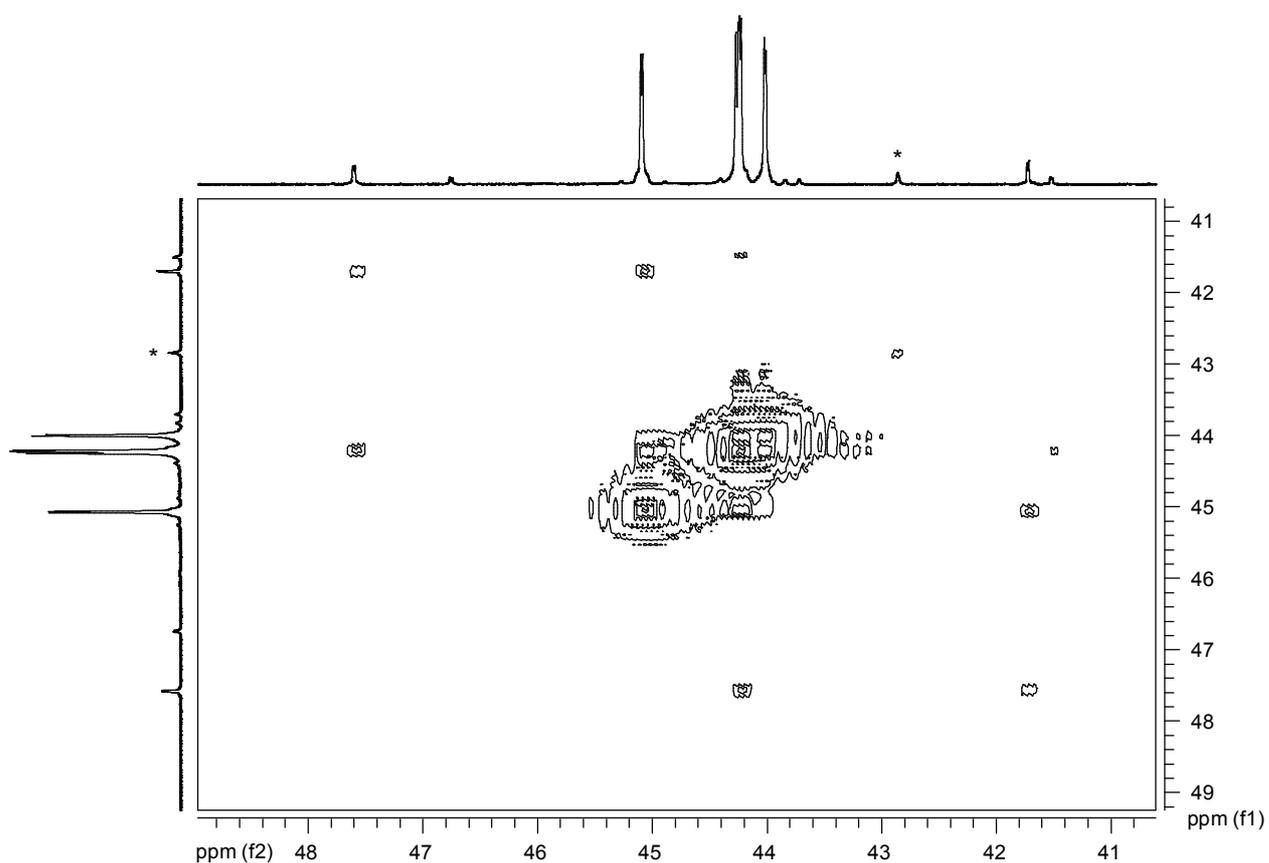


Figure S3. Part of the ^{31}P - ^{31}P COSY NMR spectrum of **1** (121.5 MHz, acetone- d_6 , 298 K, * denote admixtures).

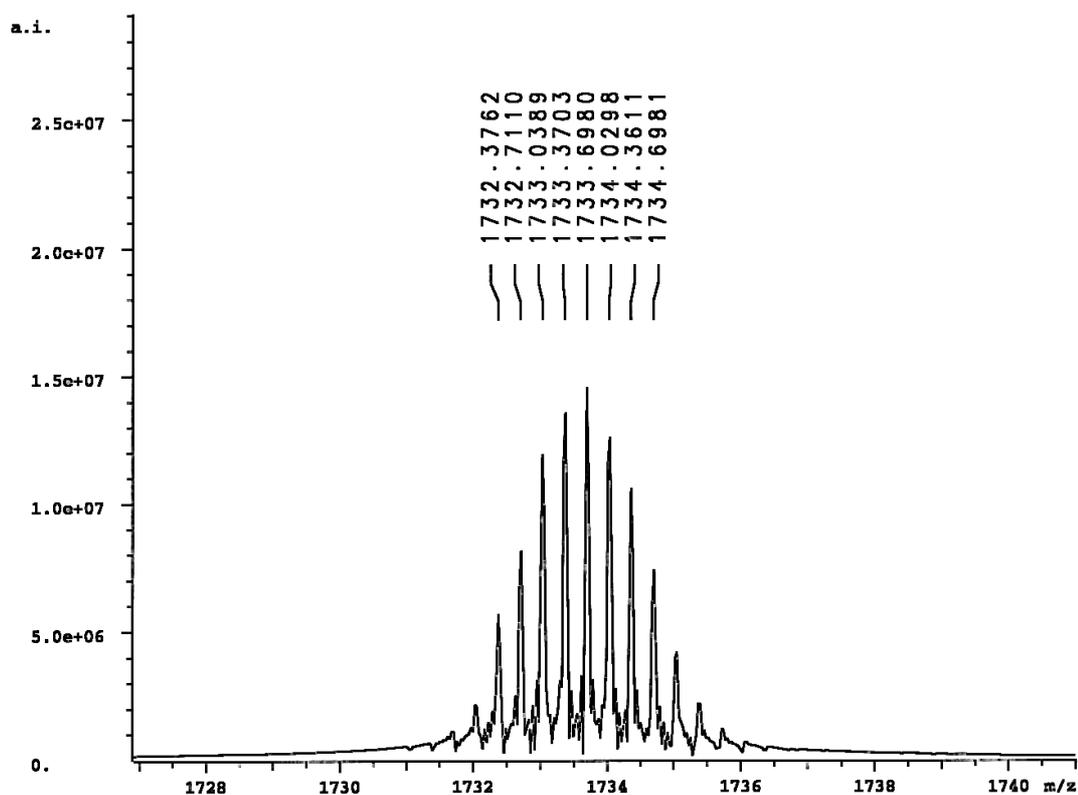


Figure S4. ESI-MS spectrum of the $[\text{Au}_9\text{Ag}_6(\text{C}_2\text{Ph})_{12}(\text{PPh}_2(\text{C}_6\text{H}_4)_2\text{PPh}_2)_3]^{3+}$ trication (**2-3PF₆**).

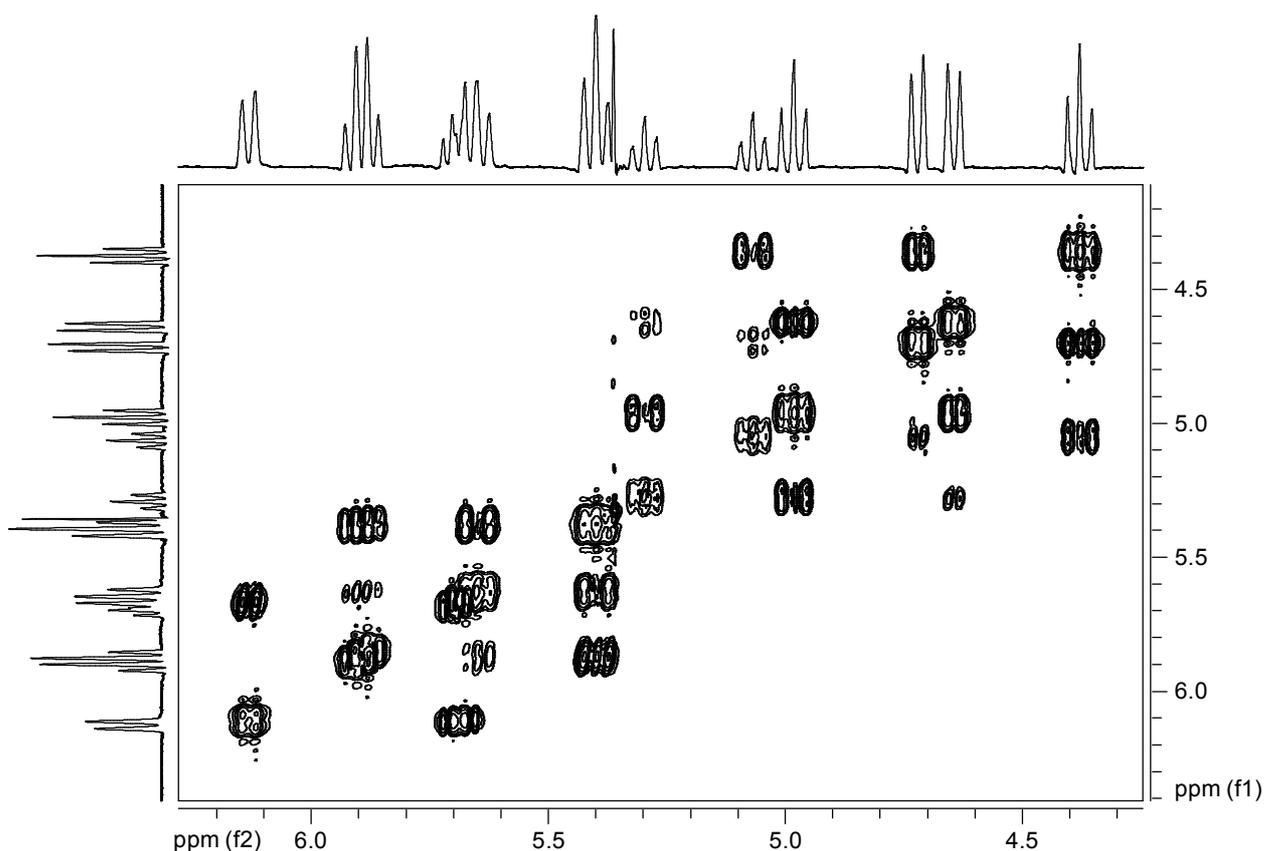


Figure S5. ^1H - ^1H COSY spectrum of **2** (300 MHz, acetone- d_6 , 298 K).

Computational supporting information

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

$[\{\text{Au}_3\text{Ag}_2(\text{C}_2\text{Ph})_6\}\text{Au}_3\text{Ag}(\text{C}_2\text{Ph})(\text{PPh}_2\text{C}_6\text{H}_4\text{PPh}_2)_3]^{2+}$ (**1**)

20.24529076032767	-1.70677463711860	34.08552051798487	au
24.49321390313253	-1.62116963147052	30.38849095944542	au
16.07263317426684	1.50652060370656	39.51497682627924	au
11.74264389067288	3.14209815724689	44.94999632907903	au
14.20190780389700	-5.29468083006894	36.40036081172630	au
11.23168903433587	-9.90590514508994	35.25397879546098	au
14.83331352218216	-0.08011222658770	34.23490598412410	ag
18.67305862754836	-3.41272021898793	39.22911887923791	ag
18.09550703820258	4.68229422911871	43.52519735282967	ag
25.93886820088878	2.56206952238960	31.04830100607903	p
24.17081367776812	-5.89182780956963	29.09239807874362	p
21.89579975765730	6.74453099598758	41.89186196481879	p
7.826771140920193	1.44920398109263	43.88915319507559	p
8.33964479400616	-9.68587830103094	38.66216432776987	p
13.33507674752642	-11.21480873381434	31.52755662197709	p
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24.13913546650468	5.58028273730183	24.09865379193291	c
23.96688841299163	4.86718247884872	22.14923446441339	h
23.76033964660058	8.15911941538717	24.61731345909139	c
23.29753197617919	9.47738252913183	23.07201726997616	h
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23.65814382734591	11.07176062780711	27.52589036219098	h
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24.72053846429072	8.10293258686240	31.03316675675867	h
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30.77495710386559	0.45725807127922	31.77911406403743	c
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33.42792394490635	0.50388875089035	31.87671248854527	c
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25.73510707192607	5.07482997540011	38.46475675316282	c
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26.60730653125648	4.21913290842472	36.10601586309989	c
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26.60190971537646	-7.82778008341824	30.65854752398738	c
27.16011427665714	-7.36055616448094	33.22955831621355	c
26.14885028523320	-5.86400456461851	34.27466998865083	h
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27.91293982587764	-9.74670378609697	29.34714642314512	c

27.51477379453888	-10.11024828685079	27.33602165205703	h
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17.10708154493981	0.25161297458316	29.18123721811059	c
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13.73764779475194	2.47862020177342	22.20451001275438	c
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22.46078096055394	-2.94072659399954	36.90890812815177	c
24.09706172510847	-3.64481316486613	38.46221191744339	c
26.02973594011378	-4.43806902953067	40.17601838989886	c
26.00839605824193	-6.90575611053250	41.24430586916448	c
24.45349373112526	-8.20566647190358	40.76709415870143	h
27.93554066495788	-7.67458633578534	42.89290186201810	c
27.88562047571805	-9.59260401025942	43.70506080820989	h
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31.43814355364342	-6.63231125467026	44.79885205765348	h
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11.21565818191231	11.13134215871101	30.37161382739742	c
10.55392306312888	12.66332639428975	29.12470206793384	h
10.96264793874419	11.35345000184649	33.00903283889500	c
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11.79550432619372	9.40960277115017	34.60464928997887	c
11.60638914135103	9.59049406594487	36.66993624465961	h
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19.56147116389931	-2.23893977333930	46.71925064548235	c
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22.99736315040063	-3.84829839461140	45.09029370582707	h
22.81335111744296	-4.45720677431064	49.15471740434590	h
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21.42185397491889	-4.07721411608608	51.38888610843964	c
22.14709927756521	-4.79002711012723	53.20731621594675	h
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18.00578817699387	-2.47573611982352	53.04060365144088	h
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16.35518577701040	-0.85216284249143	48.90948865245951	h
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7.97251456947961	-1.26454281031891	31.61001131901030	c
5.74031406213145	-2.70766263962409	31.20145499014595	c
5.64102170319571	-4.65742403546935	31.92738831662715	h
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1.96789034565668	-2.81629471974910	29.59688830716922	h
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5.99522314151397	2.26312452683799	29.34978684248178	c
6.10435502203947	4.21429719532439	28.62833707192309	h
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9.77140966136632	2.39027939741300	30.97048865009324	h
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21.42892228688721	-14.35879368117005	42.32728832807394	c
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21.45319878701677	-13.96986309457708	44.95818490357338	c
22.41737172006473	-15.32999378810224	46.20752421575232	h
20.24050488733383	-11.83718409708799	45.98620442351798	c
20.25965746943855	-11.51600404242338	48.04454020507711	h
19.00248492278655	-10.10750210719542	44.40766293134229	c
18.05794007374735	-8.43752155320010	45.21421341394446	h

[{Au₃Ag₂(C₂Ph)₆}Au₃(PPh₂C₆H₄PPh₂)₃]²⁺ (1a)

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-6.49121176902091	6.66845844014592	1.50634554337503	au
-0.54379675546041	-2.33703991369815	-3.32238677392687	au
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1.88562816294737	2.47502085954157	-1.90531432206520	ag
-2.01861442889062	-2.65219034577404	2.02012703278933	ag
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9.52261835336242	-3.87225362348778	3.28965205268581	p
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3.23746553991128	-7.65510851816335	-6.71218143652822	p
-4.50535517213885	8.30729621148567	5.22893366678452	p
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12.87138441473806	9.86095955253603	11.80579582978194	h
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5.90849700083974	9.28647865814906	-4.70688708739360	c
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5.20685231789487	2.82992249794447	-5.12471805604858	h
3.68861158701558	10.02513578263646	-5.96343618495833	c
7.20469256339107	10.73636668197312	-3.96145874144060	h
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21.12268650970970	14.60869666560926	20.73997426003162	h
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22.40672682322367	17.46534466739805	8.65083268977008	h
26.93186950167326	18.36569550737030	7.65675210774783	h
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29.23735366085786	12.08813638792946	12.42757445763975	h
24.71134235606035	11.18283289072744	13.43276575176891	h
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3.42786710870559	13.06401810852836	23.07519273145714	h
7.45160096552484	12.05153164878093	20.80783939707765	h

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18.07636781563306	0.84786956085623	0.20779019654586	c
18.13089144727389	-1.54941148631225	-1.00778107944709	c
20.19538555337337	2.47496753572648	-0.08118311638803	c
20.22409837756106	-2.28115925166548	-2.45600476188998	c
16.49763418608146	-2.82575957762781	-0.81623320162352	h
22.28505869468448	1.72169352038195	-1.52415976416518	c
20.18026761762287	4.33798023881474	0.84766491029041	h
22.31079503035342	-0.65443815213488	-2.71867095099378	c
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23.95539010317742	-1.23752695999881	-3.85679194329287	h
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22.75556542548404	8.32997463803474	6.61683236314964	c
20.45508919426662	9.17097699401726	2.69080978068968	c
25.03953402617387	8.80256965107553	5.36400060771736	c
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22.75174227432923	9.64141106622369	1.45854816914107	c
18.65064866823048	9.30533867955586	1.66156536559823	h
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26.83270167205108	8.67437070827112	6.41419571289035	h
22.75257001919524	10.16390696558002	-0.55825844076274	h
26.84994101265717	9.84129880774814	1.81421668324173	h
22.65304560969085	5.77897212959644	15.36732065323800	c
23.98190932590314	7.48214524778299	16.97009017289342	c
24.00953335020597	3.78046633803462	14.19308918799744	c
26.58093405186084	7.17983011964012	17.38018683956704	c
22.93686888220450	9.03684821427456	17.87769683605431	h
26.607452883926830	3.49771279929169	14.61763162504816	c
22.99122954134365	2.44338329397970	12.96407119438833	h
27.90118495416454	5.19056777288906	16.20841791141691	c
27.58888705269765	8.50512275696985	18.63246966382756	h
27.63091402959218	1.92742082183139	13.71128264685455	h
29.94302148256658	4.95268944193968	16.54501894466625	h
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19.89494334669087	-4.23342697783591	8.86087269395428	c
21.37077508631522	-0.35666813442015	6.81009768272527	c
21.95458759122550	-5.54632552463543	7.83687055122180	c
18.50554556660749	-5.20962907364343	10.06498344796173	h
23.42273042100964	-1.68898640831653	5.79826991437878	c
21.12294766637804	1.66915415568641	6.39716298925395	h
23.72268372076174	-4.28208000456641	6.30433734752209	c
22.18172455135407	-7.57935142366101	8.23144979320648	h
24.79455619214334	-0.69693242718746	4.58633751982649	h
25.33381795996895	-5.32378855907218	5.49310582770634	h
1.63563255129153	3.78090630659058	4.16628503632363	c
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-0.02198955164038	2.21288330700381	5.58632101487461	c
-2.04903420856207	5.59755076464157	2.01916584020103	c
1.83808534119801	6.68970697844995	1.24579474876575	h
-2.64256480858430	2.35493131100898	5.22781300258123	c
0.77940696566002	0.88761007809399	6.97623809109338	h
-3.66736159379735	4.04225267603790	3.44591109786778	c
-2.84087646951877	6.91343535718217	0.61129578141231	h
-3.90150886919205	1.13004606743292	6.34800312402340	h
-5.72905183945545	4.14220244073236	3.16460283793178	h
3.25713663694377	1.68277579499136	13.21970402914410	c
2.28488199714324	-0.49441106815467	14.46169296247414	c
1.55313633536358	3.63146408190287	12.50050598786943	c
-0.30572503187304	-0.70423879728710	14.96333596055170	c
3.59322790082238	-2.00607305623533	15.03830597105470	h
-1.03609126934829	3.39747790488032	13.00680860472499	c
2.28624416525894	5.32579835245541	11.53844117675409	h
-1.97467578614865	1.23548638887219	14.23762789481594	c
-1.03271122671549	-2.39738184198754	15.93395651458955	h
-2.33276120896343	4.92123285380340	12.43018572684592	h
-4.01242068145460	1.05948248672227	14.63329361125728	h

{[Au₁₀Ag₁₂(C₂Ph)₂₀]Au₃(PPh₂(C₆H₄)₃PPh₂)₃]⁵⁺ (3)

-0.79583619108621	-16.77762497299080	8.69233815157566	au
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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
0.30324328336904	-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
0.04678424842269	11.41968706265787	7.12753668623850	au
3.74164971868121	8.10646929809167	4.95425479288262	ag
-3.43271331304524	8.58133952692121	4.10406242406870	ag
3.38515029716418	0.14078937280071	5.32234578689576	ag
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3.43858895020789	-8.09812882840686	5.45126508962883	ag
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-2.73756623417734	-0.08046585546433	-9.33243081170726	ag
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0.00632841106812	18.59550739986516	6.88457882972623	p
1.23388121227204	5.10841414951534	-17.70514456897443	p
1.04597208663240	-3.86802724816938	-19.45035360591010	p
-1.08060969785262	-19.23978917189358	4.90575979841283	p
6.38068286804789	-18.17252086455921	17.30576840792099	c
1.21536047219865	8.74944218047072	16.12247929593770	c
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5.19625434268510	-15.93204181537820	18.10412085108456	c
-0.37958981523076	10.14030325012156	14.49415852867805	c
-2.92018726203790	-10.48728903560953	14.39434739771723	c
-0.77053512687625	-11.74998141103418	13.44458340589804	c
-2.17699836349017	6.14304018339919	13.01765708110783	c
-2.91809866276193	-7.84867300427762	14.70860496997471	c
2.38935272193055	14.59949778927369	16.33674741527505	c
3.11473557519294	-14.96982992119300	16.76692738685627	c
2.19587476752155	-16.25934331811661	14.61399258946080	c
4.80172082714120	14.67122876472359	15.18965526699350	c
5.49208658777317	-19.44516876088983	15.15050748533788	c
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1.36246925402395	-7.65966582380551	13.13101590816511	c
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6.65670020339847	16.04260730293259	19.17376437792144	c
4.25904672127027	15.98459078903742	20.31519080139705	c
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4.79213991169859	-25.79384220908124	4.98667691294221	c
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3.44248054830660	-25.14789510187245	7.18467933735010	c
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-7.39659613366450	-18.75705408166215	14.80555486919394	c
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-8.72013710671747	-20.70530185526797	5.39054974681289	c
-3.15433432329978	14.63605249169460	16.19768281232206	c
-5.41278896066786	-17.50434421211941	18.76329143757310	c
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2.12438431380389	15.26521591842085	18.90745311995323	c
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