

Supporting Information

Assembly of the heterometallic Au(I)-M(I) (M = Cu, Ag) clusters containing the dialkyne-derived diphosphines: synthesis, luminescence and theoretical studies

Igor O. Koshevoy,^{a*} Polina V. Ostrova,^{a,b} Antti J. Karttunen,^a Alexei S. Melnikov,^c Mikhail A. Khodorkovskiy,^c Matti Haukka,^a Janne Jänis,^a Sergey P. Tunik^{b*} and Tapani Pakkanen^{a*}

^a University of Eastern Finland, Joensuu, Finland.

^b St.-Petersburg State University, Department of Chemistry, St.-Petersburg, Russia.

^c St.-Petersburg State University, Department of Physics, St.-Petersburg, Russia.

E-mail: igor.koshevoy@uef.fi; stunik@inbox.ru; tapani.pakkanen@uef.fi

X-ray Structure Determination of **6**.

The crystal of **6** was diffracting weakly. Furthermore, the crystal was unstable and some solvent of crystallization was missing. Due to the poor data quality the structure is used SOLELY as supporting material. The *Denzo-Scalepak*¹ program packages 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 the data. Structural refinements were carried out using *SHELXL-97*.² Only two acetone molecules were included in the final structure model. These molecules were disordered over two sites with equal occupancies. Considerable amount of solvent of crystallization was missing. Due to the large voids (485 Å³), calculation of the contribution of missing solvent to the calculated structure factors by using the SQUEEZE routine of PLATON⁵ was not successful. Because of the low data quality a large number of geometric and displacement constraints and restraints were used. One of the PF₆ anions was disordered over two sites with equal occupancy. Several phenyl rings were at least slightly disordered, but no disorder models were used in the final refinement. Hydrogen atoms were positioned geometrically and constrained to ride on their parent atoms, with C-H = 0.95-0.98 Å³, and U_{iso} = 1.2-1.5 U_{eq}(parent atom). The crystallographic details for **6** is summarized in Table S1.

1. Z. Otwinowski, W. Minor, in *Macromolecular Crystallography, part A*, eds. J. Carter, C. W. and R. M. Sweet, Academic Press, New York, 1997, vol. 276, pp. 307-326.
2. G. M. Sheldrick, *Acta Crystallogr., Sect. A*, 2008, **A64**, 112-122.
3. L. J. Farrugia, *J. Appl. Cryst.*, 1999, **32**, 837-838.
4. G. M. Sheldrick, *SADABS-2008/1 - Bruker AXS area detector scaling and absorption correction*, Bruker AXS, Madison, Wisconsin, USA, 2008.

5. A. L. Spek, PLATON, A Multipurpose Crystallographic Tool, Utrecht University, Utrecht, The Netherlands, 2005.

Table S1. Crystallographic data for **6**.

6	
empirical formula	C ₂₀₄ H ₁₄₄ Au ₉ Cu ₆ F ₁₈ O ₂ P ₉
fw	5401.86
temp (K)	100(2)
λ (Å)	0.71073
cryst syst	Monoclinic
space group	C2/c
<i>a</i> (Å)	43.1517(5)
<i>b</i> (Å)	25.1541(3)
<i>c</i> (Å)	39.4692(5)
α (deg)	90
β (deg)	110.1053(6)
γ (deg)	90
<i>V</i> (Å ³)	40230.9(8)
<i>Z</i>	8
ρ_{calc} (Mg/m ³)	1.784
μ (Mo K α) (mm ⁻¹)	7.292
No. reflns.	153801
Unique reflns.	34041
GOOF (<i>F</i> ²)	1.020
<i>R</i> _{int}	0.0623
<i>R</i> 1 ^a (<i>I</i> ≥ 2 σ)	0.0907
w <i>R</i> 2 ^b (<i>I</i> ≥ 2 σ)	0.2577

^a $RI = \Sigma||F_o| - |F_c||/\Sigma|F_o|$. ^b $wR2 = [\Sigma[w(F_o^2 - F_c^2)^2]/\Sigma[w(F_o^2)^2]]^{1/2}$.

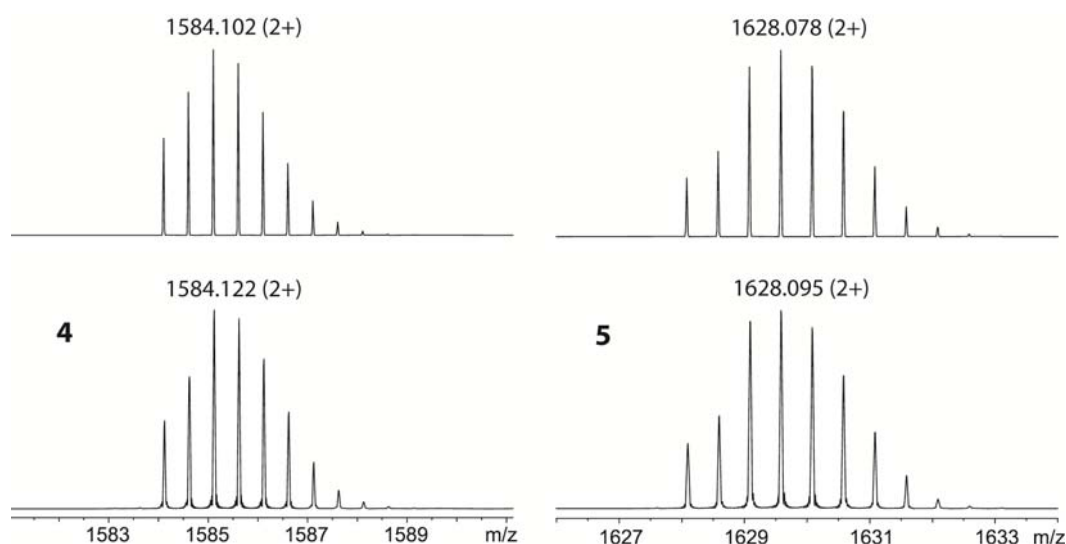


Figure S1. ESI-MS spectra of the $[\text{Au}_6\text{M}_2(\text{C}_2\text{Ph})_6(\mathbf{1})_3]^{2+}$ (*M* = Cu, **4**; Ag, **5**) dications (**4**-2PF₆, **5**-2ClO₄). Top – calculated, bottom – experimental.

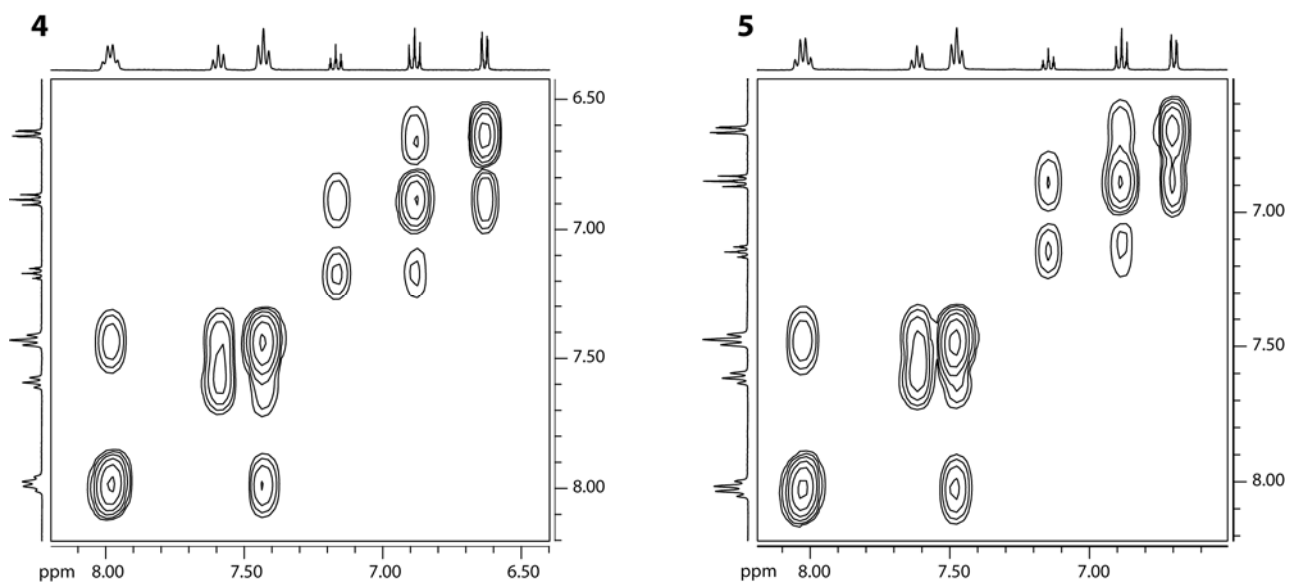


Figure S2. ^1H - ^1H COSY NMR spectra of **4** and **5**, acetone- d_6 , 298 K.

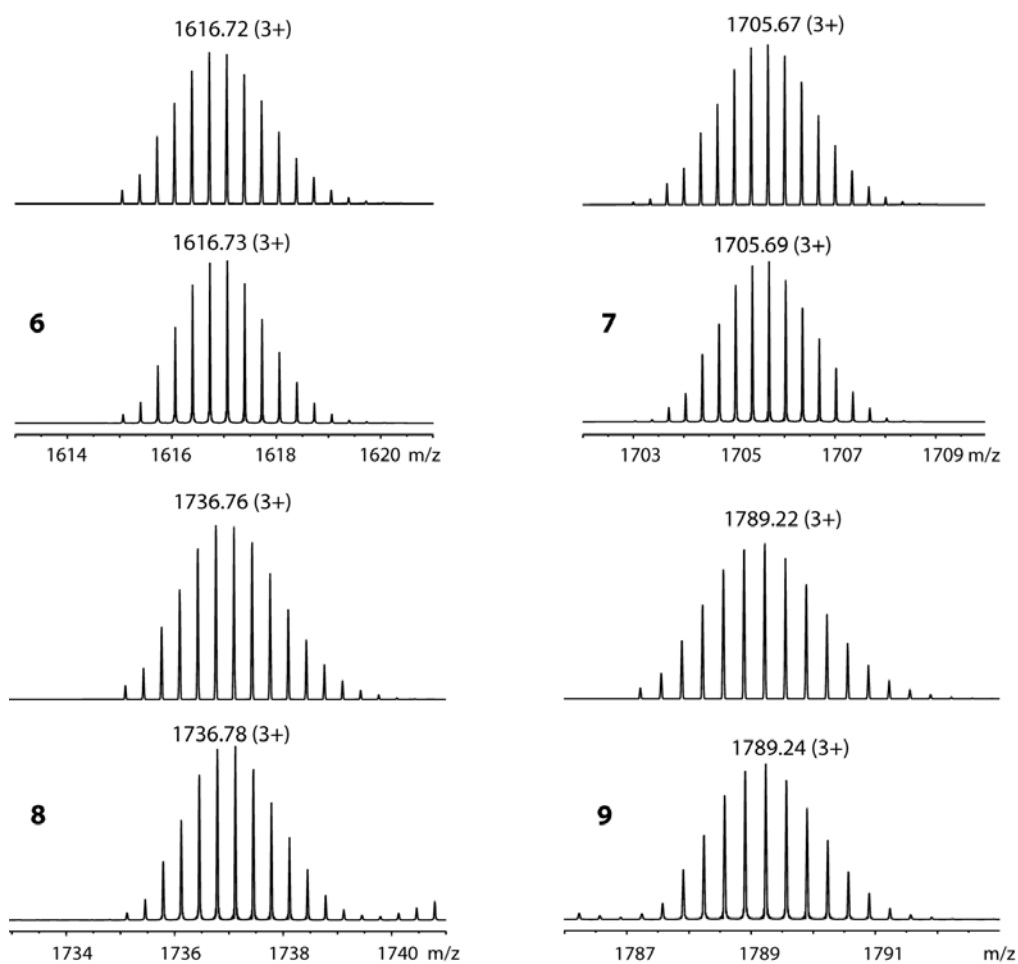


Figure S3. ESI-MS spectra of the $[\text{Au}_9\text{M}_6(\text{C}_2\text{C}_6\text{H}_4\text{X})_{12}(\mathbf{2})_3]^{3+}$, $\text{M} = \text{Cu}$, $\text{X} = \text{H}$ (**6**); $\text{X} = \text{OMe}$ (**8**); $\text{X} = \text{NMe}_2$ (**9**). $\text{M} = \text{Ag}$, $\text{X} = \text{H}$ (**7**) trications (**6-9**- 3PF_6). Top – calculated, bottom – experimental.

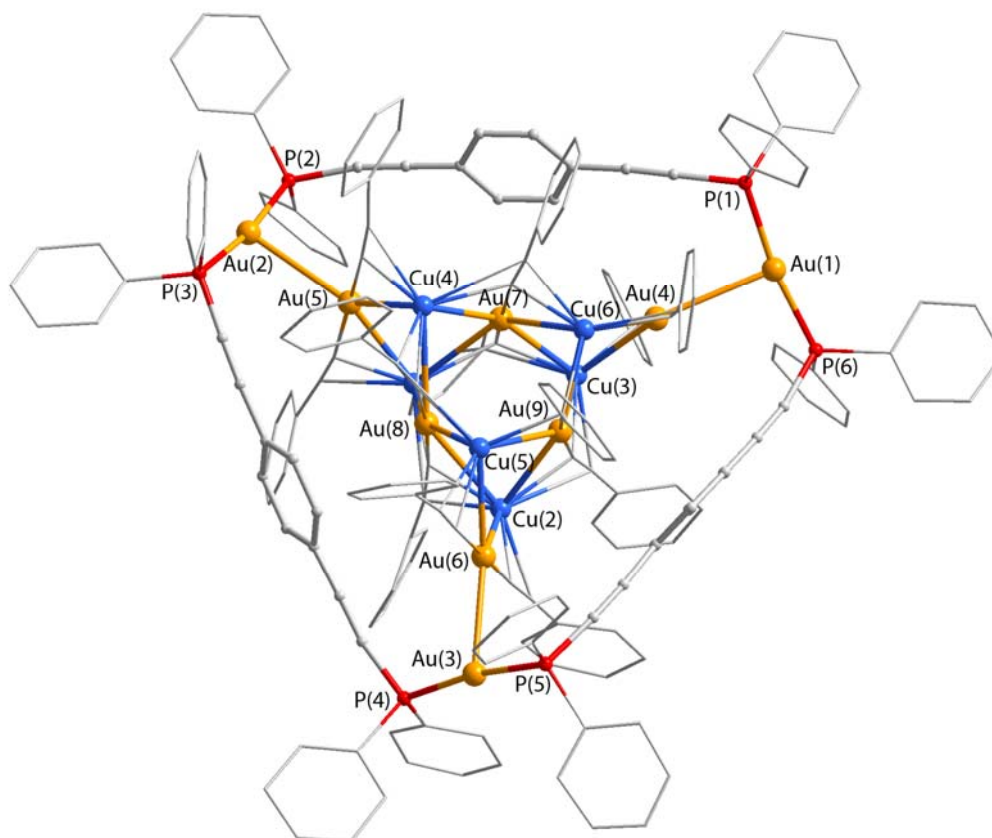


Figure S4. Molecular view of the dication **6**. Hydrogen atoms omitted for clarity. Selected interatomic distances (Å) are Au(1)-Au(4) = 3.0820(11), Au(2)-Au(5) = 3.0277(11), Au(3)-Au(6) = 3.0509(11), Au(4)-Cu(6) = 2.824(3), Au(4)-Cu(3) = 2.888(3), Au(5)-Cu(1) = 2.789(3), Au(5)-Cu(4) = 2.895(3), Au(6)-Cu(5) = 2.838(3), Au(6)-Cu(2) = 2.876(3), Au(7)-Cu(4) = 2.750(3), Au(7)-Cu(3) = 2.763(3), Au(7)-Cu(1) = 3.055(3), Au(7)-Cu(6) = 3.071(3), Au(7)-Au(8) = 3.2449(12), Au(7)-Au(9) = 3.2463(13), Au(8)-Cu(1) = 2.774(3), Au(8)-Cu(5) = 2.788(4), Au(8)-Cu(4) = 2.990(3), Au(8)-Cu(2) = 3.025(3), Au(8)-Au(9) = 3.2267(13), Au(9)-Cu(6) = 2.768(3), Au(9)-Cu(2) = 2.768(3), Au(9)-Cu(5) = 3.029(3), Au(9)-Cu(3) = 3.040(3).

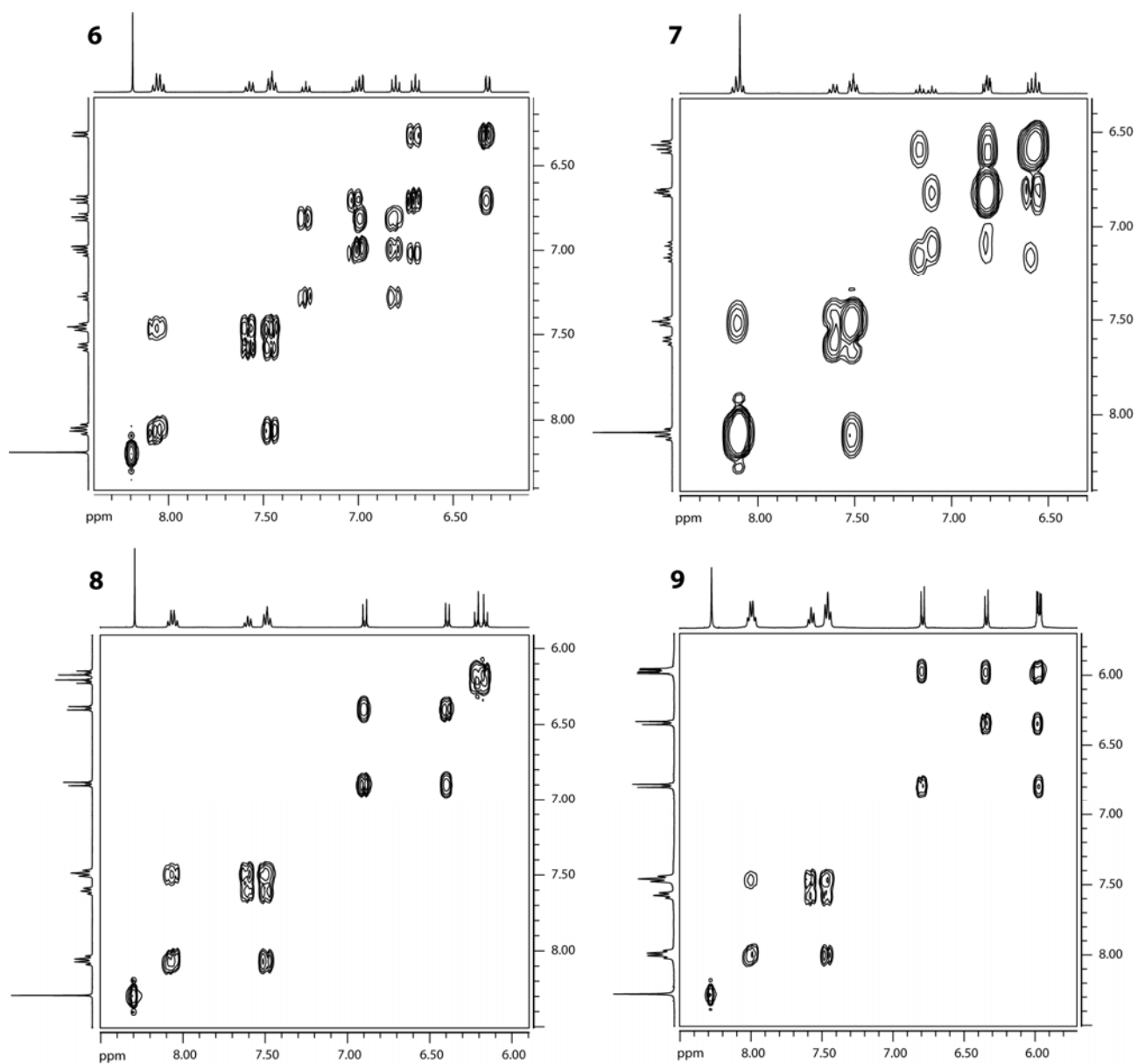


Figure S5. ^1H - ^1H COSY NMR spectra of **6-9** (aromatic region), acetone- d_6 , 298 K.

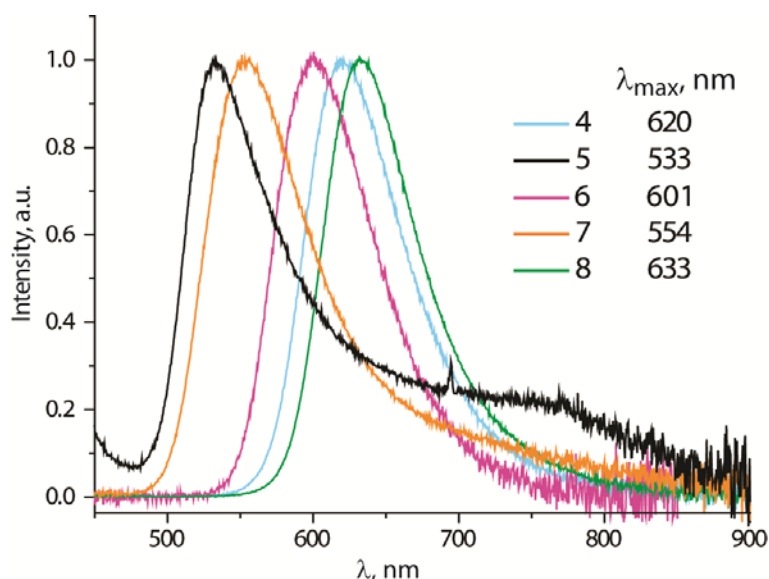


Figure S6. Normalized emission spectra of **4–8** in solid state, $\lambda_{\text{ex}} = 385$ nm, 298 K.

Table S2. Selected interatomic distances obtained for the studied complexes at the BP86-DFT level of theory.^{a,b}

Interatomic distance (Å)	4	5	6	7	8	9	10	Cu- analogue of 10
R(Au _{belt} –Au _{central})	2.93	2.90	3.13–3.23	2.97	3.08	2.98	2.90	4.05
R(Au _{central} –Au _{central})	3.54	3.95	3.29–3.75	3.75–4.13	3.35–3.71	3.36–3.68	3.62–4.20	3.19–3.69
R(Au–Cu/Ag)	2.84	2.97	2.77–3.06	2.88–3.02	2.79–2.91	2.79–2.95	2.84–3.12	2.70–2.95

^a Structure **6** optimized starting from the supplementary crystal structure given above. Corresponding experimental values: R(Au_{belt}–Au_{central}) = 3.03–3.08 Å; R(Au_{central}–Au_{central}) = 3.23–3.74 Å; R(Au–Cu/Ag) = 2.76–3.04 Å.

^b During the structural optimization of the largest D_3 -symmetric complex **10** and its hypothetical Cu-analogue, the two phenyl groups of the centermost [Au(C₂Ph)₂] rod were replaced with *tert*-butyl groups to allow optimization in D_3 -symmetry (with the phenyl groups the symmetry is lowered to C_{2v}). The bulkiness of a *tert*-butyl group is comparable to a freely rotating phenyl group.

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

[Au₆Cu₂(C₂Ph)₆(PPh₂-C₂-C₂-PPh₂)₃]²⁺ (**4**)

1.92808269193635	3.33953718362790	0.00000000000000	au
1.92808269193635	-3.33953718362790	0.00000000000000	au
-3.85616538387261	0.00000000000000	0.00000000000000	au
4.69240177854251	8.12747828996206	0.00000000000000	au
4.69240177854251	-8.12747828996206	0.00000000000000	au
-9.38480355708483	0.00000000000000	0.00000000000000	au
0.00000000000000	0.00000000000000	3.72629071678020	cu
0.00000000000000	0.00000000000000	-3.72629071678020	cu
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1.49211828647330	-11.23451513934270	0.02830557189701	p
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-10.47543465310829	-4.32504522813401	-0.02830557189701	p
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1.73602497266333	3.55415140705111	-3.79503132579169	c
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5.74507420626724	4.05094077182757	9.58654994139296	c
1.80889292777635	5.17957690757374	12.97557414258947	c
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13.73235656160648	4.87473161312989	1.98329811295985	h
12.20855430170442	13.59486179109489	5.40257549198430	h
16.75479774994908	6.75795188799829	5.08880356061527	h
16.01055748516729	11.12094281150581	6.79601301019865	h
2.26177507759650	9.20222875609277	5.46862513246615	h
0.62205307688064	16.61840330106909	2.23876703052801	h
2.22541831955637	11.09240016305971	9.78803818752303	h
0.60848695376010	18.48574846999101	6.57438411602766	h
1.40291264585691	15.73978028466812	10.35918164181087	h
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-2.64453686720165	14.32993544274200	-1.98329811295985	h
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-2.52484086241507	17.88905643072538	-5.08880356061527	h
1.62574024621437	19.42602091665885	-6.79601301019865	h
6.83847633541377	6.55986905289140	-5.46862513246615	h
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12.92959325357959	9.08484813293659	-10.35918164181087	h
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9.15322821889787	-11.71358927049405	-2.32220595668930	h
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-17.87777282287881	-3.77548727321036	-5.40257549198430	h
-14.22995688753406	-11.13110454272712	-5.08880356061527	h
9.28906961112770	-1.28233388597755	0.06171367627697	c
-5.75506852695408	-7.40340331776977	0.06171367627697	c
-3.53400108417366	8.68573720374742	0.06171367627697	c
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9.28906961112770	1.28233388597755	-0.06171367627697	c

0.0000000000000000	0.0000000000000000	-3.58017019901180	ag
0.0000000000000000	0.0000000000000000	3.58017019901180	ag

[Au₉Cu₆(C₂Ph)₁₂(PPh₂-C₂-C₆H₄-C₂-PPh₂)₃]³⁺ (6)

-11.17840617269061	-8.32899939483191	-0.25750834386766	au
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-6.44863420535321	-4.73288466933477	0.23024816769688	au
-0.75607058520573	7.94850412630735	0.00465147217409	au
7.29600139046086	-3.32038555529584	-0.06210488111608	au
-3.37435832965185	1.53587399267681	0.18484593119340	au
2.82451041245772	2.17352687799670	-0.06668276176741	au
0.28866432609753	-3.49174720335681	0.06944304765574	au
-0.02380893158586	4.31159069837370	3.81101818162632	cu
3.70223089043091	-2.06710089619107	3.76917503216523	cu
-3.64757353280797	-2.07949378844178	3.90634972842624	cu
-1.00042839556849	4.19202274066170	-3.63881699694558	cu
3.94635452140238	-1.36183726637209	-3.77695728402302	cu
-3.37623836819268	-2.72978711417877	-3.63888290077448	cu
-14.22182025486093	-5.50315548120696	-1.89193056179432	p
-5.56857556298812	14.22909008201228	1.70140880965166	p
2.75857553936881	15.22721268967586	-0.77580875166959	p
15.25949766653030	-2.56249799792922	1.47251094729670	p
11.94563555641659	-9.87223139398731	-2.04603202286274	p
-9.51869601702482	-12.21585156725091	1.17986130674092	p
-17.28396467839014	-5.83830786926877	-0.27201410276084	c
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-19.45646975482570	-4.10702290739309	-3.33557366996287	h
-21.83456438321936	-5.16176947958929	-0.15685377525231	c
-23.57808526517377	-4.47441461674460	-1.06589761696667	h
-21.93324935133867	-6.24943430528617	2.26759207550677	c
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-19.71026223116656	-7.12904477025947	3.42423060209647	c
-19.78519185377681	-7.98924512573916	5.31970147016962	h
-17.38392612780834	-6.92769154355845	2.15945800544120	c
-15.63881539686205	-7.62543642860758	3.06418212761462	h
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-13.76075107952398	-2.27913769450347	-6.52484132147702	h
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-16.28651316972083	-8.94178399499109	-8.54737296317989	c
-17.00109946245951	-10.81502167944881	-9.11141042389609	h
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-13.44195735636807	-2.29588666831157	-1.62258321929471	c
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-11.86473658101518	2.36062308838699	-0.61245448470575	c
-10.04139772824764	2.65487995195347	1.33597405660543	c
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5.72841490841443	17.86670560145057	2.85372099147143	c
6.86341450602755	16.12951512758703	3.02394177569311	h
6.33135525910058	20.00108797375706	4.31653080537973	c
7.94579772182202	19.92522407011159	5.63049551881074	h
4.89541721221621	22.22289049792450	4.09742439790801	c
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6.57931790804337	20.04587328673577	-7.94437686779341	h
3.32131120366070	17.82931484908914	-9.14856006879641	c
3.44868616420481	18.45429228681079	-11.13183810469768	h
1.40389312610136	16.14880325236658	-8.40547823908174	c
0.02108600547417	15.44556939062124	-9.79521572427632	h
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-0.26942822891097	14.03026800718618	-5.29723395430656	h
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19.17570324245349	-0.21121896554483	-1.37878134077722	c
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-6.27328238685505	-12.62151538063319	0.65055217438187	c
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-10.02028159509445	-16.85017149483442	4.31332763147784	h
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-10.69939378096290	-9.33775691667149	10.07598587458261	h
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[Au₉Cu₆(C₂C₆H₄OMe)₁₂(PPh₂-C₂-C₆H₄-C₂-PPh₂)₃]³⁺ (8)

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-3.65079940215786	0.00000000000000	0.00000000000000	au
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-3.81882374940187	15.21544291173466	0.14648657020058	p
-11.26754821669315	-10.91491983542473	0.14648657020058	p
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-3.81882374940187	-15.21544291173466	-0.14648657020058	p
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[Au₉Cu₆(C₂C₆H₄NMe₂)₁₂(PPh₂-C₂-C₆H₄-C₂-PPh₂)₃]³⁺ (9)

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1.83135439553634	3.17199885973354	0.00000000000000	au
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-2.44169767694410	-17.32268838617687	17.13306469372351	h
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-8.20762917504309	13.50785895722719	16.18837284351593	h
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7.66529503866543	1.83822005969015	-17.37099719794205	h
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13.75569755023978	-7.88093189393306	-15.22249237539990	h
16.22273704274283	-6.54677197649339	-17.13306469372351	h
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5.45975430526206	-8.96552233073462	-16.64786739712358	h
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-13.78103936579884	-10.77591640968346	-17.13306469372351	h
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-8.20762917504309	-13.50785895722719	-16.18837284351593	h

[Au₁₃Ag₁₂(C₂Ph)₂₀(PPh₂-C₂-(C₆H₄)₂-C₂-PPh₂)₃]⁵⁺ (10)

2.41584062814484	4.24046512883474	-3.31415293692061	ag
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-4.88027083948151	0.02805317341530	3.31415293692061	ag
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-6.28356974946036	-4.05050853514878	0.00336786991403	au
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21.97340741673759	5.42214583966159	-2.72942877462258	c
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11.97971699667754	-3.38382865122101	13.49093022925574	h
10.76351004453981	-1.95110897302449	9.16207410541834	h
10.01854372021453	-7.29559708417298	15.35223866055505	h
6.82660560840387	-9.75571966478973	12.83114879057607	h
5.60879597953283	-8.32965182258097	8.50551578363093	h
2.30257970339773	-12.93005554459040	6.97941150912559	h
2.86742394677781	-14.98513668252842	11.21496448385050	h
0.98535970849795	-12.99435744468671	15.09549990122336	h
-1.50168069807347	-8.96748522606284	14.70741742777898	h
-2.08573476348478	-6.93148125138153	10.48807868121946	h
0.00000000000000	0.00507922479723	9.10976128931320	c
2.14568534239299	-0.77336095235552	10.42924644223669	c
-2.14495112709858	0.78807689225919	10.42649129712399	c
2.14580987534515	-0.76995388952150	13.06486805888988	c
3.83591775505344	-1.39193201831693	9.39061028958666	h
-2.14449063863452	0.79336559319825	13.06291457339728	h
-3.83613261691475	1.40198849828927	9.38682987356056	h
0.00030090109214	0.01428700986602	14.38221162298150	c
3.83658152135762	-1.38462868034760	14.10503269762524	h
-3.83569506972594	1.41156092491266	14.10059131208964	h
0.00088119819303	0.01691671495802	16.46030410721775	h
0.00000000000000	-0.00507922479723	-9.10976128931320	c
-1.14165706722279	-1.98478481867832	-10.42443255388578	c
1.14112267156967	1.96951997002483	-10.43130420281731	c
-1.14098557193873	-1.99037871038784	-13.06005037218944	c
-2.03986271985562	-3.54197450225452	-9.38199897767445	h
1.14032813621710	1.96514893899316	-13.06772910425507	h
2.040718822488560	3.53015647627289	-9.39543810521917	h
-0.00023409927334	-0.01447052117079	-14.38221130172805	c
-2.03977656724122	-3.55133788737309	-14.09641779523338	h
2.03951880859669	3.52267864099245	-14.10920319091969	h
-0.00003749216648	-0.01923440736822	-16.46030006320383	h

[Au₁₃Cu₁₂(C₂Ph)₂₀(PPh₂-C₂-(C₆H₄)₂-C₂-PPh₂)₃]⁵⁺ (a hypothetical Cu-analogue of complex 10)

2.14988302884649	3.65301598145308	-3.53285717837571	cu
8.48792300025160	-0.06199889085817	3.80552065982922	cu
2.14988302884649	-3.65301598145308	3.53285717837571	cu
2.08866308310030	3.68836141904257	3.53285717837571	cu

8.48792300025160	0.06199889085817	-3.80552065982922	cu
2.08866308310030	-3.68836141904257	-3.53285717837571	cu
-4.23854630091940	0.03534524861688	-3.53285717837571	cu
-4.23854630091940	-0.03534524861688	3.53285717837571	cu
-4.29765409976687	-7.31975742175454	3.80552065982922	cu
-4.29765409976687	7.31975742175454	-3.80552065982922	cu
-4.19026890048473	-7.38175631261270	-3.80552065982922	cu
-4.19026890048473	7.38175631261270	3.80552065982922	cu
5.76661985902782	-3.00940738593649	-0.05290477281627	au
-5.48953328458768	-3.48933565169967	-0.05290477281627	au
-0.27708657444014	6.49874303763616	-0.05290477281627	au
-5.48953328458768	3.48933565169967	0.05290477281627	au
-0.27708657444014	-6.49874303763616	0.05290477281627	au
5.76661985902782	3.00940738593649	0.05290477281627	au
-5.93704481032211	10.28326317095062	0.00000000000000	au
11.87408962064422	0.00000000000000	0.00000000000000	au
-5.93704481032211	-10.28326317095062	0.00000000000000	au
19.53582548943390	0.00000000000000	0.00000000000000	au
-9.76791274471695	16.91852115826212	0.00000000000000	au
-9.76791274471695	-16.91852115826212	0.00000000000000	au
-6.17125482161537	-19.52229183778801	-0.36967956447189	p
-13.82117328803305	15.10560936911646	-0.36967956447189	p
19.99242810964842	4.41668246867155	-0.36967956447189	p
-13.82117328803305	-15.10560936911646	0.36967956447189	p
19.99242810964842	-4.41668246867155	0.36967956447189	p
-6.17125482161537	19.52229183778801	0.36967956447189	p
21.67137324480893	-5.26766035035296	3.29246984132666	c
21.67137324480893	5.26766035035296	-3.29246984132666	c
-6.27375885829626	21.40178982797941	3.29246984132666	c
-15.39761419754006	16.13412947762645	-3.29246984132666	c
-15.39761419754006	-16.13412947762645	3.29246984132666	c
-6.27375885829626	-21.40178982797941	-3.29246984132666	c
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12.19619948711820	-10.12825498339364	-1.96373045386603	c
11.19768520603663	-8.72934231674182	2.32373103005789	c
9.92228485045760	-11.47033659324289	-2.04147340902772	c
8.94656860338839	-10.10717036406596	2.23300603430850	c
8.250839093345545	-11.48844489391126	0.05003654649177	c
-13.98230116469877	-6.78017730499023	0.22115729494819	c
-14.86942591682263	-5.49809109835306	-1.96373045386603	c
-13.15867480111952	-5.33280884775680	2.32373103005789	c
-14.89474541241410	-2.85778256404782	-2.04147340902772	c
-13.22635042928087	-2.69437057540528	2.23300603430850	c
-14.07470480417200	-1.40121378616383	0.05003654649177	c
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11.19768520603663	8.72934231674182	-2.32373103005789	c
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9.92228485045760	11.47033659324289	2.04147340902772	c
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4.97246037298388	14.32811915729071	-2.04147340902772	c
1.96098959508289	14.06215116449862	2.32373103005789	c
2.67322624073181	15.62634608174670	-1.96373045386603	c
1.11934488179919	15.49911667937052	0.22115729494819	c
1.11934488179919	-15.49911667937052	-0.22115729494819	c
2.67322624073181	-15.62634608174670	1.96373045386603	c
1.96098959508289	-14.06215116449862	-2.32373103005789	c
4.97246037298388	-14.32811915729071	2.04147340902772	c
4.27978201486510	-12.80154075049863	-2.23300603430850	c
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5.92605567491415	3.32696376832067	3.91017974019205	c
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-0.00088110370672	0.01890005812353	16.52788708210681	h
0.00000000000000	-0.00507922479723	-9.17734772240109	c
-1.14165706722279	-1.98478481867832	-10.49201898697366	c
1.14112267156967	1.96951997002483	-10.49889063590519	c
-1.14098557193873	-1.99037871038784	-13.12763680527733	c
-2.03986271985562	-3.54197450225452	-9.44958541076233	h
1.14032813621710	1.96514893899316	-13.13531553734295	c
2.04071882248560	3.53015647627289	-9.46302453830705	h
-0.00023409927334	-0.01447052117079	-14.44979773481593	c
-2.03977656724122	-3.55133788737309	-14.16400422832126	h
2.03951880859669	3.52267864099245	-14.17678962400757	h
-0.00003749216648	-0.01923440736822	-16.52788649629171	h