

Supporting information

Reversible protonation of the amine-functionalized luminescent Au-Cu clusters: characterization, photophysical and theoretical studies

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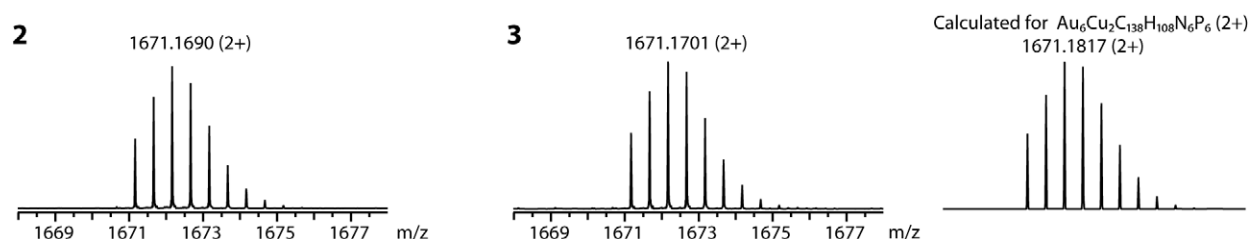


Figure S1. ESI-MS spectra of the $[\{Au_3Cu_2(C_2C_6H_4R)_6\}Au_3(PPh_2C_6H_4PPh_2)_3]^{2+}$ ($R = 4-NH_2, 3-NH_2$) dications (**2**, **3**-2PF₆).

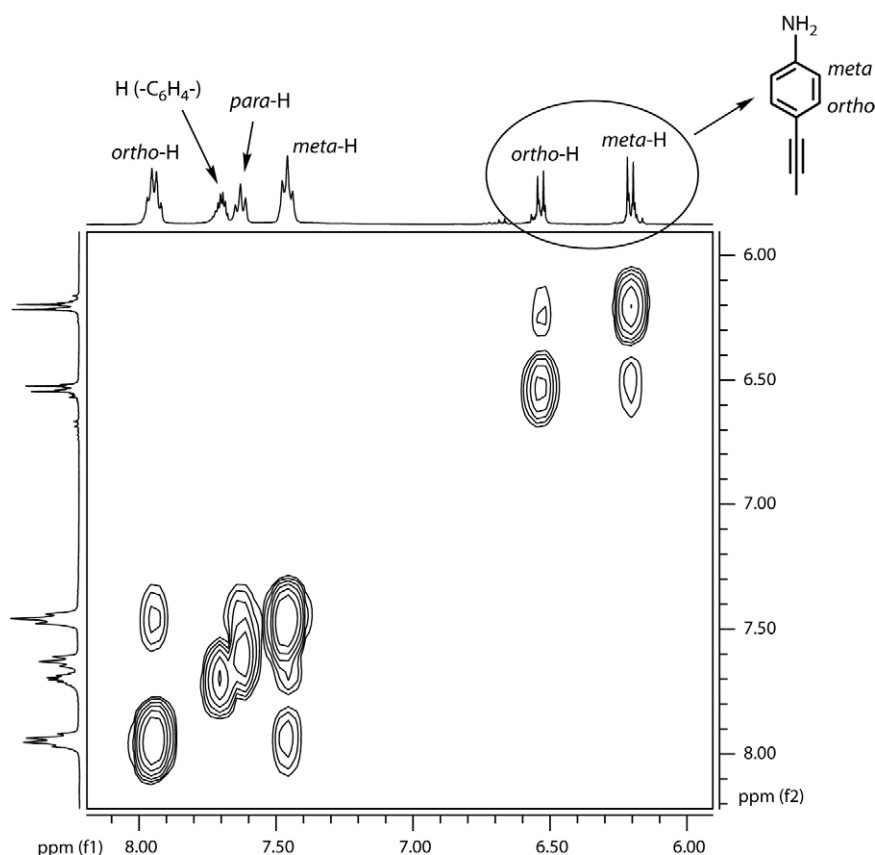


Figure S2. 1H - 1H COSY spectrum of **2**, 400 MHz, 298 K, acetone- d_6 .

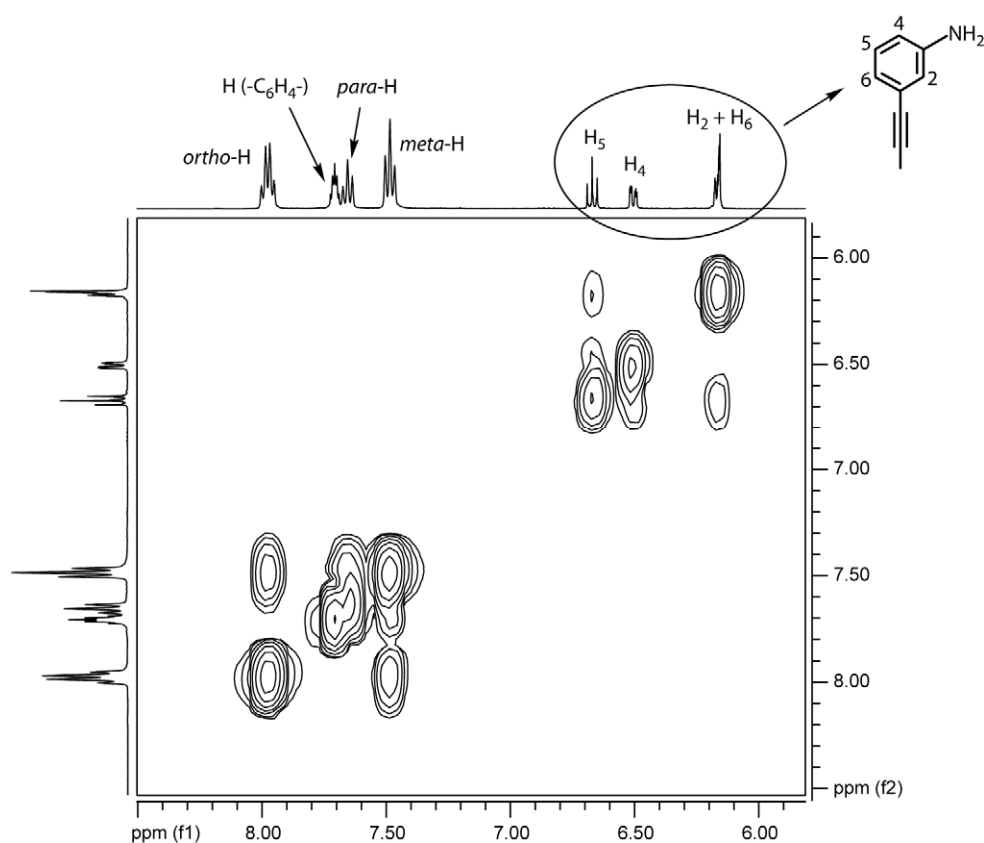


Figure S3. ^1H - ^1H COSY spectrum of **3**, 400 MHz, 298 K, acetone- d_6 .

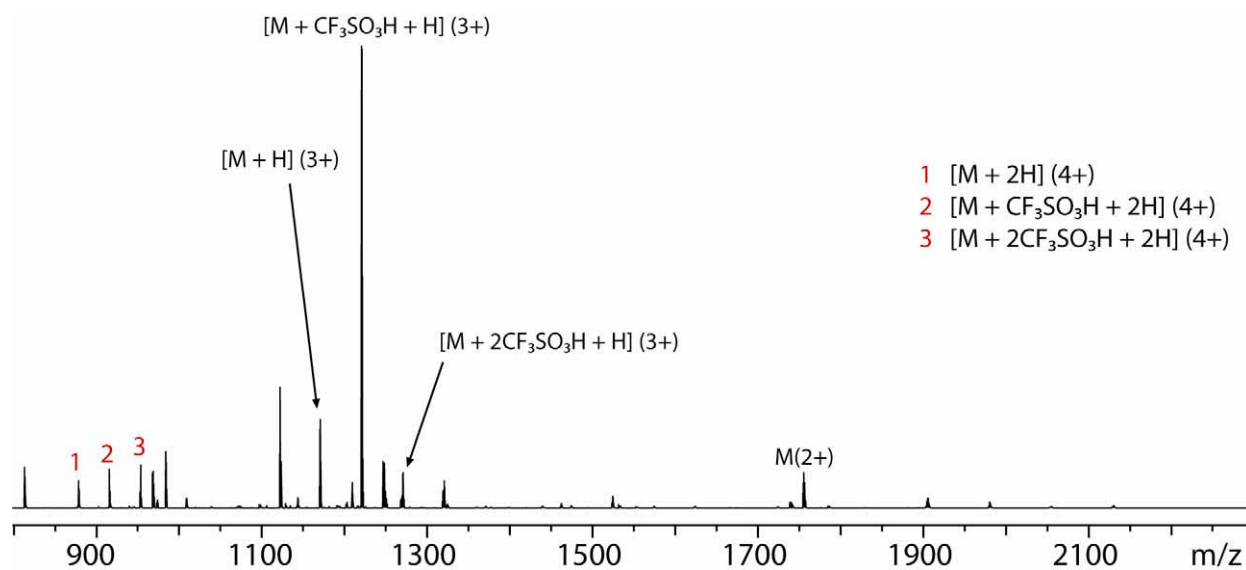


Figure S4. ESI-MS spectrum of **1** in the presence of excess of $\text{CF}_3\text{SO}_3\text{H}$.

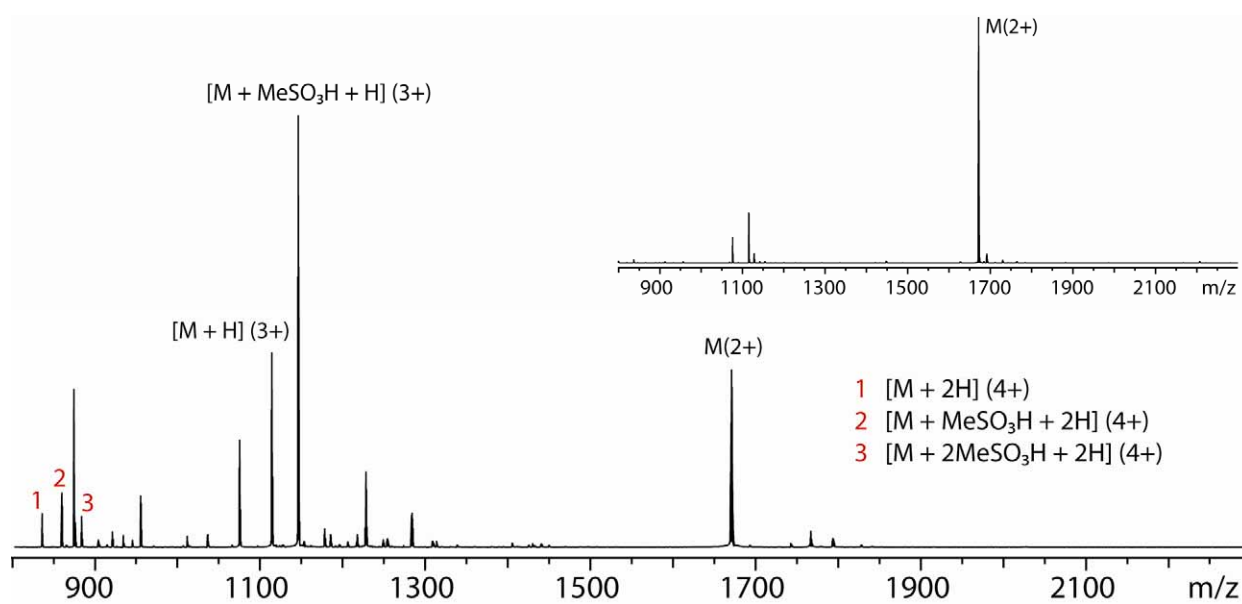


Figure S5. ESI-MS spectrum of **2** in the presence of excess of MeSO_3H . Inset – spectrum of **2** under acid-free conditions.

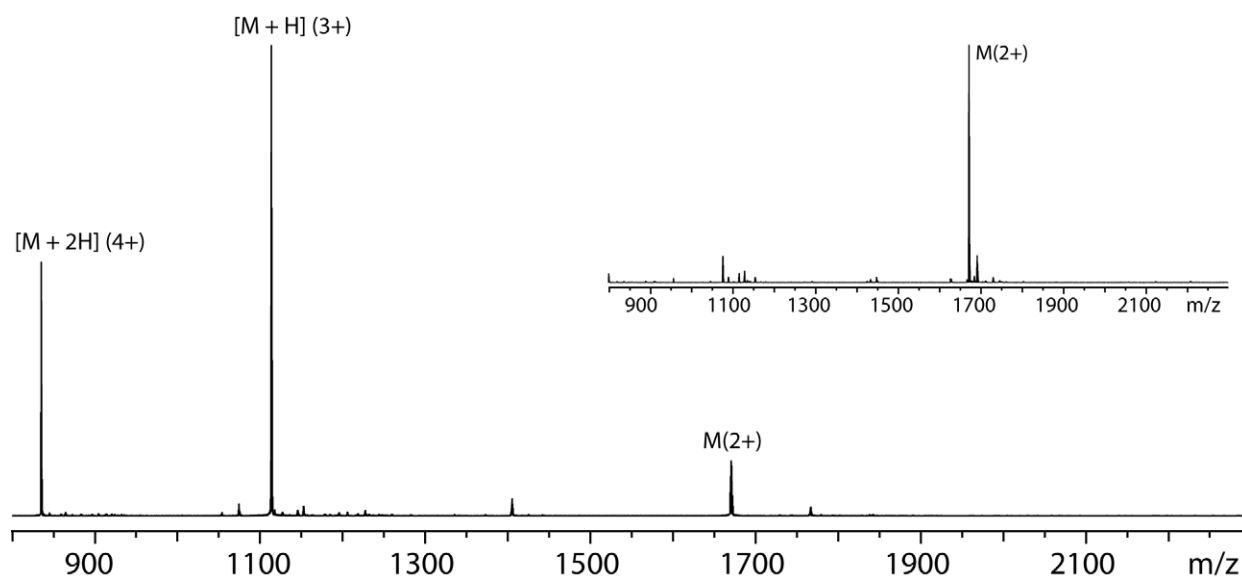


Figure S6. ESI-MS spectrum of **3** in the presence of excess of MeSO_3H . Inset – spectrum of **3** under acid-free conditions.

a = molar ratio complex : acid

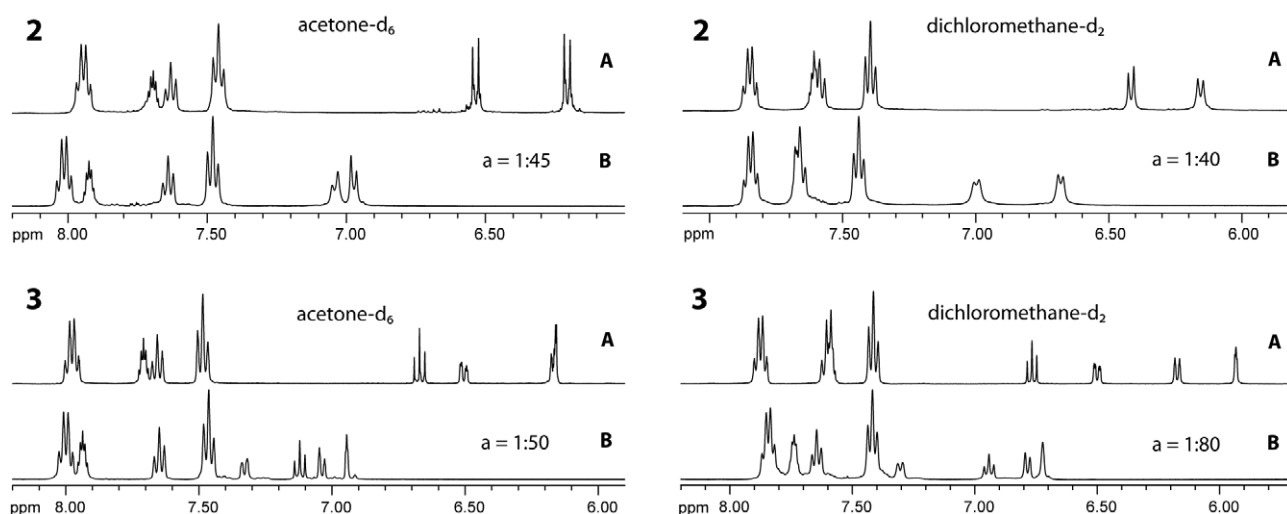


Figure S7. ^1H NMR spectra of **2** and **3** in the aromatic region: (A) under acid-free conditions; (B) in the presence of MeSO_3H , 400 MHz, 298 K.

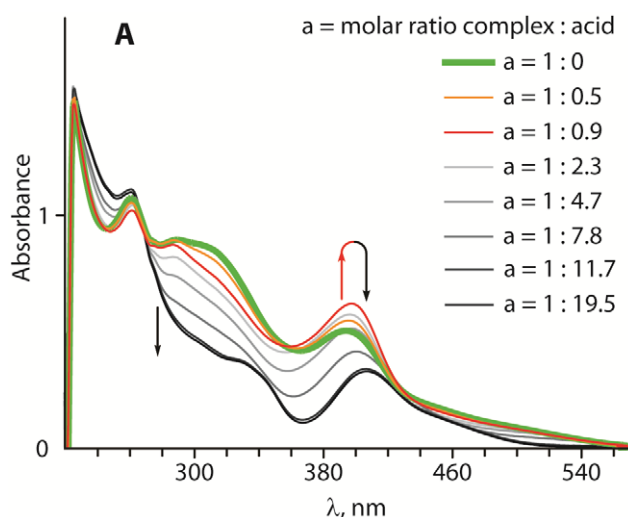


Figure S8 Changes of UV-vis absorption $C(\mathbf{1}) = 7.4 \cdot 10^{-6}$ M of **1** in CH_2Cl_2 upon addition of MeSO_3H , room temperature.

Computational results

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

Identification code	1
Empirical formula	$\text{C}_{154}\text{H}_{142}\text{Au}_6\text{Cu}_2\text{F}_{12}\text{N}_6\text{O}_2\text{P}_8$
Formula weight	3893.38
Temperature	100(2) K
Wavelength	0.71073 Å
Crystal system	Triclinic
Space group	P 1

Unit cell dimensions	a = 15.4033(2) Å b = 16.4211(3) Å c = 17.0705(2) Å	$\alpha = 77.2833(8)^\circ$ $\beta = 66.7103(9)^\circ$ $\gamma = 63.2520(8)^\circ$
Volume	3536.91(9) Å ³	
Z	1	
Density (calculated)	1.828 Mg/m ³	
Absorption coefficient	6.653 mm ⁻¹	
F(000)	1884	
Crystal size	0.20 x 0.18 x 0.11 mm ³	
Theta range for data collection	1.66 to 27.48°	
Index ranges	-19 ≤ h ≤ 19, -21 ≤ k ≤ 21, -22 ≤ l ≤ 22	
Reflections collected	70826	
Independent reflections	30295 [R(int) = 0.0280]	
Completeness to theta = 27.48°	99.5 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.4830 and 0.3221	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	30295 / 119 / 1764	
Goodness-of-fit on F ²	1.071	
Final R indices [I > 2sigma(I)]	R1 = 0.0267, wR2 = 0.0552	
R indices (all data)	R1 = 0.0316, wR2 = 0.0566	
Absolute structure parameter	0.090(3)	
Largest diff. peak and hole	1.107 and -1.090 e.Å ⁻³	

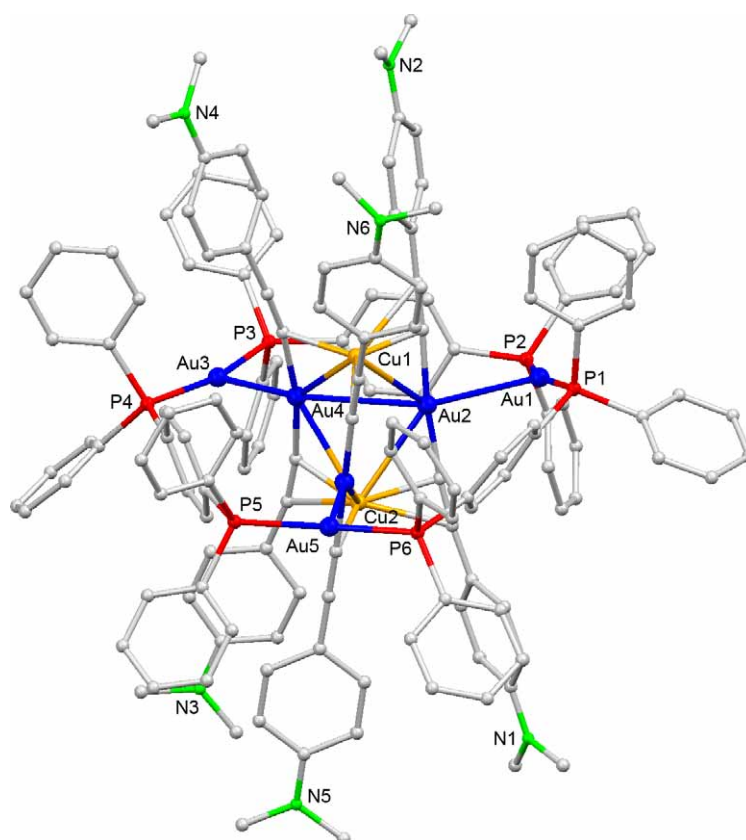


Figure S9. Molecular structure of the dication **1**. Counterions and hydrogen atoms omitted for clarity. Selected interatomic distances (Å) are: Au(1)-P(1) 2.3192(14), Au(1)-P(2) 2.3289(14), Au(1)-Au(2) 2.8449(3), Au(2)-Au(4) 3.2000(3), Au(3)-P(3) 2.3091(14), Au(3)-P(4) 2.3095(15), Au(3)-Au(4) 2.8209(3), Au(4)-Cu(1) 2.7361(7), Au(4)-Cu(2) 3.0492(7), Au(5)-P(6) 2.3171(14), Au(5)-P(5) 2.3300(15), Au(5)-Au(6) 2.8429(3), Au(6)-Cu(2) 2.7335(7), Au(6)-Cu(1) 3.1433(7).

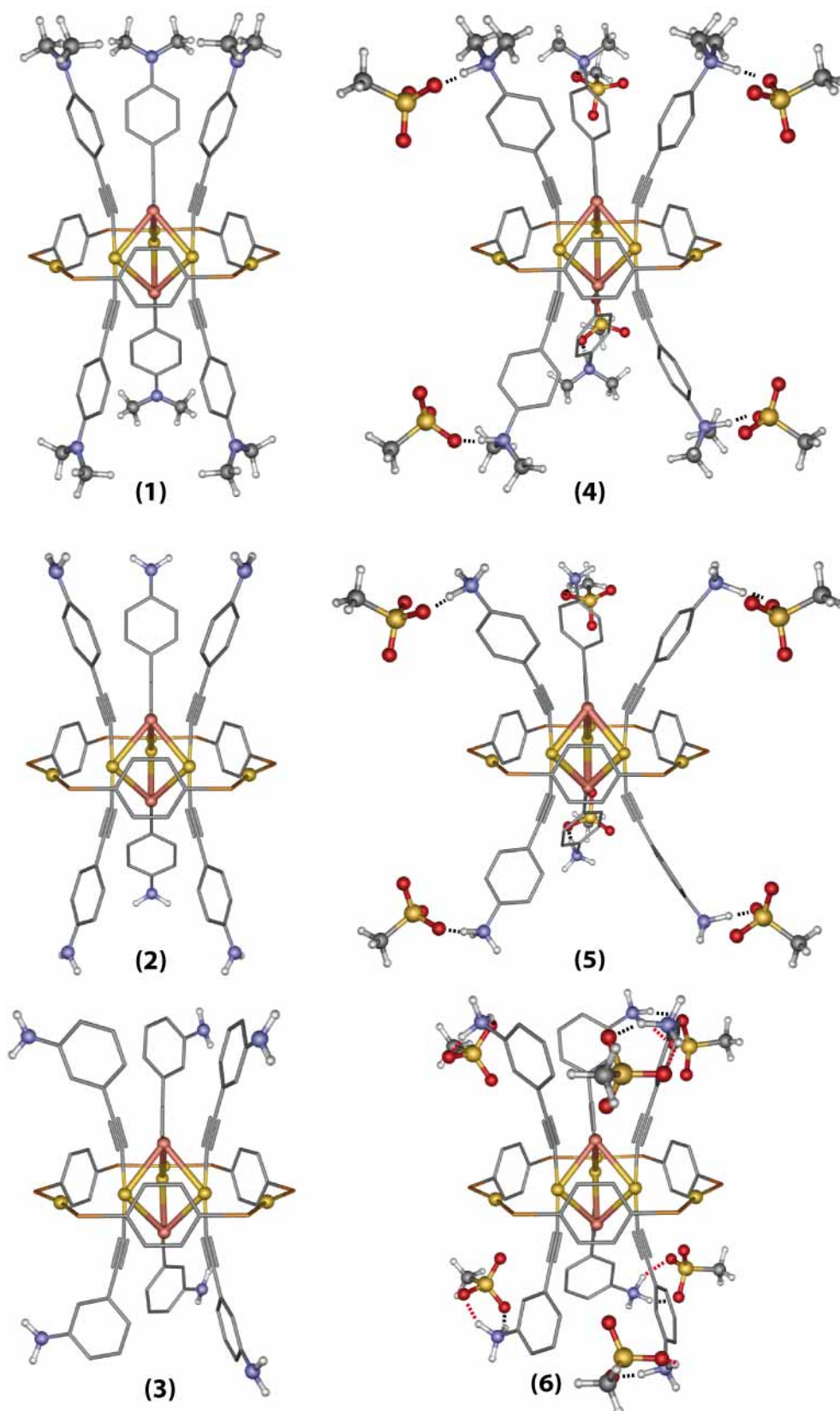


Figure S10. Optimized structures of the $\text{Au}^{\text{I}}\text{-Cu}^{\text{I}}$ clusters 1–6. Most hydrogen atoms and all diphosphine-based phenyl rings have been omitted for clarity. Primary $\text{O}\cdots\text{H}^+$ contacts are illustrated with black dots, while the secondary $\text{O}\cdots\text{H}^+$ contacts in the cluster 6 are shown with red dots.

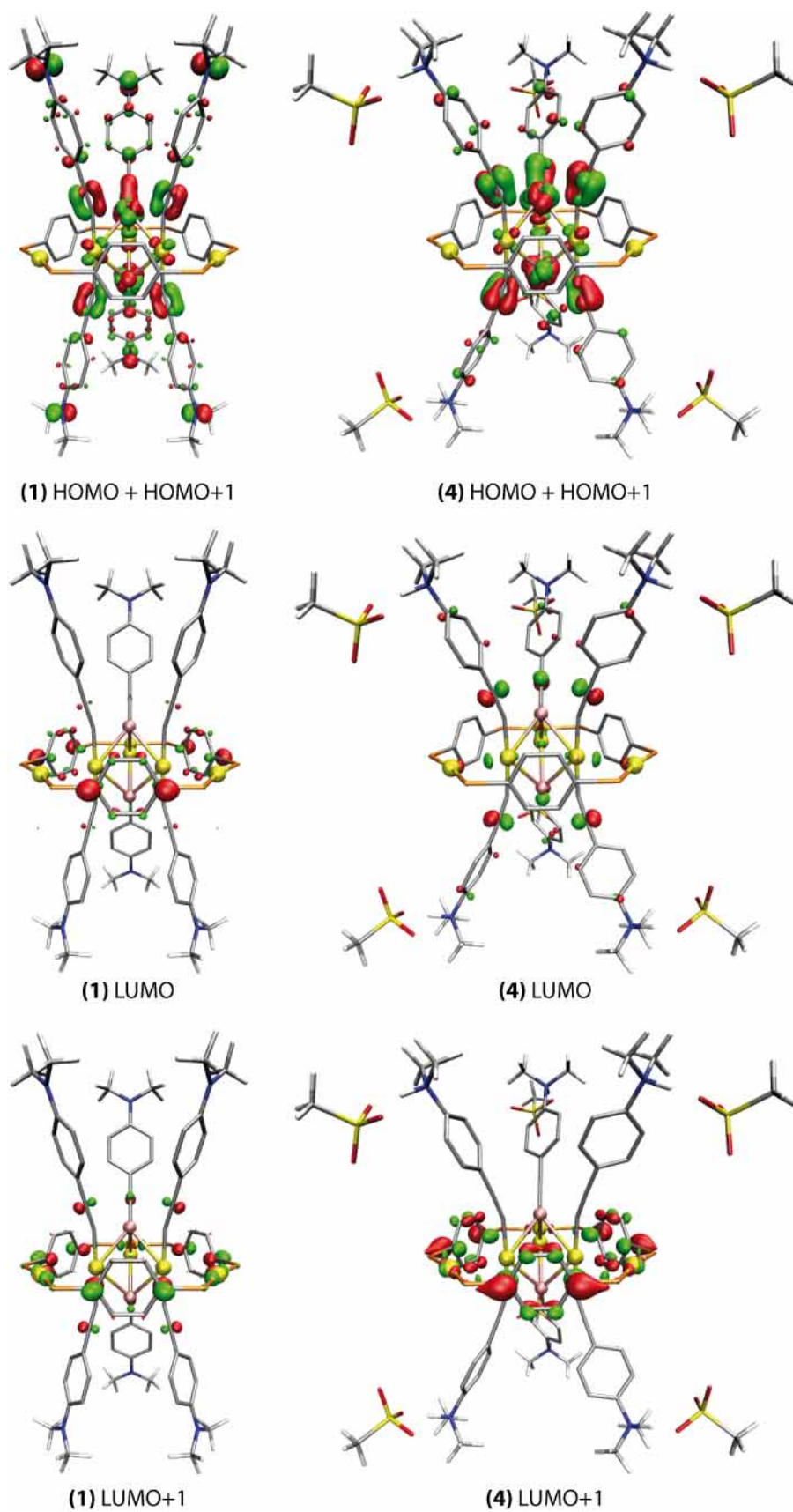


Figure S11. Frontier molecular orbital isodensity plots for the Au^I-Cu^I clusters **1** and **4** (isodensity value 0.04 a.u.). Most hydrogen atoms and all diphosphine-based phenyl rings have been omitted for clarity.

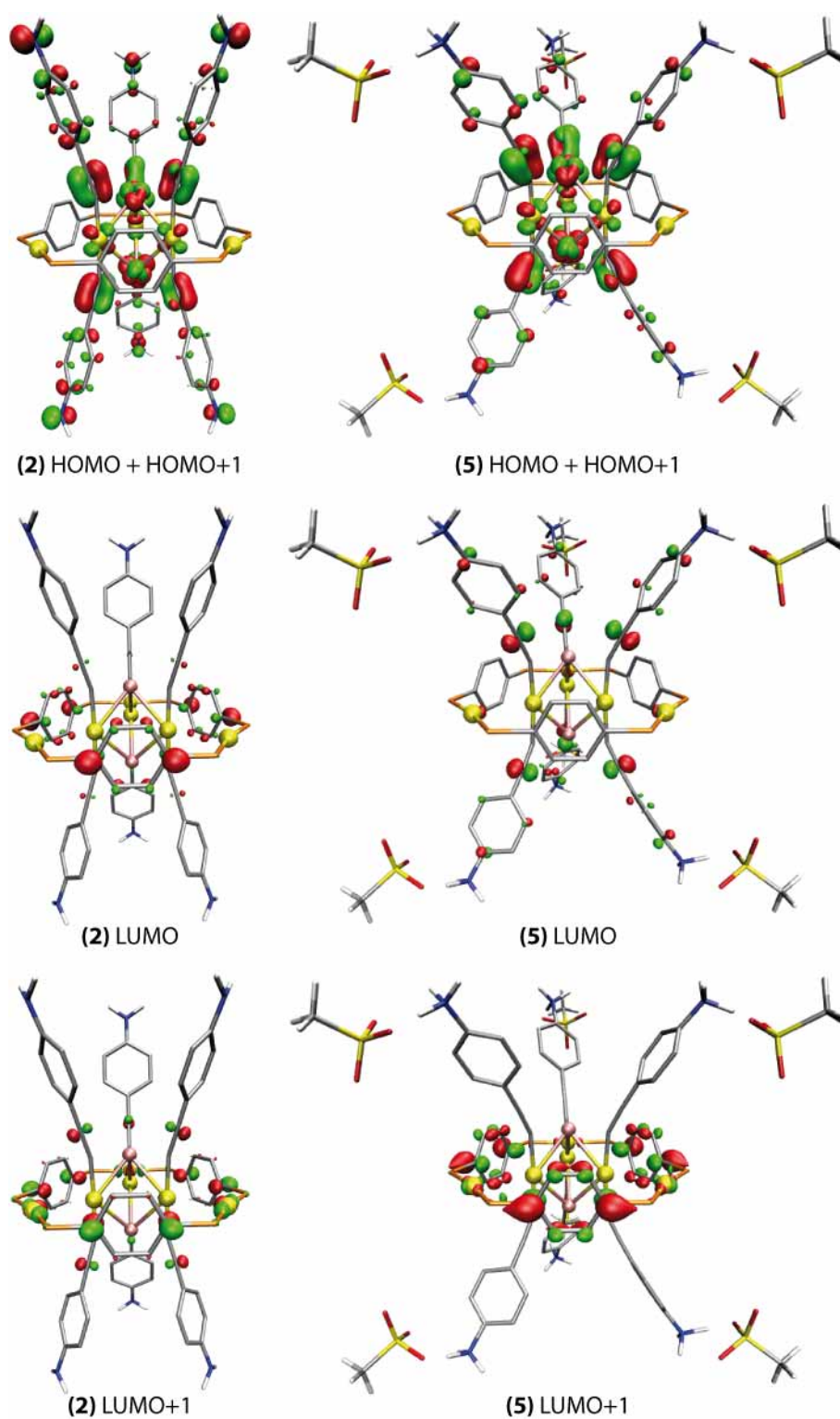


Figure S12. Frontier molecular orbital isodensity plots for the Au^I-Cu^I clusters **2** and **5** (isodensity value 0.04 a.u.). Most hydrogen atoms and all diphosphine-based phenyl rings have been omitted for clarity.

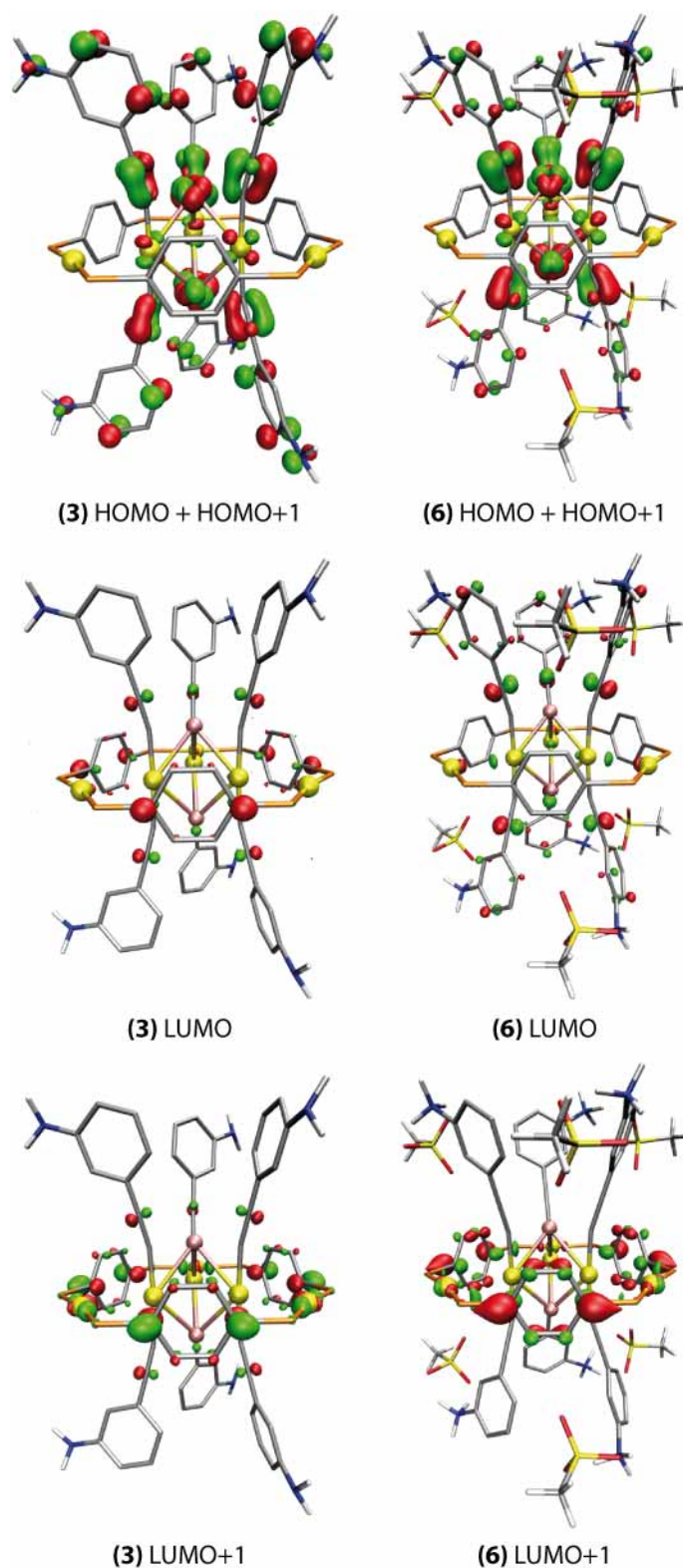


Figure S13. Frontier molecular orbital isodensity plots for the Au^I-Cu^I clusters **3** and **6** (isodensity value 0.04 a.u.). Most hydrogen atoms and all diphosphine-based phenyl rings have been omitted for clarity.

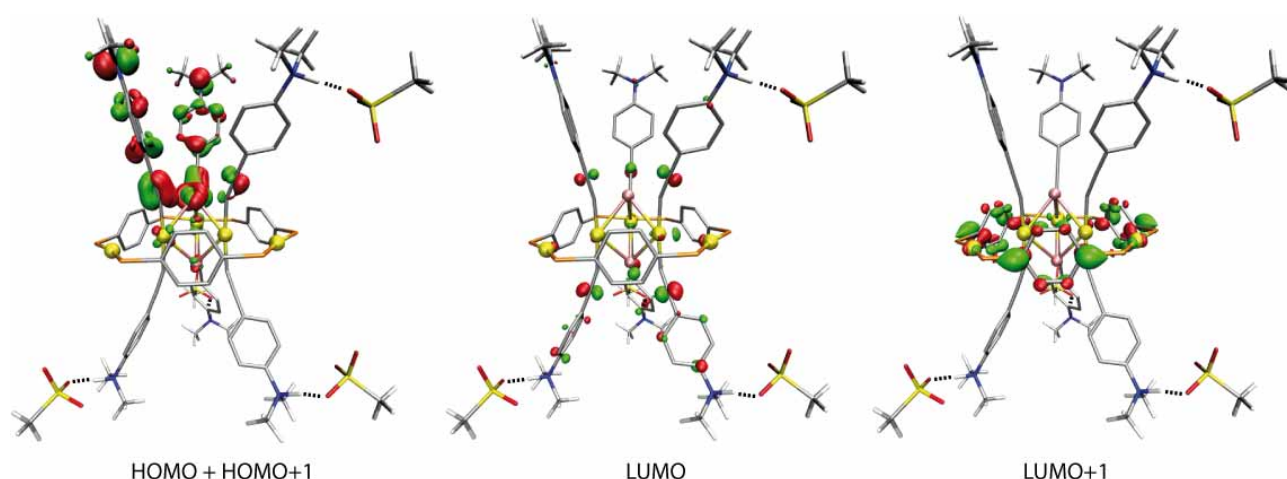
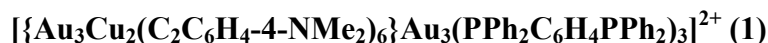


Figure S14. Frontier molecular orbital isodensity plots for the four-fold protonated Au^I-Cu^I cluster 1*(MeSO₃H)₄ (isodensity value 0.04 a.u.). Most hydrogen atoms and all diphosphine-based phenyl rings have been omitted for clarity.

Additional computational data

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



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13.11254421635751	8.84326232120555	-7.13126324387817	c
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8.72155865616405	16.73858766455002	14.50232543225089	c
7.01606206953773	14.20305329096789	13.00121496153075	s
10.76401171910811	16.46245180678178	14.13452718787767	h
8.30943480741082	16.70048296622583	16.55445725602999	h
4.29336024142759	14.40379007996884	13.68751445894946	o
8.26361746784664	11.81881764821214	14.37186298239161	o
7.67351317522905	14.23262913947484	10.27237429788146	o
6.89776222046193	10.27167704054679	14.83679431659083	h
-20.06891588614936	-2.28754868349371	13.63257764609754	h
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-18.85682146905523	-0.81620247544101	14.50232543225089	c
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7.67351317522905	14.23262913947484	-10.27237429788146	o
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6.89776222046193	10.27167704054679	-14.83679431659083	h
-12.34441436681393	0.83779879191111	-14.83679431659083	h
5.44665214635193	-11.10947583245808	-14.83679431659083	h

[{Au₃Cu₂(C₂C₆H₄-4-NH₂)₆*(MeSO₃H)₆}Au₃(PPh₂C₆H₄PPh₂)₃]²⁺ (5)

0.12845709242563	3.65487815789049	0.00000000000000	au
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0.62078611243310	9.24340818325105	0.00000000000000	au
7.69463324802787	-5.15932063530921	0.00000000000000	au
-8.31541936046093	-4.08408754794197	0.00000000000000	au
0.00000000000000	0.00000000000000	3.68580990792576	cu
0.00000000000000	0.00000000000000	-3.68580990792576	cu
-3.83666805778155	9.94926771086618	0.00000000000000	p
5.12175839803643	9.41685452731307	0.00000000000000	p
5.59435604537741	-9.14400014840236	0.00000000000000	p
10.53465261555308	-1.65198185150599	0.00000000000000	p
-10.71611444341380	-0.27285437891070	0.00000000000000	p
-6.69798455777158	-8.29728585936022	0.00000000000000	p
0.08440597689761	3.89114363721679	3.83574800288087	c
3.32762625115507	-2.01866953883303	3.83574800288087	c
-3.41203222805268	-1.87247409838378	3.83574800288087	c
0.08440597689761	3.89114363721679	-3.83574800288087	c
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13.87610913999920	-7.87018359908455	-5.37423858842262	c
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6.98117570421962	-10.19552188386670	-4.08915610213611	h
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15.81323332589419	-7.19461658767705	-5.73707572518803	h
9.38274647678625	-11.86838293392989	-7.81146001609531	h
13.82093032680483	-10.39561369433687	-8.62745325873041	h
-0.02082999983025	-12.92535031918133	-2.70565726487854	c
-2.37875149227305	-13.96257829485274	-3.40454686738626	c
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-4.13334491239722	-13.30203320652161	-2.49886468085703	h
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4.05309330953829	-13.05087687083574	-3.28814509608231	h
-0.27598681463130	-16.72555529562149	-6.42810631774112	c
-4.34708960820320	-16.63205742192200	-5.81683403522973	h
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-0.38023083047933	-18.20197375891838	-7.89423639365627	h
-11.76419490677440	-4.81004513376580	-2.79526860137296	c
-12.74139950857398	-2.95774163784100	-4.44434637016519	c
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-14.22263662102173	-3.65748360003469	-6.53492613923755	c
-12.32016880837859	-0.94811456620350	-4.08915610213611	h
-13.75383349925445	-8.08197122138251	-5.37423858842262	c
-11.47451242216475	-8.87916245497946	-2.07162297204234	h
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15.37696243148140	0.32397764819152	9.45000299259656	h
14.21106619566662	-0.21342311303011	11.10603904884954	c
11.14472582554817	1.24065256272254	10.73576280425666	s
13.94351191593315	-2.29030338073304	11.17447039398613	h
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11.59428523846237	4.07287674200722	10.72960767519313	o
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10.02796726568718	0.23250278632301	8.34460396913527	o
8.02769926013032	2.95616282936403	13.91026042543254	h
-7.96905408933288	13.15485127460608	9.45000299259656	h
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-6.92070326019443	12.41385589682462	11.10603904884954	c
-7.29036293547220	-12.20043278379452	11.10603904884954	c

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-6.57396173801769	5.47411007853235	13.91026042543254	h
-1.45373752211260	-8.43027290789638	13.91026042543254	h
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