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Reactivity of $[Re_3(\mu\text{-}H)_3(\mu_3\text{-ampy})(CO)_9]^-$. Preparation of Heteronuclear Re_3Au Carbonyl Cluster Complexes Containing Face-Capping and Edge-Bridging Gold Atoms

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*Summary: The reaction of the trirhenium anionic cluster $[Re_3(\mu\text{-}H)_3(\mu_3\text{-ampy})(CO)_9]^-$ (**1**; Hampy = 2-amino-6-methylpyridine) with the cationic gold fragment $[Au(PPh_3)]^+$ gives the neutral heteronuclear adducts $[Re_3\{\mu_3\text{-}Au(PPh_3)\}(\mu\text{-}H)_3(\mu_3\text{-ampy})(CO)_9]$ (**2**) and $[Re_3\{\mu\text{-}Au(PPh_3)\}(\mu_3\text{-}H)(\mu\text{-}H)_2(\mu_3\text{-ampy})(CO)_9]$ (**3**). In compound **2**, each hydride ligand spans an edge of the Re_3 triangle, which is capped by the gold atom, whereas in compound **3**, the gold atom spans a Re_2 edge, two hydride ligands span the two symmetry-related Re_2 edges, and the remaining hydride ligand caps the Re_2Au triangle.*

Introduction

Gold has little tendency to form hydride complexes. In fact, although a few heteronuclear complexes containing $M(\mu\text{-}H)Au$ fragments (M = Nb, Cr, Mo, W, Mn, Re, Ru, Rh, Ir, Pt) have been described,[1] no complexes of any type, homonuclear or heteronuclear, containing terminal hydride ligands attached to gold atoms have yet been reported. A nice example that points out the low tendency of gold to form hydride complexes, when compared to that of the other group 11 metals, is portrayed by the cluster complexes $[Ru_4\{M(PPh_3)\}(\mu\text{-}H)_3(CO)_{12}]$ (M = Cu, Ag, Au). While the compounds with M = Cu, Ag have the $M(PPh_3)$ fragment capping a triangular Ru_3 face with the hydrides capping the three Ru_2−M triangles,[2] the gold derivative has the $Au(PPh_3)$ fragment spanning a Ru−Ru edge with the three hydrides attached only to ruthenium atoms.[2a,b,3]

As far as heteronuclear complexes containing Re−Au bonds are concerned, only a few examples have been described. The organometallic derivatives are all carbonyl complexes, comprising trinuclear Re_2Au,[4,5] tetranuclear Re_3Au,[6] Re_2Au_2,[5] and $ReAu_3$,[7,8] pentanuclear Re_2Au_3[5a] and $ReAu_4$,[8] heptanuclear Re_6Au,[9] and octanuclear Re_7Au[10] complexes, but none of them contain hydride ligands (bridging or terminal) attached to the gold atoms. In addition, a few heteronuclear rhenium−gold complexes without metal−carbon bonds have been reported, and some contain hydride ligands bridging Re−Au bonds.[1b,d,11]

We have recently reported the synthesis of the anionic trihydride trirhenium carbonyl cluster $[HNEt_3][Re_3(\mu\text{-}H)_3(\mu_3\text{-ampy})(CO)_9]$ ($[HNEt_3]$**1**; Hampy = 2-amino-6-methylpyridine).[12] In this compound, each hydride ligand spans a different Re−Re edge of the Re_3 triangle, which is capped by the ampy ligand (Scheme 1). Its anionic nature (which makes it more prone to react with electrophilic reagents), the presence of the three hydride ligands, and the lack of previous examples of heteronuclear Re−Au carbonyl complexes in which the gold atoms interact with hydride ligands prompted us to study the reaction of compound **1** with the cationic gold fragment $[Au(PPh_3)]^+$.

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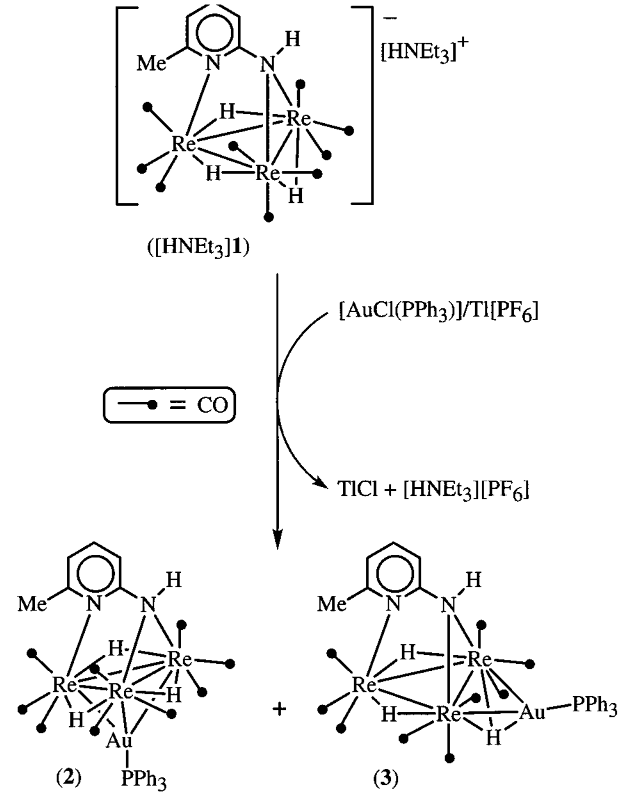
Scheme 1



Results and Discussion

Treatment of a THF solution of the anionic cluster compound **1** with the cationic fragment $[Au(PPh_3)]^+$ (prepared in situ from $[AuCl(PPh_3)]$ and $Tl[PF_6]$) gave a mixture of compounds from which two heteronuclear Re_3Au clusters, **2** and **3** (Scheme 1), in a 1:2 ratio, were isolated. The isomeric character of both compounds was unequivocally assigned after obtaining their microanalyses and FAB mass spectra.

The $^{31}P\{^1H\}$ and ^1H NMR spectra of both compounds indicated the presence of the PPh_3 and ampy ligands. The observation of two hydride resonances, in a 1:2 integral ratio, in the ^1H NMR spectra of both compounds suggested the presence of three hydride ligands and the existence of a symmetry plane within the molecules (C_s symmetry). Interestingly, while both hydride resonances of compound **2**, δ −9.92 (s, 1 H) and −10.01 (d, $J_{H-P} =$ 1.9 Hz, 2 H) ppm, appear at chemical shifts typical of edge-bridging hydride ligands in saturated trirhenium carbonyl complexes ($\delta < -9$ ppm),[12–14] only one of the hydride resonances of compound **3** appears in the same region, δ −10.24 (s, 2 H) ppm; the other is observed at an unusual chemical shift, δ −5.34 (d, $J_{H-P} =$ 58.8 Hz, 1 H) ppm. In trirhenium carbonyl cluster chemistry, such high-frequency hydride resonances have only been observed for edge-bridging hydrides in unsaturated clusters[6,13,14] or for terminal hydrides.[15] In addition, chemical shifts in the range −3 to −6 ppm have been reported for the bridging hydrides of some heteronuclear Re−Au nonorganometallic complexes.[11]

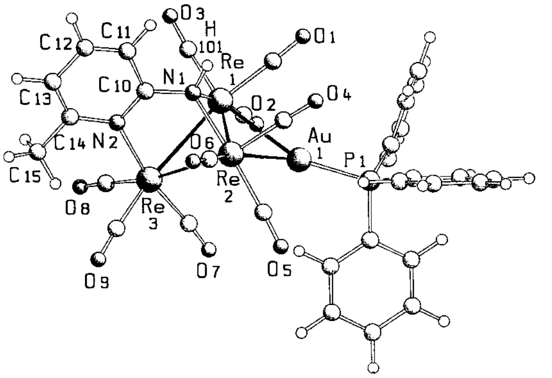


Figure 1. Molecular structure of $[Re_3\{\mu\text{-}Au(PPh_3)\}(\mu_3\text{-}H)(\mu\text{-}H)_2(\mu_3\text{-ampy})(CO)_9]$ (**3**).

The very small J_{H-P} couplings observed in the hydride resonances of compound **2** suggest that the hydrides are far away from the phosphine ligand, in other words, that the gold atom of **2** is not attached to any of the hydrides. Interestingly, the large coupling observed in the signal at δ −5.34 ppm of compound **3** ($J_{H-P} =$ 58.8 Hz) clearly indicates that the hydride ligand responsible for this resonance is bonded to the gold atom. The large value of this J_{H-P} coupling is in agreement with a *transoid* arrangement of the hydride and phosphine ligands on the gold atom.[1]

All these data strongly (but not unambiguously) support the structures proposed for both cluster complexes in Scheme 1. In the case of complex **2**, an alternative structure having the $Au(PPh_3)$ group spanning the same edge as the NH moiety of the ampy ligand, with the three hydrides bridging Re−Re edges, cannot be completely ruled out. However, we propose that **2** has the $Au(PPh_3)$ capping the Re_3 face because (a) if the three hydrides are only attached to rhenium atoms in edge-bridging equatorial positions (as suggested by the NMR spectrum), the $Au(PPh_3)$ group should be in an axial position (bridging or face-capping), (b) there is a considerable amount of experimental evidence available that suggests that, excluding sterical arguments, the energy difference between the edge-bridging and the face-capping bonding modes for $Au(PPh_3)$ groups in metal clusters is very small,[1b,16] and (c) in the resulting structure of **2**, the face-capping position is less crowded than the axial edge-bridging position. Unfortunately, we could not get crystals of this compound suitable for an X-ray diffraction study, which would definitely determine its structure.

Compound **3** was studied by X-ray diffraction methods. Its molecular structure is shown in Figure 1. Relevant bond distances and angles are given in Table 1. The $Re_3(\mu_3\text{-ampy})(CO)_9$ unit is reminiscent of that of the parent anion **1**. The $Au(PPh_3)$ group spans the Re(1)−Re(2) edge in such a way that the Re(1)−Re(2)−Au(1) plane forms a dihedral angle of 113.2(1)° with the Re_3 plane. This results in a lengthening by ca. 0.08 Å of the Re(1)−Re(2) distance, with respect to that of **1**, as a consequence of the larger size of the bridgehead

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Table 1. Relevant Bond Lengths (Å) and Angles (deg) in $[Re_3\{\mu\text{-}Au(PPh_3)\}(\mu_3\text{-}H)(\mu\text{-}H)_2(\mu_3\text{-}ampy)(CO)_9$ (3)

Au(1)−Re(1)	2.884(1)	Re(1)−Re(2)−Re(3)	61.79(3)
Au(1)−Re(2)	2.930(1)	Re(1)−Re(3)−Re(2)	56.66(2)
Au(1)−P(1)	2.250(4)	Re(2)−Re(1)−Re(3)	61.56(3)
Re(1)−Re(2)	3.008(1)	Re(1)−Au(1)−Re(2)	62.30(3)
Re(1)−Re(3)	3.173(1)	Au(1)−Re(1)−Re(3)	93.32(3)
Re(2)−Re(3)	3.166(1)	Au(1)−Re(2)−Re(3)	92. 57(3)
Re(1)−N(1)	2.219(1)	Au(1)−Re(1)−Re(2)	59.61(3)
Re(2)−N(1)	2.178(1)	Au(1)−Re(2)−Re(1)	58.09(3)
Re(3)−N(2)	2.200(1)	Re(1)−Au(1)−P(1)	147.3(1)
Mean Re−C	1.93(3)	Re(2)−Au(1)−P(1)	148.8(1)
Mean C−O	1.14(3)		

atom (gold vs hydrogen).[17] Unfortunately, the hydride ligands could not be directly located on the basis of the X-ray diffraction data. As far as we are aware, only one structural characterization of a Re_3Au carbonyl cluster has been previously reported, namely, $[PPh_4][Re_3\{\mu_3\text{-}Au(PPh_3)\}(\mu\text{-}H)_3(CO)_9]$.[6] This unsaturated compound has the gold atom capping the Re_3 triangle, with the hydrides spanning the Re−Re edges.

To have some clues about the position of the hydride ligands in **3**, we decided to use XHYDEX,[18] a computer program based on potential energy minima calculations, widely used for assigning the position of hydrides of heavy metal polynuclear complexes from diffraction data.[19] The structure of minimum energy corresponded to one having two hydrides spanning the Re(1)−Re(3) and Re(2)−Re(3) edges in approximately the same positions as in the parent anion **1**, with the remaining hydride attached to the gold atom in a terminal position, *cis* to the PPh_3 ligand, pointing away from the ampy ligand. As this situation is in conflict with the NMR data (the J_{H-P} coupling constant is in excellent agreement with a *transoid* H−Au−P arrangement), we also carried out potential energy calculations assigning to the problematic hydride a capping position on the Re_2Au face (Scheme 1). This structure, in which the hydride and phosphine ligands are in a *transoid* arrangement, was only 2 kcal/mol less stable than that having the hydride in a terminal position on the Au atom. The energy difference between the two possible structures being so small, we feel inclined to propose, on the basis of the NMR data, that, at least in solution, compound **3** adopts the structure depicted in Scheme 1, which contains a hydride capping a Re_2Au triangle.

Searching in the Cambridge Crystallographic Database, looking for compounds containing gold and problematic hydride ligands (as far as their location is concerned), we found an Os_3Au cluster, namely, $[Os_3\{\mu\text{-}Au(PPh_3)\}(\mu\text{-}H)\{\mu_3\text{-}Ph_2PCH_2PPh(C_6H_4)\}(CO)_8][PF_6]$,[20] which has the gold atom bridging one edge of the Os_3 triangle. The hydride ligand could not be located by diffraction methods (the structure was obtained using synchrotron radiation), but it was assigned to a bridging position between two osmium atoms on the basis of NMR data (no coupling is observed between the hydride and the phosphorus atom of the PPh_3 ligand). We carried out XHYDEX calculations on this compound, finding that the energy of the structure having the hydride bridging two Os atoms was similar to that of other structure having the hydride in a terminal position on the gold atom.

The origin of the formation of the two isomeric products **2** and **3** in the reaction of **1** with $[Au(PPh_3)]^+$ seems to be of kinetic character, being related to the attack of the $[Au(PPh_3)]^+$ fragment to the rhenium atoms Re(1) and Re(2) via either side of the Re(1)−H−Re(2) plane of compound **1**. In fact, compounds **2** and **3** do not interconvert into each other in solution at room temperature (they are stable and static in the NMR time scale) or in refluxing THF (wherein **3** is stable and **2** decomposes into a mixture of many products). This is surprising in view of the usual behavior exhibited by gold−phosphine units in heteronuclear clusters.[1] The characerization of two isomers of the compound $[Ru_5C\{Au(PEt_3)\}(NO)(CO)_{13}]$ represented the first example of skeletal isomerism (edge-bridging or face-capping gold−phosphine unit) involving a heteronuclear gold cluster.[21]

Concluding Remarks

In conclusion, this paper reports two novel organometallic Re_3Au cluster complexes, comprising the first example within this family of compounds in which a hydride ligand is attached to the gold atom.

Experimental Section

General Data. Solvents were dried over sodium diphenyl ketyl (THF, hydrocarbons) or calcium hydride (dichloromethane) and distilled under nitrogen prior to use. Compounds $[HNEt_3]$-**1**[12] and $[AuCl(PPh_3)]$[22] were prepared by published procedures; all other reagents were purchased as analytically pure samples. Instrumentation was as previously reported.[12]

Synthesis of Compounds 2 and 3. $Tl[PF_6]$ (44.4 mg, 0.127 mmol) was added to a solution of $[HNEt_3]$**1** (100 mg, 0.089 mmol) and $[AuCl(PPh_3)]$ (58.1 mg, 0.112 mmol) in THF (10 mL). A white precipitate (TlCl) was immediately observed. The reaction mixture was stirred at room temperature for 30 min. The solid was removed by filtration through Celite. The yellow-orange filtrate was concentrated to ca. 2 mL and was layered on preparative silica gel TLC plates. Elution with 1:1 dichloromethane−hexane afforded two bands, yellow-brown (first) and yellow (second), which were extracted with dichloromethane. The extracts were evaporated to dryness and the residues were crystallized from dichloromethane−pentane to give compound **3** from the first band (45 mg, 37%) and compound **2** from the second band (22 mg, 18%). *Analytical and spectroscopic data for **2***: Anal. Found: C, 28.88; H, 1.97; N, 1.93. Calcd for $C_{33}H_{25}AuN_2O_9PRe_3$ (MW 1380.14): C, 28.72; H, 1.83; N, 2.03. FAB-MS (*m/z*): 1380 $[M^+]$. IR (CH_2Cl_2): 2029 (w), 2002 (s), 1995 (s), 1967 (w), 1938 (w), 1913 (vs), 1895 (s, br) cm^{-1}. ^1H NMR ($CDCl_3$): δ 7.4−6.7 (m, 16 H, 3 Ph + 1 ampy-C*H*), 6.48 (d, 7.7 Hz, 1 H, ampy-C*H*), 6.41 (d, 7.7 Hz, 1 H, ampy-C*H*), 4.37 (s, br, 1 H, ampy-N*H*), 2.69 (s, 3 H, ampy-Me), −9.92 (s, 1 H, μ-H), −10.01 (d, 1.9 Hz, 2 H, μ-H) ppm. $^{31}P\{^1H\}$ NMR ($CDCl_3$): δ 59.0 (s) ppm. *Analytical and spectroscopic data for **3***: Anal. Found: C, 28.75; H, 1.90; N, 1.96. Calcd for $C_{33}H_{25}AuN_2O_9PRe_3$ (MW 1380.14): C, 28.72; H, 1.83; N, 2.03. FAB-MS (*m/z*): 1380 $[M^+]$. IR (CH_2Cl_2): 2042 (w), 2015 (vs), 1991 (w), 1981 (w), 1942 (m), 1922 (s, br) cm^{-1}. ^1H NMR ($CDCl_3$): δ 7.7−7.5 (m, 15 H, 3 Ph), 7.35 (t, 7.7 Hz, 1 H, ampy-C*H*), 6.70 (d, 7.7 Hz, 1 H, ampy-C*H*), 6.56 (d, 7.7 Hz, 1

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Table 2. Crystallographic and Refinement Data for 3

formula	$C_{33}H_{25}AuN_2O_9PRe_3$
fw	1380.09
cryst syst	triclinic
space group	$P\bar{1}$
a, Å	13.075(3)
b, Å	13.822(2)
c, Å	14.381(2)
α, deg	72.41(2)
β, deg	86.13(1)
γ, deg	64.06(1)
volume, Å^3	2221.5(7)
Z	2
F(000)	1256
D_{calcd}, g/cm^3	2.063
μ, mm^{-1}	11.518
radiation (λ, Å)	Mo Kα (0.71069)
monochromator	graphite
cryst size, mm	0.18 × 0.15 × 0.15
temp, K	293(2)
θ limits, deg	3.00−23.0
min/max h, k, l	−14/14, −14/15, 0/15
no. of reflns collected	6333
no. of unique reflns	6052
no. of reflns with $I > 2\sigma(I)$	4448
no. of refined params	407
GOF (on F^2)	1.003
R_1 (on F, $I > 2\sigma(I)$)	0.0625
wR_2 (on F^2, all data)	0.1786

H, ampy-C*H*), 4.50 (s, br, 1 H, ampy-N*H*), 2.76 (s, 3 H, ampy-Me), −5.34 (d, 58.8 Hz, 1 H, μ_3-*H*), −10.24 (s, 2 H, μ-H) ppm. ^{31}P{^{1}H} NMR (CDCl$_3$): δ 55.9 (s) ppm. ^{13}C{^{1}H} NMR (CD$_2$Cl$_2$): δ(CO) 195.95 (1 C) 190.85 (2 C), 190.29 (2 C), 185.21 (2 C), 183.69 (2 C); δ(ampy) 179.87, 160.65, 138.56, 119.24, 113.00, 31.27 ppm.

Crystal Structure of Compound 3. X-ray diffraction data collection was carried out on a Nonius CAD-4 diffractometer equipped with a graphite monocromator (Mo Kα radiation, λ = 0.71069 Å). Crystal data and details of measurements are summarized in Table 2. Diffraction data were corrected for absorption by azimuthal scanning of high-χ reflections (min, max transmission 0.58, 1.00). SHELXL97[23a] was used for structure solution and refinement based on F^2. SCHAKAL97[23b] was used for the graphical representation of the results. All non-H atoms were refined anisotropically. The H atoms bound to C atoms were added in calculated positions and were refined riding on the corresponding C atoms. The hydride-H atom positions were calculated with XHYDEX.[18]

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Supporting Information Available: Tables of crystallographic and refinement data, bond distances and angles, fractional atomic coordinates, anisotropic thermal parameters, and an ORTEP picture of compound **3**. This material is available free of charge via the Internet at http://pubs.acs.org.

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