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Electrophilic Additions of $AuPR_3^+$ and $Au_2(\mu\text{-}L)^{2+}$ (L = Diphosphine) Units to the $[Fe_3(CO)_{11}]^{2-}$ Anion

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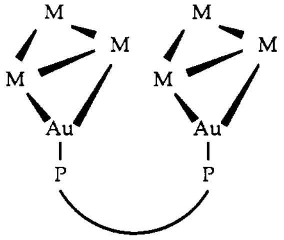
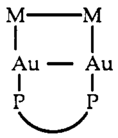
Summary: The reaction of $(PPh_4)_2[Fe_3(CO)_{11}]$ *with* $(AuCl)_2(\mu\text{-}L)$ *(L = dppm, dppe) or* $AuClPR_3$ *(R = Ph, Me) in tetrahydrofuran invariably gives mixed iron/gold clusters in which the gold atoms occupy edge-bridging positions, giving an* Fe_3Au *butterfly arrangement. The complex* $(PPh_4)[Fe_3Au(CO)_{11}(PPh_3)]$ *(**3**) has been characterized by X-ray crystallography. Crystals of **3** are monoclinic, of space group* $P2_1/c$ *with unit cell parameters of* $a = 17.532(2)$ Å, $b = 13.433(1)$ Å, $c = 21.373(3)$ Å, $\beta = 95.01(2)°$, $Z = 4$, *and* $V = 5014(2)$ Å^3. *The nature of the resulting products is rationalized in terms of previous MO calculations.*

Introduction

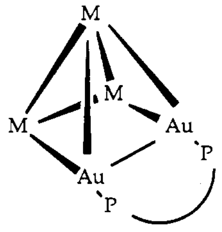
Heteronuclear clusters containing one group-11 metal fragment are well-known.[1] However, the use of bimetallic cationic fragments, such as $[Au_2(\mu\text{-}Ph_2P(CH_2)_nPPh_2)]^{2+}$ ($n = 1$–3), has been more limited despite the fact that they are particularly suitable for linking carbonyl cluster anions as a method of generating higher nuclearity cluster complexes. To date, the digold moieties can link two cluster units in a bis-μ_2 mode (A)[2] or can incorporate them into a preformed cluster. In this case, the two gold atoms of the chelating phosphine can (i) link two adjacent transition metal atoms, via a 2c–2e bond, giving a metallocycle compound (B),[3] (ii) bridge two adjacent edges of one of the triangular faces of the polyhedra (C),[4] and (iii) cap adjacent faces of the starting transition metal cluster in a μ_3 mode (D).[5] Another more complex structural framework is found in the compound $[Ru_5Au_2C(CO)_{14}(\mu\text{-}dppm)]$,[2] in which an Ru_2Au triangular face is capped by a gold phosphine fragment (E).[6] Interestingly, the only report describing an equilibrium between A and B involves the reaction between the tetrametallic $[Fe_2Au_2(CO)_8(\mu\text{-}dppe)]$ cluster (B) with 1 equiv of $[Fe_2(CO)_8]^{2-}$ to afford the hexametallic compound $(NEt_4)_2[\{Fe_2Au(CO)_8\}_2(\mu\text{-}dppe)]$ (A),[3] which, in turn, reverts to the starting tetrametallic cluster after reaction with 1 equiv of $[(AuCl)_2(\mu\text{-}dppe)]$. Surprisingly, the nearly square cluster $[Fe_2Au_2(CO)_8(\mu\text{-}dppm)]$ was inert to such a process. These findings prompted us to study the reaction between the cations $[Au_2(\mu\text{-}L)]^{2+}$ (L = bidentate phosphine) and the anion $[Fe_3(CO)_{11}]^{2-}$ in order to determine whether the formal substitution of one carbonyl group from $[Fe_2(CO)_8]^{2-}$ by one $Fe(CO)_4$ fragment would result in percebtible changes in reactivity.



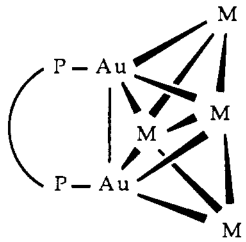
A



B



C



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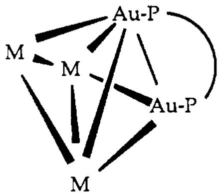

E

The reaction of $(PPh_4)_2[Fe_3(CO)_{11}]$ and $(AuCl)_2(\mu\text{-}L)$ (L = dppm, dppe) in tetrahydrofuran (thf) at −10 °C afforded a dark red solution from which the complexes $(PPh_4)_2[\{Fe_3Au(CO)_{11}\}_2(\mu\text{-}L)]$ were isolable along with $(PPh_4)Cl$ according to (1).

The nature of the final products was not dependent on the molar ratio of the reagents, and consequently, attempts to obtain pentametallic species containing an Fe_3Au_2 core by using a large excess of the gold derivative were unsuccessful. On the other hand, the reaction of the trimetallic iron anion and an excess of the monogold halide

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⊗ Abstract published in *Advance ACS Abstracts,* April 1, 1994.

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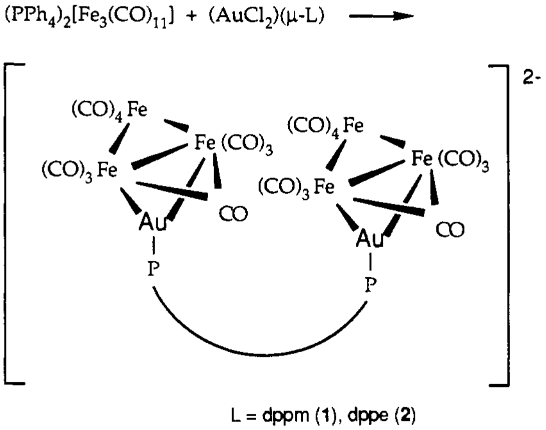

(1)

$\mathrm{AuClPPh_3}$ (1:3 molar ratio) gave the tetranuclear compound $\mathrm{(PPh_4)[Fe_3Au(CO)_{11}(PPh_3)]}$ (**3**) and no trace of the expected pentametallic $\mathrm{[Fe_3Au_2(CO)_{11}(PPh_3)_2]}$ cluster was detected. The analogous $\mathrm{(PPh_4)[Fe_3Au(CO)_{11}(PMe_3)]}$ (**4**) was also the only product synthesized from the much less sterically congested gold derivative $\mathrm{AuClPMe_3}$. The new metal clusters were characterized analytically and spectroscopically (see Experimental Section). Compounds **1**–**4** show an identical ν(CO) pattern in the IR spectrum, indicating that the metal framework is the same in all cases. Although we were not able to obtain single crystals of **1** or **2**, the X-ray crystal structure determination of **3** confirmed the proposed structure. The structure of the anion of **3**, $\mathrm{[Fe_3Au(CO)_{11}(PPh_3)]^-}$, is shown in Figure 1, while selected bond and angle parameters are listed in Table 1. The metal core consists of an $\mathrm{Fe_3Au}$ butterfly arrangement in which the $\mathrm{AuPPh_3}$ fragment bridges one edge of the $\mathrm{Fe_3}$ triangle. The resulting butterfly angle for this cluster is 120.1°, analogous to that reported for $\mathrm{[Fe_3Au(CO)_9(PPh_3)(\mu_3\text{-}SPr^i)]}$ (123.9°).[7] The iron-gold distances [2.653(1), 2.632(1) Å] compare very well with those recently reported for the thiolate-bridged clusters $\mathrm{[Fe_3Au(CO)_9(PPh_3)(\mu_3\text{-}SPr^i)]}$ [2.653(4), 2.645(5) Å][7] and $\mathrm{[Fe_2Au(CO)_7(PPh_3)(\mu\text{-}SPr^i)]}$ [2.644(1), 2.69(5) Å].[8] However, they are longer than those found in the 2c–2e metal–metal bonded complexes $\mathrm{[Fe_2Au_2(CO)_8(\mu\text{-}dppm)]}$ [2.534(2) and 2.527(2) Å][3] and $\mathrm{[AuFe\{Si(OMe)_3\}(CO)_3(\mu\text{-}dppm)]_2{\cdot}CH_2Cl_2}$ [2.535(3) and 2.562(3) Å],[9] as expected.

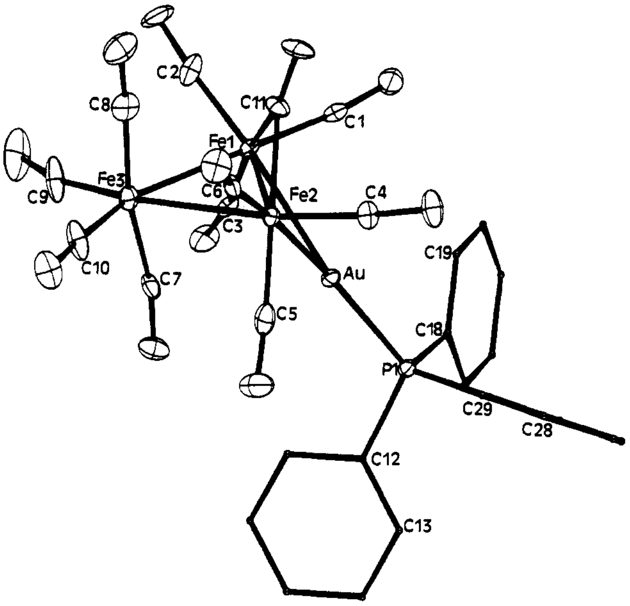

Figure 1. View of the molecular structure of the anion $\mathrm{[Fe_3Au(CO)_{11}(PPh_3)]^-}$ together with the atomic numbering scheme.

Table 1. Selected Bond Distances (Å) and Angles (deg) for 3

Fe(1)–Au	2.653(1)	C(9)–Fe(3)	1.767(3)
Fe(2)–Au	2.632(1)	C(10)–Fe(3)	1.723(3)
P(1)–Au	2.281(1)	O(1)–C(1)	1.150(3)
Fe(2)–Fe(1)	2.632(1)	O(2)–C(2)	1.161(4)
Fe(3)–Fe(1)	2.682(1)	O(3)–C(3)	1.158(3)
C(1)–Fe(1)	1.737(2)	O(4)–C(4)	1.176(3)
C(2)–Fe(1)	1.752(3)	O(5)–C(5)	1.161(4)
C(3)–Fe(1)	1.803(2)	O(6)–C(6)	1.164(3)
C(11)–Fe(1)	1.924(2)	O(7)–C(7)	1.177(3)
Fe(3)–Fe(2)	2.652(1)	O(8)–C(8)	1.126(4)
C(4)–Fe(2)	1.718(2)	O(9)–C(9)	1.132(4)
C(5)–Fe(2)	1.769(3)	O(10)–C(10)	1.208(4)
C(6)–Fe(2)	1.773(2)	O(11)–C(11)	1.161(2)
C(11)–Fe(2)	1.983(2)		
C(7)–Fe(3)	1.759(2)		
C(8)–Fe(3)	1.761(3)		
Fe(2)–Au–Fe(1)	59.7(1)		
P(1)–Au–Fe(1)	149.4(1)	O(5)–C(5)–Fe(2)	175.1(2)
P(1)–Au–Fe(2)	150.6(1)	O(6)–C(6)–Fe(2)	171.3(2)
Fe(2)–Fe(1)–Au	59.7(1)	O(7)–C(7)–Fe(3)	169.4(2)
Fe(3)–Fe(1)–Au	97.0(1)	O(8)–C(8)–Fe(3)	176.9(2)
Fe(3)–Fe(1)–Fe(2)	59.9(1)	O(9)–C(9)–Fe(3)	166.0(4)
O(1)–C(1)–Fe(1)	172.4(2)	O(10)–C(10)–Fe(3)	173.7(2)
O(2)–C(2)–Fe(1)	173.4(2)	Fe(2)–C(11)–Fe(1)	84.7(1)
O(3)–C(3)–Fe(1)	170.6(2)	O(11)–C(11)–Fe(1)	139.7(2)
O(4)–C(4)–Fe(2)	174.5(2)	O(11)–C(11)–Fe(2)	135.1(2)

From the results reported here, it is noteworthy that, in contrast to the behavior described for the $\mathrm{[Fe_2(CO)_8]^{2-}}$ anion, the reaction of the analogous salt $\mathrm{(PPh_4)_2[Fe_3(CO)_{11}]}$ with the digold derivatives $\mathrm{(AuCl)_2(\mu\text{-}L)}$ only led to compounds containing two independent metal clusters, in which the gold atoms invariably occupy an edge-bridging position, joined by the bridging ligand. These observations are in agreement with MO calculations in that only an unbridged $\mathrm{[Fe_2(CO)_8]^{2-}}$ fragment is compatible with a structure containing terminal Fe–Au bonds.[3] Instead of this, a $\mathrm{[Fe_2(CO)_6(\mu\text{-}CO)_2]^{2-}}$ fragment is compatible with the bridging $\mathrm{[AuL]^+}$ fragment found in the triangular species $\mathrm{[Fe_2Au(CO)_6(\mu\text{-}CO)_2(PPh_3)]^-}$,[10] or in the clusters $\mathrm{[Fe_2Au(CO)_6(\mu\text{-}CO)(PPh_3)(\mu\text{-}PhC{=}CHPh)]}$,[11] $\mathrm{[Fe_2Au(CO)_6(\mu\text{-}CO)(PPh_3)(\mu\text{-}SPr^i)]}$,[8] and $\mathrm{[Fe_2Au(CO)_6(\mu\text{-}CO)(PPh_3)(\mu\text{-}PPh_2)]}$,[12] which can be formally seen as the result of the substitution of one bridging carbonyl from the $\mathrm{[Fe_2Au(CO)_6(\mu\text{-}CO)_2(PPh_3)_2]^-}$ for a three-electron L ligand. As has been clearly described,[13] this is easily understod by viewing unbridged $\mathrm{[Fe_2(CO)_8]^{2-}}$ as two vertex-sharing $\mathrm{[Fe(CO)_4]^-}$ octahedra with an Fe–Fe bond (Chart 1). The two base sites can coordinate to the digold unit. On the other hand, $\mathrm{[Fe_2(CO)_6(\mu\text{-}CO)_2]^{2-}}$ is considered as two edge-sharing octahedra, thereby producting a "bridging" electron pair for coordination to a single gold fragment. In fact, $\mathrm{[Fe_2(CO)_6(\mu\text{-}CO)_2]^{2-}}$ is related to $\mathrm{Fe_2(CO)_9}$ with an electron pair formally replacing a bridging CO.

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Chart 1

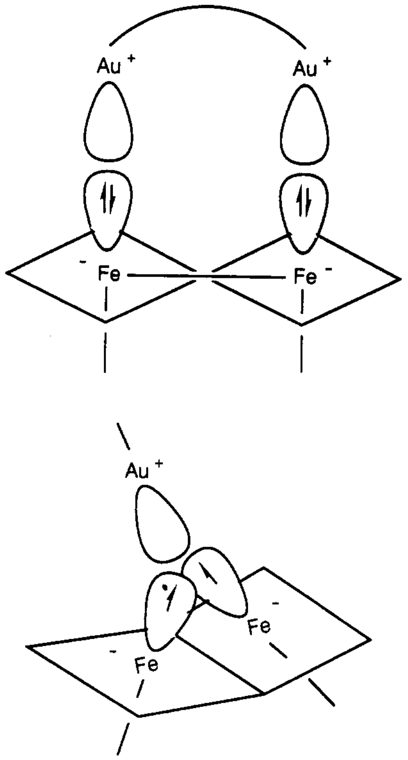

Table 2. Crystallographic Data for 3

formula	$Fe_3AuP_2O_{11}C_{53}H_{35}$		
fw	1274.3	Z	4
space group	$P2_1/c$	D_{calc}, g cm^{-1}	1.687
a, Å	17.532(2)	μ(Mo Kα)	40.03
b, Å	13.433(1)	T, °C	22
c, Å	21.373(3)	λ, cm^{-1}	0.710 69
β, deg	95.01 (2)	R	0.029
V, Å^3	5014(2)	R_w	0.031

Experimental Section

All manipulations were performed under an atmosphere of prepurified N_2 with use of standard Schlenk techniques, and all solvents were distilled from appropriate drying agents. Elemental analyses of C and H were carried out at the Institut de Bio-Orgânica de Barcelona. Infrared spectra were recorded in THF solutions on an FT-IR 520 Nicolet spectrophotometer. $^{31}P\{^1H\}$ NMR spectra were obtained on a Bruker WP 80SY spectrometer (δ(85% H_3PO_4) = 0.0 ppm). The compound $(PPh_4)_2[Fe_3(CO)_{11}]$ was prepared as described previously for the salt $(PPN)_2[Fe_3(CO)_{11}]$.[14] The complexes $(AuCl)_2(\mu$-dppm), $(AuCl)_2(\mu$-dppe), and $AuClPMe_3$ were synthesized and isolated as solids from AuCl(tht)[15] solutions by adding the appropriate amount of the corresponding phosphine.

Preparation of $(PPh_4)_2[\{Fe_3Au(CO)_{11}\}_2(\mu$-L)] (1, L = dppm; 2, L = dppe). Details of the synthesis of 1 also apply to 2. Solid $(AuCl)_2(\mu$-dppm) (0.24 g, 0.28 mmol) was added to a suspension of $(PPh_4)_2[Fe_3(CO)_{11}]$ (0.66 g, 0.57 mmol) in THF (30 mL) at −20 °C; the mixture turned dark red immediately and was stirred for 0.5 h and filtered, and the solvent was reduced to 10 mL. After the addition of 10 mL of cold hexane to the filtrate, dark red microcrystals were deposited, which were collected by filtration and recrystallized from CH_2Cl_2/hexane. Yield: 0.58 g, 80%. Spectroscopic data for 1 are as follows: IR (THF, cm^{-1}) ν(CO) stretch 2038 (m), 1969 (s), 1937 (w), 1914 (w), 1735 (w); $^{31}P\{^1H\}$ NMR (−20 °C, THF, δ (ppm)) 48.3 (*P*Au), 23.2 (*P*Ph$_4^+$). Anal. Calcd for $(PPh_4)_2[\{Fe_3Au(CO)_{11}\}_2(\mu$-dppm)]: C, 47.4; H, 2.57. Found: C, 47.3; H, 2.41. Spectroscopic and analytical data for the compound $(PPh_4)_2[\{Fe_3Au(CO)_{11}\}_2(\mu$-dppe)] (**2**): 0.47 g, 65% yield; IR (THF, cm^{-1}) ν(CO) stretch 2039 (m), 1969 (s), 1938 (w), 1914 (w), 1740 (w); $^{31}P\{^1H\}$ NMR (−20 °C, THF, δ (ppm)) 53.9

Table 3. Final Atomic Coordinates (×10000) for $C_{53}H_{35}AuFe_3O_{11}P_2$

	x/a	y/b	z/c	B_{eq}[a]
Au	19483(1)	10891(2)	18540(1)	3.59(0)
Fe(1)	31910(2)	13767(3)	12521(2)	3.68(1)
Fe(2)	25257(2)	28878(3)	17689(2)	3.45(1)
Fe(3)	26496(3)	29093(4)	5412(2)	4.68(1)
P(1)	10935(4)	213(6)	22407(2)	3.34(2)
C(1)	3575(1)	549(2)	1819(1)	4.54(9)
C(2)	3933(2)	1332(2)	755(1)	6.84(14)
C(3)	2608(1)	446(2)	833(1)	4.49(9)
C(4)	2583(1)	2748(2)	2571(1)	5.05(10)
C(5)	1517(2)	3009(2)	1711(1)	6.77(14)
C(6)	2799(1)	4155(2)	1728(1)	5.05(10)
C(7)	1791(1)	2233(2)	552(1)	5.34(10)
C(8)	3541(2)	3510(2)	678(1)	6.91(13)
C(9)	2800(3)	2418(3)	−205(1)	10.47(21)
C(10)	2209(2)	4025(2)	341(1)	8.25(16)
C(11)	3608(1)	2462(2)	1760(1)	4.92(9)
O(1)	3906(1)	49(2)	2186(1)	7.74(11)
O(2)	4450(1)	1221(2)	457(1)	10.23(14)
O(3)	2282(1)	−110(1)	493(1)	7.48(10)
O(4)	2674(1)	2702(1)	3122(1)	8.25(11)
O(5)	862(1)	3153(2)	1652(1)	9.62(14)
O(6)	2998(1)	4980(1)	1783(1)	8.04(11)
O(7)	1181(1)	1867(1)	476(1)	7.44(10)
O(8)	4116(2)	3890(2)	737(1)	10.30(16)
O(9)	3024(2)	2020(2)	−619(1)	15.33(22)
O(10)	1907(2)	4829(2)	260(1)	10.49(15)
O(11)	4188(1)	2723(1)	2011(1)	7.57(9)
P(2)	32266(4)	−26626(6)	−3944(4)	3.25(2)
C(12)	182(1)	−119(1)	1791(1)	4.66(1)
C(13)	−508(1)	−258(1)	2058(1)	4.66(1)
C(14)	−1181(1)	−431(1)	1675(1)	4.66(1)
C(15)	−1163(1)	−464(1)	1024(1)	4.66(1)
C(16)	−473(1)	−324(1)	757(1)	4.66(1)
C(17)	199(1)	−151(1)	1140(1)	4.66(1)
C(18)	1453(1)	−1235(1)	2313(1)	6.67(1)
C(19)	2244(1)	−1379(1)	2381(1)	6.67(1)
C(20)	2544(1)	−2341(1)	2399(1)	6.67(1)
C(21)	2053(1)	−3160(1)	2348(1)	6.67(1)
C(22)	1262(1)	−3016(1)	2280(1)	6.67(1)
C(23)	962(1)	−2053(1)	2262(1)	6.67(1)
C(24)	839(1)	359(1)	3025(1)	5.18(1)
C(25)	641(1)	−350(1)	3459(1)	5.18(1)
C(26)	463(1)	−53(1)	4055(1)	5.18(1)
C(27)	483(1)	954(1)	4216(1)	5.18(1)
C(28)	681(1)	1663(1)	3781(1)	5.18(1)
C(29)	859(1)	1365(1)	3186(1)	5.18(1)
C(30)	3388(1)	−3894(1)	−683(1)	4.99(1)
C(31)	2800(1)	−4438(1)	−1003(1)	4.99(1)
C(32)	2951(1)	−5371(1)	−1250(1)	4.99(1)
C(33)	3691(1)	−5761(1)	−1176(1)	4.99(1)
C(34)	4280(1)	−5217(1)	−856(1)	4.99(1)
C(35)	4129(1)	−4283(1)	−609(1)	4.99(1)
C(36)	3786(1)	−2524(1)	329(1)	5.47(1)
C(37)	3777(1)	−3261(1)	788(1)	5.47(1)
C(38)	4184(1)	−3125(1)	1372(1)	5.47(1)
C(39)	4601(1)	−2253(1)	1497(1)	5.47(1)
C(40)	4611(1)	−1516(1)	1038(1)	5.47(1)
C(41)	4204(1)	−1651(1)	454(1)	5.47(1)
C(42)	2239(1)	−2472(1)	−301(1)	5.41(1)
C(43)	1949(1)	−2517(1)	286(1)	5.41(1)
C(44)	1166(1)	−2404(1)	334(1)	5.41(1)
C(45)	672(1)	−2246(1)	−205(1)	5.41(1)
C(46)	962(1)	−2201(1)	−792(1)	5.41(1)
C(47)	1745(1)	−2314(1)	−840(1)	5.41(1)
C(48)	4045(1)	−2033(1)	−1375(1)	5.04(1)
C(49)	4271(1)	−1332(1)	−1804(1)	5.04(1)
C(50)	3974(1)	−369(1)	−1805(1)	5.04(1)
C(51)	3451(1)	−106(1)	−1378(1)	5.04(1)
C(52)	3224(1)	−807(1)	−950(1)	5.04(1)
C(53)	3521(1)	−1771(1)	−948(1)	5.04(1)

[a] $B_{eq} = {}^8/_3 U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$.

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(*P*Au), 23.2 (*P*PPh_4^+). Anal. Calcd for $(PPh_4)_2[\{Fe_3Au(CO)_{11}\}_2(\mu\text{-dppe})]$: C, 47.6; H, 2.65. Found: C, 48.1; H, 2.55.

Preparation of $(PPh_4)[Fe_3Au(CO)_{11}(PR_3)]$ (3, R = Ph; 4, R = Me). These complexes were similarly obtained by adding $AuClPPh_3$ (0.28 g, 0.57 mmol) or $AuClPMe_3$ (0.16 g, 0.57 mmol) to a suspension of $(PPh_4)_2[Fe_3(CO)_{11}]$ (0.66 g, 0.57 mmol) in THF (30 mL) at −20 °C; the workup was as above. Spectroscopic data for **3** are as follows. IR (THF, cm^{-1}) ν(CO) stretch 2038 (m), 1968 (s), 1942 (w), 1913 (m), 1738 (m); $^{31}P\{^1H\}$ NMR (−20 °C, THF, δ (ppm)) 54.7 (*P*Au), 23.3 (*P*Ph_4^+). Anal. Calcd for $(PPh_4)[Fe_3Au(CO)_{11}(PPh_3)]$: C, 49.95; H, 2.74. Found: C, 49.30; H, 2.85. Spectroscopic and analytical data for the compound $(PPh_4)[Fe_3Au(CO)_{11}(PMe_3)]$ (**4**): 0.41 g, 66% yield; IR (THF, cm^{-1}) ν(CO) stretch 2039 (m), 1968 (s), 1941 (w), 1913 (m), 1738 (m); $^{31}P\{^1H\}$ NMR (−20 °C, THF, δ (ppm)) 20.4 (*P*Au), 23.2 (*P*Ph_4^+). Anal. Calcd for $(PPh_4)[Fe_3Au(CO)_{11}(PMe_3)]$: C, 41.9; H, 2.27. Found: C, 41.6; H, 2.22.

X-ray Structure Determination. A summary of the crystal data for compound **3** is given in Table 2. A prismatic crystal (0.1 × 0.1 × 0.2 mm) was selected and mounted on an Enraf-Nonius CAD4 diffractometer. Unit cell parameters were determined for automatic centering of 25 reflections ($12 \leq \theta \leq 21°$) and refined by the least-squares method. Intensities were collected with graphite monochromatized Mo Kα radiation, using the $\omega/2\theta$ scan technique. A total 9579 reflections were measured in the range $2 \leq \theta \leq 30°$, 5190 of which were assumed as observed by applying the condition $I \geq 2.5\sigma(I)$. Three reflections were measured every 2 h as orientation and intensity control; significant intensity decay was not observed. Lorentz–polarization and absorption corrections (maximum and minimum transmission factors 1.2–0.7) were applied.

The structure was solved by Patterson synthesis, using the SHELXS computer program[16] and refined by the full-matrix least-squares method, with the SHELX76 computer program.[17] The function minimized was $\sum w(|F_o| - |F_c|)^2$, where $w = [\sigma^2(F_o) + 0.012|F_o|]^{-1}$; *f*, *f′* and *f″* were taken from ref 18. Phenyl rings were refined as "rigid groups" with C–C 1.396 Å and C–C–C bond angle 120° and using an overall anisotropic coefficient for each phenyl group. The final *R* factor was 0.029 (R_w = 0.031) for all observed reflections. The number of parameters refined was 310. The maximum shift/estimated standard deviation was 0.1. Maximum and minimum peaks in the final difference synthesis were +0.3 and −0.3 e Å^{-3}, respectively. Atomic coordinates are listed in Table 3.

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Supplementary Material Available: A table of anisotropic thermal parameters (Table SI) and a complete list of bond distances and angles (Table SII) (3 pages). Ordering information is given on any current masthead page.

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