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Effect of Diphosphine Ligands on the Metal Framework of Carbido Heteronuclear Cluster Compounds: X-ray Structure of $[Fe_4Au_2C(CO)_{12}(\mu\text{-dppm})]\cdot C_7H_8$

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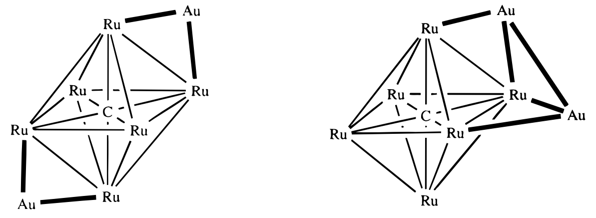
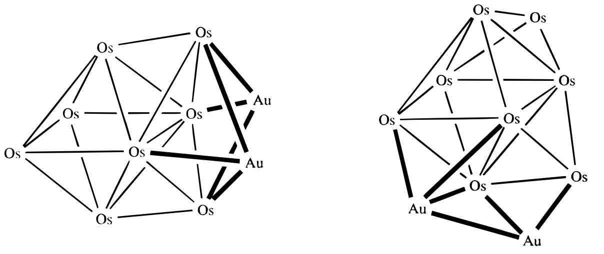
Summary: The reaction of $(NEt_4)_2[Fe_6C(CO)_{16}]$ *and* $(AuCl)_2(\mu\text{-}L)$ *(L = diphosphine) depends on the nature of the diphosphine. Unexpectedly, for L = dppm (dppm = bis(diphenylphosphino)methane), the cluster* $[Fe_4Au_2C(CO)_{12}(dppm)]$ *(**1**) was formed, whereas for L = dppe (dppe = 1,2-bis(diphenylphosphino)ethane) the reaction gave the 14-metal cluster* $(Et_4N)_2[\{Fe_6AuC(CO)_{16}\}_2(\mu\text{-dppe})]$*, in which two* Fe_6CAu *fragments are linked by the bridging ligand. Remarkably, **1** is a skeletal isomer of the previously published* $[Fe_4Au_2C(CO)_{12}(PEt_3)_2]$*.*

Although there are few reports on metal clusters dealing with skeletal isomers, defined as those compounds that have the same stoichiometry but differing skeletal geometries in the solid state,[1] their number has increased in the past few years. Most studies in this area involve compounds of low nuclearity, displaying triangular,[2] spiked-triangular,[3] tetrahedral,[4] planar-rhombohedral,[5] square-planar,[6] or butterfly arrangements,[7] but examples of systems showing more complex structures have recently been published. Two good examples are the compounds $(PPN)_2[Re_7IrC(CO)_{23}]$[8] and $[Ru_6Pt_3(CO)_{18}(\mu_3\text{-}\eta^6\text{-}ArC_2H_4Ar)(\mu_3\text{-}H)_4]$ (Ar = Ph, Tol),[9] from which two skeletal isomers were isolated. The factors that induce a compound to adopt a specific isomeric form are still under discussion; for instance, electronic factors have been invoked to account for the folding away from planarity in a series of Ru_4 butterfly clusters[10] and for the different (butterfly/tetrahedral) arrangement of the isoelectronic clusters $[PtOs_3(\mu\text{-}H)_2(CO)_{10}(PCy_3)(L)]$ (L = CO and CH_2, respectively)[11] and $[Fe_4(\mu\text{-}A)(CO)_{13}]^-$ (A = H, $HgCH_3$, $AuPR_3$, $CuPR_3$).[12] However, steric factors, resulting from the nature of the ligands directly bonded to the metal core atoms, seem to have an important role. This is particularly true for clusters containing the relatively rigid $Au_2(dppm)^{2+}$ metal ligand. Thus, for example, the structure of $[Ru_6Au_2C(CO)_{16}(PMePh_2)_2]$ consists of a doubly edge bridged octahedron which are *trans*,[13] whereas the dppm analogue, $[Ru_6Au_2C(CO)_{16}(dppm)]$, shows the geometry[14] depicted in Chart 1. More recently, the markedly different structure of $[Os_8Au_2(CO)_{22}(dppb)]$ (dppb = 1,4-bis(diphenylphosphino)butane),[15] as compared with that exhibited by the isomer $[Os_8Au_2(CO)_{22}(PPh_3)_2]$[16] (Chart 1), has been reported.

Chart 1



$[Ru_6C(CO)_{16}(AuPMePh_2)_2]$ $[Ru_6C(CO)_{16}Au_2(dppm)]$



$[Os_8(CO)_{22}(AuPPh_3)_2]$ $[Os_8(CO)_{22}(Au_2dppb)]$

[⊗] Abstract published in *Advance ACS Abstracts,* December 1, 1995.

(1) Brian, C. E.; Hall, K. P.; Mingos, D. M. P. *J. Chem. Soc., Chem. Commun.* **1984**, 290.

(2) See for example: (a) Bender, R.; Braunstein, P.; Tiripicchio, A.; Tiripicchio Camellini, M. *Angew. Chem., Int. Ed. Engl.* **1985**, *24*, 861. (b) Peng, J. J.; Horng, K. M.; Cheng, P. S.; Chi, Y.; Peng, S. M.; Lee, G. H. *Organometallics* **1994**, *13*, 2365.

(3) See for example: Einstein, F. W. B.; Johnston, V. J.; Pomeroy, R. K. *Organometallics* **1990**, *9*, 2754.

(4) See for example: (a) Xiao, C.; Vittal, J. J.; Puddephatt, R. J. *J. Chem. Soc., Chem. Commun.* **1993**, 167. (b) Park, J. T.; Woo, B. W.; Chung, J. H.; Shim, S. C.; Lee, J. H.; Lim, S. S.; Suh, I. H. *Organometallics* **1994**, *13*, 3384.

(5) See for example: (a) Braunstein, P.; de Meric de Bellefon, C.; Bouaoud, S. E.; Grandjean, D.; Halet, J. F.; Saillard, J. Y. *J. Am. Chem. Soc.* **1991**, *113*, 5282. (b) Williams, P. D.; Curtis, M. D.; Duffi, D. N.; Butler, W. M. *Organometallics* **1983**, *2*, 165.

(6) Albano, V. G.; Calderoni, F.; Iapalucci, M. C.; Longoni, G.; Monari, M. *J. Chem. Soc., Chem. Commun.* **1995**, 433.

(7) See for example: (a) Adams, R. D.; Wang, S. *Organometallics* **1985**, *4*, 1902. (b) Adams, R. D.; Wang, S. *J. Am. Chem. Soc.* **1987**, *109*, 924. (c) Chi, Y.; Wu, F.; Liu, B.; Wang, C.; Wang, S. *J. Chem. Soc., Chem. Commun.* **1989**, 873.

(8) Ma, L.; Wilson, S. R.; Shapley, J. R. *J. Am. Chem. Soc.* **1994**, *116*, 787.

(9) Adams, R. D.; Barnard, T. S.; Li, Z.; Wu, W.; Yamamoto, J. H. *J. Am. Chem. Soc.* **1994**, *116*, 9103.

(10) Carty, A. J.; McLaughlin, S. A.; van Wagner, J.; Taylor, N. J. *Organometallics* **1982**, *1*, 1013.

(11) Farrugia, L. J.; Green, M.; Hankey, D. R.; Murray, M.; Orpen, A. G.; Stone, F. G. A. *J. Chem. Soc., Dalton Trans.* **1985**, 177.

(12) Horwitz, C. P.; Holt, E. M.; Brock, C. P.; Shriver, D. F. *J. Am. Chem. Soc.* **1985**, *107*, 8147.

(13) Bunkhall, S. R.; Holden, H. D.; Johnson, B. F. G.; Lewis, J.; Pain, G. N.; Raithby, P. R.; Taylor, M. J. *J. Chem. Soc., Chem. Commun.* **1984**, 25.

(14) Bayley, P. J.; Beswick, M. A.; Lewis, J.; Raithby, P. R.; Ramirez de Arellano, M. C. *J. Organomet. Chem.* **1993**, *459*, 293.

(15) Akhter, Z.; Ingham, S. L.; Lewis, J.; Raithby, P. R. *J. Organomet. Chem.* **1994**, *474*, 165.

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With the object of increasing the knowledge of this field, in this paper we report the unexpected formation of the cluster [$Fe_4Au_2C(CO)_{12}$(dppm)], which exhibits skeletal isomerism with the previously published [$Fe_4Au_2C(CO)_{12}(PEt_3)_2$].[17]

The reaction of $(NEt_4)_2[Fe_6C(CO)_{16}]$ and $(AuCl)_2$(dppm) in tetrahydrofuran (THF) at 0 °C gave a dark-green solution from which the complex [$Fe_4Au_2C(CO)_{12}$(dppm)] (**1**) was isolated in moderate yield after filtration. The pathway for the formation of **1** seems to involve the formation of the species [$Fe_6AuC(CO)_{16}$(μ-dppm)AuCl]$^-$ (**2a**) (IR (ν(CO)) 2040 (m), 1988 (s), 1965 (m) cm^{-1}; ^{31}P NMR spectrum $\delta(P) = 52.3$ and 20.0, $J(P-P) = 39.5$ Hz) in the first step, which, after stirring for several hours, yields **1**. Attempts to obtain the analogous dppe complex [$Fe_4Au_2C(CO)_{12}$(dppe)], from $(NEt_4)_2[Fe_6C(CO)_{16}]$ and $(AuCl)_2$(dppe), were unsuccessful because the intermediate [$Fe_6AuC(CO)_{16}$(μ-dppe)AuCl]$^-$ (**2b**) (IR (ν(CO)) 2042 (m), 1990 (s), 1945 (sh), 1823 (w) cm^{-1}; ^{31}P NMR spectrum $\delta(P) = 55.4$ and 32.7, $J(P-P) = 64.5$ Hz) incorporated another $[Fe_6C(CO)_{16}]^{2-}$ fragment to give a mixture of **2b** and the 14-metal cluster [{$Fe_6AuC(CO)_{16}$}$_2$(μ-dppe)]$^{2-}$, **3**, rather than undergoing the partial cleavage of the metal core observed during the formation of **1**. It seems evident that the higher bite angle of the fragment Au(dppe)Au as compared with that of the analogous dppm is responsible for the inability of the asymmetric intermediate **2b** to yield the hexanuclear [$Fe_4Au_2C(CO)_{12}$(dppe)] cluster. The dramatic influence of the nature of the diphosphine has also recently been detected in another series of mixed iron/gold cluster compounds.[18] It is worth noting that the structure proposed for **3**, in which two metal fragments are linked by a chelating ligand, is not unusual in the chemistry of mixed-metal clusters.[18,19]

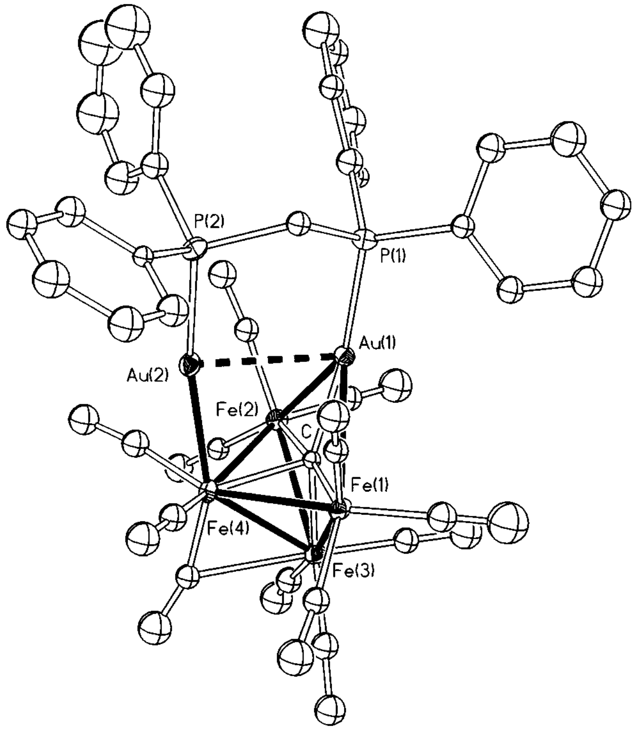

Figure 1. View of the molecular structure of the complex **1** together with the atomic numbering scheme.

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

Fe(1)−Fe(3)	2.647(3)	Au(1)−C	2.070(12)
Fe(1)−Fe(4)	2.653(3)	Fe(1)−C	1.881(13)
Fe(3)−Fe(4)	2.571(3)	Fe(3)−C	1.922(12)
Fe(2)−Fe(3)	2.650(3)	Fe(4)−C	1.973(14)
Fe(2)−Fe(4)	2.643(3)	Fe(2)−C	1.840(13)
Au(1)−Fe(1)	2.831(2)	Au(1)−P(1)	2.271(3)
Au(1)−Fe(2)	2.825(2)	Au(2)−P(2)	2.282(4)
Au(2)−Fe(4)	2.560(2)		
Au(1)−Au(2)	2.860(2)		

Fe(1)−Fe(3)−Fe(2)	89.13(9)	Fe(1)−C−Fe(2)	174.4(8)
Fe(1)−Fe(3)−Fe(4)	61.11(8)	Fe(1)−C−Fe(3)	88.2(5)
Fe(1)−Fe(4)−Fe(2)	89.15(9)	Fe(1)−C−Fe(4)	87.0(5)
Fe(1)−Fe(4)−Fe(3)	60.85(9)	Fe(1)−C−Au(1)	91.4(5)
Fe(2)−Fe(3)−Fe(4)	60.79(8)	Fe(2)−C−Fe(3)	89.6(5)
Fe(2)−Fe(4)−Fe(3)	61.08(8)	Fe(2)−C−Fe(4)	87.7(6)
Fe(3)−Fe(1)−Fe(4)	58.04(8)	Fe(2)−C−Au(1)	92.4(5)
Fe(3)−Fe(2)−Fe(4)	58.12(8)	Fe(3)−C−Fe(4)	82.6(5)
Fe(1)−Au(1)−Fe(2)	82.16(7)	Fe(3)−C−Au(1)	161.2(8)
Fe(1)−Au(1)−Au(2)	79.47(6)	Fe(4)−C−Au(1)	116.1(6)
Fe(2)−Au(1)−Au(2)	79.84(6)	C−Au(1)−Au(2)	78.0(4)
Fe(4)−Au(2)−Au(1)	78.34(5)	P(1)−Au(1)−C	170.2(4)
Au(1)−Fe(1)−Fe(3)	91.87(8)	P(1)−Au(1)−Fe(1)	133.91(10)
Au(1)−Fe(1)−Fe(4)	77.39(7)	P(1)−Au(1)−Fe(2)	141.55(10)
Au(1)−Fe(2)−Fe(3)	91.92(8)	P(1)−Au(1)−Au(2)	78.0(4)
Au(1)−Fe(2)−Fe(4)	77.65(7)	P(2)−Au(2)−Fe(4)	169.64(11)
Au(1)−Au(2)−Fe(4)	78.34(5)	P(2)−Au(2)−Au(1)	93.65(10)
Au(2)−Fe(4)−Fe(1)	88.50(7)		
Au(2)−Fe(4)−Fe(2)	89.05(7)		
Au(2)−Fe(4)−Fe(3)	135.34(9)		

The structure of **1** is shown in Figure 1, and selected bond and angle parameters are listed in Table 1. The four Fe atoms define a butterfly configuration while the Au(1) atom bridges the two wingtip metal atoms Fe(1) and Fe(2), and the Au(2) atom asymmetrically bridges the butterfly created by the Fe(1), Fe(2), and Fe(4) face and the Au(1) atom. The carbido carbon atom, C, lies approximately at the center of the distorted square defined by the Fe(1), Fe(2), Fe(3), and Au(1) atoms. The Fe_4Au_2 metal framework has no precedent in the literature. The Au(2)−Fe(4) distance (2.560(2) Å) is markedly shorter than those for Au(1)−Fe(2) and Au(1)−Fe(1) (2.825(2) and 2.831(2) Å, respectively) and compares well with the values reported for clusters containing terminal Fe−Au bonds.[18,20] The Au−Au bond length, 2.860(2) Å, is comparable to that found in metallic gold (2.884 Å)[21] and shorter than that published in the cluster [$Fe_2Au_2(CO)_8$(dppm)],[18] so a bonding interaction could be assumed, even if the gold atom Au(2) seems almost linearly coordinated to the Fe(4) and P(2) atoms, as indicated by the Fe(4)−Au(2)−P(2) angle, 169.64(11)°. The C(carbido)−Fe(wingtip) distances are ca. 0.08 Å shorter than the distance to the hinge Fe atoms. The Fe(2)−C−Fe(1) angle is 174.4(8)° with the carbide slightly displaced toward the Au(1) atom. If we ignore the Au(2) atom, the resulting metal skeleton $Fe_4CAu(1)$ is remarkably similar to that found for the clusters [$Fe_4AuC(\mu\text{-}H)(CO)_{12}(PPh_3)$][17] and [$Fe_3CoAuC(CO)_{12}(PPh_3)$].[22]

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[16] Johnson, B. F. G.; Lewis, J.; Nelson, W. J. H.; Raithby, P. R.; Vargas, M. D. *J. Chem. Soc., Chem. Commun.* **1983**, 608.

[17] Johnson, B. F. G.; Kaner, D. A.; Lewis, J.; Raithby, P. R.; Rosales, M. J. *J. Organomet. Chem.* **1982**, *231*, C59.

[18] Alvarez, S.; Rossell, O.; Seco, M.; Valls, J.; Pellinghelli, M. A.; Tiripicchio, A. *Organometallics* **1991**, *10*, 2309.

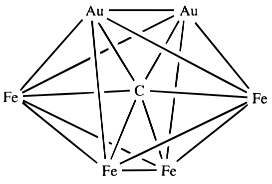
[19] (a) Amoroso, A. J.; Edwards, A. J.; Johnson, B. F. G.; Lewis, J.; Al-Mandhary, M. R.; Raithby, P. R.; Saharan, V. P.; Wong, W. T. *J. Organomet. Chem.* **1993**, *443*, C11. (b) Rossell, O.; Seco, M.; Reina, R.; Font-Bardía, M.; Solans, X. *Organometallics* **1994**, *13*, 2129. (c) Deng, H.; Shore, S. G. *Organometallics* **1991**, *10*, 3486.

[20] Braunstein, P.; Knorr, M.; Tiripicchio, A.; Tiripicchio Camellini, M. *Inorg. Chem.* **1992**, *31*, 3685.

[21] Pearson, W. D. *Lattice Spacings and Structure of Metals and Alloys*; Pergamon Press: London, 1957.

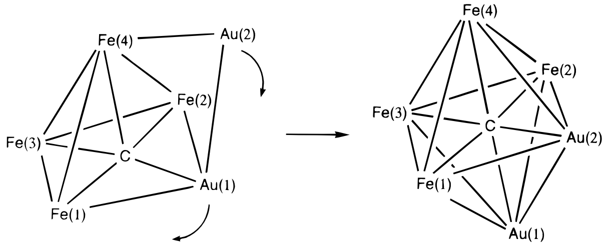
[22] Thöne, C.; Vahrenkamp, H. *J. Organomet. Chem.* **1995**, *485*, 185.

Chart 2



$[Fe_4Au_2C(CO)_{12}(PEt_3)_2]$

Scheme 1



Interestingly, **1** can be considered as a skeleton isomer of $[Fe_4Au_2C(CO)_{12}(PEt_3)_2]$.[17] In this, the six metal atoms define a distorted octahedron with the carbide carbon, C, lying approximately at its center (Chart 2). Formally, the skeletons of **1** and $[Fe_4Au_2C(CO)_{12}(PEt_3)_2]$ can interconvert to each other by moving the Au_2 fragment, (i) allowing the angle C−Fe(4)−Au(2) to bend from 87.8(4)° (dppm isomer) to about 45° (PEt_3 isomer) and (ii) creating the Au(1)−Fe(3), Au(2)−Fe(1), and Au(2)−Fe(2) bonds (Scheme 1). By comparison of the structures of these two isomers, it follows that the steric constraints imposed by the backbone of the diphosphine in **1** forces its metal atoms to adopt a closer structure. This can also be seen by comparison of the Au−Au distances: 2.860(2) Å in **1** and 3.017(1) Å in $[Fe_4Au_2C(CO)_{12}(PEt_3)_2]$. However, in order to explain the different geometry exhibited by the two skeletal isomers, other factors such as the steric hindrance caused by the presence of bulky substituents in the diphosphine should not be ruled out.

Experimental Section

All manipulations were performed under an atmosphere of prepurified N_2 with 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-Orgànica de Barcelona. Infrared spectra were recorded in THF solutions on an FT-IR 520 Nicolet spectrophotometer. ^1H and $^{31}P\{^1H\}$ NMR spectra were obtained on a Bruker WP 80SY spectrometer (δ(85% H_3PO_4) = 0.0 ppm). $^{13}C\{^1H\}$ NMR spectra were obtained on a Varian Unity-300 MHz spectrometer. The complexes $(AuCl)_2$(dppm) and $(AuCl)_2$(dppe) were synthesized and isolated as solids from AuCl(tht) solutions[23] by adding the appropriate amount of the corresponding phosphine. The compound $(NEt_4)_2[Fe_6C(CO)_{16}]$ was synthesized as described previously.[24]

Synthesis of $[Fe_4Au_2C(CO)_{12}(dppm)]$ (1). Solid $(ClAu)_2$dppm (0.28 g, 0.33 mmol) and $TlBF_4$ (0.19 g, 0.66 mmol) were added to a solution of $(NEt_4)_2[Fe_6C(CO)_{16}]$ (0.37 g, 0.36 mmol) in THF (50 mL) at 0 °C. The solution was stirred for 24 h and then filtered through Celite and evaporated to dryness. The residual solid was extracted twice with toluene (2 × 25 mL), and the resulting solution was concentrated to 10 mL and cooled overnight. Green crystals of **1** were obtained. Yield: 133 mg, 30%. IR (THF, cm^{-1}): ν(CO) stretch 2061 (s), 2017 (vs), 1994 (s), 1954 (m), 1929 (sh), 1903 (sh). $^{31}P\{^1H\}$ NMR (220 K, THF, δ (ppm)): 47.4 (d), 40.6 (d), J_{P-P} = 73.6 Hz. $^{13}C\{^1H\}$ NMR (295 K, acetone-d_6, δ (ppm)): 451 (t) (carbide, $^{2+3}J_{C-P}$ = 27 Hz), 217 (4CO), 214 (8CO). Anal. Calc: C, 33.8; H, 1.63. Found: C, 33.9; H, 1.65.

Table 2. Crystallographic Data for $1\cdot C_7H_8$

formula	$Au_2Fe_4P_2O_{12}C_{45}H_{30}$	Z	4
fw	1441.96	D_{calc}, g·cm^{-3}	2.061
space group	$P2_1/c$	μ(Mo Kα), mm^{-1}	7.636
a, Å	20.210(8)	T, K	150(2)
b, Å	13.761(7)	λ, Å	0.71073
c, Å	17.835(8)	R	0.0793
β, deg	110.48(5)	R_w	0.1556
V, Å^3	4647(4)		

Synthesis of $(NEt_4)_2[\{Fe_6C(CO)_{16}Au\}_2(dppe)]$ (3). To a solution of $(NEt_4)_2[Fe_6C(CO)_{16}]$ (85 mg, 0.08 mmol) in THF (20 mL) at −10 °C were added solid $(ClAu)_2$dppe (70 mg, 0.08 mmol) and $TlBF_4$ (47 mg, 0.16 mmol). After the mixture was stirred for 2 h, another 1 equiv of $(NEt_4)_2[Fe_6C(CO)_{16}]$ (85 mg) was added. The solution was stirred for 1 h and then filtered through Celite and evaporated to 10 mL. The addition of 25 mL of hexane gave black microcrystals of **3** after cooling overnight. Yield: 90mg, 42%. IR (THF, cm^{-1}): ν(CO) stretch 2042 (m), 1990 (vs), 1945 (m), 1823 (w), 1802 (w). $^{31}P\{^1H\}$ NMR (220 K, THF, δ (ppm)): 55.8. Anal. Calc: C, 34.5; H, 2.42; N, 1.06. Found: C, 34.2; H, 2.44; N, 1.05.

X-ray Structure Determination. Crystal data for **1**: $C_{38}H_{22}O_{12}P_2Fe_4Au_2\cdot C_7H_8$, M_r = 1441.96 (including solvate molecule), monoclinic, space group $P2_1/c$ (No. 14), a = 20.210(8) Å, b = 13.761(7) Å, c = 17.835(8) Å, β = 110.48(5)°, V = 4647(4) Å^3, Z = 4, D_c = 2.061 g cm^{-3}, μ(Mo Kα) = 7.636 mm^{-1} (graphite monochromated), λ = 0.710 73 Å, F(000) = 2752. The data were collected at 150(2) K on a Stöe Stadii-4 diffractometer fitted with an Oxford cryosystems low-temperature device,[25] using the ω scan mode (2θ = 5−45°). Corrections for Lorentz and polarization effects were applied, and absorption corrections were performed using the program DIFABS[26] (maximum and minimum corrections 1.625 and 0.771). A total of 10 042 reflections were measured within the range $0 \rightarrow h \rightarrow 21, -14 \rightarrow k \rightarrow 0, -19 \rightarrow l \rightarrow 18$ and averaged to yield 6091 unique reflections (R_{int} = 0.0881) of which 4731 were judged as observed by the criterion that $F_o^2 > 2\sigma(F_o^2)$. The structure was solved by a combination of direct methods and Fourier techniques.[27] Hydrogen atoms were placed in calculated positions and were refined using a *riding* model. Anisotropic thermal motion was assumed for the gold, iron, and phosphorus atoms only. A molecule of toluene solvate was located in a difference electron density Fourier synthesis and was included in subsequent refinement. Full-matrix least-squares refinement[28] on F_o^2 for 6084 data and 301 parameters converged to w$R2$ = 0.1556 (all data), conventional $R1$ = 0.0565 (observed data), $(\Delta/\sigma)_{max}$ = 0.001, and GOF = 1.047. The function minimized was $\sum w(F_o^2 - F_c^2)^2$, with $w = 1/[\sigma^2(F_o^2) + (0.0873P)^2 + 17.871P]$, where $P = (F_o^2 + 2F_c^2)/3$ and σ was obtained from counting statistics. A final difference electron density Fourier synthesis revealed maximum and minimum residual electron density peaks of 2.60 and −1.77 e Å^{-3}, which were in close proximity to the gold atoms.

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Supporting Information Available: Tables of atom positional and thermal parameters, anisotropic thermal parameters, and bond distances and angles (3 pages). Ordering information is given on any current masthead page.

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[23] Usón, R.; Laguna, A. *Organomet. Synth.* **1986**, *3*, 324.

[24] Hill, E. W.; Brandley, J. S. *Inorg. Synth.* **1987**, *27*, 182.

[25] Cosier, J.; Glaser, A. M. *J. Appl. Crystallogr.* **1986**, *19*, 105.

[26] Walker, N.; Stuart, D. *Acta Crystallogr.* **1983**, *A39*, 158.

[27] Sheldrick, G. M. SHELXTL/PC, release 4.3. Siemens Analytical X-ray Instruments Inc., Madison, WI, 1992.

[28] Sheldrick, G. M. SHELXL-93, Program for structure refinement, University of Göttingen, 1993.