The $[Fe_3(\mu_3\text{-}CR)(CO)_{10}]^-$ Cluster Anions as Building Blocks for the Synthesis of Mixed-metal Clusters. Part 1. Synthesis of Mixed Clusters $[MFe_3(\mu_3\text{-}CMe)(CO)_{10}(PPh_3)]$ (M = Cu or Au) and Crystal Structure of $[CuFe_3(\mu_3\text{-}CMe)(CO)_{10}(PPh_3)]$†

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The reaction of $[Fe_3(\mu_3\text{-}CMe)(\mu_3\text{-}CO)(CO)_9]^-$ with $[MCl(PPh_3)]$ (M = Cu or Au) yields the complexes $[MFe_3(\mu_3\text{-}CMe)(CO)_{10}(PPh_3)]$. The structure of $[CuFe_3(\mu_3\text{-}CMe)(CO)_{10}(PPh_3)]$ has been determined by *X*-ray diffraction. Crystals are monoclinic, space group $P2_1$ with $Z = 2$ in a unit cell with $a = 11.828(2)$, $b = 15.605(3)$, $c = 8.939(2)$ Å, and $\beta = 107.85(2)°$. The structure was solved and refined to *R* and *R'* values of 0.036 using 2 821 reflections. It consists of a butterfly metal-atom core (interplanar angle 133.3°) with the copper atom occupying a 'wing-tip' site. The CMe ligand triply bridges the triangle of iron atoms. On the opposite site of the triangle a carbonyl ligand bridges one of the basal iron atoms and the iron at the 'wing-tip' site. This ligand also interacts with the third iron and the copper atom. Each iron is surrounded by three terminal carbonyl groups and non-bonding contacts are also apparent between the three terminal carbonyl groups bonded to the basal iron atoms and the copper atom. In the case where M = Au, i.r. data in the ν(CO) stretching region suggest a weaker interaction between the carbonyl groups and the gold atom. No catalytic activity of the two mixed clusters toward hydrogenation of 1-heptene was observed.

We have shown recently that the cluster anion $[HFe_3(CO)_{11}]^-$ reacts readily with acetylene leading to $[Fe_3(\mu_3\text{-}CMe)(\mu_3\text{-}CO)(CO)_9]^-$ (**1**) at room temperature.[1] Due to its anionic nature, this cluster seemed a good candidate to build new mixed-metal cluster systems by reaction with transition-metal halide complexes. Moreover, the presence of a capping ethylidyne ligand seemed propitious to maintain the initial trinuclear iron unit, avoiding or limiting the frequently encountered problem of metal redistribution during the synthesis of mixed-metal clusters.[2]

In a first approach we have checked the reactivity of (**1**) toward $[MCl(PPh_3)]$ (M = Cu or Au) complexes. The field of mixed-metal clusters containing Au[3] and to a lesser extent Cu is now well developed,[4] particularly the similar bonding characteristics of the $Au(PR_3)^+$ fragment and hydride which are now well established. We have made this study because in complex (**1**) there is a carbonyl ligand bridging the iron triangle[1] and as the gold or copper centres are unsaturated we expected an interaction between this carbonyl group and these metal centres and perhaps an activation of the carbonyl ligand.

Indeed the i.r. spectra in the ν(CO) stretching region in the solid state for the isolated $[CuFe_3(\mu_3\text{-}CMe)(CO)_{10}(PPh_3)]$ (**2**) showed a broad band centred at 1 650 cm^{-1} which was not detected for the similar gold complex. A crystal structure determination was undertaken for complex (**2**) in order to obtain precise knowledge of the bonding interactions.

Experimental

All reactions were performed under a nitrogen atmosphere. Proton n.m.r. spectra were recorded on a Bruker WH90 spectrometer, i.r. spectra on a Perkin-Elmer 225 spectrometer for hexadecane solutions or KBr pellets in the ν(CO) stretching region. Mass spectra were recorded on a Varian MAT 311-A instrument using 70-eV (*ca.* 1.12×10^{-18} J) electron-impact ionization. Elemental analyses were performed in this laboratory.

Preparation of $[CuFe_3(\mu_3\text{-}CMe)(CO)_{10}(PPh_3)]$ (**2**).—The salt $[PPh_4][Fe_3(\mu_3\text{-}CMe)(CO)_{10}]$ (2 g) dissolved in acetone (10 cm^3) was treated with $[CuCl(PPh_3)]$ (0.880 g) and $NaBPh_4$ (0.840 g). The mixture was stirred under nitrogen for 1 h. Acetone was then eliminated *in vacuo* and the residue was extracted with pentane containing 10% CH_2Cl_2 until the solution was colourless. After evaporation of these extracts, crystallization from CH_2Cl_2–MeOH (1:1) at −20 °C gave $[CuFe_3(\mu_3\text{-}CMe)(CO)_{10}(PPh_3)]$ as black crystals (1.2 g, 60% yield). I.r. [hexadecane solution, ν(CO)]: 2 075m, 2 027s, 2 003m, 1 982m, 1 942w, and 1 800w, br cm^{-1}. ^1H N.m.r. (CD_2Cl_2): 7.57, 7.51 (PPh_4), and 4.25 p.p.m. (Me). Mass spectrum: m/z 800 (^{63}Cu) (Found: C, 44.6; H, 2.20. Calc. for $C_{30}H_{18}CuFe_3O_{10}P$: C, 44.95; H, 2.25%).

Preparation of $[AuFe_3(\mu_3\text{-}CMe)(CO)_{10}(PPh_3)]$ (**3**).—The same procedure was used starting with $[PPh_4][Fe_3(\mu_3\text{-}CMe)(CO)_{10}]$ (2 g), $[AuCl(PPh_3)]$ (1.212 g), and $NaBPh_4$ (0.840 g). Crystallization at −20 °C from CH_2Cl_2–MeOH (1:1) gave black crystals (1.5 g, 65% yield) of $[AuFe_3(\mu_3\text{-}CMe)(CO)_{10}(PPh_3)]$. I.r. [hexadecane solution, ν(CO)]: 2 068m, 2 016s, 1 998m, 1 977m, 1 943w, and 1 800w cm^{-1}. ^1H N.m.r. $[(CD_3)_2CO]$: 7.79, 7.65 (PPh_4) and 4.36 p.p.m. (Me). Mass spectrum: $m/z = 934$ (Found: C, 38.5; H, 2.0. Calc. for $C_{30}H_{18}AuFe_3O_{10}P$: C, 38.55; H, 1.95%).

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† 1,2-μ-Carbonyl-1,1,1,2,2,2,3,3,3-nonacarbonyl-μ_3-ethylidyne-1,3-triphenylphosphinecuprio-*triangulo*-tri-iron.

Supplementary data available (No. SUP 56334, 6 pp.): thermal parameters, H-atom co-ordinates, complete bond lengths and angles, least-squares planes. See Instructions for Authors, *J. Chem. Soc., Dalton Trans.*, 1985, Issue 1, pp. xvii—xix. Structure factors are available from the editorial office.

Non-S.I. unit employed: atm = 101 325 Pa.

Table 1. Fractional atomic co-ordinates with estimated standard deviations (e.s.d.s) in parentheses for complex (**2**)

Atom	*x*	*y*	*z*	Atom	*x*	*y*	*z*
Cu	0.720 08(7)	3/4	0.738 99(11)	O(10)	0.967 6(8)	0.963 3(5)	1.258 4(9)
Fe(1)	0.893 97(8)	0.853 63(8)	0.770 06(11)	C(11)	1.033 7(6)	0.780 5(5)	0.853 6(10)
Fe(2)	0.923 08(8)	0.701 48(9)	0.894 26(13)	C(12)	1.137 4(7)	0.767 3(6)	0.796 1(11)
Fe(3)	1.019 85(8)	0.833 55(9)	1.053 93(12)	P	0.530 18(14)	0.729 52(12)	0.612 4(2)
C(1)	0.848 9(6)	0.827 8(6)	0.974 0(9)	C(13)	0.440 9(5)	0.823 1(4)	0.625 2(4)
O(1)	0.762 8(4)	0.838 1(4)	1.010 2(6)	C(14)	0.353 6(5)	0.855 0(4)	0.495 9(4)
C(2)	0.984 9(7)	0.945 2(5)	0.774 9(10)	C(15)	0.289 1(5)	0.926 7(4)	0.511 2(4)
O(2)	1.041 1(7)	1.003 5(4)	0.768 6(10)	C(16)	0.311 8(5)	0.966 6(4)	0.655 8(4)
C(3)	0.758 2(8)	0.914 4(5)	0.713 8(10)	C(17)	0.399 1(5)	0.934 7(4)	0.785 2(4)
O(3)	0.675 6(6)	0.957 7(5)	0.675 3(9)	C(18)	0.463 6(5)	0.863 0(4)	0.769 9(4)
C(4)	0.889 3(7)	0.825 0(6)	0.574 4(10)	C(19)	0.456 2(3)	0.637 8(3)	0.667 9(6)
O(4)	0.888 1(7)	0.806 4(5)	0.448 9(8)	C(20)	0.522 0(3)	0.564 3(3)	0.721 4(6)
C(5)	1.043 2(7)	0.629 3(5)	0.976 3(10)	C(21)	0.468 1(3)	0.493 0(3)	0.761 7(6)
O(5)	1.120 7(6)	0.583 8(4)	1.024 3(10)	C(22)	0.348 3(3)	0.495 0(3)	0.748 5(6)
C(6)	0.819 5(7)	0.653 5(6)	0.985 9(13)	C(23)	0.282 5(3)	0.568 4(3)	0.695 0(6)
O(6)	0.761 1(6)	0.621 2(5)	1.050 4(11)	C(24)	0.336 5(3)	0.639 8(3)	0.654 7(6)
C(7)	0.876 9(9)	0.649 8(6)	0.710 2(13)	C(25)	0.507 8(4)	0.712 8(3)	0.403 6(6)
O(7)	0.859 0(8)	0.615 8(5)	0.592 4(9)	C(26)	0.426 2(4)	0.654 8(3)	0.314 1(6)
C(8)	1.165 2(8)	0.874 5(7)	1.071 4(10)	C(27)	0.417 0(4)	0.642 4(3)	0.157 4(6)
O(8)	1.257 4(6)	0.900 8(7)	1.083 4(9)	C(28)	0.489 6(4)	0.687 9(3)	0.090 2(6)
C(9)	1.061 7(8)	0.756 7(6)	1.210 0(10)	C(29)	0.571 3(4)	0.745 9(3)	0.179 7(6)
O(9)	1.090 3(8)	0.712 4(5)	1.314 1(9)	C(30)	0.580 4(4)	0.758 4(3)	0.336 4(6)
C(10)	0.988 6(8)	0.914 5(5)	1.180 6(10)				

Catalysis Experiments.—All experiments were carried out in a stainless-steel autoclave (100 cm^3) in which a glass vessel was inserted. Chromatographic analysis of the reactions was performed on an Intersmat IGC 120 F apparatus using a 2-m Carbowax column (46 mm diameter) operating at 25 °C. Complex (**2**) or (**3**) (50 mg) was dissolved in dichloromethane (15 cm^3) and 1-heptene (2 cm^3). The solution was pressurized under 10 atm of hydrogen and heated to 60 °C.

X-Ray Crystallography for Complex (**2**).—*Crystal data*. $C_{30}H_{18}CuFe_3O_{10}P$, $M = 800.1$, monoclinic, space group $P2_1$ (C_2^2, no. 4), $a = 11.828(2)$, $b = 15.605(3)$, $c = 8.939(2)$ Å, $\beta = 107.85(2)°$, $U = 1\,570.5$ Å^3, $Z = 2$, $D_c = 1.69$ g cm^{-3}, $F(000) = 800$, Mo-K_α radiation, $\lambda = 0.710\,69$ Å, $\mu = 21.3$ cm^{-1}, 293 K.

Data collection. A black plate-like crystal of dimensions 0.175 (010, 0$\bar{1}$0) × 0.185 (101, $\bar{1}$0$\bar{1}$) × 0.175 (10$\bar{1}$, $\bar{1}$01) mm (distances from centre to faces) was centred on an Enraf–Nonius CAD4 diffractometer equipped with a graphite monochromator. The unit-cell dimensions were determined from the setting angles of 25 reflections in the range $10 < \theta < 13°$. A unique data set ($1.5 < \theta < 27°$, $\pm h, k, l$) was recorded as described previously[5] by the θ—2θ scan technique (scan width $0.75 + 0.35 \tan\theta$, scan speed 1.1—10.1° min^{-1}). The intensities of three standard reflections monitored every 2 h showed no significant variation during data collection. 3 542 Independent reflections were obtained then corrected for Lorentz and polarization effects,[6] 2 821 with $I > 3\sigma(I)$ being considered 'observed' and used in the least-squares refinement after solution of the structure and absorption corrections[7] (maximum, minimum transmission factors 0.61, 0.47).

Structure determination. The systematic absences 0*k*0 ($k \neq 2n$) were compatible with space groups $P2_1$ or $P2_1/m$. *E*-Value statistics indicated an acentric distribution of atoms which implied space group $P2_1$, a choice verified by the subsequent refinement. The MULTAN system[8] was used to locate the Cu, Fe, and P atoms. Subsequent full-matrix least-squares refinement and interpretation of Fourier difference maps using SHELX[9] enabled all non-hydrogen atoms in the structure to be located; these were refined anisotropically, except those of the phenyl rings which refined as isotropic rigid groups (C–C 1.385 Å). Hydrogen atoms were located on a Fourier difference map and introduced in the calculations with a constrained geometry (C–H 0.95 Å) and fixed isotropic thermal parameters, $U_H = 0.07$ Å^2. Neutral atom scattering factors were used, those for the non-hydrogen atoms being corrected for anomalous dispersion (f', f'').[10] Both enantiomeric forms were tested. Unit weights were applied and gave satisfactory weight analysis. In the last full-matrix least-squares refinement cycle no shifts were greater than 0.06 times the estimated standard deviation and the final *R* was 0.036 ($R' = 0.036$). A final Fourier difference map showed no excursion of electron density greater than 0.3 e Å^{-3}. All calculations were performed on a VAX-11/730 DEC computer. Atomic co-ordinates are listed in Table 1.

Results and Discussion

The reaction of $[PPh_4][Fe_3(\mu_3\text{-}CMe)(CO)_{10}]$ with $[MCl(PPh_3)]$ (M = Cu or Au) has been studied under different conditions: direct reaction in refluxing acetone, using $TlBF_4$ as a chloride abstractor, or adding $NaBPh_4$ in acetone at room temperature. The latter method gave the best results and is described in the Experimental section. The function of $NaBPh_4$ is to exchange the PPh_4^+ by Na^+, the salt PPh_4BPh_4 being nearly insoluble in the acetone solution. Yields greater than 60% have thus been obtained for $[CuFe_3(\mu_3\text{-}CMe)(CO)_{10}(PPh_3)]$ (**2**) and $[AuFe_3(\mu_3\text{-}CMe)(CO)_{10}(PPh_3)]$ (**3**). Both complexes afford a violet solution, but (**2**) is not very stable in solution, slowly depositing metallic copper and giving unidentified products. The complexes are stable in the solid state.

Mass spectra and analyses confirm the formulations. Examination of the i.r. spectra of complexes (**2**) and (**3**) in solution shows a very similar pattern in the terminal CO stretching region, but in the bridging region the situation is quite different. In both cases there is a band near 1 800 cm^{-1} but this band is weaker and broader for (**2**) than for (**3**) [60 cm^{-1} at half-height for (**2**) and 30 cm^{-1} for (**3**)]. Moreover this difference is more pronounced in the solid state; for complex (**2**) in the bridging region the band is very broad with a maximum at 1 650 cm^{-1} (*ca.* 200 cm^{-1} at half-height) and for (**3**) the band remains quite narrow, with a maximum at 1 785 cm^{-1}.

These observations suggest that the two complexes have quite

Table 2. Selected bond lengths (Å) and angles (°) with e.s.d.s in parentheses for [$CuFe_3(\mu_3\text{-}CMe)(CO)_{10}(PPh_3)$]

Fe(1)–Fe(2)	2.599(2)	Fe(1)–C(3)	1.798(9)	Fe(2)–Fe(1)–Fe(3)	59.98(4)	Fe(3)–C(11)–C(12)	131.5(5)
Fe(1)–Fe(3)	2.537(1)	Fe(1)–C(4)	1.790(9)	Fe(1)–Fe(2)–Fe(3)	58.82(4)	Fe(1)–C(2)–O(2)	175.9(8)
Fe(2)–Fe(3)	2.568(2)	Cu ··· C(3)	2.626(8)	Fe(1)–Fe(3)–Fe(2)	61.20(4)	Fe(1)–C(3)–O(3)	175.4(8)
Cu–Fe(1)	2.564(1)	Fe(2)–C(5)	1.785(7)	Fe(1)–Cu–Fe(2)	61.82(4)	Fe(1)–C(4)–O(4)	179.0(8)
Cu–Fe(2)	2.494(1)	Fe(2)–C(6)	1.830(11)	Cu–Fe(1)–Fe(2)	57.78(3)	Fe(2)–C(5)–O(5)	177.8(9)
Cu–P	2.207(2)	Fe(2)–C(7)	1.762(11)	Cu–Fe(2)–Fe(1)	60.40(3)	Fe(2)–C(6)–O(6)	175.5(8)
Fe(1)–C(1)	2.090(8)	Cu ··· C(6)	2.633(10)	Fe(1)–Cu–P	143.47(6)	Fe(2)–C(7)–O(7)	173.1(10)
Fe(2) ··· C(1)	2.356(9)	Cu ··· C(7)	2.499(10)	Fe(2)–Cu–P	153.67(6)	Fe(3)–C(8)–O(8)	179.5(10)
Fe(3)–C(1)	1.930(7)	Fe(3)–C(8)	1.795(9)	Fe(1)–C(1)–O(1)	133.8(5)	Fe(3)–C(9)–O(9)	175.9(8)
Cu ··· C(1)	2.499(7)	Fe(3)–C(9)	1.791(9)	Fe(3)–C(1)–O(1)	142.5(6)	Fe(3)–C(10)–O(10)	178.6(9)
C(1)–O(1)	1.170(10)	Fe(3)–C(10)	1.808(9)	Fe(1)–C(1)–Fe(3)	78.2(3)	Cu–P–C(13)	111.7(2)
Fe(1)–C(11)	1.958(7)	P–C(13)	1.827(6)	Fe(1)–C(1)–Fe(2)	71.3(3)	Cu–P–C(19)	118.1(1)
Fe(2)–C(11)	1.912(8)	P–C(19)	1.825(5)	Cu–C(1)–O(1)	86.6(4)	Cu–P–C(25)	110.6(2)
Fe(3)–C(11)	2.025(9)	P–C(25)	1.822(5)	Fe(1)–C(11)–Fe(2)	84.4(3)		
C(11)–C(12)	1.483(13)	C–O	1.137(12) (mean)	Fe(1)–C(11)–Fe(3)	79.1(3)		
Fe(1)–C(2)	1.781(9)			Fe(1)–C(11)–C(12)	129.8(6)		

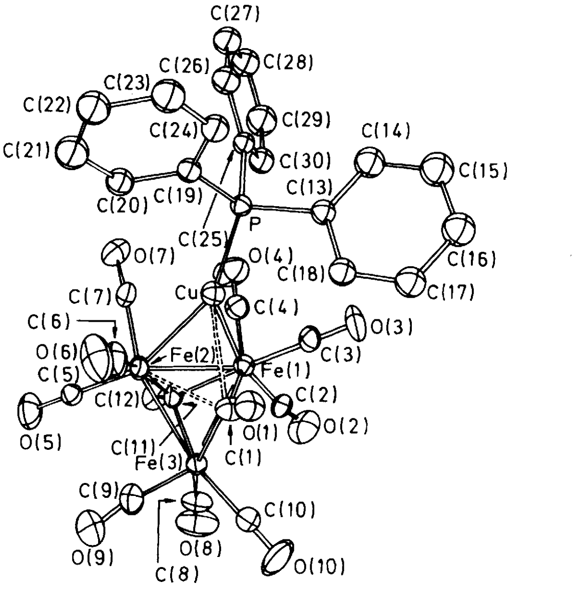

Figure. Structure of complex (**2**) showing the atomic numbering scheme with ellipsoids at the 40% probability level

similar structures (from the i.r. spectra in the terminal stretching region) but that in complex (**2**) there are strong interactions between the bridging carbonyl ligand (and perhaps other terminal carbonyl ligands) and the copper centre (from the i.r. spectra in the bridging stretching region). To confirm this hypothesis, a single crystal *X*-ray analysis of (**2**) was undertaken. The structure is shown in the Figure with the atomic numbering scheme. Selected bond lengths and angles are given in Table 2.

The three Fe atoms define the vertices of a triangle, one edge of which is bridged by the Cu atom, to give a butterfly arrangement. The dihedral angle between the Fe(1)Fe(2)Fe(3) and the Fe(1)Fe(2)Cu planes is 133.3°. This value is quite high compared with those found in similar complexes [$AuRu_3(\mu\text{-}COMe)(CO)_{10}(PPh_3)$] (117°)[3a] or [$CuOs_3H_3(CO)_{10}(PPh_3)$] (107.8°)[4a] and is certainly a consequence of the steric effect due to the presence of the bridging C(1)–O(1) group. The ethylidyne group remains μ_3-bonded as in the starting anion[1] but its mode of bonding is less symmetrical than in the similar complex [$Fe_3(\mu_3\text{-}CH)(CO)_{10}$]$^-$.[11] The longer distance is Fe(3)–C(11) and this is certainly a consequence of the presence of the Cu atom which bridges the Fe(1)–Fe(2) bond. The C(11)–C(12) bond is nearly perpendicular to the Fe(1)Fe(2)Fe(3) plane [angle between C(11)–C(12) and the perpendicular to the iron plane is 2.2°]. The C(1)–O(1) bridging carbonyl ligand is nearly symmetrically bonded to Fe(1) and Fe(3). We have previously shown[1] that in the starting anion (**1**) there is an equilibrium between [$Fe_3(\mu_3\text{-}CMe)(\mu_3\text{-}CO)(CO)_9$]$^-$ (**1a**) and [$Fe_3(\mu_3\text{-}CMe)(\mu\text{-}CO)(CO)_9$]$^-$ (**1b**) in solution and that in the solid state only (**1a**) or (**1b**) exists depending on the associated cation. In complex (**2**) the doubly bridging situation is favoured but interactions with Fe(3) and Cu are also evident from the distances between C(1) and the two metals. Moreover the O(1)–C(1)–Cu angle of 86.6(4)° suggests a weak interaction of O(1) with Cu. Similar interactions have been found with mixed-metal clusters containing Au or Cu[3,4,12] and are generally attributed to the electronically unsaturated nature of these metal centres. This explanation has sometimes been questioned, steric constraints being invoked[13] to explain short non-bonding contacts, but in our case electronic interactions seem substantiated by the i.r. spectra in the ν(CO) stretching region.

Interaction of the copper centre with the C(3), C(6), and C(7) carbons of the carbonyl groups is also evident and could explain the diffuse nature of the CO stretching band of complex (**2**) in the bridging region in solution or in the solid state. These interactions are not accompanied by great changes in the linearity of the M–C–O bonds, but nevertheless the greater change observed for the carbonyl group C(7)O(7), which interacts most strongly with the copper centre, suggests incipient semi-bridging. The other carbonyl groups of the molecules do not show any special feature. The Cu atom bridges slightly asymmetrically the Fe(1)–Fe(2) bond, the shortest distance being to Fe(1) which is bonded to the bridging carbonyl ligand. The Cu–P distance is short but in the same range as that found in [$\{CuH(PPh_3)\}_6$][14] and the P atom is slightly out of the Fe(1)Fe(2)Cu plane [0.227(2) Å].

The copper-bridged Fe(1)–Fe(2) bond is the longest Fe–Fe bond of the triangle but is shorter than the hydrogen-bridged iron–iron bonds of [$Fe_3(\mu\text{-}H)_3(\mu_3\text{-}CMe)(CO)_9$] [2.618(2) Å].[15] The shortest bond is Fe(1)–Fe(2) which is bridged by C(1)O(1).

To summarize, the addition of the $Cu(PPh_3)^+$ cation to the [$Fe_3(\mu_3\text{-}CMe)(CO)_{10}$]$^-$ anion preserves the mode of bonding of the bridging ethylidyne and the bridging carbonyl ligands. This is in contrast with the structure proposed for [$Fe_3H(\mu\text{-}CMe)(CO)_{10}$] from spectroscopic data, where there is no CO bridge and the ethylidyne ligand bridges two metal atoms.[16] A similar structure was also proposed for the related [$Fe_3H(\mu\text{-}CH)(CO)_{10}$].[11]

Furthermore, comparison of the spectroscopic data for complexes (**2**) and (**3**) suggests that for the latter the structure of the metallic core is nearly the same, but that the interactions of the carbonyl ligands with the gold centre are weaker than those with the copper centre.

Taking into account the unsaturated nature of the copper and gold centres, we have studied whether complex (**2**) or (**3**) has catalytic activity for the hydrogenation of alkenes. Under our conditions (30 atm of H_2, 60 °C), neither hydrogenation nor isomerization occurs, but the complexes are transformed. We have tried to identify the products and first determined whether the presence of alkenes was necessary to induce this transformation. It was found that the reactions observed are the result of the interaction of the complexes with hydrogen. In the case of complex (**2**) two products are formed. One, $[Fe(CO)_4(PPh_3)]$, was identified by comparison of its i.r. spectrum with an authentic sample.[17] For the other, mass spectrometry ($m/z = 774$) and proton n.m.r. data [$\delta = 7.49$ (15 H), 4.46 (3 H), and -21.70 p.p.m. (2 H, d, $J_{PH} = 8$ Hz)] suggest the formulation $[CuFe_3H_2(CMe)(CO)_9(PPh_3)]$. A related compound $[AuRu_3(\mu\text{-}H)_2(\mu_3\text{-}COMe)(CO)_9(PPh_3)]$ has also been obtained by the reaction of hydrogen with $[AuRu_3(\mu_3\text{-}COMe)(CO)_{10}(PPh_3)]$.[3a] Complex (**3**) reacts more slowly with hydrogen, and the i.r. spectrum in the ν(CO) stretching region indicates the formation of new compounds, none of which could be isolated and identified.

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