The Heteronuclear Cluster Chemistry of the Group 1B Metals. Part 5.[1] *X*-Ray Crystal Structure and Dynamic Behaviour of the Heptanuclear Mixed-metal Cluster $[Au_3Ru_4(\mu\text{-}H)(CO)_{12}(\mu\text{-}Ph_2PCH_2PPh_2)(PPh_3)]$ †

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A single-crystal *X*-ray diffraction study of the mixed-metal gold cluster $[Au_3Ru_4(\mu\text{-}H)(CO)_{12}(\mu\text{-}Ph_2PCH_2PPh_2)(PPh_3)]$ (**1**) reveals a different core geometry to the bicapped trigonal-bipyramidal structure adopted by the closely related species $[Au_3Ru_4(\mu_3\text{-}H)(CO)_{12}(PPh_3)_3]$ (**2**). The metal skeleton of (**1**) may be described as a distorted Au_2Ru_3 square pyramid with two adjacent triangular faces capped by the metal atoms of a $Ru(CO)_3$ and a $Au(PPh_3)$ unit. The $Ph_2PCH_2PPh_2$ group bridges the Au–Au edge of the square base of the pyramid, the hydrido ligand bridges a Ru–Ru edge of the capping $Ru(CO)_3$ unit, and each ruthenium atom is bonded to three terminal carbonyl groups. Knowledge of the structure of (**1**) allows further insight into the nature of the dynamic behaviour that (**1**) and (**2**) exhibit in solution at ambient temperature.

Although mixed-metal cluster compounds containing one or more $Au(PR_3)$ (R = alkyl or aryl) groups are currently attracting a great deal of attention,[1–3] detailed investigations of similar species with bidentate diphosphines remain rare.[1,4,5] The structure of $[Au_3Ru_4(\mu\text{-}H)(CO)_{12}(\mu\text{-}Ph_2PCH_2PPh_2)(PPh_3)]$ (**1**) has been determined to investigate the relationship between its skeletal geometry and that of its tris(monodentate phosphine) analogue $[Au_3Ru_4(\mu_3\text{-}H)(CO)_{12}(PPh_3)_3]$ (**2**).[3] The work was prompted by the recent observation that when two PPh_3 groups in the hexanuclear species $[Au_2Ru_4(\mu_3\text{-}H)(\mu\text{-}H)(CO)_{12}(PPh_3)_2]$ (**3**) are replaced by one $Ph_2PCH_2PPh_2$ ligand in $[Au_2Ru_4(\mu_3\text{-}H)(\mu\text{-}H)(CO)_{12}(\mu\text{-}Ph_2PCH_2PPh_2)]$ (**4**), the metal core structure of the cluster is altered from a capped trigonal bipyramid to a capped square pyramid.[5,6] Previously, because of the close similarity between the i.r. and ^1H n.m.r. spectroscopic data of the heptanuclear clusters (**1**) and (**2**), it had been thought that (**1**) adopted the same bicapped trigonal-bipyramidal metal core geometry as that earlier established for its tris(monodentate phosphine) analogue (**2**) by *X*-ray diffraction study.[3]

Results and Discussion

The *X*-ray structure analysis shows that the Au_3Ru_4 skeleton of (**1**) is not based on a trigonal-bipyramidal geometry as originally thought,[3] but may be described as a distorted Au_2Ru_3 square pyramid, capped on two adjacent triangular faces by a $Ru(CO)_3$ and a $Au(PPh_3)$ unit. The molecular configuration of (**1**) is illustrated in the Figure, and selected interatomic distances and bond angles are summarised in Table 1.

The basal atoms Au(1), Au(2), Ru(2), and Ru(3) and the apical atom Ru(1) define the square pyramid and the Au(1)Ru(1)Ru(2) and Ru(1)Ru(2)Ru(3) faces of this Au_2Ru_3 unit are capped by Au(3) and Ru(4), respectively. The $Ph_2PCH_2PPh_2$ ligand bridges the two gold atoms Au(1) and Au(2), which form one edge of the square base, the hydrido ligand bridges one edge, Ru(3)–Ru(4), of the Ru_4 tetrahedral cap, and each ruthenium atom is bonded to three essentially linear carbonyl groups [M–C–O 165(2)—177(3)°]. There are several short Au $\cdots$ C(carbonyl) intramolecular contacts [2.51(3)—2.84(4) Å]; similar short distances have been observed previously between carbonyl ligands and gold atoms in other heteronuclear cluster compounds.[1,7–9]

Although the Au(1)–Au(3) edge of the Au_2Ru_2 tetrahedral cap is unbridged, whereas the Au(1)–Au(2) edge of the square pyramid is bridged by the bidentate diphosphine ligand, there is only a slight difference between the two Au–Au separations [2.749(2) and 2.758(2) Å respectively]. Both of these Au–Au distances are markedly shorter than those found in the tris(monodentate phosphine) analogue (**2**) [2.822(2) and 2.838(2) Å][3] and it is interesting that in the gold–ruthenium diphosphine clusters $[Au_2Ru_4(\mu_3\text{-}H)(\mu\text{-}H)(CO)_{12}(\mu\text{-}Ph_2PCH_2PPh_2)]$ and $[Au_2Ru_3(\mu_3\text{-}S)(CO)_9(\mu\text{-}Ph_2PCH_2PPh_2)]$ the Au–Au edges bridged by the $Ph_2PCH_2PPh_2$ ligand are also considerably longer [2.823(1)[5] and 2.802(1)[1] Å respectively] than the Au–Au distances in (**1**). The Au–Ru distances observed in (**1**) [2.762(3)—2.920(2) Å] cover a similar range to those found in (**2**) [2.825(2)—3.011(3) Å],[3] but the individual bond lengths are slightly shorter in the former cluster. As observed in (**2**),[3] the unbridged Ru–Ru edges of the Ru_4 tetrahedron in (**1**) [Ru(1)–Ru(4), Ru(2)–Ru(3), Ru(2)–Ru(4), and Ru(3)–Ru(4)] are shorter [2.774(3)—2.931(3) Å] than the Ru–Ru distances bridged by the gold atoms [Ru(1)–Ru(2) 3.044(3) and Ru(1)–Ru(3) 2.967(4) Å].

The molecular configuration of (**1**) clearly exhibits a difference in the mode of bonding of the two basal gold atoms spanned by the bidentate diphosphine group. Both Au atoms are bonded to the apical atom Ru(1) but, in addition, Au(1) is also bonded to the capping atom Au(3). This difference is reflected in different Au–P bond lengths [Au(1)–P(1) 2.287(8) and Au(2)–P(2) 2.307(8) Å]. Although the differences in these Au–P distances are of low significance, the larger value, that to the uncapped gold atom [Au(2)], is very close to the Au–P bond lengths found in the structurally related hexanuclear cluster (**4**) [2.304(3) and 2.309(3) Å],[10] where both gold atoms exhibit similar uncapped basal environments to that adopted by Au(2).

The metal core geometry of (**1**) is very closely related to the capped square-pyramidal structure adopted by the hexanuclear cluster (**4**),[5] with one $AuRu_2$ face of the latter being capped by a $Au(PPh_3)$ group in the heptanuclear species. Interestingly, the

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† 1,2-μ-[Bis(diphenylphosphino)methane]-4,4,4,5,5,5,6,6,6,7,7,7-dodecacarbonyl-6,7-μ-hydrido-3-triphenylphosphine-*cyclo*-1,2,3-trigold-4,5,6,7-tetraruthenium (2 *Au–Au*) (6 *Au–Ru*) (6 *Ru–Ru*).

Supplementary data available: see Instructions for Authors, *J. Chem. Soc., Dalton Trans.*, 1988, Issue 1, pp. xvii—xx.

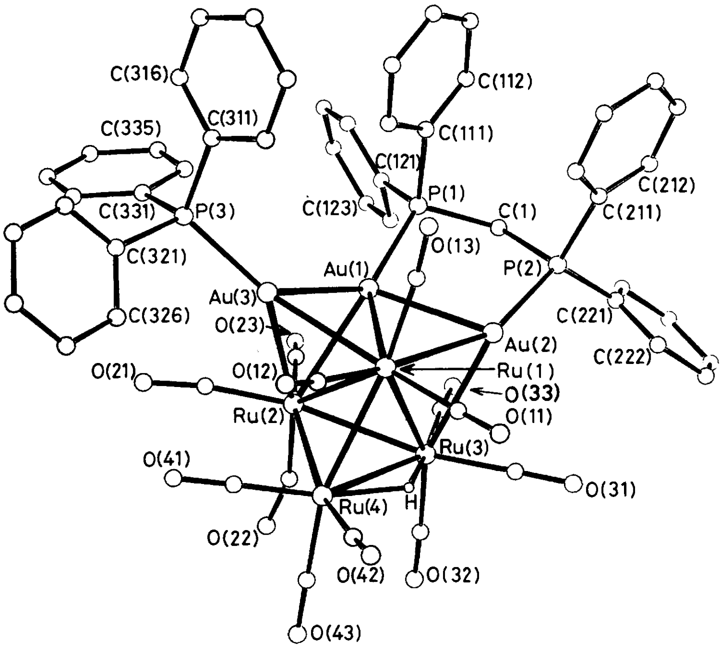
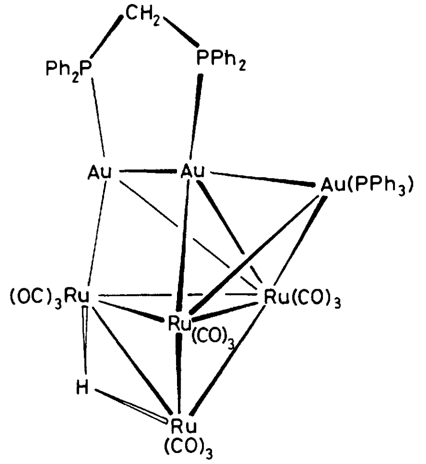
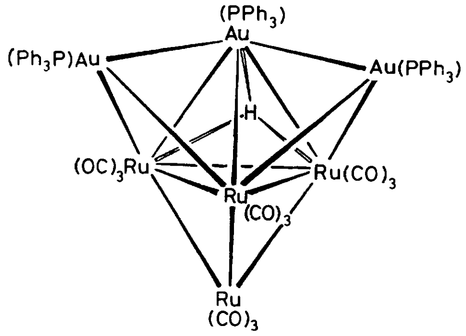


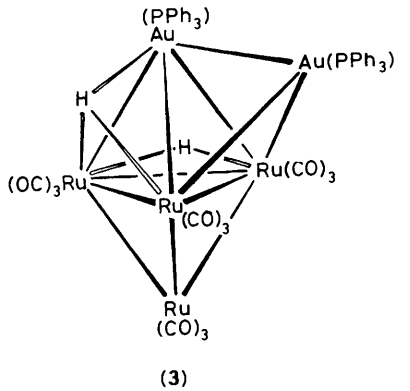
Figure. The molecular structure of $[Au_3Ru_4(\mu\text{-}H)(CO)_{12}(\mu\text{-}Ph_2PCH_2PPh_2)(PPh_3)]$ (**1**) showing the bicapped square-pyramidal metal framework. The carbonyl C-atoms have the same numbers as the oxygens to which they are bonded



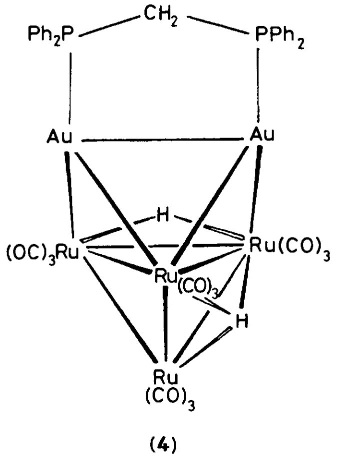
(**1**)



(**2**)



(**3**)



(**4**)

Table 1. Selected bond lengths (Å), and angles (°), with estimated standard deviations in parentheses, for $[Au_3Ru_4(\mu\text{-}H)(CO)_{12}(\mu\text{-}Ph_2PCH_2PPh_2)(PPh_3)]$ (**1**)

Au(1)–Au(2)	2.758(2)	Au(1)–Au(3)	2.749(2)	Ru(3)–Ru(4)	2.931(4)	Ru(3)–C(31)	1.83(4)
Au(1)–Ru(1)	2.920(2)	Au(1)–Ru(2)	2.781(2)	Ru(3)–C(32)	1.86(3)	Ru(3)–C(33)	1.88(3)
Au(1)–P(1)	2.287(8)	Au(2)–Ru(1)	2.762(3)	Ru(3)–H	1.857	Ru(4)–C(41)	1.94(4)
Au(2)–Ru(3)	2.869(2)	Au(2)–P(2)	2.307(8)	Ru(4)–C(42)	1.91(3)	Ru(4)–C(43)	1.84(3)
Au(3)–Ru(1)	2.812(3)	Au(3)–Ru(2)	2.898(3)	Ru(4)–H	1.845	P(1)–C(1)	1.87(3)
Au(3)–P(3)	2.305(9)	Ru(1)–Ru(2)	3.044(3)	P(1)–C(111)	1.798(23)	P(1)–C(121)	1.82(3)
Ru(1)–Ru(3)	2.967(4)	Ru(1)–Ru(4)	2.847(3)	P(2)–C(1)	1.80(3)	P(2)–C(211)	1.779(21)
Ru(1)–C(11)	1.85(3)	Ru(1)–C(12)	1.92(3)	P(2)–C(221)	1.82(3)	P(3)–C(311)	1.801(18)
Ru(1)–C(13)	1.90(3)	Ru(2)–Ru(3)	2.840(4)	P(3)–C(321)	1.795(23)	P(3)–C(331)	1.800(24)
Ru(2)–Ru(4)	2.774(3)	Ru(2)–C(21)	1.92(4)	Ru–CO	1.82(3)—1.94(4)	Mean Ru–CO	1.86(3)
Ru(2)–C(22)	1.82(3)	Ru(2)–C(23)	1.89(3)				

Au(3)–Au(1)–Au(2)	115.6(1)	Ru(1)–Au(1)–Au(2)	58.1(1)	Au(3)–Ru(2)–Au(1)	57.9(1)	Ru(1)–Ru(2)–Au(1)	60.0(1)
Ru(1)–Au(1)–Au(3)	59.4(1)	Ru(2)–Au(1)–Au(2)	101.6(1)	Ru(1)–Ru(2)–Au(3)	56.4(1)	Ru(3)–Ru(2)–Au(1)	78.3(1)
Ru(2)–Au(1)–Au(3)	63.2(1)	Ru(2)–Au(1)–Ru(1)	64.5(1)	Ru(3)–Ru(2)–Au(3)	114.8(1)	Ru(3)–Ru(2)–Ru(1)	60.4(1)
P(1)–Au(1)–Au(2)	90.0(2)	P(1)–Au(1)–Au(3)	132.6(2)	Ru(4)–Ru(2)–Au(1)	117.3(1)	Ru(4)–Ru(2)–Au(3)	95.4(1)
P(1)–Au(1)–Ru(1)	140.0(2)	P(1)–Au(1)–Ru(2)	153.2(2)	Ru(4)–Ru(2)–Ru(1)	58.4(1)	Ru(4)–Ru(2)–Ru(3)	62.9(1)
Ru(1)–Au(2)–Au(1)	63.9(1)	Ru(3)–Au(2)–Au(1)	78.2(1)	Ru(2)–Ru(3)–Ru(1)	63.2(1)	Ru(4)–Ru(3)–Au(2)	113.8(1)
Ru(3)–Au(2)–Ru(1)	63.6(1)	P(2)–Au(2)–Au(1)	98.8(2)	Ru(4)–Ru(3)–Ru(1)	57.7(1)	Ru(4)–Ru(3)–Ru(2)	57.4(1)
P(2)–Au(2)–Ru(1)	156.3(3)	P(2)–Au(2)–Ru(3)	131.6(2)	Ru(2)–Ru(4)–Ru(1)	65.6(1)	Ru(3)–Ru(4)–Ru(1)	61.8(1)
Ru(1)–Au(3)–Au(1)	63.3(1)	Ru(2)–Au(3)–Au(1)	58.9(1)	Ru(3)–Ru(4)–Ru(2)	59.6(1)	C(41)–Ru(4)–Ru(1)	105.0(9)
Ru(2)–Au(3)–Ru(1)	64.4(1)	P(3)–Au(3)–Au(1)	131.1(2)	C(111)–P(1)–Au(1)	114.9(7)	C(111)–P(1)–C(1)	108(1)
P(3)–Au(3)–Ru(1)	152.9(2)	P(3)–Au(3)–Ru(2)	141.3(2)	C(121)–P(1)–Au(1)	118.9(7)	C(121)–P(1)–C(1)	101(1)
Au(2)–Ru(1)–Au(1)	58.0(1)	Au(3)–Ru(1)–Au(1)	57.3(1)	C(211)–P(2)–Au(2)	119.0(8)	C(211)–P(2)–C(1)	105(1)
Au(3)–Ru(1)–Au(2)	113.4(1)	Ru(2)–Ru(1)–Au(1)	55.5(1)	C(221)–P(2)–Au(2)	117.2(8)	C(221)–P(2)–C(1)	101(1)
Ru(2)–Ru(1)–Au(2)	95.2(1)	Ru(2)–Ru(1)–Au(3)	59.2(1)	C(321)–P(3)–Au(3)	113.4(7)	C(321)–P(3)–C(311)	104.6(9)
Ru(3)–Ru(1)–Au(1)	74.2(1)	Ru(3)–Ru(1)–Au(2)	60.0(1)	C(331)–P(3)–Au(3)	110.4(9)	C(331)–P(3)–C(311)	106(1)
Ru(3)–Ru(1)–Au(3)	113.5(1)	Ru(3)–Ru(1)–Ru(2)	56.4(1)	C(311)–P(3)–Au(3)	115.7(8)	Mean Ru–C–O	174(3)
Ru(4)–Ru(1)–Au(1)	110.7(1)	Ru(4)–Ru(1)–Au(2)	120.1(1)	Ru–C–O	165(2)—177(3)		
Ru(4)–Ru(1)–Au(3)	95.7(1)	Ru(4)–Ru(1)–Ru(2)	56.1(1)				
Ru(4)–Ru(1)–Ru(3)	60.5(1)	C(11)–Ru(1)–Au(1)	140(1)				

same structural relationship is also observed between the metal frameworks of (**2**) and (**3**), with one $AuRu_2$ face of the capped trigonal-bipyramidal core[6] of the hexanuclear species (**3**) being capped by an extra $Au(PPh_3)$ fragment in the heptanuclear cluster (**2**).[3] Thus, replacing two PPh_3 groups attached to gold atoms in (**2**) and (**3**) by a $Ph_2PCH_2PPh_2$ ligand causes a very similar distortion of the metal core geometry in both cases.

The tris(monodentate phosphine) heptanuclear cluster (**2**) is known to exhibit interesting dynamic behaviour in solution at ambient temperatures. Previous variable-temperature ^1H and $^{31}P\text{-}\{^1H\}$ n.m.r. studies indicate that the metal framework is undergoing an intramolecular rearrangement which exchanges the three gold atoms between the two distinct coinage metal sites in the ground-state structure.[3] Similar studies on the analogous heptanuclear cluster (**1**) show that the two phosphorus nuclei of the $Ph_2PCH_2PPh_2$ group are equivalent on the n.m.r. time-scale.[3] As this observation is inconsistent with the ground-state structure of $[Au_3Ru_4(\mu\text{-}H)(CO)_{12}(\mu\text{-}Ph_2PCH_2PPh_2)(PPh_3)]$ (**1**) and no low-energy pathways for direct scrambling of phosphorus ligands have been reported, it would seem that the metal skeleton of (**1**) is also undergoing an intramolecular rearrangement in solution and that the fluxional process exchanges the environments of the two gold atoms attached to the bidentate diphosphine group between the two distinct types observed at Au(1) and Au(2).

A restricted Berry pseudo-rotation mechanism has previously been proposed,[1,5,6,8] for the intramolecular metal core rearrangements exhibited by a variety of heteronuclear Group 1B metal clusters which have a trigonal-bipyramidal M_2Ru_3 (M = Cu, Ag, or Au) unit incorporated into their metal skeletons. In the present work it has been shown that $[Au_3Ru_4(\mu\text{-}H)(CO)_{12}(\mu\text{-}Ph_2PCH_2PPh_2)(PPh_3)]$ (**1**) has a solid-state structure with a metal core markedly distorted from the bicapped trigonal-bipyramidal geometry, observed in its analogue $[Au_3Ru_4(\mu_3\text{-}H)(CO)_{12}(PPh_3)_3]$ (**2**), towards a bicapped square-pyramidal arrangement of the type required as an intermediate in the above mechanism. This observation provides further evidence for the theory that metal core rearrangements are also implicated in the dynamic behaviour of these clusters. In the case of the tris(monodentate phosphine) cluster (**2**), which has two distinct phosphorus environments in the solid state, a series of site interchanges between the central and each of the outer positions must be invoked to explain the equivalence of the phosphorus atoms. It is reasonable to propose that this Au_3Ru_4 core rearrangement, like that of the M_2Ru_3 clusters, proceeds by a series of metal core site exchanges involving a Berry pseudo-rotation exchange of two gold atoms, but here further accompanied by a concomitant movement of the third gold atom to an equivalent site bridging a different face. In the case of (**1**) it is the two phosphorus atoms of a bidentate ligand that are equivalent and this restricts the mobility of the two gold atoms they bridge. In the original report of the n.m.r. data insufficient evidence was available to conclude whether the $Au(PPh_3)$ group in (**1**) was involved in the rearrangement.[3] The structure established in the *X*-ray analysis makes most probable a series of polyhedral rearrangements which interchange the third gold atom, Au(3) in the Figure, between the site capping the $Au(1)Ru_2$ face to one capping a $Au(2)Ru_2$ face, thus making the environments of Au(1) and Au(2) equivalent on the n.m.r. time-scale. This could proceed by a successive change in bonding of the Au(3) atom between face-capping and edge-bridging sites, known to be relatively similar in energy,[11–13] accompanied by an overall rearrangement of the framework by a Berry pseudo-rotation mechanism.

Table 2. Fractional atomic co-ordinates, with estimated standard deviations in parentheses, for $[Au_3Ru_4(\mu\text{-}H)(CO)_{12}(\mu\text{-}Ph_2PCH_2PPh_2)(PPh_3)]$ (**1**)

Atom	*x*	*y*	*z*	Atom	*x*	*y*	*z*
Au(1)	0.095 44(8)	0.160 80(4)	0.215 92(9)	C(116)	0.032 6(12)	0.158 6(7)	−0.013 4(14)
Au(2)	−0.041 71(8)	0.097 67(5)	0.246 57(8)	C(111)	−0.026 4(12)	0.195 4(7)	0.010 0(14)
Au(3)	0.244 23(8)	0.106 03(5)	0.194 73(8)	C(122)	0.007 3(15)	0.316 8(10)	0.192 0(14)
Ru(1)	0.122 32(16)	0.048 20(9)	0.268 96(16)	C(123)	0.023 0(15)	0.372 2(10)	0.192 7(14)
Ru(2)	0.226 91(16)	0.142 12(9)	0.366 43(16)	C(124)	0.048 3(15)	0.396 6(10)	0.121 7(14)
Ru(3)	0.072 42(16)	0.104 50(10)	0.418 40(15)	C(125)	0.057 8(15)	0.365 7(10)	0.049 8(14)
Ru(4)	0.235 03(17)	0.039 97(9)	0.439 50(16)	C(126)	0.042 1(15)	0.310 4(10)	0.049 0(14)
P(1)	−0.005 1(6)	0.214 1(3)	0.123 9(5)	C(121)	0.016 8(15)	0.285 9(10)	0.120 1(14)
P(2)	−0.160 0(6)	0.150 1(3)	0.176 8(6)	C(212)	−0.306 0(14)	0.152 3(8)	0.036 0(14)
P(3)	0.332 8(5)	0.117 0(3)	0.095 0(5)	C(213)	−0.350 1(14)	0.138 8(8)	−0.049 3(14)
C(11)	0.058 2(21)	−0.009 9(13)	0.296 9(20)	C(214)	−0.311 6(14)	0.102 4(8)	−0.097 8(14)
O(11)	0.017 0(16)	−0.046 0(10)	0.309 7(16)	C(215)	−0.229 1(14)	0.079 4(8)	−0.061 1(14)
C(12)	0.221 3(23)	0.002 0(13)	0.264 8(21)	C(216)	−0.185 1(14)	0.092 9(8)	0.024 1(14)
O(12)	0.274 2(15)	−0.026 9(9)	0.252 3(14)	C(211)	−0.223 5(14)	0.129 3(8)	0.072 7(14)
C(13)	0.064 6(22)	0.042 4(13)	0.148 6(22)	C(221)	−0.240 4(14)	0.170 0(10)	0.240 5(14)
O(13)	0.040 7(16)	0.034 2(10)	0.072 0(16)	C(222)	−0.274 5(14)	0.128 2(10)	0.282 7(14)
C(21)	0.344 0(25)	0.153 4(13)	0.346 3(22)	C(223)	−0.338 3(14)	0.139 0(10)	0.331 2(14)
O(21)	0.416 5(16)	0.161 7(9)	0.343 2(15)	C(224)	−0.367 9(14)	0.191 6(10)	0.337 6(14)
C(22)	0.257 6(23)	0.159 4(13)	0.482 7(23)	C(225)	−0.333 8(14)	0.233 4(10)	0.295 3(14)
O(22)	0.287 5(18)	0.172 5(10)	0.556 1(18)	C(226)	−0.270 0(14)	0.222 7(10)	0.246 8(14)
C(23)	0.190 5(20)	0.214 0(12)	0.338 0(19)	C(311)	0.282 2(12)	0.099 4(7)	−0.016 8(9)
O(23)	0.181 3(17)	0.260 8(10)	0.337 1(16)	C(312)	0.224 9(12)	0.055 3(7)	−0.034 1(9)
C(31)	−0.025 7(25)	0.063 5(14)	0.421 4(24)	C(313)	0.187 0(12)	0.040 8(7)	−0.120 6(9)
O(31)	−0.094 6(19)	0.040 5(11)	0.429 3(17)	C(314)	0.206 4(12)	0.070 5(7)	−0.189 8(9)
C(32)	0.094 2(21)	0.122 3(12)	0.536 6(21)	C(315)	0.263 6(12)	0.114 6(7)	−0.172 6(9)
O(32)	0.111 2(16)	0.132 3(9)	0.610 6(17)	C(316)	0.301 5(12)	0.129 1(7)	−0.086 1(9)
C(33)	0.019 5(20)	0.172 6(12)	0.391 4(19)	C(321)	0.433 5(11)	0.078 2(7)	0.120 7(14)
O(33)	−0.012 9(14)	0.215 1(8)	0.380 5(13)	C(322)	0.489 0(11)	0.072 3(7)	0.061 9(14)
C(41)	0.355 1(22)	0.039 8(12)	0.420 3(20)	C(323)	0.564 8(11)	0.040 0(7)	0.083 9(14)
O(41)	0.425 2(16)	0.037 2(9)	0.410 0(14)	C(324)	0.585 1(11)	0.013 6(7)	0.164 7(14)
C(42)	0.222 8(21)	−0.035 7(13)	0.453 4(20)	C(325)	0.529 6(11)	0.019 5(7)	0.223 5(14)
O(42)	0.217 1(18)	−0.082 3(11)	0.451 5(17)	C(326)	0.453 9(11)	0.051 8(7)	0.201 5(14)
C(43)	0.277 0(23)	0.046 3(13)	0.558 8(23)	C(331)	0.367 2(17)	0.186 1(7)	0.092 6(14)
O(43)	0.299 7(18)	0.051 5(10)	0.634 8(18)	C(332)	0.455 4(17)	0.203 7(7)	0.109 8(14)
C(1)	−0.114 0(19)	0.214 4(10)	0.157 5(19)	C(333)	0.474 3(17)	0.258 5(7)	0.107 3(14)
C(112)	−0.098 2(12)	0.214 7(7)	−0.053 3(14)	C(334)	0.405 0(17)	0.295 8(7)	0.087 5(14)
C(113)	−0.111 0(12)	0.197 2(7)	−0.140 0(14)	C(335)	0.316 9(17)	0.278 3(7)	0.070 3(14)
C(114)	−0.052 0(12)	0.160 4(7)	−0.163 4(14)	C(336)	0.298 0(17)	0.224 4(7)	0.072 9(14)
C(115)	0.019 8(12)	0.141 0(7)	−0.100 1(14)	H	0.121 25	0.038 41	0.458 80

Experimental

The cluster $[Au_3Ru_4(\mu\text{-}H)(CO)_{12}(\mu\text{-}Ph_2PCH_2PPh_2)(PPh_3)]$ (**1**) was prepared as previously described[3] and crystals suitable for *X*-ray diffraction studies were grown slowly, by layer diffusion, from a dichloromethane–light petroleum (b.p. 40—60 °C) solution at −20 °C.

Crystal Data.—$C_{55}H_{38}Au_3O_{12}P_3Ru_4$, $M = 1\,978$, monoclinic, space group $P2_1/n$, $a = 15.412(2)$, $b = 24.835(3)$, $c = 15.613(2)$ Å, $\beta = 102.87(2)°$, $U = 5\,832.57$ Å^3, $F(000) = 3\,688$, $\mu(\text{Mo-}K_\alpha) = 82.76$ cm^{-1}, $Z = 4$, and $D_c = 2.25$ g cm^{-3}.

The methods of data collection, data processing, and absorption correction used for (**1**) were similar to those described previously.[14] The crystal selected for data collection had dimensions 0.44 × 0.48 × 0.21 mm. A scan width of 0.8° was used to collect data in the θ range 3—25°. 360 Azimuthal scan data were used in absorption correction and relative transmission factors varied from 1.000 to 0.810. Equivalent reflections were merged to give 5 998 data with $I/\sigma(I) > 3.0$.

Structure Solution and Refinement.[15]—The structure of (**1**) was solved from a Patterson synthesis in which two gold atoms were located. The remaining metal and non-hydrogen atoms were found from subsequent difference Fourier syntheses. The hydrido ligand site was deduced from potential energy minimisation calculations,[16] and was included in the structure factor calculations with a thermal parameter U of 0.08 Å^2, but not refined. Blocked full-matrix refinement of the atomic positional and thermal parameters converged to final R and R' values of 0.0443 and 0.0442 respectively. The phenyl groups were treated as rigid hexagons [d(C–C) = 1.395 Å] and the hydrogen atoms were included in the structure factor calculations at calculated positions [d(C–H) = 1.08 Å] with fixed thermal parameters of 0.08 Å^2. Anisotropic thermal parameters were assigned to the metal, and P atoms during the final cycles of refinement.

Fractional atomic co-ordinates are listed in Table 2. Additional material available from the Cambridge Crystallographic Data Centre comprises thermal parameters, H-atom co-ordinates, and intermolecular distances.

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