Unusual coupling of diynes and CO on an Ru_3-cluster: crystal structure of $Ru_3\{\mu_3\text{-}CPhCC(O)C(SiMe_3)C(C{\equiv}CSiMe_3)CCPh\}(\mu\text{-dppm})(\mu\text{-}CO)(CO)_6$

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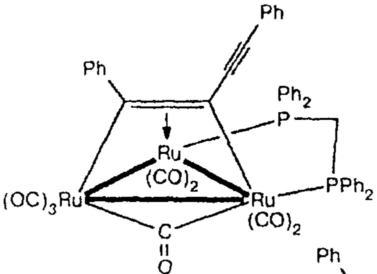
Among several products isolated in the reaction between $Ru_3(\mu_3\text{-}PhC_2C{\equiv}CPh)(\mu\text{-dppm})(CO)_8$ and $Me_3SiC{\equiv}CC{\equiv}CSiMe_3$ was the $Ru_3\{\mu_3\text{-}CPhCC(O)C(SiMe_3)C(C{\equiv}CSiMe_3)CCPh\}(\mu\text{-dppm})(CO)_7$ complex formally obtained by coupling of the two diynes and a CO molecule on the Ru_3 framework. Other products were $Ru_4(\mu_4\text{-}PhC_2C{\equiv}CPh)(\mu\text{-dppm})(CO)_{10}$ and the already known complex $Ru_3\{\mu_3\text{-}PhCCHCC(C_6H_4)\}(\mu\text{-dppm})(CO)_8$.

Key words: ruthenium, carbonyl clusters, alkyne complexes; diynes, silylalkynes; coupling of diynes and carbon monoxide.

Over the last few years, we have studied the reactions of 1,3-diynes with carbonyl clusters of ruthenium with the aim of producing complexes in which all four carbon atoms of the diyne unit interact with the metal cluster.[1–7] However, we have found that the reaction between $Ru_3(CO)_{12}$ or $Ru_3(\mu\text{-dppm})(CO)_{10}$ and diynes, such as $RC{\equiv}CC{\equiv}CR$ (R = Ph or $SiMe_3$), which was carried out under mild conditions, gave complexes of well-known types in which the diyne acts as a monoalkyne, and the second C≡C triple bond remains uncoordinated. Among other reactions, we have studied the addition of ynophilic reagents, such as $Co_2(CO)_8$, which produced a variety of products, including the bow-tie $Co_2Ru_3(\mu_5\text{-}PhC_2C_2Ph)(\mu\text{-}CO)_2(CO)_{12}$ cluster[4] and the $Co_2Ru_3(\mu_4\text{-}C_2Ph)(\mu_3\text{-}C_2Ph)(\mu\text{-dppm})(CO)_{10}$ complex in which the central C—C bond of the diyne ligand has been cleaved.[5] We have also found a series of conventional coupling reactions between the cluster-bound diyne and other monoalkynes (these results will be described elsewhere[7]).

Results and Discussion

In the present work, we describe the unusual coupling reaction that occurs between $Ru_3(\mu_3\text{-}PhC_2C{\equiv}CPh)(\mu\text{-dppm})(CO)_8$ (**1**)[2] and $Me_3SiC{\equiv}CC{\equiv}CSiMe_3$ to give the $Ru_3\{\mu_3\text{-}CPhCC(O)C(SiMe_3)C(C{\equiv}CSiMe_3)CCPh\}(\mu\text{-dppm})(CO)_7$ complex (**2**). Other products isolated in this reaction were identified as alteration products of cluster **1** and included $Ru_4(\mu_4\text{-}PhC_2C{\equiv}CPh)(\mu\text{-dppm})(CO)_{10}$ complex (**3**) and the already known $Ru_3\{\mu_3\text{-}PhCCHCC(C_6H_4)\}(\mu\text{-dppm})(CO)_8$ complex (**4**).[2] These complexes were obtained by heating cluster **1** with $Me_3SiC{\equiv}CC{\equiv}CSiMe_3$ in refluxing THF for 2 h



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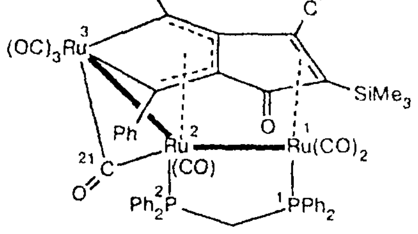

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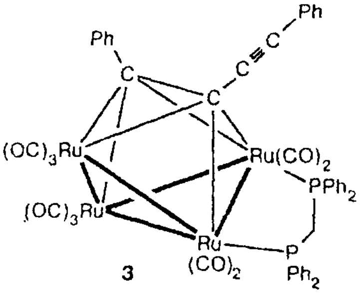

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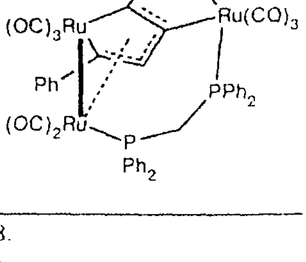

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followed by separation of the products by preparative thin-layer chromatography.

Complex **2** forms red crystals. The IR spectrum of **2** contains a weak band at 2146 cm^{-1} assigned to a ν(C≡C) absorption from the uncoordinated C≡C triple bond and an absorption band at 1893 cm^{-1} from the semi-bridging CO group (see below). Six terminal ν(CO) bands are also present. The ^{1}H NMR spectrum contains two signals at δ 0.01 and 0.18 assigned to two different SiMe$_3$ groups, a multiplet at δ 3.51 from the CH$_2$ group of the dppm ligand, and a multiplet at δ 6.27—7.59 assigned to the various Ph groups. The highest mass ion peak in the FAB mass spectrum corresponds to [M−CO]$^+$ and fragment ions formed by loss of up to six CO groups and a phenyl group are also observed.

The molecular structure of complex **2** was established by a single-crystal X-ray diffraction analysis. A plot of molecule **2** is shown in Fig. 1. The principal geometric parameters of **2** are listed in Table 1. The three Ru atoms form an open array (the Ru(1)—Ru(2) and Ru(3)—Ru(2) bond lengths are 3.161(2) and 2.720(2) Å, respectively; the Ru(1)—Ru(2)—Ru(3) angle is 124.09(6)°) with the Ru(1)—Ru(2) vector bridged by the dppm ligand (Ru(1)—P(1), 2.350(5) Å; Ru(2)—P(2), 2.377(4) Å). The Ru(2)—Ru(3) bond is semi-bridged by the C(21)O group (the Ru(2)—C(21) and Ru(3)—C(21) bond lengths are 1.90 and 2.48(2) Å, respectively, the Ru(2)—C(21)—O(21) angle is 158(1)°). The six terminal CO groups are coordinated to the Ru atoms.

The organic ligand consists of a five-membered ring, whose C(4) and C(5) atoms are attached to the Ru(1) atom by a π-type interaction (Ru(1)—C(4) and Ru(1)—C(5) bond lengths are 2.13 and 2.25(1) Å, respectively). These carbon atoms have the C≡CSiMe$_3$

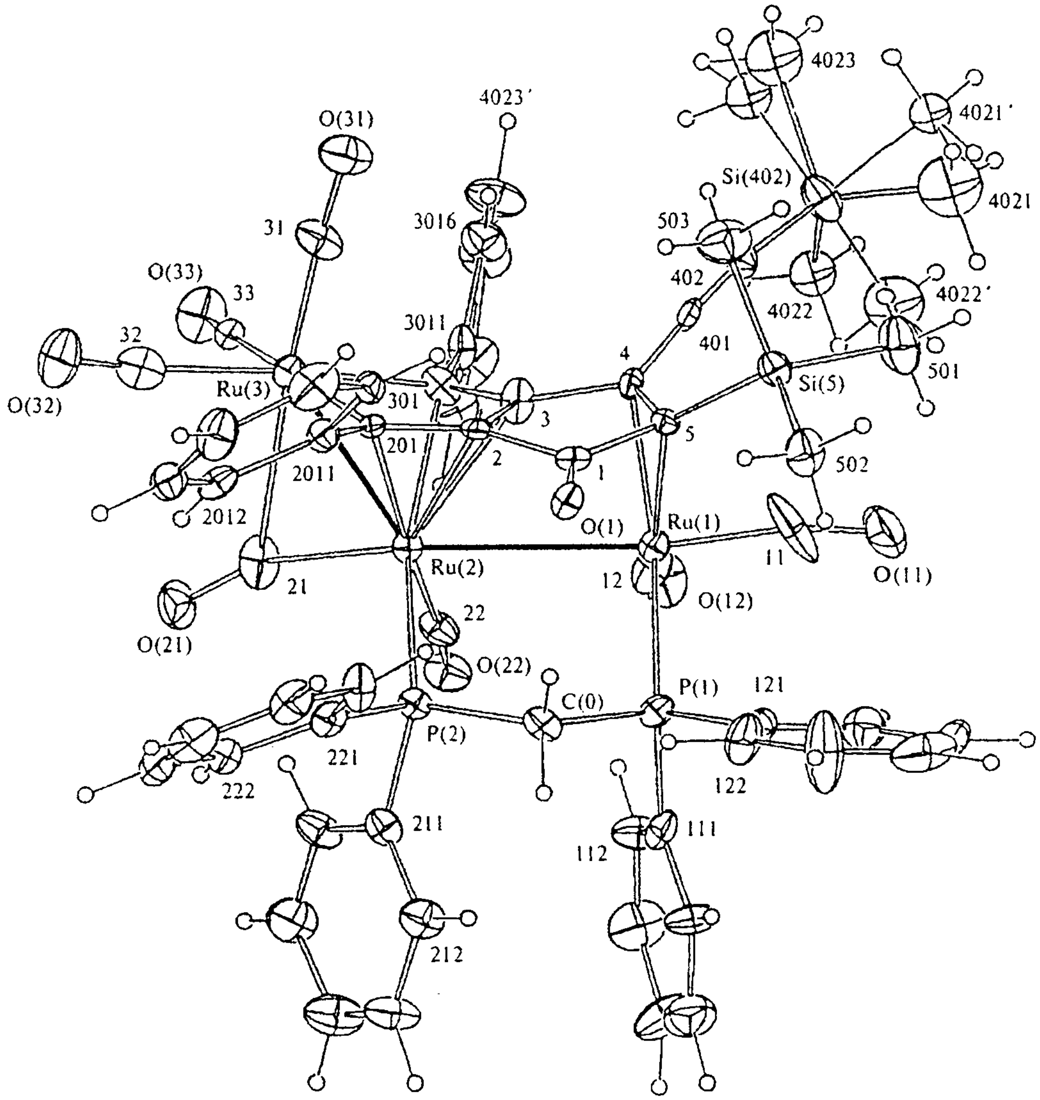

Fig. 1. Overall view of the Ru$_3${μ$_3$-CPhCC(O)C(SiMe$_3$)C(C≡CSiMe$_3$)CCPh}(μ-dppm)(μ-CO)(CO)$_6$ molecule (**2**) and the atomic numbering scheme (the C symbols are omitted for clarity).

Table 1. Selected bond lengths (*d*) and bond angles (ω) in complex **2**

Bond	*d*/Å	Bond	*d*/Å	Bond	*d*/Å	Angle	ω/deg
Ru(1)—Ru(2)	3.161(2)	Ru(2)—C(2)	2.28(2)	C(1)—O(1)	1.25(1)	Ru(1)—Ru(2)—Ru(3)	124.09(6)
Ru(2)—Ru(3)	2.720(2)	Ru(2)—C(3)	2.34(2)	C(1)—C(2)	1.53(1)	Ru(2)—Ru(1)—P(1)	90.6(1)
Ru(1)—P(1)	2.350(5)	Ru(2)—C(201)	2.33(1)	C(2)—C(3)	1.38(2)	Ru(1)—Ru(2)—P(2)	87.7(1)
Ru(2)—P(2)	2.377(4)	Ru(2)—C(301)	2.25(2)	C(2)—C(201)	1.38(2)	C(201)—Ru(3)—C(301)	79.3(5)
Ru(1)...C(1)	2.76(1)	Ru(3)—C(201)	2.11(1)	C(3)—C(4)	1.46(2)	Ru(1)—P(1)—C(0)	109.6(5)
Ru(1)...C(3)	2.57(1)	Ru(3)—C(301)	2.13(1)	C(3)—C(301)	1.45(2)	Ru(2)—P(2)—C(0)	113.4(5)
Ru(1)—C(4)	2.13(1)	P(1)—C(0)	1.80(1)	C(4)—C(5)	1.45(2)	P(1)—C(0)—P(2)	116.5(7)
Ru(1)—C(5)	2.25(1)	P(2)—C(0)	1.85(1)				

Note. Supplementary data, which involve the remaining bond lengths and bond angles and isotropic thermal parameters of the atoms in the structure of complex **2** are available from the Cambridge Crystallographic Data Centre, Cambridge CB2 1EZ, UK.

and $SiMe_3$ substituents, respectively. The C(2) and C(3) atoms are a part of the second five-membered ring containing also the C(201) and C(301) atoms (each carries the Ph group) and the Ru(3) atom (the Ru(3)—C(201) and Ru(3)—C(301) bond lengths are 2.11 and 2.13(1) Å, respectively). The C(1) atom is bonded to the O(1) atom thereby forming a carbonyl group. The Ru(3), C(201), C(2), C(3), and C(301) atoms form a five-membered ruthenole (ruthenacyclopentadiene) ring fused to the cyclopentadienone part of the ligand, and are attached to the Ru(2) atom *via* the four carbon atoms (the Ru(2)—C(ring) bond lengths are in the 2.25—2.48(2) Å range). There is no significant bonding between the C(1) and Ru(1) or Ru(2) atoms (the Ru(1)—C(1) and Ru(1)—C(2) distances are 2.76 and 3.32(1) Å, respectively).

Complex **2** is thus related to the well-established complexes of the type $Ru_2(\mu\text{-}C_4R_2R'_2)(CO)_6$ (*e.g.*, R = R′ = CO_2Me,[8] CH_2OH;[9] 1,4-R_2 = $(C_2H_4OH)_2$, 2,3-R'_2 = Et_2;[9] 1,4-R_2 = Fc_2, 2,3-R'_2 = H_2;[10] 1,3-R_2 = Ph_2, 2,5-R'_2 = $(C{\equiv}CPh)_2$ [1]) with the formation of an additional five-membered ring by the C(1), C(4), and C(5) atoms. These atoms are attached to the third Ru atom, which is also linked to the Ru(2) atom through the μ-dppm ligand. The Ru(2)—Ru(3) separation is similar to those found for these above-mentioned complexes, and each Ru atom has an 18-count. On the other hand, the attached ligands donate only eight electrons to the Ru(1) atom (2 CO + PR_3 + C=C), so that we interpret the elongated Ru(1)—Ru(2) separation as being consistent with a donor bond from the Ru(2) atom to the Ru(1) atom, which allows the latter to attain the expected 18-electron configuration.

The formation of complex **2** can be account for, formally at least, by the combination of the PhC_4Ph ligand of complex **1** with a molecule of $Me_3SiC{\equiv}CC{\equiv}CSiMe_3$ and a molecule of CO. It is interesting that the entering ligands became attached to the central two C atoms of the initial diphenylbutadiyne, thus allowing the terminal carbon atoms to chelate the Ru(3) atom. Only one of the two C≡C triple bonds of the silylated diyne entered into the reaction thus making possible further expansion of the cluster by reactions with suitable ynophiles.

The formation of cyclopentadienones from alkynes on metal carbonyls has been known for many years, but this appears to be the first occasion when this type of coupling of alkynes and CO afforded the metallabicyclo[3.3.0]octatrienone system has been described.

Two other products of this reaction contain no added silylated diyne and were identified as $Ru_4(\mu_4\text{-}PhC_2C{\equiv}CPh)(\mu\text{-dppm})(CO)_{10}$ (**3**) and $Ru_3\{\mu_3\text{-}PhCCHCC(C_6H_4)\}(\mu\text{-dppm})(CO)_8$ (**4**). Complex **3** is related to the crystallographically characterized $Ru_4(\mu_4\text{-}PhC_2C{\equiv}CPh)(CO)_{12}$ complex[1] by substitution of two CO groups (one on a hinge Ru atom and the other on a wing-tip Ru atom) for the dppm ligand. Complex **4** has been described previously[2] and was identified by comparison with the previous sample.

In conclusion, we have shown that complexes of novel structural types can be obtained by reactions of η^2-diyne metal clusters with another diyne, which in this case, however, reacts as a monoalkyne. There are some resemblances between complex **2** and product **5**, which was obtained by coupling of the second molecule of $W(C{\equiv}CC{\equiv}CH)(CO)_3Cp$ with the $Ru_3(\mu\text{-}H)\{\mu_3\text{-}C_2C{\equiv}C[W(CO)_3Cp]\}(CO)_9$ complex[11] in that the noncoordinated C≡C triple bond of the precursor enters into the coupling reaction. These observations reveal possible pathways for activation and functionalization of diynes on metal clusters. Further work will explore this possibility.

Experimental

The general experimental details are similar to those described in Ref. 1. Complex **1** and $Me_3SiC{\equiv}CC{\equiv}CSiMe_3$ were prepared according to cited methods.[1,12]

Reaction of complex 1 with $Me_3SiC{\equiv}CC{\equiv}CSiMe_3$. A solution of $Ru_3(\mu_3\text{-}PhC_2C{\equiv}CPh)(\mu\text{-dppm})(CO)_8$ (68 mg, 0.061 mmol) and $Me_3SiC{\equiv}CC{\equiv}CSiMe_3$ (24 mg, 0.122 mmol) in THF (10 mL) was refluxed for 2 h. At this time, a TLC analysis showed the presence of several products, but no starting material remained. After cooling, the solvent was removed, and the residue was separated by preparative TLC

Table 2. Nonhydrogen positional and isotropic displacement parameters for the structure of **2**

Atom	*x*	*y*	*z*	U_{eq}/Å^2
Ru(1)	0.8009(1)	0.6626(1)	0.60285(3)	0.0405(5)
Ru(2)	1.0598(1)	0.64069(9)	0.62608(3)	0.0375(5)
Ru(3)	1.1781(1)	0.5643(1)	0.69006(3)	0.0420(5)
C(11)	0.654(2)	0.657(2)	0.5938(7)	0.13(1)
O(11)	0.5591(9)	0.648(1)	0.5821(3)	0.117(8)
C(12)	0.792(1)	0.527(1)	0.5782(5)	0.070(8)
O(12)	0.7724(9)	0.4492(9)	0.5632(3)	0.084(6)
C(21)	1.211(1)	0.610(1)	0.6325(5)	0.071(8)
O(21)	1.2952(9)	0.603(1)	0.6253(3)	0.081(6)
C(22)	1.019(1)	0.556(1)	0.5840(4)	0.056(7)
O(22)	1.0056(9)	0.5003(9)	0.5608(3)	0.067(5)
C(31)	1.147(1)	0.555(1)	0.7315(4)	0.064(8)
O(31)	1.119(1)	0.547(1)	0.7569(3)	0.091(6)
C(32)	1.335(1)	0.613(1)	0.7127(5)	0.070(8)
O(32)	1.4206(9)	0.632(1)	0.7270(3)	0.103(7)
C(33)	1.225(1)	0.412(1)	0.6898(4)	0.042(6)
O(33)	1.247(1)	0.3232(9)	0.6882(3)	0.090(6)
P(1)	0.8117(4)	0.8044(3)	0.5637(1)	0.048(2)
C(111)	0.805(1)	0.778(1)	0.5182(4)	0.055(7)
C(112)	0.823(1)	0.678(1)	0.5070(4)	0.072(9)
C(113)	0.822(2)	0.659(2)	0.4724(5)	0.11(1)
C(114)	0.801(2)	0.742(2)	0.4488(5)	0.10(1)
C(115)	0.781(2)	0.846(2)	0.4593(5)	0.11(1)
C(116)	0.780(2)	0.865(1)	0.4949(4)	0.075(8)
C(121)	0.712(1)	0.912(1)	0.5611(4)	0.056(7)
C(122)	0.737(1)	1.018(1)	0.5717(5)	0.080(9)
C(123)	0.661(2)	1.096(2)	0.5689(7)	0.14(1)
C(124)	0.558(2)	1.073(2)	0.5559(5)	0.11(1)
C(125)	0.552(1)	0.972(2)	0.5443(5)	0.09(1)
C(126)	0.602(1)	0.891(1)	0.5470(5)	0.072(8)
C(0)	0.938(1)	0.879(1)	0.5807(4)	0.045(7)
P(2)	1.0660(3)	0.7978(3)	0.5912(1)	0.042(2)
C(211)	1.098(1)	0.777(1)	0.5487(4)	0.052(7)
C(212)	1.064(1)	0.851(1)	0.5220(4)	0.069(8)
C(213)	1.095(2)	0.838(1)	0.4912(5)	0.09(1)
C(214)	1.149(2)	0.749(2)	0.4856(5)	0.09(1)
C(215)	1.185(2)	0.674(1)	0.5127(5)	0.09(1)
C(216)	1.159(1)	0.690(1)	0.5435(4)	0.629(8)
C(221)	1.166(1)	0.907(1)	0.6088(4)	0.048(7)
C(222)	1.271(1)	0.896(1)	0.6048(4)	0.057(7)
C(223)	1.348(1)	0.984(1)	0.6197(5)	0.078(9)
C(224)	1.318(1)	1.071(1)	0.6358(5)	0.079(8)
C(225)	1.218(1)	1.078(1)	0.6404(5)	0.071(8)
C(226)	1.142(1)	0.994(1)	0.6277(5)	0.063(8)
C(1)	0.898(1)	0.7979(9)	0.6589(3)	0.032(6)
O(1)	0.9126(7)	0.8989(7)	0.6577(2)	0.044(4)
C(2)	0.992(1)	0.715(1)	0.6687(3)	0.039(6)
C(201)	1.105(1)	0.720(1)	0.6818(3)	0.037(6)
C(2011)	1.164(1)	0.821(1)	0.6966(4)	0.043(6)
C(2012)	1.273(1)	0.845(1)	0.6938(4)	0.049(6)
C(2013)	1.332(1)	0.933(1)	0.7107(4)	0.065(8)
C(2014)	1.287(1)	1.003(1)	0.7308(5)	0.078(9)
C(2015)	1.187(1)	0.981(1)	0.7350(5)	0.079(9)
C(2016)	1.121(1)	0.892(1)	0.7174(4)	0.047(6)
C(3)	0.941(1)	0.614(1)	0.6615(4)	0.048(7)
C(301)	1.013(1)	0.522(1)	0.6631(4)	0.051(7)
C(3011)	0.981(1)	0.404(1)	0.6597(4)	0.052(7)
C(3012)	0.983(1)	0.343(1)	0.6313(5)	0.068(8)
C(3013)	0.957(2)	0.228(1)	0.6305(5)	0.09(1)
C(3014)	0.939(2)	0.184(1)	0.6618(5)	0.09(1)
C(3015)	0.934(2)	0.245(1)	0.6876(5)	0.09(1)
C(3016)	0.955(1)	0.356(1)	0.6880(5)	0.072(8)
C(4)	0.822(1)	0.623(1)	0.6569(4)	0.035(6)
C(401)	0.753(1)	0.534(1)	0.6636(4)	0.041(6)
C(402)	0.691(1)	0.465(1)	0.6680(4)	0.047(7)
Si(402)	0.5979(4)	0.3530(4)	0.6665(2)	0.071(2)
C(4021)*	0.473(4)	0.407(4)	0.665(1)	0.15(2)
C(4022)*	0.600(3)	0.269(3)	0.631(1)	0.08(1)
C(4023)*	0.640(4)	0.264(4)	0.708(1)	0.13(2)
C(4021′)*	0.493(3)	0.404(3)	0.6918(9)	0.06(1)
C(4022′)*	0.523(4)	0.328(4)	0.620(1)	0.12(2)
C(4023′)*	0.664(3)	0.241(3)	0.695(1)	0.09(1)
C(5)	0.795(1)	0.739(1)	0.6543(3)	0.032(5)
Si(5)	0.6848(4)	0.8041(4)	0.6699(1)	0.049(2)
C(501)	0.551(1)	0.734(1)	0.6567(6)	0.09(1)
C(502)	0.666(1)	0.949(1)	0.6558(5)	0.069(8)
C(503)	0.738(2)	0.800(2)	0.7190(5)	0.09(1)
Cl(2)*	0.537(2)	0.572(2)	0.4927(7)	0.34(2)
Cl(1)*	0.515(2)	0.363(2)	0.5168(5)	0.25(1)
C(01)*	0.447(3)	0.475(3)	0.508(1)	0.10(1)

* The site occupancy factor of the atom is 0.5.

(silica gel, a 3 : 7 acetone—hexane mixture) into several bands from which the following products were further purified and characterized.

1. Recrystallization (CH_2Cl_2—EtOH) of the red solid residue obtained from the band with R_f 0.50 gave red crystals of $Ru_3\{\mu_3$-CPhCC(O)C(SiMe$_3$)C(C≡CSiMe$_3$)CCPh$\}$(μ-dppm)(CO)$_7$ · 0.5 CH_2Cl_2 (**2**) (9.1 mg, 11.4%). Found (%): C, 53.43; H, 3.87. $C_{59}H_{50}O_8P_2Ru_3Si_2 \cdot 0.5\ CH_2Cl_2$. Calculated (%): C, 52.93; H, 3.78. M = 1309. IR (*cyclo*-C_6H_{12}), ν/cm^{-1}: 2146 w (C≡C); 2079 s, 2019 m, 2010 vs, 1978 m, 1968 (sh), 1948 w, 1843 w (br) (CO). ^{1}H NMR ($CDCl_3$), δ: 0.01 (s, 9 H, SiMe$_3$); 0.18 (s, 9 H, SiMe$_3$); 3.51 (t, 2 H, CH$_2$, J_{PH} = 11 Hz), 6.27—7.59 (m, 30 H, Ph). FAB MS, *m/z* 1281 [M−CO]$^+$, 1253—1113 [M−*n*CO]$^+$ (*n* = 2−6), 1036 [M−7CO−Ph]$^+$.

2. The product from band 3 (R_f 0.33) was recrystallized (CH_2Cl_2—MeOH) to give yellow crystals of $Ru_3\{\mu_3$-CPhCHCC(C_6H_4)$\}$(μ-dppm)(CO)$_8$ (**4**) (4 mg, 6%) identified by a comparison of its IR spectrum in the ν(CO) region with that of an authentic sample.

3. Band 4 (purple, R_f 0.27) gave very dark red crystals (from C_6H_6—hexane or CH_2Cl_2—EtOH) of Ru_4(μ_4-PhC$_2$C≡CPh)(μ-dppm)(CO)$_{10}$ (**3**) (8 mg, 10%) (this compound will be described elsewhere[7]).

Two other complexes were separated (band 2, pale yellow, R_f 0.38, and band 5, red-orange, R_f 0.20), but they have not yet been identified.

X-ray diffraction analysis of complex 2. Crystals of the $Ru_3\{\mu_3$-CPhCC(O)C(SiMe$_3$)C(C≡CSiMe$_3$)CCPh$\}$(μ-dppm)(CO)$_7$ · 0.5CH$_2$Cl$_2$ ($C_{59}H_{50}O_8P_2Ru_3Si_2 \cdot 0.5CH_2Cl_2$) (**2**), M = 1350.85, are monoclinic, at −22 °C *a* = 12.654 Å, *b* = 12.227(5) Å, *c* = 39.42(2) Å, β = 105.95(3)°, *V* = 5865(7) Å^3, space group $P2_1/c$, *Z* = 4, d_{calc} = 1.530 g cm^{-3}; *F*(000) = 5568, μ(Mo-Kα) = 9.6 cm^{-1}, specimen 0.23×0.37×0.22 mm;

$A^*_{min,max}$ = 1.22, 1.28; $2\theta_{max}$ = 50°. A total of 10069 independent reflections were measured (monochromatic Mo-Kα radiation, λ = 0.71073 Å) of which 5185 reflections with $I > 3\sigma(I)$ were considered as observed and were used in the full-matrix least-squares refinement (after the Gaussian absorption correction was applied). The anisotropic thermal parameters of the nonhydrogen atoms were refined (Table 2). The hydrogen atoms were placed in calculated positions and were taken into account in the calculations of F_{theor}. The conventional residuals R and R_w based on $|F|$ were 0.077 and 0.077, respectively. The statistical weights $\sigma^2(I) = \sigma^2(I_{diff}) + 0.0004\sigma^4(I_{diff})$ were used in the refinement. Calculations were carried out using the XTAL 3.4 program package.[13] Complex scattering factors of the neutral atoms were employed. The pertinent results for complex **2** are shown in Fig. 1 and listed in Table 1. The precision of the determination is adversely affected by three factors: (1) rotational disorder of one of the $SiMe_3$ groups, which was modeled as a group distributed over two sites of equal occupancy after trial refinemnet; (2) occupation of a lattice cavity about a crystallographic inversion center by residues modeled as a disordered CH_2Cl_2 molecule of solvation; (3) the effects of profile overlap arising from the longest *c* axis (a possible source of systematic errors (care!)).

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