The 18-electron rule requires metal–metal bonds in **2**–**4** which, if present, necessitate significant deformations of the π-ligands from planarity.[1c] A single-crystal X-ray diffraction study confirms this for **2** (Figure 1), its structure contrasting with that of $[(\eta^5\text{-}C_5H_5)Ru(CO)_2]_2$, **5**.[4]

The chemistry of **2** (and also **3** and **4**)[2] is quite distinct from that of **5**.[5] For example, iodine cleaves the Ru–Ru bond to the diiodide, which when treated with LiEt$_3$BH even at −60 °C gives **2** and none of the dihydride,[2] indicating the possible operation of a fast intramolecular H$_2$-extrusion process, not available to $(\eta^5\text{-}C_5H_5)Ru(CO)_2H$.[6] **2**, although thermally inert, undergoes photosubstitution by alkynes (including ethyne) to give ("parallel")[7] $\mu_2\eta^2$-alkyne tricarbonyl complexes, characterized by a dirutheniacyclobutene nucleus and one bridging carbonyl group.[2] In the absence of added ligands irradiation of yellow **2** in THF with 350-nm or sunlight leads to rapid formation of the new, thermally unstable, colorless compound **6**.[3] X-ray analysis (Figure 2) reveals the occurrence of a remarkable (and possibly unprecedented)[8] dinuclear oxidative addition to an sp^2–sp^2 hydrocarbon single bond.

Structures related to **6** have been postulated as precursors to (fulvalene)dimetal complexes,[1m–t] the former reacting by the thermal reverse of the **2** → **6** interconversion, although reported cases have involved metal hydride species at some stage of their formation. Nevertheless, **6** thermally reverts cleanly to **2** (THF, room temperature) following first-order kinetics ($E_a = 21.9 \pm 1.0$ kcal mol^{-1}, log $A = 11.7$). This also occurs sharply in the crystalline state (208 °C; $\Delta G = -29.8 \pm 1.5$ kcal mol^{-1} by DSC). The **2** → **6** → **2** sequence represents a new photochemical energy storage cycle.

A crossover experiment involved a 1:1 mixture of **2** and 90% deuterated **2**.[9] The absence of any crossover was ascertained by mass spectral analyses of the resulting **6** and of subsequently regenerated **2**. This rules out the presence of monomeric (metal–carbene?)[10] intermediates in either process.

Although the exact mechanistic details of the reported reactions are not understood, it is clear that hydrides are not necessary for their occurrence. It is tempting to postulate initial photocleavage of the Ru–Ru bond,[4b] followed by rotation and rearrangement, possibly involving bridging cyclopentadienylidenes.[11] Interestingly, **2** converts to **6** in the presence of chlorinated solvents, normally excellent traps for 17-electron metal centers,[12] making this pathway less attractive. An alternative is a concerted process through a tetrahedral transition state.[1o]

Acknowledgment. This work was supported by the Director, Office of Energy Research, Office of Basic Energy Sciences, Chemical Sciences Division of the U.S. Department of Energy under Contract DE-AC03-76SF00098. The crystal structure analyses were performed by Dr. F. J. Hollander, U.C. Berkeley X-ray Crystallographic Facility. K.P.C.V. is a Camille and Henry Dreyfus Teacher-Scholar (1978–1983).

Supplementary Material Available: Spectral properties of all new compounds, details of the X-ray analysis, a listing of positional and thermal parameters, and tables of bond lengths and angles of **2** and **6** (9 pages). Ordering information is given on any current masthead page.

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[3] **1**: dark red cubes, 80%; mp 97–98 °C. **2**: yellow plates, 78%; mp 288–290 °C; ^{1}H NMR (200 MHz, acetone-d_6) δ 5.90 (dd, 4 H, J = 2.2, 2.1 Hz), 4.40 (dd, 4 H, J = 2.1, 2.1 Hz); IR (CH$_2$Cl$_2$) ν_{CO} 2020 (vs), 1952 (vs) cm^{-1}; UV λ_{max} (THF) 243 nm (log ϵ 3.99), 273 (4.04), 329 (3.85), 388 sh (3.18). **3**: purple crystals, 60%; mp 279–280 °C (lit.[1a] mp not reported). **4**: orange flakes, 18%; mp 256–258 °C. **6**: colorless plates, 62%; mp 208 °C (isomerization point to **2**); ^{1}H NMR (CDCl$_3$) δ 5.39 (dd, 4 H, J = 2.1, 2.1 Hz), 4.68 (dd, 4H, J = 2.1, 2.1 Hz); IR (KBr) ν_{CO} 2000 (vs), 1960 (vs) cm^{-1}; UV λ_{max} (THF) 239 nm (log ϵ 3.90), 286 sh (3.18).

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Phosphine Ligands for the Construction of Polynuclear Complexes. 1. Bis(diphenylphosphinomethyl)phenylphosphine Complexes of Palladium(II) and Rhodium(I)

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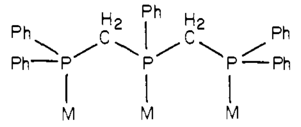
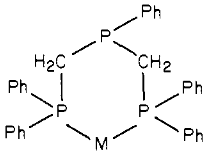
Binuclear transition-metal complexes bridged by phosphine ligands such as bis(diphenylphosphino)methane (dpm)[1] and 2-(diphenylphosphino)pyridine (Ph$_2$Ppy)[2] have been shown to provide a rich array of structural forms and an extensive reaction chemistry. We have become interested in obtaining complexes containing linear or nearly linear arrays of several metal atoms and have chosen to examine the coordinating properties of several polyphosphines related to dpm and Ph$_2$Ppy. Here we describe complexes formed from bis(diphenylphosphinomethyl)phenylphosphine (dpmp).[3] This ligand represents an elaboration of the structural elements found in dpm. It has the potential for binding three metal centers in a row, A, or forming a chelate ring about one metal ion, B. In the cases described here, the interaction of this ligand with two different d^8 metal ions, Pd(II) and Rh(I), leads to the formation of both structural types.

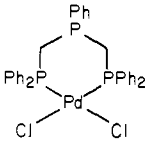


A



B

The reaction of equimolar quantities of dpmp with bis(benzonitrile)palladium(II) chloride in dichloromethane solution followed by the addition of ethyl ether yields the cream-colored crystalline complex **1** in 76% yield. The ^{31}P{^{1}H} NMR spectrum



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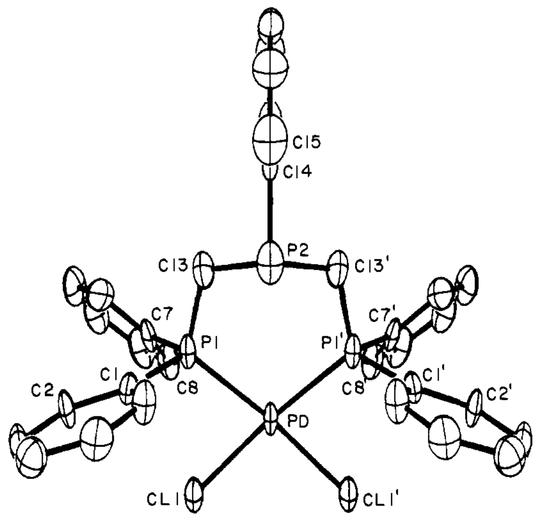
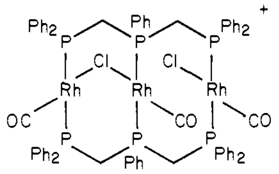


Figure 1. Perspective drawing of (dpmp)PdCl$_2$. Some significant interatomic distances (Å) and angles (deg) are as follows: Pd–Cl, 2.354 (3); Pd–P(1), 2.259 (3); Pd···P(2), 3.893 (5); Cl–Pd–Cl, 88.7 (2); P–Pd–P, 99.3 (2).

of **1** consists of a doublet at 18.3 ppm due to the two terminal phosphorus atoms and a triplet at −40.7 ppm due to the internal phosphorus atom (^2J(P,P) = 62 Hz). The structure of **1** as determined by an X-ray crystallographic study is shown in Figure 1.[4] A mirror plane passes through the palladium atom, the central phosphorus atom, and the edge of the central phenyl group. Only the terminal phosphorus atoms are coordinated to the palladium atom. The central phosphorus atom is 3.893 (5) Å from the palladium atom and is not bonded to it. The chelate ring has adopted a boat conformation.[5]

The reaction of dpmp with dicarbonylrhodium(I) chloride dimer in dichloromethane/methanol mixtures yields a deep rose-colored solution from which the cation **2** has been isolated in 40% yield as the Cl$^-$, BPh$_4^-$, and PF$_6^-$ salts. This cation possesses only terminal carbonyl groups as shown by the infrared spectra of the tetraphenylborate salt both as a solid (ν(CO) 1988, 1972, 1965, 1958 sh cm^{-1}) and in dichloromethane solution (ν(CO) 1985, 1970, 1965 sh, 1955 sh cm^{-1}).



2

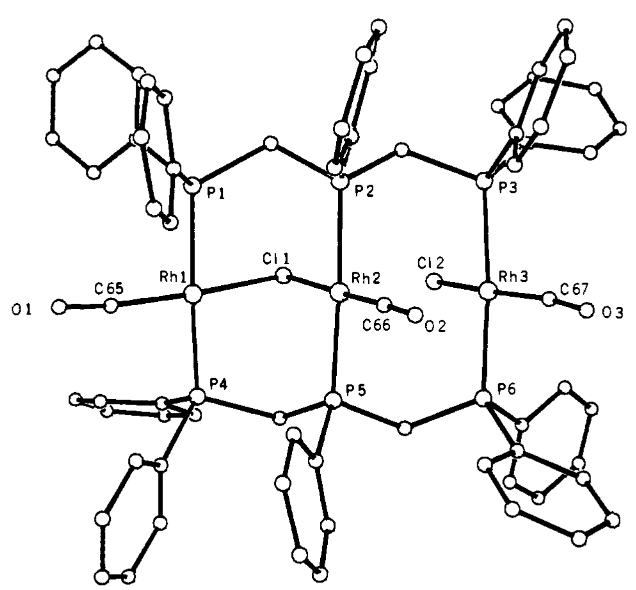
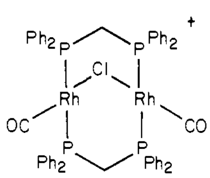
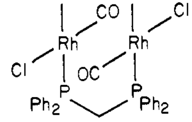


Figure 2. Perspective drawing of [(μ-dpmp)$_2$Rh$_3$(μ-Cl)Cl(CO)$_3$]$^+$. Some significant interatomic distances (Å) and angles (deg) are as follows: Rh(1)···Rh(2), 3.164 (3); Rh(2)···Rh(3), 3.180 (3); Rh(1)–Cl(1), 2.383 (6); Rh(2)–Cl(1), 2.401 (6); Rh(3)–Cl(2), 2.376 (6); Rh(1)–C(65), 1.87 (2); Rh(2)–C(66), 1.80 (3); Rh(3)–C(67), 1.83 (2); Rh(1)–P(1), 2.339 (7); Rh(1)–P(4), 2.324 (7); Rh(2)–P(2), 2.318 (7); Rh(2)–P(5), 2.324 (7); Rh(3)–P(3), 2.328 (7); Rh(3)–P(6), 2.313 (7); Rh(3)···Cl(1), 3.476 (6); Rh(1)···Rh(2)···Rh(3), 157.4 (1); C(65)–Rh(1)–Cl(1), 171.5 (7); C(66)–Rh(2)–Cl(1), 178.3 (7); C(67)–Rh(3)–Cl(2), 167.5 (8).

The details of the geometry of **2** have been obtained by X-ray crystallography.[6] The structure of the cation is shown in Figure 2. It consists of a slightly bent array of three rhodium atoms that are bridged by two triphosphine ligands. Each rhodium atom is connected to a terminal carbonyl group. One chloride ligand bridges Rh(1) and Rh(2), while the other chloride acts as a terminal ligand. There exist close correspondences between structural portions of **2** and previously characterized, dpm bridged, binuclear rhodium(I) complexes. Thus, the left side Rh$_2$P$_4$(μ-Cl)(CO)$_2$ unit in **2** is similar to the A-frame cation **3**, which has Rh···Rh 3.1520 (8) Å and Rh–Cl–Rh 82.38 (5)°.[7] The Rh$_2$P$_4$(CO)$_2$Cl$_2$ unit on the right side of **2** is related to the face-to-face rhodium dimers such as **4**, which has a Rh···Rh separation of 3.2386 (5) Å.[8] While the Rh···Rh separations in **2**–**4** are all longer than expected for Rh–Rh single bonds, the electronic interaction between the metal ions in **2** is demonstrated by the position of the proximity shifted[9] absorption band, which occurs at 560 nm for **2** relative to 442 nm for **3**[10] and 450 nm for **4**.[9]



3



4

The ^{31}P{^{1}H} NMR spectrum of [(dpmp)$_2$Rh$_3$(μ-Cl)Cl(CO)$_3$][BPh$_4$] indicates that there are only two chemically unique phosphorus environments, δ(interior) 15.3 and δ(terminal) 23.0, in dichloromethane solution. The spectrum remains invarient over

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[4] Single crystals of (dpmp)PdCl$_2$·CH$_2$Cl$_2$ were grown by diffusion of ethyl ether into a dichloromethane solution of the complex. They belong to the space group $P2_1/m$(No. 11) with a = 9.864 (1) Å, b = 17.513 (8) Å, c = 10.706 (10) Å, β = 112.85 (6)°, Z = 2, at 140 K. R = 7.3% for 2021 reflections with $I > 3\sigma(I)$ and 214 parameters.

[5] The uncoordinated phosphorus atom in **1** and in other chelated complexes of dpmp can bind other metal centers to give bi- and trinuclear complexes. Guimerans, R. R.; Olmstead, M. M.; Balch, A. L., to be submitted for publication.

[6] Single crystals of [(μ-dpmp)$_2$Rh$_3$(μ-Cl)Cl(CO)$_3$]Cl·1.5C$_2$H$_2$Cl$_4$ were grown by diffusion of ethyl ether into a tetrachloroethane solution of the complex. They belong to the space group $P2_1/c$(No. 14) with a = 12.821 (3) Å, b = 25.206 (14) Å, c = 23.233 (11) Å, β = 99.67 (3)°, Z = 4, at 140 K. R = 11.8% for 5522 reflections with $I > 2\sigma(I)$ and 493 parameters. A severely disordered molecule of C$_2$H$_2$Cl$_4$ and poor counting statistics (4160 reflections were rejected with $I < 2\sigma(I)$), which resulted from difficulty in obtaining a good quality crystal, have thwarted our attempts to achieve a better R value. We are attempting to obtain a more suitable crystal of the cation and a full report will appear.

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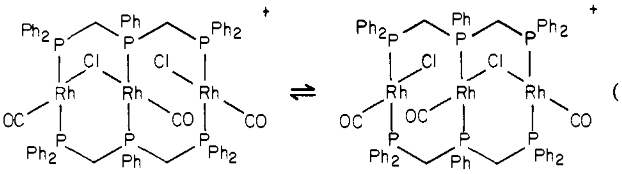
the temperature range +25 to −70 °C. To account for the equivalence of all four of the terminal phosphorus atoms, we propose that, in solution, the chloride ligands undergo rapid bridge–terminal exchange as shown in eq 1. This operation requires only minimal geometric displacements of the carbonyl group on Rh(2) and the chloride ligands. Note that the nonbonded Rh(2)···Cl(2) separation is only 1.087 Å longer than the Rh(2)–Cl(1) bond length.



(1)

Preliminary results indicate that $[(\mu\text{-dpmp})_2Rh_3(\mu\text{-Cl})Cl(CO)_3]^+$ has a rich reaction chemistry. Adducts are readily formed with sulfur dioxide and activated acetylenes. Although **3** readily and reversibly adds carbon monoxide in the trough between the two rhodium centers to form the double A-frame, $[(\mu\text{-dpm})_2Rh_2(\mu\text{-CO})(\mu\text{-Cl})(CO)_2]^+$,[10] carbon monoxide (at 1 atm) does not add to the similarly sized trough in **2**.

Acknowledgment. We thank the National Science Foundation (CHE 7924575) for support. Acquisitions of the X-ray diffractometer and NMR spectrometer used in these studies were made possible through NSF equipment grants.

Registry No. 1, 84751-01-9; $\mathbf{2}^+Cl^-\cdot CH_2Cl_2$, 84751-03-1; $\mathbf{2}^+BPh_4^-$, 84774-75-4; $\mathbf{2}^+PF_6^-$, 84774-76-5; bis(benzonitrile)palladium(II) chloride, 14220-64-5; dicarbonylrhodium(I) chloride dimer, 14523-22-9.

Supplementary Material Available: List of atomic fractional coordinates and thermal parameters for $(dpmp)PdCl_2\cdot CH_2Cl_2$ and $[(\mu\text{-dpmp})_2Rh_3(\mu\text{-Cl})Cl(CO)_3]Cl\cdot 1.5C_2H_2Cl_4$ and the $^{31}P\{^1H\}$ NMR spectrum of **2** (4 pages). Ordering information is given on any current masthead page.

Preparation and Reactivity of a Dimeric Ruthenium μ-Methylene Complex with No Metal–Metal Bond: Crystal and Molecular Structure of $[(\eta^5\text{-}C_5H_5)Ru(CO)_2]_2(\mu\text{-}CH_2)$

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Received November 12, 1982

Transition-metal methylene complexes have attracted attention because of their proposed role in catalytic reactions and their novel structural and chemical features.[1] All known bridging methylene complexes contain either a metal–metal bond or an additional bridging ligand.[2] Indeed, it has been postulated that these are necessary structural features to stabilize μ-methylene complexes.[1] Here we report the isolation and characterization of a methylene complex without these features. We have found that this compound is far more reactive than analogous alkyl or metal–metal bonded μ-methylene complexes; for example, it readily inserts CO, affording a complex containing the $RuC(O)CH_2Ru$ fragment.

The μ-methylene complex $[CpRu(CO)_2]_2(\mu\text{-}CH_2)$ (**1**) was prepared by reacting $Na[CpRu(CO)_2]$ with 0.5 equiv of methylene chloride at −35 °C in THF and isolated by warming the reaction mixture to room temperature, evaporating the solvent, and recrystallizing from hexane (eq 1).[3] The complex decomposes at room temperature over a 24 h period but can be kept indefinitely at −35 °C. Use of $CH_2(OTs)_2$ instead of CH_2Cl_2 gave **1** in lower yield.

$$Na[CpRu(CO)_2] + {}^1/_2CH_2Cl_2 \rightarrow {}^1/_2\underset{\mathbf{1}}{Cp(CO)_2Ru(CH_2)Ru(CO)_2Cp} \quad (1)$$

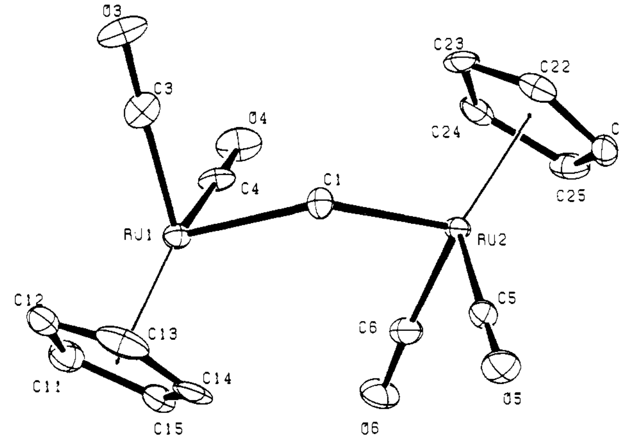
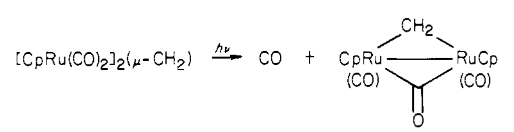


Figure 1. ORTEP drawing of $[(\eta^5\text{-}C_5H_5)Ru(CO)_2]_2(\mu\text{-}CH_2)$. The vibrational ellipsoids are drawn at the 50% probability level.

The structure[4] of **1** in Figure 1 shows that the two ruthenium atoms are symmetrical about the μ-CH_2 group. The Ru–C distance of 2.18 Å (av) is slightly longer than those in $[CpRu(CO)]_2(\mu\text{-CO})(\mu\text{-CHCH}_3)$ (2.08 Å) and $[CpRu(CO)]_2[\mu\text{-C}(CH_3)_2][\mu\text{-C}(CH_3)_2]$ (2.11 Å).[5] In these complexes, however, the Ru–Ru distance of 2.7 Å and the ∠Ru–C–Ru of 80° indicate a metal–metal bond. In **1**, which lacks this bond, the Ru–Ru distance is 3.8 Å (av) and the ∠Ru–C–Ru is 123°. This metal–metal separation is greater than that found in $[Pt_2Cl(CH_2PPh_3)](\mu\text{-dppm})_2(\mu\text{-}CH_2)$.[2a] The ∠Ru–C–Ru is somewhat larger than expected for the sp[3] hybridization. This large angle and relatively long Ru–C distance suggests steric crowding about the methylene group, which may be responsible for its unusual reactivity.

Photolysis of **1** gave an 80% yield of the cis and trans isomers of the metal–metal bonded μ-methylene dimer $[CpRu(CO)]_2(\mu\text{-}CH_2)(\mu\text{-CO})$ (**2**), previously prepared from $(Ph)_3P{=}CH_2$ and $[CpRu(CO)]_2(\mu\text{-CO})(\mu\text{-C(O)C}_2(Ph)_2)$.[6] A similar decarbonylation with concomitant metal–metal bond formation occurs for $[CpFe(CO)_2]_2[\mu\text{-Si(H)(CH}_3)]$.[7] Carbonylation of **1** occurs readily (40 psig at room temperature) to give $[CpRu(CO)_2]_2(\mu\text{-}CH_2C(O))$ (**3**; ~80%).[8] Authentic **3** was prepared in low yield



$$[CpRu(CO)_2]_2(\mu\text{-}CH_2) \xrightarrow{h\nu} CO + \underset{(CO)}{CpRu}\overset{CH_2}{\underset{\underset{O}{\overset{\|}{C}}}{\text{———}}}\underset{(CO)}{RuCp} \quad (2)$$

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† Contribution No. 3160.

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(3) ^1H NMR (C_6D_6) δ 4.80 (10 H, s), 2.30 (2 H, s); ^{13}C NMR (CD_2Cl_2) δ 203.50 (CO), 90.41 (C_5H_5, $J_{C\text{-}H} = 177.9$ Hz, $J_{C\text{-}H'} = 6.8$ Hz), −37.42 (CH_2, $J_{C\text{-}H} = 137.2$ Hz). Anal. Calcd (found): C, 39.30 (39.24); H, 2.62 (2.75).

(4) Crystal data for $Ru_2C_{15}H_{12}O_4$: monoclinic, $P2/c$, $a = 19.020$ (3) Å, $b = 6.239$ (1) Å, $c = 20.046$ (4) Å, $\beta = 108.63$ (1)°, $V = 2254.1$ Å^3, ρ (calcd) = 2.026 g/cm^3, $Z = 6$, $\mu = 19.834$ cm^{-1}, crystal faces ±[100, 0.10; −102, 0.0083; −210, 0.350], 3421 reflections with $I > 2\sigma(I)$ (−100 °C), analytically corrected for absorption, 258 variables, $R = 0.058$, $R_w = 0.061$. One molecule lies in a general position while a second half molecule is related to itself by the 2-fold axis.

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