

have been considered as adopting a semibridging mode.¹⁴ The Os-I bond length of 2.732 (2) Å agrees with other reported values for terminal iodide groups.

The structural parameters for bridged and unbridged bonds [2.951 (2) and 2.799 (2) Å, respectively] ought to be compared with the values reported for other isoelectronic species such as [H₂Os₄(CO)₁₂]²⁻ [2.934 (4), 2.798 (4) Å], [H₃Os₄(CO)₁₂]⁻¹⁵ [2.949 (2), 2.798 (2) Å], and [H₃Os₄(C-O)₁₁NO]⁶ [2.925 (2), 2.799 (2) Å] showing substantial agreement. However, a similar comparison with the values reported for 1 [3.010 (3), 2.877 (1) Å, respectively]^{3d} shows that a general lengthening in the metal-metal bonds is observed on passing from a tetrahedral to a butterfly arrangement of the metal core. Longer bonds were also observed for the butterfly-shaped [H₃Os₄(CO)₁₂(μ-NO)] [2.997 (5), 2.853 (5) Å].¹⁶

It is interesting to speculate about why the actual asymmetric stereochemistry is preferred to a more symmetric C_s configuration. A tentative rationalization could be that one H atom prefers coordination to the more electron-rich Os(1), leaving two nonequivalent sites for the second one. One of the sites [Os(2)-Os(3), Os(2)-Os(4)] is twice as probable as the other [Os(3)-Os(4)]. The statistically favored position could thus be preferred when the molecule is "frozen" in the crystal.

The facile reversible⁸ structural transformation herein described (Scheme 1) is certainly enhanced by the availability of different bonding modes for the halide ligand; this allows a modification of the iodide from a doubly bridging, 3e donor to a terminal, 1e donor ligand and vice versa.

Three examples of similar metal framework transformations in Fe₄ clusters have been confirmed by structural characterization of the species involved, viz., [Fe₄(CO)₁₃]²⁻ (tetrahedral), which reversibly yields [HFe₄(CO)₁₃]⁻ (butterfly) on protonation,^{10d} and [Fe₄(CO)₁₂(COCH₃)]⁻ (tetrahedral), which produces Fe₄X(CO)₁₂(COCH₃) (X = H,^{10e} AuPEt₃^{10g}) (butterfly) by reaction with acid and PEt₃ AuCl, respectively. Also, the existence of both tetrahedral and butterfly isomers of [XFe₄(CO)₁₃]⁻ (X = H,^{10f} AuPEt₃^{10g}) in solution has been proposed on the basis of NMR data. All these rearrangements are accompanied by interconversion of terminal and bridging (or η) carbonyl ligands.

However, to our knowledge, this is the first report of such a reversible structural transformation assisted by a halide ligand. This has important mechanistic implications in a number of reactions of metal clusters, including catalytic processes, which are known to be promoted by halides, and particularly iodide.⁴ Preliminary studies on the reactivity of 2 toward the electrophilic reagents [AuPPh₃]⁺ and NO⁺ indicate that its behavior is considerably different from that of the isoelectronic [H₃Os₄(C-O)₁₂]⁻.

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Registry No. 1, 67966-16-9; 2, 98331-78-3.

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Supplementary Material Available: Crystal data and details of measurement, Figure 2, showing two different orientations of the anion [H₂Os₄(CO)₁₂(I)]⁻ (2), Table I, fractional atomic coordinates and thermal parameters, Table II, anisotropic thermal parameters, Table III, bond distances, Table IV, bond angles, Table V, contacts, and listings of observed and calculated structure factors for 2 (53 pages). Ordering information is given on any current masthead page.

Skeletal Reactivity of Metallophosphorus Clusters. *nido*-Ru₄(CO)₁₀(μ-CO)₂[PhPC(Ph)C(Ph)] and *closo*-Ru₄(CO)₉(μ-CO)₂(PhP)[C(Ph)C(Ph)] via Acetylene Incorporation into the Nido Framework of the Phosphinidene Cluster Ru₄(CO)₁₃(μ₃-PPh)

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Summary: Reaction of the butterfly phosphinidene complex Ru₄(CO)₁₃(μ₃-PPh) with diphenylacetylene affords two metal-carbon-phosphorus clusters, *nido*-Ru₄(CO)₁₀(μ-CO)₂[P(Ph)C(Ph)C(Ph)] and *closo*, pentagonal-bipyramidal Ru₄(CO)₉(μ-CO)₂(μ₃-PPh)(μ₄-η²-PhC₂Ph), characterized by X-ray diffraction and having metal, carbon, and phosphorus atoms in the skeletal framework.

The metallophosphorus clusters,¹ molecules which include both metal and phosphorus atoms in a polyhedral framework are members of a potentially vast class of mixed transition metal-main group compounds.² Such molecules are currently of structural and chemical interest because they offer the opportunity to evaluate the validity of current models of bonding for mixed polynuclear systems³

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(2) Between the extremes of the homonuclear transition-metal clusters and main-group cages such as the polyhedral boranes lie mixed species such as the metalboranes and carboranes, carbido, nitrido, phosphido, and sulfido clusters, and numerous metal-hydrocarbon π-complexes in which the carbon atoms can be considered as skeletal. For general surveys of types see: (a) O'Neill, M. E.; Wade, K. In "Metal Interactions with Boron Clusters"; Grimes, R. N. Ed.; Plenum Press: New York, 1982. (b) "Transition Metal Clusters"; Johnson, B. F. G., Ed.; Wiley: New York, 1980. (c) Vahrenkamp, H. *Adv. Organomet. Chem.* 1983, 22, 169. (d) Adams, R. D.; Horvath, I. T. *Prog. Inorg. Chem.* 1985, 33, 127.

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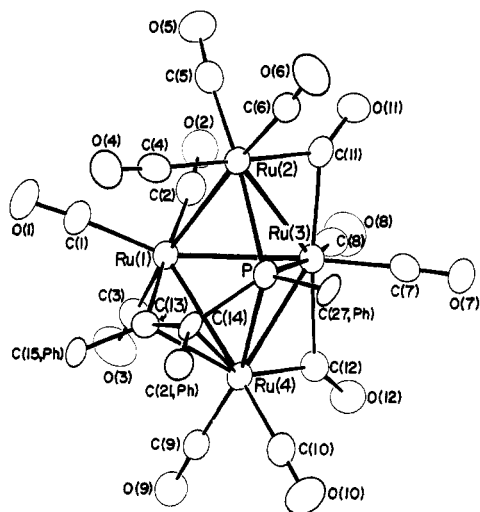


Figure 1. A perspective view of the structure of one independent molecule (molecule A) of $\text{Ru}_4(\text{CO})_{10}(\mu\text{-CO})_2[\text{P}(\text{Ph})\text{C}(\text{Ph})\text{C}(\text{Ph})]$, **2**, showing the atomic numbering. Structural parameters for molecules A and B are virtually identical. Important bond lengths (Å) and angles (deg) not in the text are (molecule A) as follows: $\text{Ru}(1)\text{-Ru}(2) = 2.890$ (1), $\text{Ru}(1)\text{-Ru}(3) = 2.879$ (1), $\text{Ru}(1)\text{-Ru}(4) = 2.881$ (1), $\text{Ru}(2)\text{-Ru}(3) = 2.878$ (1), $\text{Ru}(3)\text{-Ru}(4) = 2.762$ (1), $\text{Ru}(2)\cdots\text{Ru}(4) = 4.926$ (1), $\text{C}(13)\text{-C}(14) = 1.385$ (12), $\text{Ru}(1)\text{-C}(13) = 2.168$ (8), $\text{Ru}(4)\text{-C}(13) = 2.270$ (8), $\text{Ru}(4)\text{-C}(14) = 2.311$ (9) Å; $\text{C}(14)\text{-C}(13)\text{-C}(15) = 119.7$ (5), $\text{C}(13)\text{-C}(14)\text{-C}(21) = 125.3$ (5).

and to improve on the degradative instability of homonuclear metal compounds. Although the μ_3 - or μ_4 -phosphinidene group is usually thought of as a (stabilizing) face bridging ligand, it is clear from structural studies and electron counting that PR units may be considered as an integral part of the skeletal framework. As such and given the polarity of the metal-phosphorus bonds, cluster reactions implicating μ_3 -PR or μ_4 -PR groups are to be expected. Of particular significance in this context are butterfly complexes since the related M_4C cluster carbides⁴ and nitrides⁵ have a well-developed and intriguing chemistry. We now report that $\text{Ru}_4(\text{CO})_{13}(\text{PPh})$, **1**, a molecule with a phosphinidene group coordinated between the wings of an Ru_4 butterfly undergoes skeletal expansion reactions with diphenylacetylene leading to the incorporation of a two-carbon fragment into the cluster framework. These reactions reveal a new and potentially significant aspect of metallophosphorus cluster chemistry and open a novel route to an interesting class of ternary (M, P, C) clusters. The products *nido*- $\text{Ru}_4(\text{CO})_{10}(\mu\text{-CO})_2[\text{PhPC}(\text{Ph})\text{C}(\text{Ph})]$ and *closo*- $\text{Ru}_4(\text{CO})_9(\mu\text{-CO})_2(\text{PhP})(\text{C}(\text{Ph})\text{C}(\text{Ph}))$ have seven-vertex polyhedral structures which can be rationalized within the framework of the skeletal electron counting (SEC) rules.

Treatment of **1**⁵ (0.5 g) in benzene (50 mL) with diphenylacetylene (1 equiv) at 60 °C for 2 h followed by column chromatography (Florisil, eluant heptane/benzene) afforded two fractions which on crystallization at -5 °C in heptane/toluene gave red **2** (40%) (IR (C_6H_{12}) 2094 m, 2059 vs, 2048 vs, 2041 sh, 2020 sh, 2010 s, 1986 m cm^{-1} ; ³¹P

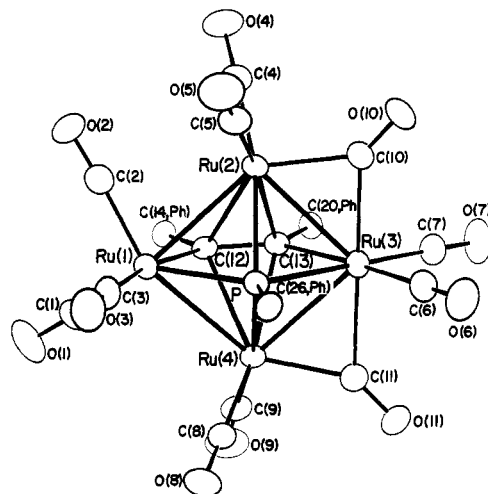
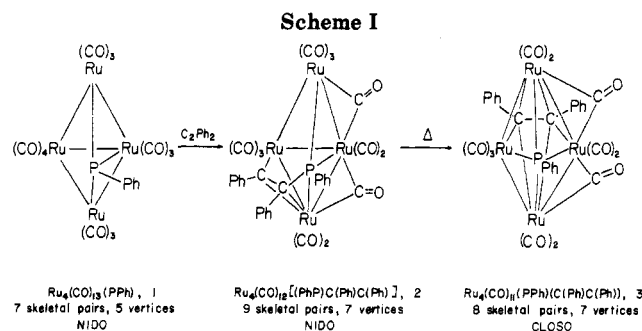


Figure 2. The molecular structure of $\text{Ru}_4(\text{CO})_9(\mu\text{-CO})_2(\text{PPh})\text{C}(\text{Ph})\text{C}(\text{Ph})$, **3**, drawn so as to illustrate the pentagonal-bipyramidal skeleton. Important bond distances (Å) and angles (deg) are as follows: $\text{Ru}(1)\text{-Ru}(2) = 2.8515$ (5), $\text{Ru}(1)\text{-Ru}(4) = 2.8746$ (5), $\text{Ru}(2)\text{-Ru}(3) = 2.7432$ (5), $\text{Ru}(3)\text{-Ru}(4) = 2.7777$ (5), $\text{Ru}(1)\cdots\text{Ru}(3) = 4.1204$ (5), $\text{Ru}(2)\cdots\text{Ru}(4) = 3.7651$ (5), $\text{Ru}(1)\text{-P} = 2.359$ (1), $\text{Ru}(2)\text{-P} = 2.431$ (1), $\text{Ru}(3)\text{-P} = 2.395$ (1), $\text{Ru}(4)\text{-P} = 2.416$ (1), $\text{C}(12)\text{-C}(13) = 1.402$ (6); $\text{C}(14)\text{-C}(12)\text{-C}(13) = 117.5$ (2), $\text{C}(20)\text{-C}(13)\text{-C}(12) = 119.9$ (2).



NMR (C_7D_8 , 223 K, 101.2 MHz) $\delta +229.3$) and dark red **3** (IR (C_6H_{12}) 2085 s, 2055 vs, 2043 vs, 2033 vs, 2020 s, 2008 m, 1986 m, 1975 sh cm^{-1} ; ³¹P NMR (C_7D_8 , 233 K) $\delta +352$). The structures of both molecules were solved by single-crystal X-ray diffractometry.⁶ Compound **2** (Figure 1) has the molecular formula $\text{Ru}_4(\text{CO})_{10}(\mu\text{-CO})_2[\text{PPhC}(\text{Ph})\text{C}(\text{Ph})]$ and is derived from the precursor **1** by loss of a single CO group, coupling of one carbon atom of the acetylene with the phosphorus atom of the phosphinidene and the other carbon atom with one of the ruthenium atoms in the hinge of the butterfly (Scheme I). The five Ru-Ru bonds in **1**⁵ are retained in **2**, and the PPh fragment originally strongly

(6) Crystal data for **2**: $\text{Ru}_4\text{PO}_{11}\text{C}_{32}\text{H}_{15}\cdot 0.5\text{C}_7\text{H}_8\cdot 0.25\text{C}_6\text{H}_6$; $M_r = 1092.32$; triclinic, space group $P\bar{1}$; $a = 12.158$ (2), $b = 17.591$ (2), $c = 21.741$ (3) Å; $\alpha = 112.19$ (1), $\beta = 92.20$ (1), $\gamma = 113.08$ (1)°; $Z = 4$; $\rho_{\text{meas}} = 1.87$, $\rho_{\text{calc}} = 1.877$ g cm^{-3} ; $F(000) = 2106$; $\mu(\text{Mo K}\alpha) = 16.0$ cm^{-1} . The structure was solved by Patterson and Fourier methods using 7102 observed ($I \geq 3\sigma(I)$) (10166 measured) reflections measured on a Syntex P21 diffractometer to R and R_w values of 0.034 and 0.039, respectively.

There are two independent molecules in the asymmetric unit. Distances quoted in the text are averages of the values in the two molecules. Crystal data for **3**: $\text{Ru}_4\text{PO}_{11}\text{C}_{31}\text{H}_{15}$; $M_r = 998.71$; triclinic, space group $P\bar{1}$; $a = 9.393$ (2), $b = 9.519$ (1), $c = 19.556$ (2) Å; $\alpha = 79.15$ (1), $\beta = 84.33$ (1), $\gamma = 71.07$ (1)°; $Z = 2$; $\rho_{\text{meas}} = 2.02$, $\rho_{\text{calc}} = 2.043$ g cm^{-3} ; $F(000) = 960$, $\mu(\text{Mo K}\alpha) = 19.05$ cm^{-1} . From 5125 measured independent reflections, 4388 were considered as observed ($I \geq 3\sigma(I)$) and used to solve (Patterson, Fourier methods) and refine (full matrix least squares) the structure to convergence at R and R_w of 0.026 and 0.030, respectively. All non-hydrogen atoms were refined anisotropically in both structures with thermal parameters for **2** divided into two blocks. Further details of data collection, reduction, and refinement are given in Table I of the supplementary material. Computer programs used in the structure analysis have been described in detail elsewhere. See: Carty, A. J.; Mott, G. N.; Taylor, N. J.; Yule, J. E. *J. Am. Chem. Soc.* **1978**, *100*, 3051.

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bound to three metal atoms in the butterfly interacts strongly with Ru(2) (Ru(2)-P(av) = 2.369 Å) and more weakly with Ru(3) (Ru(3)-P(av) = 2.470 Å) and Ru(4) (Ru(4)-P(av) 2.566 Å). The new P-C(14) bond length is 1.808 (8) Å. The overall skeletal geometry is that of a nido dodecahedron with the missing vertex being of high coordination number.⁷ The second cluster 3 (Figure 2) has one less carbonyl than 2 and has a closo, pentagonal bipyramidal skeleton with two bridging carbonyl groups between axial (Ru(2) and Ru(4)) and equatorial (Ru(3)) ruthenium atoms as in 2. Whereas in 2 one of the alkyne carbon atoms was bonded to the phosphorus atom, in 3 the acetylene is attached only to the four ruthenium atoms. The equatorial belt of the pentagonal bipyramid in 3 thus consists of Ru(1), Ru(3), P, and C(12) and C(13) of the alkyne.⁸ The apparent relationship of 2 and 3 evident in their structures was confirmed by warming an authentic sample of 2 in heptane when a smooth conversion to 3 occurred. The transformation of 1 to 3 via intermediate 2 can be best viewed in terms of the SEC rules. Thus 1 a nido octahedron is converted via addition of two three-electron CR fragments and loss of a carbonyl to the seven-vertex nido dodecahedron 2 and subsequently via CO loss and molecular rearrangement to the closo seven-vertex, eight skeletal pair pentagonal bipyramid of 3. The rearrangement 2 to 3 occurs with P-C and Ru-Ru bond cleavage but not C-C cleavage. These results may have implications for skeletal rearrangement processes in other mixed-cluster systems.

Although the insertion of acetylenes into metal-phosphorus bonds in μ -PR₂-bridged binuclear systems has been reported,⁹ the formation of complexes analogous to 2 and 3 from a phosphinidene has precedent only in the very recent report by Huttner and co-workers¹⁰ of the conversion of Fe₃(CO)₁₀(μ_3 -PR) to Fe₃(CO)₁₀[PRC(R')C(R'')] and Fe₃(CO)₉(PR)[C(R')C(R'')]. All of these clusters represent a ternary (M, P, C) structural variety of which the metallocarboranes¹¹ are perhaps the best known examples. There is, in addition, a synthetic analogy between the synthesis of 2 and 3 and the generation of metallocarboranes via reaction of metalboranes with RC≡CR.¹² We are currently exploring further aspects of the reactivity of μ -PR complexes with ternary cluster formation in mind.

Acknowledgment. We are grateful to the Natural Sciences and Engineering Research Council of Canada for financial support of this work via operating grants (to A.J.C.) and a scholarship (to S.A.M.).

Note Added in Proof. Following submission of this paper we learned that similar insertion of an acetylene into the framework of the metal-sulfur cluster Os₄(CO)₁₂(μ_3 -S) has been observed (Adams, R. D., personal communication;

see also: Adams, R. D.; Wang, S., Abstract No. 366, XIIth ICOMC, Vienna, Austria, Sept 1985).

Registry No. 1, 81726-80-9; 2, 98586-80-2; 3, 98586-81-3; C₂Ph₂, 501-65-5.

Supplementary Material Available: Tables of crystal and intensity data, data collection, reduction, and refinement (Table I), atomic positions (Tables IIA-C, VI), anisotropic thermal parameters (Tables IIIA,B, VII), bond lengths and angles (Tables IV, VIII), and structure factors (Tables V, IX) for 2 and 3, respectively (79 pages). Ordering information is given on any current masthead page.

"Closed", "Half-Open", and "Open" Ferrocenes: Redox Behavior and Electron Spin Resonance of the Radical Cations[†]

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Summary: Successive opening of one or both rings of ferrocene results in a cathodic shift of the redox potential for the couple ferrocene/ferricinium, increased chemical lability, and vastly decreased *g* anisotropy in the ESR spectra of the respective complex radical cations. As opposed to closed ferricinium, ESR of the open ferricinium ions is observed at room temperature. These findings are discussed on the basis of the electronic structure as suggested by INDO MO calculations.

A characteristic property of ferrocene, 1, is the reversible one-electron oxidation to the ferricinium radical cation 1⁺.¹ ESR studies of 1⁺, however, are possible only at very low temperatures since the orbital degeneracy of the ²E_g ground state for 1⁺ results in a large *g* anisotropy and concomitant short relaxation times.^{2,3} Since metallocene chemistry recently experienced a fruitful extension via the class of "open metallocenes",⁴ a comparison of redox and ESR properties of the "closed" ferrocene with its "open" counterpart is warranted. While peripheral substitution^{5,6} (as in 2) or introduction of heteroatoms into the cyclic ligand (e.g., bis(η^5 -dicarbollide)iron(III)⁷) only brings about small changes in redox and ESR behavior of the closed bis(η^5 -cyclopentadienyl)iron, 1, we expected that opening one or both rings to form the η^5 -pentadienyl complexes 3 and 4 would constitute a major perturbation resulting in profoundly modified redox potentials and *g* factor anisotropies. In order to study the consequences of succes-

[†] Dedicated to Professor Karl Dimroth on the occasion of his 75th birthday.

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(8) Deviations from the least-squares plane containing these atoms are as Ru(1), 0.039; Ru(3), 0.014; P, -0.032; C(12), -0.045; C(13), -0.024 Å.

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