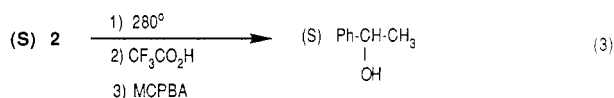
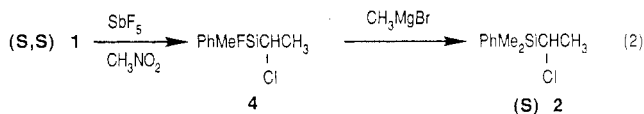


which showed a specific rotation of  $-3.46^\circ$  ( $c$  7.22, cyclohexane) (eq 2). Thermolysis-oxidation of **2** also provided



(*S*)-1-phenylethanol, this time in 86% ee (eq 3). These results, together with those shown in Scheme I, provide the first stereochemical studies carried out on the thermal rearrangement of  $\alpha$ -haloorganosilanes. The reactions proceed with inversion of configuration at carbon, a result which is inconsistent with the formation of an open carbocation intermediate and consistent with an intramolecular, simultaneous migration of the chlorine and aryl groups.

**Acknowledgment.** G.L.L. thanks the NIH-MBRS (RR-8102) for partial support of this work. The Petroleum Research Fund administered by the American Chemical Society is thanked for their support. Drs. Sydia Sandoval and J. A. Prieto are thanked for helpful discussions.

**Registry No.** (*S,S*)-1, 85082-06-0; **2**, 110079-44-2; **3a**, 110079-45-3; **3b**, 110079-46-4; **4** (isomer 1), 110079-47-5; **4** (isomer 2), 110079-48-6; (*S*)-1-phenylethanol, 1445-91-6; (*S*)-1-(1-naphthyl)ethanol, 15914-84-8.

### Differentiating Metal Centers in Homopolynuclear Systems: Use of the Oxodiphenylphosphorano ( $\mu$ -Ph<sub>2</sub>P=O) Ligand as a Versatile Bridging Group and a Comparison with Related $\mu$ -Diphenylphosphido ( $\mu$ -PPh<sub>2</sub>) Complexes

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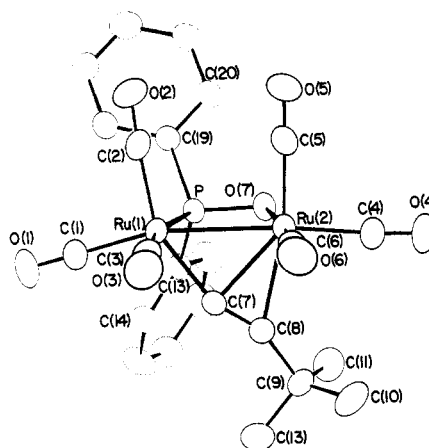
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Received May 13, 1987

**Summary:** The novel diphenylphosphidoxo complexes Ru<sub>2</sub>(CO)<sub>6</sub>( $\mu_2$ - $\eta^2$ -C≡C-*t*-Bu)( $\mu$ -Ph<sub>2</sub>P=O) and Fe<sub>3</sub>(CO)<sub>9</sub>( $\mu_3$ - $\eta^2$ -C≡C-*t*-Bu)( $\mu$ -Ph<sub>2</sub>P=O) have been synthesized from the phosphine oxide Ph<sub>2</sub>P(O)C≡C-*t*-Bu and Ru<sub>3</sub>(CO)<sub>12</sub> or Fe<sub>2</sub>(CO)<sub>9</sub>; X-ray structural analyses of Ru<sub>2</sub>(CO)<sub>6</sub>( $\mu_2$ - $\eta^2$ -C≡C-*t*-Bu)( $\mu$ -Ph<sub>2</sub>P=O) (**1**) and Fe<sub>3</sub>(CO)<sub>9</sub>( $\mu_3$ - $\eta^2$ -C≡C-*t*-Bu)( $\mu$ -Ph<sub>2</sub>P=O) (**2**) revealed phosphidoxo ligands bridging a strong metal-metal bond in **1** (Ru(1)-Ru(2) = 2.7729 (3) Å) and two noninteracting metals in **2** (Fe(1)-Fe(3) = 3.6064 (6) Å).

There are a number of reasons for anticipating an interesting chemistry for polynuclear complexes containing

(1) (a) On leave from Le Laboratoire de Chimie Organométallique, L'Université de Rennes, Campus de Beaulieu, Rennes, France. (b) We have chosen to describe these ligands simply as diorganophosphido groups to emphasize the relationship to diorganophosphido complexes. Thus the diphenylphosphido anion Ph<sub>2</sub>P<sup>-</sup> is derived from diphenylphosphine Ph<sub>2</sub>PH. The diphenylphosphidoxo anion Ph<sub>2</sub>P=O<sup>-</sup> (oxodiphenylphosphorano) relates to diphenylphosphine oxide Ph<sub>2</sub>P(O)H via deprotonation at phosphorus.



**Figure 1.** An ORTEP II plot of the molecular structure of Ru<sub>2</sub>(CO)<sub>6</sub>( $\mu_2$ - $\eta^2$ -C≡C-*t*-Bu)( $\mu$ -Ph<sub>2</sub>P=O) (**1**) showing the atomic numbering.

bridging phosphidoxo groups  $\mu$ -R<sub>2</sub>P=O;<sup>1b</sup> (i) the combination of a soft donor (P) and a hard ligand (=O) differentiates metal centers even in homobinuclear systems; (ii) the  $\mu$ -R<sub>2</sub>P=O moiety should, like the  $\mu$ -R<sub>2</sub>P group, be capable of bridging both bonding and nonbonding metals; (iii) there is evidence that phosphine oxides exert a labilizing influence on metal-carbonyl bonds.<sup>2</sup> Thus the oxygen end of the  $\mu$ -phosphidoxo ligand may exercise control over the sites of substitution in polynuclear complexes. We describe herein a facile route to polynuclear carbonyl complexes of iron and ruthenium containing a single  $\mu$ -Ph<sub>2</sub>P=O ligand. X-ray analyses of Ru<sub>2</sub>(CO)<sub>6</sub>( $\mu_2$ - $\eta^2$ -C≡C-*t*-Bu)( $\mu$ -Ph<sub>2</sub>P=O) (**1**) and Fe<sub>3</sub>(CO)<sub>9</sub>( $\mu_3$ - $\eta^2$ -C≡C-*t*-Bu)( $\mu$ -Ph<sub>2</sub>P=O) (**2**) have allowed a detailed comparison of structural parameters for closely related diphenylphosphidoxo and diphenylphosphido complexes, suggesting that  $\mu$ -Ph<sub>2</sub>PO ligands may be equally as versatile as their  $\mu$ -Ph<sub>2</sub>P counterparts. Although bridging phosphonato and phosphinito complexes are known<sup>3</sup> including several obtained by thermal degradation of phosphites,<sup>3c-e</sup> there are to our knowledge no structurally characterized examples of  $\mu$ -Ph<sub>2</sub>P=O ligands bridging strong metal-metal bonds.<sup>4</sup>

In a typical reaction diphenyl(*tert*-butylethynyl)phosphine oxide<sup>5</sup> (1.1 g, 3.9 mmol), a few drops of sodium benzophenone ketyl, and Ru<sub>3</sub>(CO)<sub>12</sub> (1.5 g, 2.3 mmol) in dry degassed THF (150 mL) were refluxed for 30 h giving an orange-red solution. Solvent was removed in vacuo and the residue chromatographed on Florisil. Elution with *n*-heptane afforded unreacted Ru<sub>3</sub>(CO)<sub>12</sub> (0.4 g); yellow **1** (1.3 g, 76%) was eluted with benzene.<sup>6</sup> The <sup>31</sup>P{<sup>1</sup>H} NMR

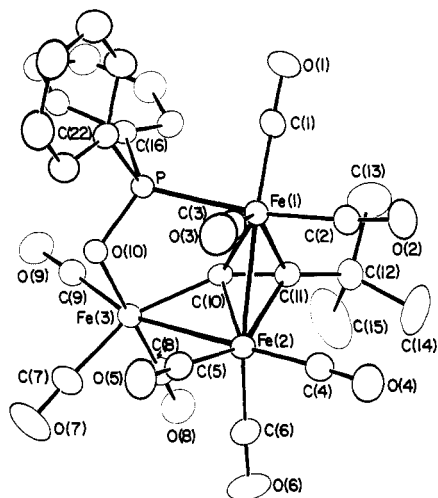
(2) Darensbourg, D. J.; Darensbourg, M. Y.; Walker, N. *Inorg. Chem.* 1981, 20, 1918.

(3) See, for example: (a) Berry, D. E.; Beveridge, K. A.; Bushnell, G. W.; Dixon, K. R.; Pidcock, A. *Can. J. Chem.* 1986, 64, 343 and references therein. (b) Duncan, J. A. S.; Hedden, D.; Roundhill, D. M.; Stephenson, T. A.; Walkinshaw, M. D. *Angew. Chem., Int. Ed. Engl.* 1982, 21, 452. (c) Burch, R. R.; Muettterties, E. L.; Thompson, M. R.; Day, V. W. *Organometallics* 1983, 2, 474. (d) Fernandez, J. M.; Johnson, B. F. G.; Lewis, J.; Raithby, P. R. *J. Chem. Soc. Dalton Trans.* 1981, 2250. (e) Bruce, M. I.; Shaw, G.; Stone, F. G. A.; *J. Chem. Soc., Dalton Trans.* 1973, 1667. (f) Klauil, W.; Otto, H.; Eberspach, W.; Bucholz, E. *Chem. Ber.* 1982, 115, 1922.

(4) A few  $\mu$ -Me<sub>2</sub>PO complexes have been synthesized via oxygen insertion into a  $\mu$ -Me<sub>2</sub>P bridge. See: Klingert, B.; Werner, H. *J. Organomet. Chem.* 1983, 252, C47.

(5) (a) Charrier, C.; Chodkiewicz, W.; Cadiot, P. *Bull. Soc. Chim. Fr.* 1966, 1002. (b) Carty, A. J.; Hota, N. K.; Ng, T. W.; Patel, H. A.; O'Connor, T. J. *Can. J. Chem.* 1971, 49, 2706.

(6) **1**: IR (C<sub>6</sub>H<sub>12</sub>)  $\nu$ (CO) 2087 (m), 2057 (s), 2021 (s), 2012 (m), 1992 (m); IR (Nujol)  $\nu$ (P=O) 980 (m) cm<sup>-1</sup>; <sup>31</sup>P NMR (C<sub>6</sub>D<sub>6</sub>)  $\delta$  +83.2; <sup>1</sup>H NMR (CDCl<sub>3</sub>)  $\delta$  7.5-7.1 (m, 10 H), 1.2 (s, 9 H).



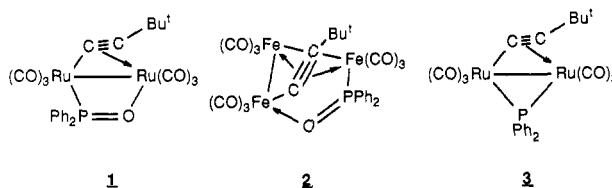
**Figure 2.** A perspective view of the structure of  $\text{Fe}_3(\text{CO})_9(\mu_3\text{-}\eta^2\text{-C}\equiv\text{C-}t\text{-Bu})(\mu\text{-Ph}_2\text{P=O})$  (**2**).

spectrum of **1** exhibits a single resonance at +83.2 ppm downfield of the shift in the intact phosphine oxide ( $\delta$  1.1). In the IR spectrum a medium band at  $980\text{ cm}^{-1}$  is shifted  $211\text{ cm}^{-1}$  to lower frequency than in the free phosphine oxide ( $\nu(\text{P=O})$   $1191\text{ cm}^{-1}$ ), indicative of phosphoryl group coordination.

Treatment of  $\text{Ph}_2\text{P(O)C}\equiv\text{C-}t\text{-Bu}$  (1.0 g, 3.5 mmol) in benzene (50 mL) with  $\text{Fe}_2(\text{CO})_9$  (2 equiv) for 5 h at ambient temperature gave, after Florisil chromatography, eluting with benzene, red-brown **2** (0.27 g, 11%). Dark red crystals of **2** were obtained from benzene at  $5^\circ\text{C}$ . Spectroscopic features<sup>7</sup> include a  $\nu(\text{P=O})$  band at  $985\text{ cm}^{-1}$  and a  $^{31}\text{P}$  resonance at +150.5 ppm.

The structures of **1** and **2** were determined by single-crystal X-ray diffraction<sup>8</sup> (Figures 1 and 2). The ruthenium dimer consists of two tricarbonylruthenium moieties joined by a strong Ru–Ru bond (Ru(1)–Ru(2) = 2.7729 (3) Å) and supported by  $\mu_2\text{-}\eta^2$ -acetylide and phosphidoxo groups. The oxygen atom of the latter is coordinated to Ru(2) with an Ru(2)–O(7) bond length of 2.135 (2) Å, a value which compares with an Ru–O distance of 2.119 (3) Å in the bridging acyl complex  $\text{Ru}_2(\text{CO})_6[\mu\text{-O=CCH=C(Ph)NEt}_2](\mu\text{-PPh}_2)$ .<sup>9</sup> The P–O bond of the  $\mu\text{-Ph}_2\text{P=O}$

ligand retains substantial multiple-bond character with the P–O(7) bond length of 1.544 (2) Å at the long end of the range for typical  $\mu$ -phosphinito complexes<sup>10</sup> but much shorter than expected for a P–O single bond.<sup>11</sup> There is a remarkable similarity between **1** and the corresponding  $\mu\text{-PPh}_2$  complex  $\text{Ru}_2(\text{CO})_6(\mu_2\text{-}\eta^2\text{-C}\equiv\text{C-}t\text{-Bu})(\mu\text{-PPh}_2)$  (**3**)<sup>12</sup>



which allows a direct analysis of the perturbation caused by replacing a soft phosphorus ligand by a hard oxygen ligand with  $\pi$ -donor capabilities. There is in fact a significant difference in the metal–acetylide  $\eta$ -bonding in the two cases with the degree of distortion of the alkynyl group from linearity being more marked in the phosphidoxo complex **1**. Bend back angles at  $\text{C}_\beta$  are  $24.0(8)^\circ$  in **1** compared to  $21.4(8)^\circ$  in **3** and the Ru(2)–C(acetylide) distances are quite significantly shorter in **1** (Ru(2)–C(7) = 2.244 (3) Å; Ru(2)–C(8) = 2.338 (4) Å) than in **3** (Ru(2)–C(7) = 2.285 (3) Å; Ru(2)–C(8) = 2.417 (3) Å). All of these parameters are indicative of stronger ruthenium–acetylide  $\pi$ -bonding in **1** where the “hard” oxygen atom is coordinated to Ru(2). There is also evidence for a ground-state influence of the  $\pi$ -donor on the Ru(2)–C(CO) bond lengths. Thus the Ru(2)–C(6) bond length in **1** (1.875 (4) Å) is fully 0.08 Å shorter than the corresponding Ru(2)–C(6) (1.955 (4) Å) value in **3** where the trans ligand is the phosphido group. At this level of accuracy this represents a remarkable bond shortening. In contrast neither the remaining Ru(2)–C(CO) distances (average 1.925 Å) nor the Ru(1)–C(CO) bond lengths (average 1.934 Å) in **1** differ significantly from their counterparts in **3** (Ru(2)–C(CO)<sub>av</sub> = 1.908 Å; Ru(1)–C(CO)<sub>av</sub> = 1.932 Å).

In the case of the trinuclear iron cluster **2** the  $\mu\text{-Ph}_2\text{P=O}$  ligand bridges an “open” edge of the  $\text{Fe}_3$  triangle (Fe(1)–Fe(3) = 3.6064 (6) Å; Fe(1)–Fe(2) = 2.6012 (6) Å; Fe(2)–Fe(3) = 2.6837 (6) Å). The acetylide is  $\sigma$ -bound to the oxygen-substituted metal Fe(3) with an  $\eta$ -interaction to Fe(1) and Fe(2). The alkyne triple bond (C(10)–C(11) = 1.287 (5) Å) and the bend back angle at  $\text{C}_\beta$  ( $37.9^\circ$ ) are larger than in **1** as expected for  $\mu_3\text{-}\eta^2$  versus  $\mu_2\text{-}\eta^2$  coordination. In solution **2** ( $\delta(^{31}\text{P})$  150.5) apparently undergoes isomerization to a second isomer with  $\delta(^{31}\text{P})$  at 178.5, the equilibrium concentration of the two species being  $\sim 2:1$ . This isomerization, which is apparently not due to P–O decoordination,<sup>13</sup> may involve either a change in bonding mode for the hydrocarbyl from  $\mu_3\text{-}\eta^2$  to  $\mu^2\text{-}\eta^2$  coupled with Fe–Fe bond closure or a rearrangement of the hydrocarbyl on the cluster face. Precedents for both of these processes are known.<sup>14</sup>

In summary we have found that alkynylphosphine oxides are a useful source of bridging phosphidoxo and acetylide groups. The  $\text{Ph}_2\text{P=O}$  group has novel properties including the ability to bridge strongly bound and nonin-

(7) **2**: IR ( $\text{C}_6\text{H}_{12}$ )  $\nu(\text{CO})$  2082 (m), 2066 (w), 2044 (s), 2023 (s), 2010 (s), 2000 (m), 1968 (m), 1957 (w); IR (Nujol)  $\nu(\text{P=O})$  985 (m)  $\text{cm}^{-1}$ ;  $^{31}\text{P}$  NMR ( $\text{C}_6\text{D}_6$ )  $\delta$  +178.5 (minor isomer), 150.5;  $^1\text{H}$  NMR ( $\text{CD}_2\text{Cl}_2$ )  $\delta$  7.5–7.4 (m, 10 H), 1.6 (s, 6 H, major), 1.3 (s, 3 H, minor).

(8) **Crystal data for 1** ( $\text{Ru}_2\text{P}_2\text{O}_7\text{C}_{24}\text{H}_{18}$ ). A pale yellow prism of dimensions  $0.23 \times 0.23 \times 0.26\text{ mm}$  grown from a heptane/benzene mixture (5:1) was selected and mounted on a Syntex  $\text{P}_2$  diffractometer. The compound crystallized in the monoclinic system, space group  $\text{P}2_1/n$ , with  $a = 9.525(1)\text{ \AA}$ ,  $b = 18.962(2)\text{ \AA}$ ,  $c = 15.039(2)\text{ \AA}$ ,  $\beta = 106.56(1)^\circ$ ,  $V = 2603.6(6)\text{ \AA}^3$ ,  $Z = 4$ ,  $\rho_{\text{calc}} = 1.665\text{ g cm}^{-3}$ , and  $\mu(\text{Mo K}\alpha) = 12.36\text{ cm}^{-1}$ . A total of 4620 independent reflections were measured by using Mo  $\text{K}\alpha$  ( $\lambda = 0.71069\text{ \AA}$ ) radiation of which 3635 satisfied the criterion  $I \geq 3\sigma(I)$ . The structure was solved via the heavy-atom and Fourier techniques and refined by full-matrix least-squares methods to a final  $R = 0.025$  and  $R_w = 0.028$  where  $R = \sum|F_o| - |F_c|/\sum|F_o|$  and  $R_w = \sum w(|F_o| - |F_c|)^2/\sum w|F_o|^2$ . Additional details of X-ray data collection, reduction, and refinement are given in Table S1. **Crystal data for 2** ( $\text{Fe}_3\text{P}_2\text{O}_7\text{C}_{27}\text{H}_{18}$ ). A red-brown crystal of dimensions  $0.21 \times 0.23 \times 0.27\text{ mm}$  grown from benzene solution was selected. The cluster crystallized in the triclinic system, space group  $\text{P}\bar{1}$ , with  $a = 9.322(1)\text{ \AA}$ ,  $b = 11.580(2)\text{ \AA}$ ,  $c = 13.396(2)\text{ \AA}$ ,  $\alpha = 80.76(1)^\circ$ ,  $\beta = 87.02(1)^\circ$ ,  $\gamma = 85.54(1)^\circ$ ,  $V = 1421.8(4)\text{ \AA}^3$ ,  $Z = 2$ ,  $\rho_{\text{calc}} = 1.611\text{ g cm}^{-3}$ , and  $\mu(\text{Mo K}\alpha) = 16.60\text{ cm}^{-1}$ . A total of 5051 independent reflections were measured of which 3867 had  $I \geq 3\sigma(I)$  and were used in the structure solution and refinement. The structure was also solved by using the heavy-atom technique. The butyl group hydrogen atoms were not included due to the considerable thermal motion of this entity. Least-squares refinement converged at  $R = 0.033$  and  $R_w = 0.039$ . Additional information is given in Table S1.

(9) Mott, G. N.; Granby, R.; MacLaughlin, S. A.; Taylor, N. J.; Carty, A. J. *Organometallics* 1983, 2, 189.

(10) See, for example: (a) Berry, D. E.; Beveridge, K. A.; Browning, J.; Bushnell, G. W.; Dixon, K. R. *Can. J. Chem.* 1986, 64, 1903. (b) Paine, R. T.; Duesler, E. N.; Moody, D. C. *Organometallics* 1982, 1, 1097.

(11) Cruickshank, D. W. J. *J. Chem. Soc.* 1961, 5486.

(12) (a) MacLaughlin, S. A.; Carty, A. J., unpublished results. (b) Carty, A. J. *Pure Appl. Chem.* 1982, 54, 113.

(13) Neither the infrared nor the  $^{31}\text{P}$  NMR data show evidence of an uncoordinated P=O bond ( $\nu(\text{P=O})$   $1191\text{ cm}^{-1}$ ;  $\delta(^{31}\text{P})$  1.1 in  $\text{Ph}_2\text{P(O)C}\equiv\text{C-}t\text{-Bu}$ ).

(14) Deeming, A. J. *J. Organomet. Chem.* 1978, 150, 123.

teracting metal centers. The oxo end of the  $\text{Ph}_2\text{P}=\text{O}$  group has a significant effect on the  $\pi$ -acid ligands coordinated to the same metal atom, strengthening the bond to the trans CO group and increasing the cis metal-alkyne interaction. Such effects may presage a useful role for such ligands in modifying and directing reactivity at polynuclear sites. These results also suggest that the entire family of P(V)-derived anions,  $[\text{R}_2\text{P}=\text{X}]^-$  ( $\text{X} = \text{O}, \text{S}, \text{CR}_2, \text{NR}$ ),<sup>15</sup> may have an interesting organo coordination chemistry.

**Acknowledgment.** We are grateful to the Natural Sciences and Engineering Research Council of Canada for support of this work.

**Registry No.** 1, 110096-54-3; 2, 110096-55-4;  $\text{Ph}_2\text{P}(\text{O})\text{C}\equiv\text{C}-t\text{-Bu}$ , 110096-56-5;  $\text{Ru}_3(\text{CO})_{12}$ , 15243-33-1;  $\text{Fe}_2(\text{CO})_9$ , 15321-51-4.

**Supplementary Material Available:** Table S1, further details on X-ray data, intensity collection, structure solution and refinement for 1 and 2, Tables S2 and S3, atomic positions (fractional  $\times 10^4$ ), Tables S4 and S5, anisotropic thermal parameters ( $\times 10^3$ ), Tables S6 and S7, bond lengths ( $\text{\AA}$ ) and angles (deg), Tables S8 and S9, hydrogen atom positions, thermal parameters, and C-H bond lengths, for 1 and 2 (13 pages); Tables S10 and S11, structure factors for 1 and 2 (40 pages). Ordering information is given on any current masthead page.

(15) Examples of formally analogous bridging ylides  $\mu\text{-R}_2\text{P}=\text{CR}_2$  have been synthesized from  $\mu$ -phosphido compounds. See, e.g.: Chau, C.-N.; Yu, Y.-F.; Wojcicki, A.; Calligaris, M.; Nardin, G.; Balducci, G. *Organometallics* 1987, 6, 308. (b) Werner, H.; Zolk, R. *Organometallics* 1985, 4, 601.

### The Unexpected Open Cluster Structure of $[1-(\eta^6\text{-MeC}_6\text{H}_4\text{-}i\text{-Pr})\text{-}2,4\text{-Me}_2\text{-}1,2,4\text{-RuC}_2\text{B}_8\text{H}_8]$

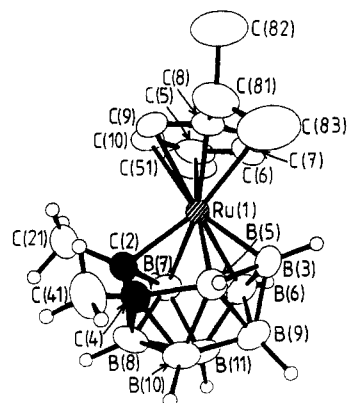
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Norman N. Greenwood,\* John D. Kennedy, and  
Mark Thornton-Pett

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Received May 14, 1987

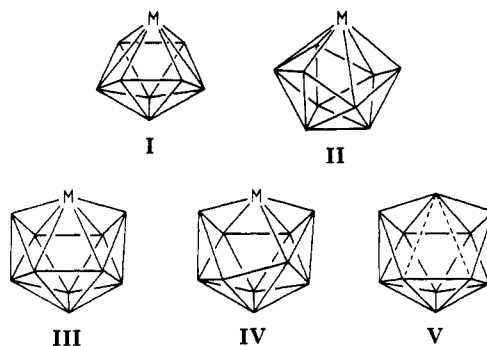
**Summary:** The structure of the title compound is found to contain a quadrilateral open face which casts some doubt on the previously assumed and generally accepted closo nature of this type of polyhedral metalladecaborane cluster.

Polyhedral metallaboranes that do not contain other cluster heteroatoms and that have the polar nine-,<sup>1,2</sup> ten-,<sup>2-4</sup> and eleven-vertex<sup>4,5</sup> configurations, I-III, respectively, are of interest<sup>6</sup> because they contravene the simple Williams-Wade<sup>7,8</sup> cluster geometry and electron-counting



**Figure 1.** Crystallographically determined molecular structure of compound 1. Selected interatomic distances from Ru(1) are as follows: to C(2), 212.4 (6), to B(3), 206.5 (6), to C(4), 268.3 (7), to B(5), 236.3 (3), to B(6), 228.9 (6), and to B(7), 228.7 (6) pm. C(2)-C(4) is 148.5 (7) pm, and the dihedral angle between the aromatic  $[\text{C}_6]$  plane and the B(5)B(6)B(7) plane is  $7.35^\circ$ . C(4) is 31.6 pm below the B(5)B(6)B(7) plane.

rules; consequently there is some discussion in the literature<sup>6,9</sup> concerning the nature of the cluster bonding in these so-called "isocloso" species.<sup>10</sup> In relation to this discussion, it has hitherto been assumed that the heteroatom-containing eleven-vertex metalladecaborane species such as  $[(\text{C}_5\text{H}_5)\text{CoC}_2\text{B}_8\text{H}_{10}]$ ,<sup>11</sup>  $[(\text{PPh}_3)_2\text{HfC}_2\text{B}_8\text{H}_{10}]$ ,<sup>12</sup>  $[(\text{C}_6\text{H}_6)\text{-RuC}_2\text{B}_8\text{H}_{10}]$ ,<sup>13a</sup> etc. do not contravene the Williams-Wade rules because (i) they have the straightforward Wadland closo counts and (ii) they are presumed to have straightforward closo deltahedral geometries that are grossly similar to III.



We now report results of a single-crystal X-ray diffraction analysis on the eleven-vertex ruthenadecaborane  $[(\text{MeC}_6\text{H}_4\text{-}i\text{-Pr})\text{RuMe}_2\text{C}_2\text{B}_8\text{H}_8]$  (compound 1) that cast serious doubt on the generality of the second of these two presumptions. This air-stable yellow compound

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(10) These "isocloso" geometries are generated by the capping of the open faces of adjacent-arachno geometries with a metal atom vertex (see ref 6).

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(13) (a) Hanusa, T. P.; Huffman, J. C.; Curtis, T. L.; Todd, L. J. *Inorg. Chem.* 1985, 24, 787. (b) Hanusa, T. P.; Huffman, J. C.; Todd, L. J. *Polyhedron* 1982, 1, 77.

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