bonded.²⁶ ¹H NMR and ¹³C NMR resonances for the methylene group of 7 are located at 1.98 and -2.3 ppm, respectively. These values compare favorably with those for the (benzofulvene)- and (fulvene)diiron hexacarbonyl complexes.^{25,27}

Our current work includes efforts to improve the yields of 6, a crystal structure, and chemical studies. We will report on these in due course.

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Registry No. 6, 124511-84-8; 7, 49596-01-2; 2,3-dihydro-1Hinden-2-one, 49596-01-2; 2-methyl-2,3-dihydro-1H-inden-2-ol, 33223-84-6; 2-methyl-1H-indene, 2177-47-1; 3-bromo-2-bromomethyl-1*H*-indene, 60858-22-2; $[\eta^4$ -5,6-bis(methylene)-1,3-cyclohexadiene]tricarbonyliron, 12181-95-2.

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¹H NMR (C₆D₆, δ): 1.85 (s, CH₂); 4.01 (t, H1,3); 4.93 (t, H5,4). ¹³C NMR (δ): 212.8 (CO); 94.5 (C2); 84.2 (C4,5); 75.6 (C1,3); -3.1 (C6). IR (cyclohexane): $\nu(CO) = 2077, 2014, 2004, 2000, 1981, 1950 \text{ cm}^{-1}$

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Synthesis and Characterization of the Chiral Cluster $Fe_3(CO)_9[\eta^3-B(H)C(H)C(Me)]$. An Unambiguous Example of a Transition-Metal-Main-Group Six-Atom Cluster

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Summary: The synthesis and structural characterization of the first example of a boracyclopropene ring coordinated to a trimetal fragment, Fe₃(CO)₂[C³-B(H)C(H)C(Me)], I, are described. The compound constitutes an example of a cluster containing three transition metals that can only be reasonably considered as a six-atom cluster containing three iron, two carbon, and one boron atoms. This particular derivative of the cluster core also constitutes an example of a chiral cluster.



Figure 1. Structure and labeling scheme of $Fe_3(CO)_9[\eta^3-B(H)-$ C(H)C(Me)], I. Atoms are shown as 30% thermal ellipsoids except hydrogen atoms, which are drawn at a fixed radius. One of two crystallographically independent but chemically similar molecules is shown.

The relationship between main-group and transitionmetal clusters is expressed in the electron-counting formalism that has been developed over the past 10 or 15 years.¹ Although the reality of these rules lies in their usefulness, the study of compounds containing both transition-metal and main-group atoms in a contiguous network serves to emphasize similarities and differences in bonding of homonuclear clusters of atoms with and without d valence orbitals. However, in these mixed main group-metal systems an ambiguity often arises in the sense that a molecule containing n metal atoms and m maingroup atoms can be equally well considered as an n + matom cluster or an n atom cluster with a η^m ligand. Herein we report the characterization of a molecule that can only be reasonably considered a six-atom cluster containing equal numbers of main-group and transition-metal atoms.

The new compound was prepared via a developing method² for the synthesis of metallaboranes and metallacarboranes via the reaction of organometallic compounds, particularly those containing negatively charged ligands, with boranes. Specifically, the new cluster was synthesized from the reaction of $[Fe(Py)_6][Fe_4(CO)_{13}]^3$ with BH_2Br . SMe₂ in toluene at 75 °C for 2 h. After removal of toluene under reduced pressure, protonation of the residues with CF₃COOH and hexane was followed by low-temperature chromatography. Fractional crystalization of the second band (brown) containing mainly $[HFe_4(CO)_{12}C]BH_2^4$ gave bright red crystals of a new compound in about 5% yield. The spectroscopic data⁵ indicate that the compound can be formulated as $Fe_3(CO)_9[\eta^3-B(H)C(H)C(Me)]$, I. Our approach usually produces compounds with either reduced

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Table I. Selected Bond Distances and Angles for I			
(a) Bond Distances, Å			
Fe(1)-Fe(2)	2.605 (2)	Fe(1)-B	2.010 (11)
Fe(2)-Fe(3)	2.552 (2)	Fe(2)-B	2.046 (11)
Fe(1)-Fe(3)	2.586 (2)	Fe(1) - C(10)	1.995 (11)
Fe(2)-C(11)	2.003 (9)	Fe(3)-C(10)	2.008 (8)
Fe(3)-C(11)	2.020 (7)	C(11)-C(10)	1.502 (14)
C(10)-B	1.596 (15)	C(11)-B	1.597 (15)
(b) Bond Angles, deg			
Fe(2)-Fe(1)-Fe(3)	58.9 (1)	Fe(2)-Fe(1)-C(10)	74.0 (3)
Fe(3)-Fe(1)-B	77.4 (3)	Fe(3)-Fe(1)-C(10)	50.0 (3)
Fe(2)-Fe(1)-B	50.6 (3)	C(10)-Fe(1)-B	47.0 (4)
Fe(1) - Fe(2) - Fe(3)	60.2 (1)	Fe(1)-Fe(2)-C(11)	74.0 (3)
Fe(3)-Fe(2)-C(11)	50.9 (2)	C(11) - Fe(2) - B	46.4 (4)
Fe(1)-Fe(2)-B	49.4 (3)	Fe(1)-Fe(3)-Fe(2)	60.9 (1)
Fe(3)-Fe(2)-B	77.6 (3)	Fe(2)-Fe(3)-C(10)	75.0 (3)
Fe(1)-Fe(3)-C(10)	49.5 (3)	Fe(1)-Fe(3)-C(11)	74.2 (3)
Fe(2)-Fe(3)-C(11)	50.3 (3)	C(10)-Fe(3)-C(11)	43.8 (4)
Fe(1)-C(10)-C(11)	106.3 (7)	Fe(3)-C(10)-C(11)	68.5 (4)
Fe(1)-C(10)-B	67.0 (6)	Fe(3)-C(10)-B	107.0 (6)
C(11)-C(10)-B	62.0 (7)	Fe(2)-C(11)-Fe(3)	78.7 (3)
Fe(2)-C(11)-C(10)	105.7 (6)	Fe(3)-C(11)-C(10)	67.7 (4)
Fe(2)-C(11)-B	68.2 (5)	Fe(3)-C(11)-B	106.4 (6)
Fe(1)-B-Fe(2)	79.9 (3)	Fe(1)-B-C(10)	66.0 (5)
Fe(2)-B-C(10)	100.3 (6)	Fe(1)-B-C(11)	101.9 (5)
Fe(2)-B-C(11)	65.4 (5)	C(10)-B-C(11)	56.1 (6)
C(10)-C(11)-C(12)	124.6 (9)	Fe(2)-C(11)-C(12)	128.4 (7)
Fe(3)-C(11)-C(12)	127.9 (6)	C(12)-C(11)-B	124.2 (6)
C(10)-C(11)-B	61.9 (7)		

carbon⁶ or inserted boron.⁷ Occasionally it has yielded compounds containing both boron and reduced carbon, e.g., $[HFe_4(CO)_{12}C]BH_2^4$ and $HFe_3(CO)_9(H_3BCH_3)$,⁷ but in these cases either the boron or the carbon atom are found in a cage substituent.

The result of X-ray crystallographic analysis has shown that the new compound contains a typical triangular $Fe_3(CO)_9$ fragment apparently η^3 -coordinated to a threemembered heterocyclic ligand containing one boron atom such that the borirene ring is almost (1.2° deviation) coplanar with the triiron plane, thereby forming a "Star of David" as shown in Figure 1.8 Selected bond distances and angles for I are given in Table I.

As free trimesitylborirene has been fully characterized,⁹ the obvious initial way of viewing I is as a borirene ligand bound η^3 to a Fe₃(CO)₉ fragment. In this regard the metal fragment in I is not unlike that in $Os_3(CO)_9(\eta^3-C_6H_6)$.¹⁰ The ring of free trimesitylborirene is an equilateral triangle with an edge length of 1.42 Å. In I the boron-carbon (1.596 (15) and 1.597 (15) Å) and carbon-carbon (1.502 (14) Å) bond distances are unequal but normal for single B-C and C-C bonds.¹¹ Coordination of π ligands usually results

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in an increase in bond distances, thus, in terms of geometric changes, consideration of I as a polyhapto ligand bound to a trimetal fragment is acceptable. However, this view of I creates a problem in counting electrons. The $Fe_3(CO)_9$ fragment requires six electrons to become saturated, while the borirene ligand has only two electrons to donate. Hence, I would constitute a presently unknown example of a 44-electron trimetal cluster. While not impossible, one would expect to observe short Fe-Fe distances reflecting an unsaturated metal framework. On the contrary, the observed Fe-Fe distances [2.605 (2), 2.586 (2), and 2.552 (2) Å] must be considered long, if anything.

Alternatively, one notes that the core of I forms a severely distorted octahedron. As the $Fe(CO)_3$ fragment is isolobal with the BH fragment, I is electronically analogous to 1,2-C₂B₄H₆.¹¹ Further I is analogous to $(C_5H_5)_3Co_3B_3$ -H₅¹² as well as Ru₄(CO)₁₂C₂R₂.¹³ Hence, I can be easily recognized as a closo cluster containing seven skeletal electron pairs.¹ For this reason, it should be considered unsaturated or "electron deficient" in the same sense that $[B_6H_6]^{2-}$ or $[Ru_6(CO)_{18}]^{2-}$ is unsaturated. Consistent with this view is the fact that the B-C and C-C distances are very similar to those reported for $1,2-C_2B_4H_6^{11}$ and the Fe-Fe distances are similar to those for $[Fe_6(CO)_{16}C]^{2-.14}$ In fact, consideration of I as a three-member ring bound η^3 to a trimetal fragment makes as much or little sense as considering $[B_6H_6]^{2-}$ (or $[Ru_6(CO)_{18}]^{2-}$) as two $[B_3H_3]^-$ (or [Ru₃(CO)₉]⁻) rings bound face-to-face.¹⁵ Clearly, in the homonuclear clusters the irreducible element of bonding is the six-member octahedral framework. Although it is hidden by the distorted nature of the octahedral core, the minimal bonding element of I, too, is a six-member closed cage.

Further, compound I constitutes an example of a chiral metallacarborane cluster. The first chiral clusters containing a triangular array of three different metal atoms with a capping main-group element were reported in 1980¹⁶ and have considerable potential significance as mechanistic probes and catlaysts.¹⁷ The use of chiral organometallic complexes as enantioselective or optical induction catalysts for asymmetric synthetic reactions has been developed in the past two decades.¹⁸ Although the formation of the chiral compound I was serendipitous rather than planned, the compound now provides an example of chirality supplied by the main-group portion of the cluster rather than by ligands on the metal¹⁹ or by different metals.¹⁶ Since each of the six vertices of I can be considered as a chiral atom, such a cluster may be even more effective than the monocapped trimetal cluster.¹⁶ However, before any applications of asymmetric reactivity can be envisioned, the

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optical resolution of the enatiomers of I must be achieved.

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Supplementary Material Available: Tables of crystal data, atom coordinates, bond distances and angles, anisotropic thermal parameters, and H atom coordinates (7 pages); tables of observed and calculated structure factors (20 pages). Ordering information is given on any current masthead page.

Core Conformational Rearrangement Accompanying Two-Center, Bimetallic Redox Reactions. Chemical and Structural Properties of the Pyrazolyl-Bridged **Rhodium Dimers** $[Rh(\eta^5-C_5Me_5)Cl(\mu-pz)]_2$ and $[Rh(\eta^5-C_sH_s)(\mu-pz)]_2$

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Summary: Reaction between $[Rh(\eta^5-C_5Me_5)Cl(\mu-pz)]_2$ (5), which exhibits a "chair" conformation for the core pyrazolyl-bridged bimetallic unit in the crystal with Rh₂ separation 4.059 (2) Å, and AgBF₄ affords $[Rh_2(\eta^5-C_5Me_5)_2(\mu$ $pz)_{2}(\mu$ -Cl) BF₄ (6), in which the cation core exists in a "boat" conformation with Rh centers 3.588 (2) Å apart; reduction (Zn/Cu) of 5 yields a purple product (7) tentatively identified as $[Rh(\eta^5-C_5Me_5)(\mu-pz)]_2$, the bright purple, reactive η^5 -C₅H₅ analogue 9 of which has been synthesized from $[Rh(\eta^5-C_5H_5)Cl(\mu-pz)]_2$ (8) and shown by using single-crystal X-ray diffraction to possess a deeply folded ("boat") geometry in which Rh-Rh = 2.657 (3) Å.

Concurrent activation of linked coordinatively unsaturated transition-metal centers, which may mediate critical steps in a range of natural and artificial catalytic cycles, is an emergent area of bimetallic chemistry for which so far only very sketchy mechanistic definition exists. We have found that in the family of diiridium(I) complexes $[IrL^{1}L^{2}(\mu-pz)]_{2}$ (1, $L^{1}L^{2}$ = COD, cycloocta-1,5-diene, pzH = pyrazole; 2, $L^1 = L^2 = CO$; 3, $L^1 = CO$, $L^2 = PPh_3$) the pyrazolyl ligands are unusually flexible in bridging, accommodating major alterations in intermetallic separation along the Ir_2 axis.¹ Thus multisite redox reactions that are accompanied by metal-metal bond formation or fracture can proceed without disruption of the integrity of the cyclic bimetallic core.² Weak metal-metal interactions characteristic³ of binuclear structures like those of 1-3 may act to constrain the geometry of the bridged framework (accounting, e.g., for the conformational rigidity of dimer 1, where COD CH₂ resonances remain distinguishable in ¹H or ¹³C NMR spectra even at elevated temperature); but whether ring inversion facilitates formation of axially substituted diiridium(II) products is not known, although it is a logical mechanistic idea. In fact to date the extensive redox chemistry¹⁻⁵ associated with



Figure 1. ORTEP drawings of (top) $[Rh(\eta^5-C_5Me_5)Cl(\mu-pz)]_2$ (5) showing the "chair" conformation about the six-membered metallacycle, (middle) the cation in $\{[Rh(\eta^5-C_5Me_5)(\mu-pz)]_2(\mu-Cl)\}BF_4$ (6) showing the "boat" conformation, and (bottom) $[Rh(\eta^{5}-C_{5}H_{5})(\mu-pz)]_{2}$ (9). Selected bond distances (Å) and angles (deg; ϕ is cp ring centroid): compound 5, Rh…Rh' 4.059 (2); Cl(1)-Rh 2.399 (2); N(1)-Rh 2.105 (7); N(2)'-Rh 2.110 (7); ϕ -Rh 1.782 (10); N(1)-Rh-Cl(1) 89.6 (2); N(2)'-Rh-N(1) 90.8 (2); N(2)'-Rh-Cl 88.5 (2); ϕ -Rh-Rh' 131.6 (3); compound 6, Rh-Rh' 3.588 (2); Cl-Rh 2.378 (2); N(1)-Rh 2.081 (7); N(2)'-Rh 2.101 (9); ϕ -Rh 1.777 (2); N(1)-Rh-Cl 88.3 (2); Rh-Cl-Rh' 97.9 (1); N(2)'-Rh-N(1) 79.9 (2); N(2)'-Rh-Cl 88.0(2); ϕ -Rh-Rh' 163.8 (2); compound 9, Rh(2)-Rh(1) 2.657 (3); N(1)-Rh(1) 2.04 (3); N(3)-Rh(1) 2.06 (2); N-(2)-Rh(2) 2.00 (2); N(4)-Rh(2) 2.02 (2); ϕ (1)-Rh(1) 1.84 (3); ϕ -(2)-Rh(2) 1.83 (3); N(1)-Rh(1)-Rh(2) 70.7 (7); N(3)-Rh(1)-Rh(2) 70.7 (7); N(3)-Rh(1)-N(1) 83.4 (9); N(2)-Rh(2)-Rh(1) 72.4 (7); N(4)-Rh(2)-Rh(1) 69.3 (6); N(4)-Rh(2)-N(2) 86.1 (10); $\phi(1)-$ Rh(1)-Rh(2) 131.7 (9); $\phi(2)-Rh(2)-Rh(1)$ 135.2 (9). Estimated standard deviations are given in parentheses.

compounds 1-3 has connected exclusively geometries that exist in "boat" conformations (including those^{4,5} that afford d_{2}^{6} products as cofacial bioctahedra). In examining further the $d_2^7: d_2^6$ redox manifold (i.e., that connecting the met-

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