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CALIFORNIA UNIV LOS ANGELES DEPT OF CHEMISTRY

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THE SYNTHESIS AND CHARACTERIZATION OF 1,1-(P(C₆H₅)₃)₂-1-H-1,2,4-ETC(U)

FEB 81 J D HEWES, C B KNOBLER, M F HAWTHORNE

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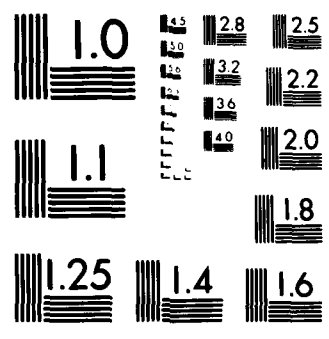
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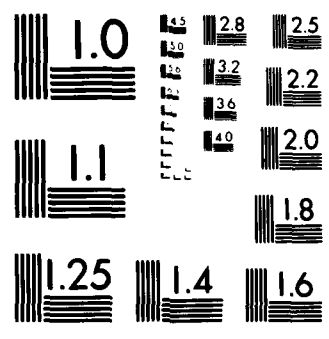
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Task No. NR 053-608 *14*

TECHNICAL REPORT NO. *9* 113 *TR-*

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Prepared for Publication
in
Chemical Communications

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Department of Chemistry
University of California
Los Angeles, California 90024

February 4, 1981

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Icosahedral hydridophosphinorhodacarboranes of general formula $C_2B_9H_{11}RhH(P(C_6H_5)_3)_2$ have been shown to catalytically hydrogenate blocked olefins^{1a,b} and an intensive study has focused on the elucidation of catalytic pathways² and the associated structural chemistry³ unique to catalytically active metallocarboranes. A successful effort to synthesize 10-⁴ and 11-⁵ vertex catalyst precursors through routes analogous to the 12-vertex synthesis, i.e., by reaction of the monoanions $C_2B_7H_{12}^-$ and $C_2B_8H_{11}^-$ with $[(P(C_6H_5)_3)_3RhCl]$, prompted an examination of synthetic routes

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toward 13-vertex supraicosahedral phosphinorhodacarboranes utilizing the previously reported⁶⁻⁸ monoanions $[9,12-C_2B_{10}H_{13}]^-$ (stable isomer) and $[C_2B_{10}H_{13}]^{-9}$ (unstable isomer) whose potential toward metallocarborane formation had not yet been exploited.

The thermally unstable $[(CH_3)_4N]^+[C_2B_{10}H_{13}]^-$ rearranges at 100°C to the stable $[(CH_3)_4N]^+[9,12-C_2B_{10}H_{13}]^-$. The structural differences between the two isomers probably exist in the position of the carbon atoms, and in the tentative assignment of a B-H-B bridge in the unstable isomer¹⁰. In addition, the reactivity of the isomers toward metal complex formation was found to differ significantly.

Refluxing methanolic solutions of the stable $[(CH_3)_4N]^+[9,12-C_2B_{10}H_{13}]^-$ with $[(P(C_6H_5)_3)_3RhCl]$ produced no metallocarboranes, as deduced from the lack of a ca. 2500 cm^{-1} B-H stretching band in the I.R. spectra of the observed products.

Reaction of the unstable $[(CH_3)_4N]^+[C_2B_{10}H_{13}]^-$ with $[(P(C_6H_5)_3)_3RhCl]$ ¹¹ produced closo-1,1-($P(C_6H_5)_3$)₂-1-H-1,2,4-RhC₂B₁₀H₁₂ (I), which was found to catalytically hydrogenate blocked olefins under mild conditions, and which also exhibited a structural chemistry different from previously reported¹²⁻¹⁴ 13-vertex metallocarboranes derived from the $C_2B_{10}H_{12}^{2-}$ ion. I was characterized by ³¹P{¹H}, ¹H, and ¹¹B NMR prior to the x-ray crystal structure analysis.

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For the x-ray crystal structure analysis, I was prepared as described and purified by recrystallizations from THF/n-heptane and THF/ethanol. Suitable crystals of monoclinic, $P2_1/n$ symmetry were grown from 1,2-dichloroethane/n-pentane by vapor diffusion. As shown in Figure 1, the cage geometry of I differs from the structure

Figure 1

of 1-(π -cyclopentadienyl)-1,2,4- $\text{CoC}_2\text{B}_{10}\text{H}_{12}$ reported by Churchill¹⁴. The most obvious feature is the lack of a bonding interaction between B(3) and B(8) with an internuclear distance of 2.166 Å; this compares with a value of 2.082 Å, which Churchill emphasized was abnormally long, and represents a significant departure from the normally triangulated polyhedral carborane systems. The B(9)···C(2) diagonal in the C(2)-B(3)-B(9)-B(8) system is 2.720 Å, in the C(2)-B(7)-B(12)-B(8) system the diagonal C(2)···B(12) is 2.841 Å, and in the B(10)-B(9)-B(3)-C(4) system the B(10)···B(3) diagonal is 2.938 Å.

The four boron atoms in the top belt of 1-(π -cyclopentadienyl)-1,2,4- $\text{CoC}_2\text{B}_{10}\text{H}_{12}$ were observed to be exactly coplanar, with C(2) above and C(4) below that plane. The structure of I varies in this respect, with small deviations from planarity observed in the top belt, but with the locations of C(2) and C(4) exhibiting greater least squares deviations from planarity than B(3), B(5), B(6), and B(7). The entire top belt is distorted to accommodate the perturbed 13-vertex system.

The structural chemistry of I is unique when compared to the systems previously reported¹²⁻¹⁴. The variable temperature ^{11}B NMR indicated no cage fluxionality, and no structural changes were observed for samples which had been refluxed in THF for 3 hrs. In fact, thermal isomerization was not observed when I was heated in vacuo as a solid to its decomposition temperature (490K) at 25° increments and followed by IR spectroscopy.

Variable temperature ^1H and $^{31}\text{P}\{^1\text{H}\}$ NMR spectra of I indicated hindered rotation of the metal vertex about the planar hexagonal bonding face of the carborane cage; this is consistent with previous conclusions based on studies of icosahedral closo-bisphosphinometallocarboranes¹⁵.

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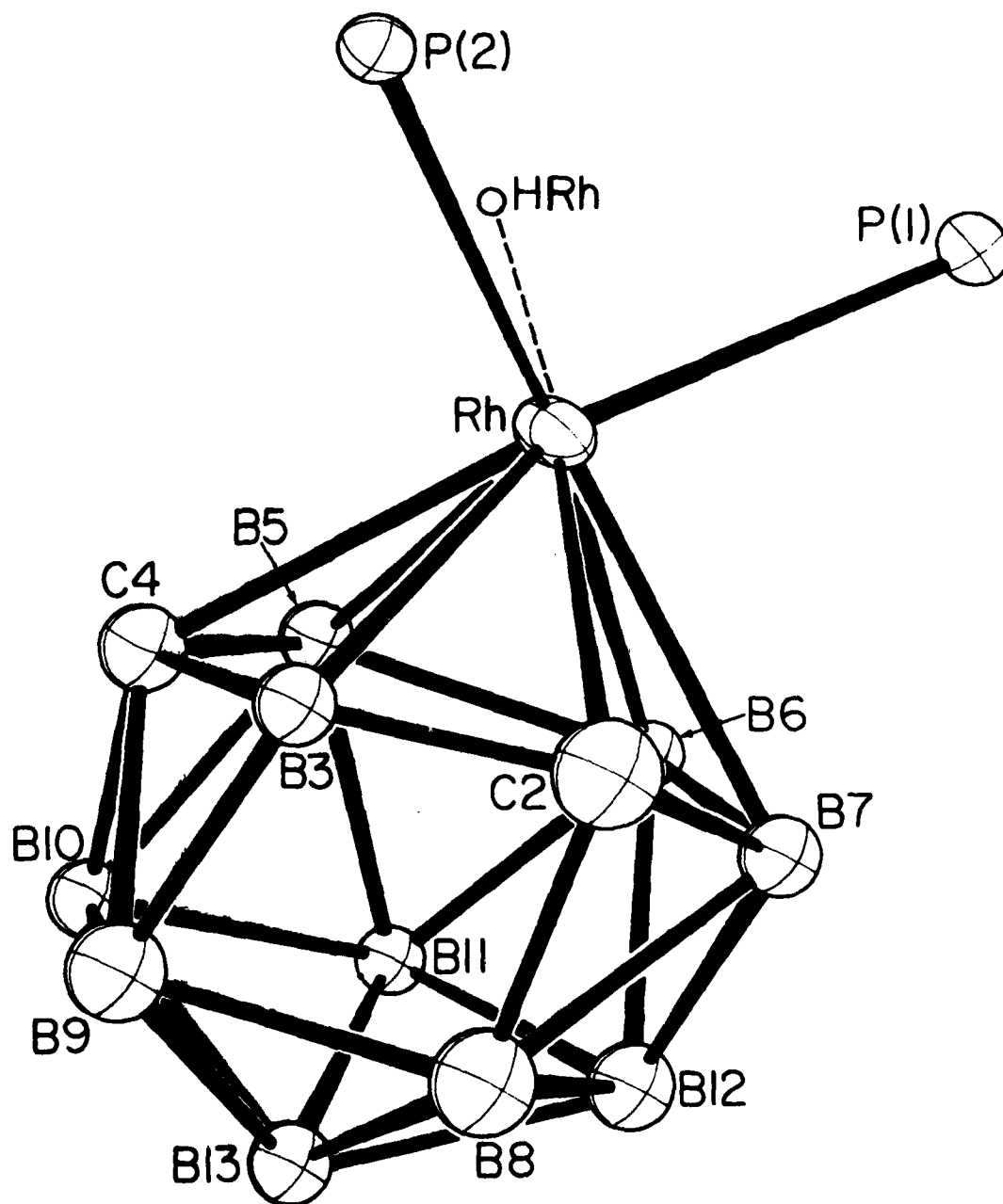
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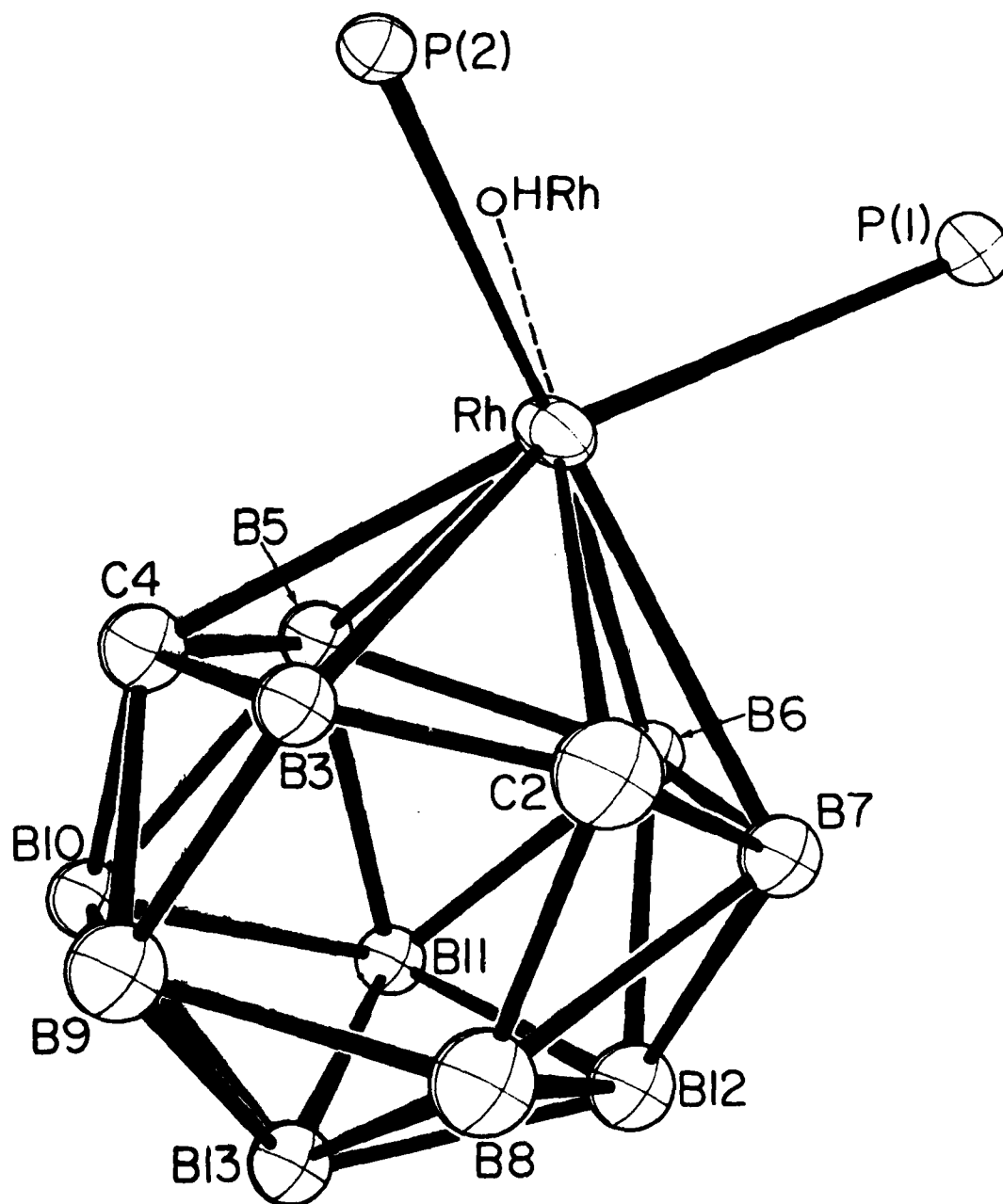
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Figure 1. The molecular structure of $1,1-(P(C_6H_5)_3)_2-1-H-1,2,4-RhC_2B_{10}H_{12} \cdot 1.5 C_2H_4Cl_2$. The molecules of solvation, the phenyl rings, and the terminal boron hydrides have been omitted for clarity. The metal hydride was located on the difference maps only.





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