# Synthesis and Molecular Structure of a Novel Monocarbon Hydridorhodacarborane: *closo*-2,7-$(PPh_3)_2$-2-H-2-Cl-1-$(NMe_3)$-2,1-$RhCB_{10}H_9$

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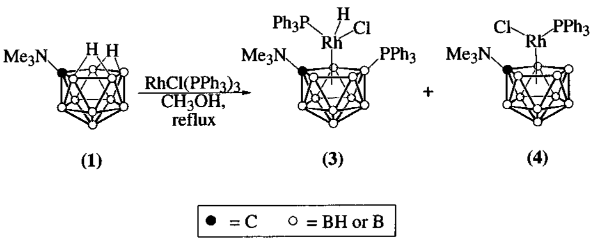
## Introduction

Since the first demonstration of the exceptional efficacy of hydridometallacarboranes derived from the 7,8- and 7,9-isomers of *nido*-$C_2B_9H_{11}{}^{2-}$ as homogeneous catalyst precursors,[2] significant advances have been achieved in the development of their unique chemistry.[3] In addition, many related hydridometallacarboranes of the platinum group metals based upon a variety of carborane derivatives have been obtained.[3c,4] We here report the synthesis and single-crystal X-ray diffraction study of a novel monocarbon hydridometallacarborane derived from *nido*-$B_{10}H_{12}CNMe_3$ (**1**), viz. *closo*-2,7-$(PPh_3)_2$-2-H-2-Cl-1-$(NMe_3)$-2,1-$RhCB_{10}H_9$ (**3**), which is the first structurally characterized mononuclear hydride complex with an icosahedral monocarbon carborane ligand.

## Results and Discussion

The reaction of **1**[5] with 1 molar equiv of $Rh(PPh_3)_3Cl$ (**2**) in hot methanol produced **3** as a yellow precipitate in 72% yield, together with a small amount of a 16-electron rhodacarborane, *closo*-2-$(PPh_3)$-2-Cl-1-$(NMe_3)$-2,1-$RhCB_{10}H_{10}$ (**4**), which was isolated from the reduced mother liquor by column chromatography on silica gel (Scheme 1). The structure of **4** was deduced from spectroscopic and analytical data. The $^{31}P\{^1H\}$ NMR spectrum of **4** exhibited one doublet at 37.3 ppm with $J_{P-Rh} = 152$ Hz due to the $PPh_3$ group attached to rhodium. Both the chemical shift and coupling constant values associated with this signal proved to be very similar to those observed in the $^{31}P\{^1H\}$ NMR spectra of the previously described 16-electron Rh(III) mononuclear monocarbon *closo*-rhodacarboranes.[6] The $^1H$ NMR spectrum of **4** revealed phenyl multiplets centered at 7.45 ppm as well as a singlet at 3.69 ppm, as expected for equivalent methyl groups in the $N(CH_3)_3$ cage substituent. The latter complex was obtained in reasonable yield when the reaction was terminated and the products were isolated after 3.5 h.[7] The latter observation suggests **4** as one of the possible intermediates leading to the formation of **3**.

**Scheme 1**



Complex **3** was obtained as an air-stable crystalline material, and its structure was elucidated as described below. The $^{31}P\{^1H\}$ NMR spectrum of **3** in $CH_2Cl_2$ contained two resonances, a sharp doublet ($\delta_{Pa} = 26.2$ ppm, $J_{P-Rh} = 104.5$ Hz) and a broadened signal ($\delta_{Pb} = 11.0$ ppm, $J_{P-B} \approx 120$ Hz) which resembled a quadruplet. These resonances were attributed to $PPh_3$ groups at rhodium and boron atoms, respectively. This was confirmed by the $^{11}B\{^1H\}$ NMR spectrum of **3** which revealed a unique sharp doublet at $-7.8$ ppm ($J_{B-P} \approx 127$ Hz) originating from the boron atom substituted by a $PPh_3$ group within a set of broad resonances derived from the BH vertices of the carborane cage. The $^1H$ NMR spectrum of **3** in $CD_2Cl_2$ exhibited complex overlapping multiplets originating from both of the $PPh_3$ groups, a singlet at 2.92 ppm from equivalent methyl groups at the $N(CH_3)_3$ substituent, and a doublet of doublets of doublets at $-9.63$ ppm due to the terminal hydride at the rhodium atom. The multiplicity of the hydride resonance observed is consistent with the coupling of hydrogen to two nonequivalent $PPh_3$ groups ($^{31}P_a$ and $^{31}P_b$) and a $^{103}Rh$ nucleus. Selective decoupling of $^{31}P_b$ nuclei caused a collapse of the hydride signal to a doublet of doublets with $J_{H-Rh} = 11.0$ Hz and $J_{H-Pa} = 46$ Hz. The small coupling constant of the $^{31}P_b$ nucleus with the hydride at the Rh center ($J_{H-P} \approx 7$ Hz) was elucidated from a second decoupling $^1H\{^{31}P_a\}$ NMR experiment and gave additional confirmation of the nature of this $PPh_3$ group.

The solid state structure of complex **3** was determined by an X-ray diffraction study which proved this species to be a *closo*-hydridorhodacarborane (Figure 1) with quaternary nitrogen and phosphorus groups attached to a monocarbon carborane ligand

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(7) In this case another new crystalline product was isolated from the reaction mixture by the procedure given in the Experimental Section. Although this compound was not available in sufficient quantities to allow complete characterization from spectroscopic data, it was concluded that the species can be regarded as a 12-vertex monocarbon rhodacarborane with $PPh_3$ ($\delta_P = 30.9$ ppm, $J_{P-Rh} = 164.2$ Hz in $CD_2Cl_2$) and $NMe_3$ ($\delta_H = 3.49$ ppm in $CD_2Cl_2$) ligands at the rhodium and at the carbon cage atoms, respectively. No other attempts, however, were made to characterize this compound.

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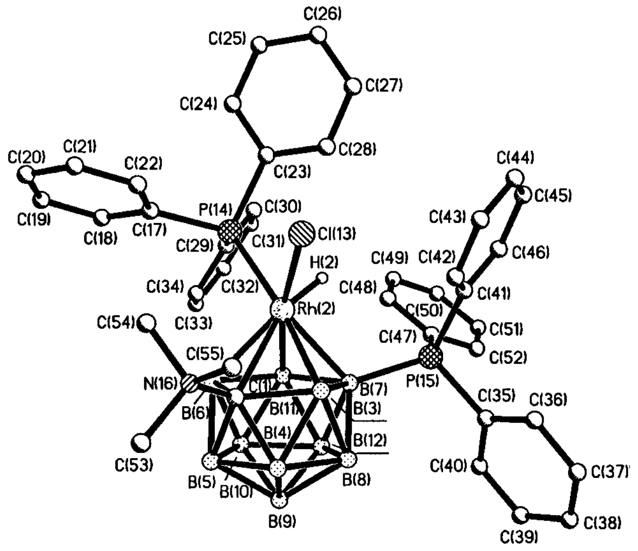

**Figure 1.** Molecular structure of complex **3**. All hydrogen atoms except H(Rh) have been omitted for clarity. Selected interatomic distances (Å) and angles (deg): Rh(2)−B(3) = 2.195(4), Rh(2)−B(6) = 2.189(4), Rh(2)−B(7) = 2.209(4), Rh(2)−B(11) = 2.178(4), Rh(2)−C(1) = 2.343(3), Rh(2)−P(14) = 2.355(1), Rh(2)−Cl = 2.449(1), Rh(2)−H(2) = 1.41(7), C(1)−N(16) = 1.561(4), B(17)−P(15) = 1.958(4); P(14)−Rh(2)−Cl(13) = 88.49(4), P(14)−Rh(2)−H(2) = 70(3), Cl(13)−Rh(2)−H(2) = 93(3).

**Table 1.** Crystallographic Data for Complex **3**

| | | | |
|---|---|---|---|
| formula | $C_{41}H_{51}B_{10}Cl_3NP_2Rh$ | $V$, Å$^3$ | 2225.2(7) |
| fw | 937.11 | $Z$ | 2 |
| cryst syst | triclinic | $d_{calcd}$, g/cm$^3$ | 1.398 |
| space group | $P\bar{1}$ (No. 2) | $\lambda$ (Mo Kα), Å | 0.71073 |
| $a$, Å | 11.460(2) | $\mu$ (Mo Kα), cm$^{-1}$ | 6.7 |
| $b$, Å | 11.649(2) | temp, °C | 20 |
| $c$, Å | 17.836(4) | $R_1$[a] | 0.0352 |
| α, deg | 108.94(3) | w$R_2$[b] | 0.1044 |
| $\beta$, deg | 94.57(3) | independent reflectn | 7819 |
| $\gamma$, deg | 95.69(3) | obsd reflectn | 5888 ($I > 2\sigma$) |

[a] $R_1 = \sum|F_o - |F_c||/\sum(F_o)$ for observed reflections. [b] $wR_2 = (\sum[w(F_o^2 - F_c^2)^2]/\sum[w(F_o^2)^2]]^{1/2}$ for all reflections.

and an 18-electron Rh center. The Rh(2)−C(1) distance of 2.343(3) Å, as in the previously studied monocarbon *closo*-2-Br-2-(PPh$_3$)-1-[(CH$_2$CH═CHCH$_3$)$_2$NH]-2,1-RhCB$_{10}$H$_{10}$,[6] is somewhat longer than the Rh−B bonds [2.178(4)−2.209(4) Å]. The Rh(2)−P(14) and the Rh(2)−Cl bond lengths of 2.355(1) and 2.449(1) Å, respectively, are in the range of the distances which are usually observed for (phosphine)rhodacarboranes containing chlorine at the metal vertex.[6,8] The B(7)−P(15) distance of 1.958(4) Å proved to be considerably longer than those observed in uncoordinated *nido*-9-PPh$_3$-7,8-C$_2$B$_9$H$_{11}$ [1.912(2) Å][9] or monocarbon *nido*-carborane 9-(PPh$_3$)-7-[((CH$_3$)$_3$Si)$_2$CH]CB$_{10}$H$_{11}$ [1.928(5) Å][10] with a PPh$_3$ substituent at the boron cage atoms.

**Table 2.** Selected Bond Lengths (Å) and Angles (deg) for Complex **3**

| | | | |
|---|---|---|---|
| C(1)−N(16) | 1.561(4) | B(6)−B(11) | 1.884(6) |
| C(1)−B(4) | 1.697(5) | B(7)−B(12) | 1.767(6) |
| C(1)−B(6) | 1.711(5) | B(7)−B(8) | 1.777(6) |
| C(1)−B(5) | 1.721(5) | B(7)−B(11) | 1.817(6) |
| C(1)−B(3) | 1.785(5) | B(7)−P(15) | 1.958(4) |
| C(1)−Rh(2) | 2.343(3) | B(8)−B(12) | 1.784(6) |
| Rh(2)−B(11) | 2.178(4) | B(8)−B(9) | 1.784(6) |
| Rh(2)−B(6) | 2.189(4) | B(9)−B(12) | 1.765(6) |
| Rh(2)−B(3) | 2.195(4) | B(9)−B(10) | 1.792(6) |
| Rh(2)−B(7) | 2.209(4) | B(10)−B(11) | 1.777(6) |
| Rh(2)−P(14) | 2.355(1) | B(10)−B(12) | 1.781(6) |
| Rh(2)−Cl(13) | 2.449(1) | B(11)−B(12) | 1.786(6) |
| B(3)−B(8) | 1.795(6) | P(14)−C(29) | 1.836(4) |
| B(3)−B(7) | 1.813(5) | P(14)−C(17) | 1.838(4) |
| B(3)−B(4) | 1.821(6) | P(14)−C(23) | 1.842(4) |
| B(4)−B(5) | 1.771(6) | P(15)−C(35) | 1.817(4) |
| B(4)−B(9) | 1.772(6) | P(15)−C(41) | 1.817(4) |
| B(4)−B(8) | 1.772(6) | P(15)−C(47) | 1.824(4) |
| B(5)−B(9) | 1.746(6) | N(16)−C(55) | 1.500(5) |
| B(5)−B(10) | 1.748(6) | N(16)−C(54) | 1.501(5) |
| B(5)−B(6) | 1.812(6) | N(16)−C(53) | 1.517(5) |
| B(6)−B(10) | 1.786(6) | | |

| | | | |
|---|---|---|---|
| N(16)−C(1)−B(4) | 112.7(3) | P(14)−Rh(2)−Cl(13) | 88.49(4) |
| N(16)−C(1)−B(6) | 123.0(3) | B(12)−B(7)−B(8) | 60.5(2) |
| B(4)−C(1)−B(6) | 113.9(3) | B(12)−B(7)−B(3) | 107.3(3) |
| N(16)−C(1)−B(5) | 114.8(3) | B(8)−B(7)−B(3) | 60.0(2) |
| B(4)−C(1)−B(5) | 62.4(2) | B(12)−B(7)−B(11) | 59.8(2) |
| B(6)−C(1)−B(5) | 63.7(2) | B(8)−B(7)−B(11) | 108.4(3) |
| N(16)−C(1)−B(3) | 121.2(3) | B(3)−B(7)−B(11) | 106.7(3) |
| B(4)−C(1)−B(3) | 63.0(2) | B(12)−B(7)−P(15) | 113.5(2) |
| B(6)−C(1)−B(3) | 108.4(3) | B(8)−B(7)−P(15) | 114.6(2) |
| B(5)−C(1)−B(3) | 112.3(3) | B(3)−B(7)−P(15) | 126.8(2) |
| N(16)−C(1)−Rh(2) | 116.4(2) | B(11)−B(7)−P(15) | 122.8(2) |
| B(4)−C(1)−Rh(2) | 119.6(2) | B(12)−B(7)−Rh(2) | 118.2(2) |
| B(6)−C(1)−Rh(2) | 63.1(2) | B(8)−B(7)−Rh(2) | 119.4(2) |
| B(5)−C(1)−Rh(2) | 119.9(2) | B(3)−B(7)−Rh(2) | 65.3(2) |
| B(3)−C(1)−Rh(2) | 62.5(2) | B(11)−B(7)−Rh(2) | 64.6(2) |
| B(11)−Rh(2)−B(6) | 51.1(2) | P(15)−B(7)−Rh(2) | 118.0(2) |
| B(11)−Rh(2)−B(3) | 83.5(2) | C(29)−P(14)−C(17) | 103.4(2) |
| B(6)−Rh(2)−B(3) | 80.6(2) | C(29)−P(14)−C(23) | 102.8(2) |
| B(11)−Rh(2)−B(7) | 48.9(2) | C(17)−P(14)−C(23) | 100.0(2) |
| B(6)−Rh(2)−B(7) | 83.9(2) | C(29)−P(14)−Rh(2) | 113.80(12) |
| B(3)−Rh(2)−B(7) | 48.62(14) | C(17)−P(14)−Rh(2) | 120.17(12) |
| B(11)−Rh(2)−C(1) | 80.87(14) | C(23)−P(14)−Rh(2) | 114.33(13) |
| B(6)−Rh(2)−C(1) | 44.21(14) | C(35)−P(15)−C(41) | 107.2(2) |
| B(3)−Rh(2)−C(1) | 46.19(13) | C(35)−P(15)−C(47) | 104.8(2) |
| B(7)−Rh(2)−C(1) | 80.24(13) | C(41)−P(15)−C(47) | 102.8(2) |
| B(11)−Rh(2)−P(14) | 98.67(12) | C(35)−P(15)−B(7) | 111.5(2) |
| B(6)−Rh(2)−P(14) | 94.28(12) | C(41)−P(15)−B(7) | 114.8(2) |
| B(3)−Rh(2)−P(14) | 171.42(10) | C(47)−P(15)−B(7) | 114.8(2) |
| B(7)−Rh(2)−P(14) | 138.10(10) | C(55)−N(16)−C(54) | 107.9(3) |
| C(1)−Rh(2)−P(14) | 125.79(9) | C(55)−N(16)−C(53) | 106.7(3) |
| B(11)−Rh(2)−Cl(13) | 170.03(11) | C(54)−N(16)−C(53) | 106.9(4) |
| B(6)−Rh(2)−Cl(13) | 135.66(11) | C(55)−N(16)−C(1) | 110.7(3) |
| B(3)−Rh(2)−Cl(13) | 90.39(11) | C(54)−N(16)−C(1) | 110.3(3) |
| B(7)−Rh(2)−Cl(13) | 121.37(11) | C(53)−N(16)−C(1) | 114.1(3) |
| C(1)−Rh(2)−Cl(13) | 100.61(9) | | |

One may assume that the attachment of the PPh$_3$ ligand to a carborane cage proceeds via an electronically unsaturated complex **4** which plays the role of an intermediate in the formation of **3** from **1** and **2**. The same mechanism has previously been suggested for the formation of some boron-substituted (phosphine)rhoda-,[8] (phosphine)ruthena-,[11] and (phosphine)nickelacarboranes.[12] The reaction of **4** with 1 molar equiv of PPh$_3$ was carried out at reflux in methanol for 2 h to produce **3**, whose structure was confirmed by its characteristic $^1$H and $^{31}$P NMR spectra, in 68% yield. This result lends strong support to the assumption that **3** arises from the reaction of **4** with PPh$_3$.

## Experimental Section

All experiments were performed under an atmosphere of dry argon in standard Schlenk equipment. Solvents were dried according to standard literature procedures[13] prior to use. The $^1$H, $^{31}$P, and $^{11}$B NMR

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spectra were obtained by using a Bruker WP-200-SY spectrometer operating at 200.13, 81.02, and 64.2 MHz, respectively, and using $SiMe_4$, $H_3PO_4$, and $BF_3{\cdot}Et_2O$ as external standards with high-field resonances being quoted as negative values. The IR spectra were obtained as KBr pellets and recorded on a Specord M-82 spectrometer. Microanalyses were carried out in the analytical laboratory of the Institute of Organoelement Compounds of the Russian Academy of Sciences.

**Preparation of *closo*-2,7-$(PPh_3)_2$-2-H-2-Cl-1-$(NMe_3)$-2,1-$RhCB_{10}H_9$ (3) and *closo*-2-$(PPh_3)$-2-Cl-1-$(NMe_3)$-2,1-$RhCB_{10}H_{10}$ (4).** (***A***) A suspension of **1** (0.10 g, 0.52 mmol) and $Rh(PPh_3)_3Cl$ (0.48 g, 0.52 mmol) in methanol (30 mL) was stirred under reflux for 16 h, and the yellow precipitate thus formed was collected by filtration, washed with methanol, and dried under vacuum to give an analytically pure complex **3** (0.32 g, 72.4%). Anal. Calcd for $C_{40}H_{49}B_{10}ClNP_2Rh$: C, 56.36; H, 5.79; B, 12.69; Cl, 4.15; P, 7.26. Found: C, 56.58; H, 5.48; B, 12.29; Cl, 4.00; P, 7.29. IR ($cm^{-1}$): $\upsilon$(B−H) 2524. $^1H$ NMR ($CD_2Cl_2$, 22 °C): $\delta$ 7.61−7.52 (m, 30H, Ph), 2.92 (s, 9H, $CH_3$), −9.63 (ddd, 1H, Rh−H). $^{31}P\{^1H\}$ NMR ($CH_2Cl_2$, 22 °C): $\delta$ 26.2 (d, $J_{P-Rh} = 104.5$ Hz), 11.0 (q-like, $J_{P-B} \approx 120$ Hz). $^{11}B\{^1H\}$ NMR ($CH_2Cl_2$, 22 °C): −2.9 (s), −7.8 (d, $J_{P-B} = 127$ Hz), −8.9 (s), −19.1 (s), −22.3 (s). The reduced mother liquor was chromatographed on silica gel with $CH_2Cl_2$ as the eluent. The deep red fraction eluted as the first band was evaporated, and the solid residue was recrystallized from a mixture of $CH_2Cl_2$/*n*-hexane to afford red microcrystalline **4** (0.022 g, 7.2%). Anal. Calcd for $C_{22}H_{34}B_{10}ClNPRh$: C, 44.78; H, 5.77; B, 18.32; Cl, 6.02. Found: C, 44.77; H, 5.35; B, 18.20; Cl, 6.89. IR ($cm^{-1}$): $\upsilon$(B−H) 2528. $^1H$ NMR ($CD_2Cl_2$, 22 °C): $\delta$ 7.63−7.32 (m, 30H, Ph), 3.69 (s, 9H, $CH_3$). $^{31}P\{^1H\}$ NMR ($CH_2Cl_2$, 22 °C): $\delta$ 37.3 (d, $J_{P-Rh} = 152$ Hz). $^{11}B\{^1H\}$ NMR ($CD_2Cl_2$, 22 °C): 8.4 (1B), 6.7 (2B), 2.9 (2B), −6.3 (1B), −18.0 (4B).

(***B***) The treatment of **1** with $Rh(PPh_3)_3Cl$ under exactly the same conditions, but interrupted after 3.5 h and worked up by an analogous procedure followed by separation of the reduced mother liquor on preparative TLC plates using $CH_2Cl_2$ as the eluent, afforded **3** (0.17 g, 38%), **4** (0.073 g, 24%), and a small quantity of an unidentified pink crystalline species (from $CH_2Cl_2$/*n*-hexane) which was characterized by $^1H$ and $^{31}P\{^1H\}$ NMR spectra only.[7]

(***C***) Freshly recrystallized $PPh_3$ (0.050 g, 0.19 mmol) was added to a stirred suspension of **4** (0.050 g, 0.085 mmol) in methanol (10 mL) which was allowed to reflux for 2 h. The yellow precipitate was filtered off and dried under vacuum to afford **3** (0.050 g, 68%).

**X-ray Diffraction Study of 3.** The crystal data are summarized in Table 1, and selected bond lengths and angles are given in Table 2. X-ray quality crystals of **3** were obtained by slow crystallization from $CH_2Cl_2$/*n*-hexane at −5 °C. Experimental data were collected on a parallelepiped, 0.3 × 0.3 × 0.2 mm, on a CAD4 Enraf-Nonius diffractometer ($T = 293$ K, graphite-monochromated Mo K$\alpha$ radiation, $(\theta - 5)/3\theta$ scan technique, $2\theta < 50°$). The structure was solved by direct methods using SHELXTL PLUS programs (PC Version) and refined by the SHELXL-93 program. The carbon atom of the $CH_2Cl_2$ solvating molecule is disordered over two positions with occupancies of 0.8 and 0.2.

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**Supporting Information Available:** Tables of crystallographic data collection parameters, atom coordinates, bond distances and angles, and anisotropic parameters and a figure for **3** (11 pages). Ordering information is given on any current masthead page.

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