Facile formation of *exo-nido*→*closo*-rearrangement products upon the replacement of PPh_3 ligands with bis(diphenylphosphino)alkanes in "three-bridge" ruthenacarborane 5,6,10-[$RuCl(PPh_3)_2$]-5,6,10-(μ-H)$_3$-10-H-*exo-nido*-7,8-$C_2B_9H_8$

D. N. Cheredilin, E. V. Balagurova, I. A. Godovikov, S. P. Solodovnikov, and I. T. Chizhevsky★

A. N. Nesmeyanov Institute of Organoelement Compounds, Russian Academy of Sciences, 28 ul. Vavilova, 119991 Moscow, Russian Federation. Fax: +7 (495) 135 5085. E-mail: chizbor@ineos.ac.ru

The replacement of the PPh_3 ligands in "three-bridge" *exo-nido*-ruthenacarborane 5,6,10-[$RuCl(PPh_3)_2$]-5,6,10-(μ-H)$_3$-10-H-*exo-nido*-7,8-$C_2B_9H_8$ with diphosphines, *viz.*, 1,3-bis(diphenylphosphino)propane (dppp) or 1,4-bis(diphenylphosphino)butane (dppb) dramatically decreases the barrier to the thermal *exo-nido*→*closo* rearrangement affording the chelate *closo*-complexes 3,3-[$Ph_2P(CH_2)_nPPh_2$]-3-H-3-Cl-*closo*-3,1,2-$RuC_2B_9H_{11}$ (n = 3 or 4) under mild conditions. In the reaction with dppp, the rearrangement is accompanied by the formation of 17-electron paramagnetic *closo*-ruthenacarborane 3,3-[$Ph_2P(CH_2)_3PPh_2$]-3-Cl-*closo*-3,1,2-$RuC_2B_9H_{11}$, which could be isolated as the main product when the reaction was carried out at 80 °C.

Key words: synthesis, diphosphine *exo-nido*- and *closo*-ruthenacarboranes, *exo-nido*→*closo* rearrangement, paramagnetic *closo*-ruthenacarborane.

The chemistry of *exo-nido*-metallacarboranes, in which the metal atom is coordinated by the *nido*-carborane ligand through a multiple two-electron three-center $(B—H)_n$...M bonds (n = 2 or 3), is of interest because these clusters can easily be transformed into new mononuclear *closo*-[1,2] or dinuclear *exo-closo*-metallacarboranes.[3—5] A detailed study of the *exo-nido*→*closo* rearrangement of metallacarboranes has been primarily performed for 16-electron "two-bridge" bis(triphenylphosphine)-*exo-nido*-rhodacarboranes.[6] It has been found that isomeric *exo-nido*- and *closo*-rhodacarboranes exist in solution as an equilibrium mixture, and for complexes containing the sterically more hindered carborane ligand, this equilibrium is normally shifted to *exo-nido* structures.[6] In the case of stronger coordination of the transition metal atom by the *nido*-carborane ligand *via* three two-electron three-center $(B—H)_3$...M bonding interactions, only 18-electron chlorobis(triphenylphosphine)-*exo-nido*-metallacarboranes containing unsubstituted carborane can be rearranged into isomeric *closo*-complexes. If M = Ru, heating is required for such a rearrangement,[1] whereas compounds with M = Os undergo the rearrangement at room temperature.[2]

In the present study, we found that the replacement of the PPh_3 ligands in "three-bridge" *exo-nido*-ruthenacarborane 5,6,10-[$RuCl(PPh_3)_2$]-5,6,10-(μ-H)$_3$-10-H-*exo-nido*-7,8-$C_2B_9H_8$ (**1**) with diphosphines [$Ph_2P(CH_2)_nPPh_2$] (n = 3 or 4) decreases the barrier to the thermal *exo-nido*→*closo* rearrangement, with the result that it proceeds in benzene at 22 °C.

Results and Discussion

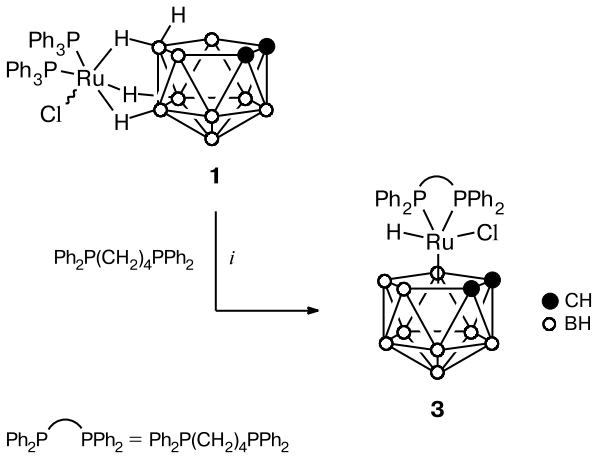
The reaction of *exo-nido*-ruthenacarborane **1** with a 10% excess of 1,4-bis(diphenylphosphino)butane (**2**, dppb) in benzene at 22 °C for 48 h afforded the *closo*-complex 3,3-(dppb)-3-H-3-Cl-*closo*-3,1,2-$RuC_2B_9H_{11}$ (**3**) (Scheme 1). After column chromatography of the reaction mixture (silica gel; C_6H_6—*n*-hexane, 1 : 1, as the eluent), product **3** was isolated as air-stable yellow crystals in 78% yield. Complex **3** is moderately soluble in C_6H_6, CH_2Cl_2, and acetone and is virtually insoluble in hydrocarbons. Upon recrystallization from a CH_2Cl_2—*n*-hexane mixture, complex **3** formed a stable solvate with CH_2Cl_2.

The composition and structure of *closo*-complex **3** were determined by elemental analysis and $^{31}P\{^1H\}$, ^1H, $^1H\{^{31}P\}$, $^{13}C\{^1H\}$, ^{11}B, and $^{11}B\{^1H\}$ NMR spectroscopy. The $^{31}P\{^1H\}$ NMR spectrum of complex **3** in CD_2Cl_2 exhibited two closely spaced singlet resonances with equal intensities at δ 37.5 and 37.45 (the spectrum of **3** in C_6D_6 consists of only one collapsed resonance), which indicates that complex **3** contains the chelate dppb ligand coordinated to the metal atom. It should be noted that the

Published in Russian in *Izvestiya Akademii Nauk. Seriya Khimicheskaya,* No. 11, pp. 2455—2459, November, 2005.

1066-5285/05/5411-2535

Scheme 1



i. C_6H_6, 22 °C, 48 h.

spectrum of the starting complex **1**, which exists as a mixture of symmetrical and asymmetrical isomeric species, shows resonances of the PPh_3 ligands at substantially lower field (at δ 50—55) as a singlet and two doublets, respectively.[7] In the ^1H NMR spectrum of complex **3**, the signals of the aliphatic moiety of the coordinated diphosphine ligand are observed as four multiplets, the intensity of each multiplet being 2H. The resonances of the methylene groups of the dppb ligand in **3** were assigned based on the $^1H\{^{31}P\}$ NMR spectrum, in which the multiplicities of the signals of the CH_2 groups directly bound to the phosphorus atoms differ substantially from those observed in the ^1H NMR spectrum, which allowed us to estimate the coupling constant for the pairs of the nonequivalent geminal protons H_A and H_B of these groups ($J_{A,B} \approx 14.5$ Hz). The assignment was confirmed by the $\{^1H—^1H\}$ COSY correlation spectrum, which shows the corresponding cross-peaks between the signals for H_A and H_B of each CH_2 group. It should also be noted that the ^1H NMR spectrum of complex **3** revealed a high-field resonance at δ −8.16 as a doublet of triplets with the coupling constants $J_1 = 27.5$ Hz and $J_2 = 6.0$ Hz characteristic of a metal hydride. In the $^1H\{^{31}P\}$ NMR spectrum, this signal is collapsed to a doublet with $J_2 = 6.0$ Hz. Hence, the coupling constants J_1 and J_2 in the initially observed doublet of triplets can be assigned to $^2J_{H,P}$ and $^3J_{H,H}$, respectively. The constant $^3J_{H,H}$ is associated with either the long-range spin-spin coupling H(Ru)...H(B) through the metal atom or the specific through-space coupling interaction between the metal hydride and the hydrogen atom of the BH group, which has been observed earlier for the structurally similar ruthenium complexes *closo*-3,3-$(PPh_3)_2$-3-H-3-Cl-3,1,2-$RuC_2B_9H_{11}$ (see Ref. 1) and 1,1-$(PPh_3)_2$-1-H-1-Cl-*closo*-1,2,3-$RuC_2B_4H_6$ (see Ref. 8).

Although complex **3** was isolated under the above-mentioned conditions as the only product, TLC monitoring of the reaction provided evidence for the formation of an apparent intermediate. The intermediate complex was isolated as a mixture with complex **3** when the reaction was terminated immediately after the disappearance of the starting reagent **1** from the reaction mixture, *i.e.*, after approximately ~2 h. This complex was further identified as 5,6,10-[RuCl(dppb)]-5,6,10-$(\mu\text{-H})_3$-10-H-*exo-nido*-7,8-$C_2B_9H_8$ (**4**). In spite of the fact that complex **4** was observed on a silica gel column as an individual band, whose R_f differs substantially from that of the final product (**3**, $R_f = 0.75$; **4**, $R_f = 0.35$), this complex appeared to be kinetically unstable since, upon storage in a benzene solution, it is readily transformed into compound **3**. This fact was unambiguously confirmed by monitoring of the ^1H NMR spectrum of this solution.

That is why the structure of *exo-nido*-complex **4** was studied only by ^1H and $^{31}P\{^1H\}$ NMR spectroscopy of a ~1 : 1 mixture of **4** with complex **3** (Fig. 1). The upfield region of the ^1H NMR spectrum from 0 to −20 ppm, where the hydride resonances of the exopolyhedral B—H...Ru bonds and the resonances of the H_{extra} atom are observed, is proved to be most informative. Analysis of the NMR data demonstrated that complex **4**, like its precursor **1**, exists as a mixture of geometric isomers having symmetric (*s*) and asymmetric (*as*) structures (see Fig. 1). We assigned the signals of these isomers by analogy with the Ru [7] and Os [9] *exo-nido*-complexes described

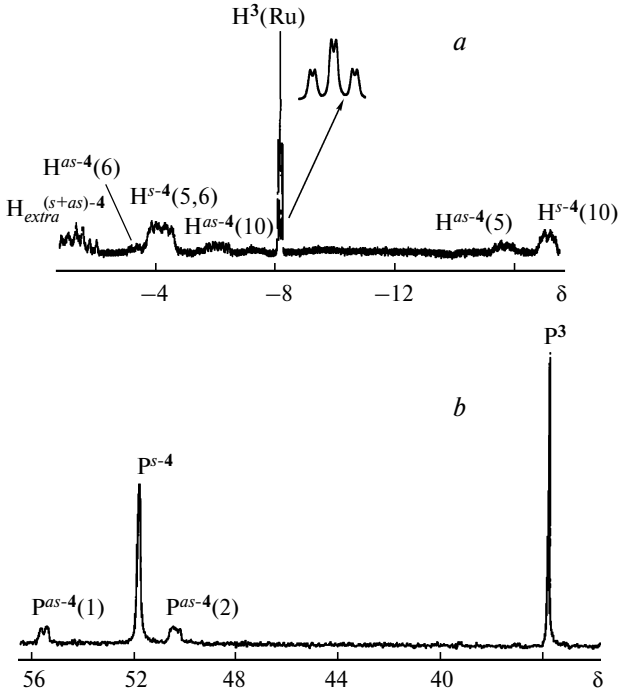
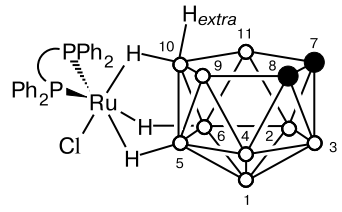
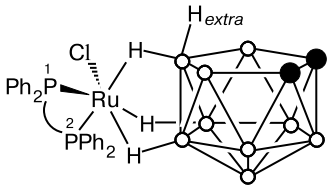


Fig. 1. ^1H NMR (*a*) and $^{31}P\{^1H\}$ NMR (*b*) spectra of a mixture of the *s* and *as* isomers of complexes **3** and **4** in CD_2Cl_2.

earlier. The presence of two isomers in a solution of **4** was confirmed also by the $^{31}P\{^1H\}$ NMR spectrum of **4** in CD_2Cl_2, which showed two sets of resonances, a singlet at δ 51.8 (*s* isomer) and two doublets of equal intensities at δ 55.5 and 50.3 with $^2J_{P,P}$ = 35 Hz (the *as* isomer).



s-**4**



as-**4**

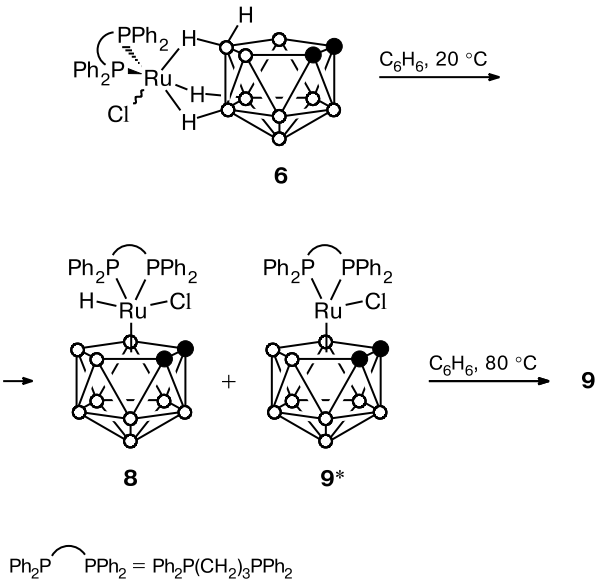
We thus established that the replacement of the triphenylphosphine ligands in complex **1** with dppb initially affords *exo-nido*-complex **4**, which is readily rearranged into the final *closo*-complex **3** at room temperature.

Taking into account this reaction pathway, we turned our attention to the *exo-nido*-complex with 1,3-bis(diphosphino)propane (**5**, dppp) as a chelating diphosphine ligand, *viz.*, 5,6,10-[RuCl(dppp)]-5,6,10-(μ-H)$_3$-10-H-*exo-nido*-7,8-$C_2B_9H_8$ (**6**), which has been studied earlier.[10] The conditions of the *exo-nido*→*closo* rearrangement of complex **6** remained unknown. In spite of the fact that *exo-nido*-complex **6** is rather stable in the solid state, in a benzene solution the B—H...Ru coordination bonds in this complex are readily cleaved in the presence of free dppp ligands to produce the 16-electron cationic species $[RuCl(dppp)_2]^+[nido\text{-}7,8\text{-}C_2B_9H_{12}]^-$ (**7**). Because of this, the synthesis of **6** by the replacement of the PPh_3 ligands in **1** with dppp always gives rise to a mixture consisting of complexes **6** and **7**.[10]

In the present study, we found that *exo-nido*-complex **6**, like complex **4**, can be rearranged under mild conditions into *closo*-3,3-(dppp)-3-H-3-Cl-3,1,2-$RuC_2B_9H_{11}$ (**8**). However, this reaction in benzene at 22 °C proceeds very slowly, and 60 h are required to achieve 100% conversion. The *exo-nido*→*closo* rearrangement is accompanied by the formation of the 17-electron paramagnetic complex *closo*-3,3-(dppp)-3-Cl-3,1,2-$RuC_2B_9H_{11}$ (**9**) as a by-product (Scheme 2).

Complexes **8** and **9** were isolated as a mixture in a total yield of 86%. We succeeded in separating these complexes into individual compounds only by TLC on Silufol

Scheme 2



* Trace amounts.

plates using a 1 : 1 benzene—hexane mixture as the eluent. The ^1H and $^{31}P\{^1H\}$ NMR spectroscopic data for diamagnetic complex **8** agree well with its postulated structure. The ^1H NMR spectrum of complex **9**, which has a number of broad unassignable peaks in the region from —14 to + 12 ppm, is characteristic of paramagnetic ruthenacarborane complexes.[11] Pure 17-electron complex **9** can be prepared in 44% yield by refluxing a mixture of complexes **8** and **9** in benzene for 3.5 h. The composition of complex **9** was confirmed by elemental analysis, and its paramagnetic structure was supported by the ESR spectrum (Fig. 2). It should be noted that paramagnetic *closo*-ruthenacarborane complexes of this type have been prepared for the first time only recently.[11] Therefore, the direct synthesis of new 17-electron complex **9** is of obvious interest. It should also be noted that *exo-nido*-ruthenacarborane **4** under analogous thermal conditions (benzene, 80 °C) does not give a paramagnetic complex structurally similar to **9**.

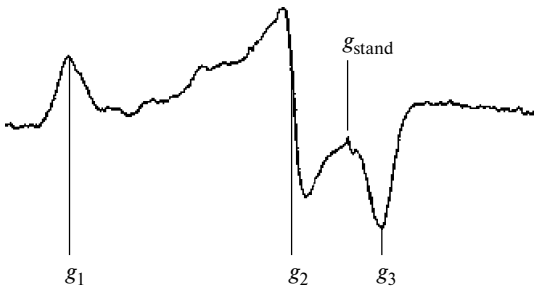


Fig. 2. ESR spectrum of 17-electron complex **9**: g_1 = 2.39, g_2 = 2.08, g_3 = 1.96.

Based on the results of the present study and earlier data,[10,11] it can be concluded that *exo-nido*-ruthenacarborane complex **1** is a convenient and readily available reagent for the synthesis of *closo*-ruthenacarboranes with various chelate diphosphines, including the synthesis of particular paramagnetic complexes of type **9** in moderate yields. In this process, the formation of *closo* products occurs through the *exo-nido*→*closo* rearrangement of diphosphine *exo-nido*-complexes.

Experimental

All reactions were carried out under argon with the use of anhydrous solvents, which were prepared according to standard procedures. The reaction products were isolated and purified by column chromatography with the use of silica gel (Merck, 230—400 mesh). Diphosphines dppb and dppp were purchased from Strem Chemicals. The NMR spectra were recorded on Bruker AMX-400 (400.13 MHz for ^{1}H, 161.98 MHz for ^{31}P, and 128.3 MHz for ^{11}B) and Bruker Avance^{TM}300 (300.13 MHz for ^{1}H) spectrometers. The ESR spectrum of complex **9** was measured on a Varian E-12A radiospectrometer. Elemental analysis was carried out in the Laboratory of Microanalysis of the A. N. Nesmeyanov Institute of Organoelement Compounds of the Russian Academy of Sciences.

3-Chloro-3,3-(1,4-diphenylphosphinobutane)-3-hydro-3,1,2-*closo*-dicarbollylruthenium (3). ***A.*** Complex **1** (0.03 g, 0.04 mmol) was added to a solution of diphosphine **2** (0.02 g, 0.05 mmol) in benzene (12 mL), and the reaction mixture was stirred at 22 °C for 48 h. The solvent was removed *in vacuo*. The residue was chromatographed on a silica gel column, complex **3** being eluted with a 1 : 1 benzene—hexane mixture. The orange crystalline product was recrystallized from an *n*-hexane—CH_2Cl_2 mixture, and analytically pure complex **3** was isolated in 77% yield.

B. *exo-nido*-Ruthenacarborane **1** (0.1 g, 0.13 mmol) was added to a solution of diphosphine **2** (0.06 g, 0.14 mmol) in benzene (15 mL). The reaction mixture was stirred under gentle reflux for 1 h. The solvent was evaporated *in vacuo*. The residue was chromatographed on a short silica gel column, complex **3** being eluted with a 1 : 1 benzene—hexane mixture. The yield of **3** was 73%.

Found (%): C, 50.63; H, 5.65; P, 8.64. $C_{30}H_{40}B_9ClP_2Ru \cdot 0.25CH_2Cl_2$. Calculated (%): C, 50.52; H, 5.85; P, 8.66. ^{1}H NMR ($CDCl_3$), δ: −8.22 (dt, 1 H, RuH, $J_{H...H-B}$ = 6.0 Hz, $J_{H,P}$ = 27.5 Hz); 1.52 (br.m, 2 H, $(PCH_2CH_A\underline{H}_B)_2$); 1.72 (br.m, 2 H, $(PCH_2C\underline{H}_AH_B)_2$); 2.50 (br.m, 2 H, $(PCH_A\underline{H}_B)_2(CH_2)_2$); 3.28 (br.s, 2 H, $C_{carb}H$); 3.45 (br.m, 2 H, $(PC\underline{H}_AH_B)_2(CH_2)_2$); 7.93—7.21 (2 t + m, 20 H, Ph). ^{1}H{^{31}P} NMR ($CDCl_3$), δ: −8.22 (d, 1 H, RuH, $J_{H...H-B}$ = 6.0 Hz); 1.52 (br.m, 2 H, $(PCH_2CH_A\underline{H}_B)_2$); 1.72 (br.m, 2 H, $(PCH_2C\underline{H}_AH_B)_2$); 2.50 (br.dt, 2 H, $(PCH_A\underline{H}_B)_2(CH_2)_2$, J = 3.5 Hz, $J_{A,B}$ = 14.5 Hz); 3.28 (br.s, 2 H, $C_{carb}H$); 3.45 (ddd, 2 H, $(PC\underline{H}_AH_B)_2(CH_2)_2$, J_1 = 3.5 Hz, J_2 = 9.0 Hz, $J_{A,B}$ = 14.5 Hz); 7.93—7.21 (2 d + m, 20 H, Ph). ^{13}C{^{1}H} NMR ($CDCl_3$), δ: 21.56 (2 C), 25.81, 26.26 ($P(CH_2)_4P$); 63.39 (C_{carb}); 127.35, 128.68, 130.20, 131.14, 133.14, 134.32 (Ph). ^{31}P{^{1}H} NMR (CD_2Cl_2), δ: 37.4 and 37.5 (both s, 1 P + 1 P). ^{11}B NMR (CD_2Cl_2), δ: −21.8 (d, 2 B, J = 135.0 Hz); −19.9 (br.d, 1 B); −8.1 (d, 5 B, J = 138.0 Hz); 5.5 (d, 1 B, J = 142.5 Hz).

A mixture of chlorodiphenylphosphinobutane-*exo-nido*-[10-hydroorthocarborane-5,6,10-tris(hydro)]ruthenium (4) and complex 3. A solution of complex **1** (0.06 g, 0.08 mmol) and diphosphine **2** (0.04 g, 0.09 mmol) in benzene (10 mL) was stirred at 22 °C until the starting complex **1** was completely consumed (~2 h, TLC control). The solvent was removed *in vacuo*. The residue was chromatographed on a silica gel column under a low pressure of an inert gas, a colored band with R_f = 0.75 being eluted with benzene (the first fraction) followed by elution of the band with R_f = 0.35 (the second fraction). After removal of the solvent *in vacuo*, pure complex **3** was isolated from the first fraction in 4% yield, and a mixture of complexes **3** and **4** in an approximate ratio of 1 : 1 was isolated from the second (major) fraction. The total yield of complexes **3** and **4** isolated from the second fraction was 89%.

Complex **4** (*s* : *as* = 2.5 : 1). ^{1}H NMR* (CD_2Cl_2), δ: −17.12 (m, *s*); −15.69 (m, *as*); −6.10 (m, *as*); −4.70 — −3.10 (m, *as* + *s*); −1.32 (m, H_{extra}, *s* + *as*); 2.25 (br.s, $C_{carb}H$, *s*); 2.20 and 2.30 (both br.s, $C_{carb}H$, *as*); 2.01, 2.41, 3.43, and 3.57 (all br.m, $PCH_2CH_2CH_2P$, *s* + *as*); 7.31—7.84 (m, Ph, *s* + *as*). ^{31}P{^{1}H} NMR, δ: 50.3 and 55.5 (both d, *as*, $J_{P,P}$ = 35 Hz); 51.8 (c, *s*).

A mixture of 3-chloro-3,3-(1,3-diphenylphosphinopropane)-3-hydro-3,1,2-*closo*-dicarbollylruthenium (8) and 3-chloro-3,3-(1,3-diphenylphosphinopropane)-3,1,2-*closo*-dicarbollylruthenium (9). A solution of complex **6** (0.035 g, 0.05 mmol), which was prepared according to a procedure described earlier,[10] in benzene (10 mL) was stirred at 22 °C for ~60 h until the starting complex **6** was completely consumed. The solvent was removed *in vacuo*. The residue was chromatographed on a silica gel column, a mixture of complexes **8** and **9** being eluted with benzene. The yield of the mixture was 86%. To identify the complexes by NMR and ESR spectroscopy, the mixture of **8** and **9** (0.008 g) was separated into individual compounds by TLC on Silufol plates with the use of a 1 : 1 benzene—hexane mixture as the eluent.

Complex **8**. ^{1}H NMR ($CDCl_3$), δ: −8.75 (dt, 1 H, RuH, $J_{H...H-B}$ = 14.0 Hz, $J_{H,P}$ = 25.0 Hz); 1.94 (br.m, 1 H, $(PCH_2)_2CH_A\underline{H}_B$); 2.38 (br.m, 1 H, $(PCH_2)_2C\underline{H}_AH_B$); 2.58 (m, 2 H, $(PCH_A\underline{H}_B)_2CH_2$); 3.44 (dt, 2 H, $(PC\underline{H}_AH_B)_2CH_2$, J_1 = 13.5 Hz, J_2 = 3.0 Hz); 3.59 (br.s, 2 H, $C_{carb}H$); 7.23—7.66 (m, 20 H, Ph). ^{31}P{^{1}H} NMR ($CDCl_3$), δ: 28.5 (s).

Paramagnetic complex **9**. A solution of a mixture of complexes **8** and **9** (0.02 g, 0.03 mmol) in benzene (7 mL) was refluxed for 3 h. The solvent was evaporated *in vacuo*. The residue was chromatographed on a silica gel column, complex **9** being eluted with benzene. The yield of **9** was 44%. Found (%): C, 51.12; H, 5.44; B, 14.29. $C_{29}H_{37}B_9ClP_2Ru$. Calculated (%): C, 50.68; H, 5.10; B, 14.23. ESR (CH_2Cl_2, g_{stand} = 2.0023, T = 77 K): g_1 = 2.3933; g_2 = 2.0796; g_3 = 1.9594.

This study was financially supported by the Russian Foundation for Basic Research (Project No. 03-03-32651).

* Only hydride resonances of isomers *s*-**4** and *as*-**4** with their assignment are given.

References

1. I. T. Chizhevsky, I. A. Lobanova, V. I. Bregadze, P. V. Petrovskii, A. V. Polyakov, A. I. Yanovsky, and Yu. T. Struchkov, *Metalloorg. Khim.,* 1991, **4**, 957 [*Organomet. Chem. USSR*, 1991, **4**, 469 (Engl. Transl.)].
2. G. D. Kolomnikova, P. V. Sorokin, I. T. Chizhevsky, P. V. Petrovskii, V. I. Bregadze, F. M. Dolgushin, and A. I. Yanovsky, *Izv. Akad. Nauk, Ser. Khim.*, 1997, 2076 [*Russ. Chem. Bull.*, 1997, **46**, 1971 (Engl. Transl.)].
3. I. A. Lobanova, I. T. Chizhevsky, P. V. Petrovskii, and V. I. Bregadze, *Izv. Akad. Nauk, Ser. Khim.*, 1996, 250 [*Russ. Chem. Bull.*, 1996, **45**, 241 (Engl. Transl.)].
4. D. D. Ellis, S. M. Couchman, J. C. Jeffery, J. M. Malget, and F. G. A. Stone, *Inorg. Chem.*, 1999, **38**, 2981.
5. I. T. Chizhevsky, I. A. Lobanova, P. V. Petrovskii, V. I. Bregadze, F. M. Dolgushin, A. I. Yanovsky, Yu. T. Struchkov, A. L. Chistyakov, I. V. Stankevich, C. B. Knobler, and M. F. Hawthorne, *Organometallics*, 1999, **18**, 726.
6. J. A. Long, T. B. Marder, P. E. Behnken, and M. F. Hawthorne, *J. Am. Chem. Soc.*, 1984, **106**, 2979.
7. I. T. Chizhevsky, I. A. Lobanova, V. I. Bregadze, P. V. Petrovskii, V. A. Antonovich, A. V. Polyakov, A. I. Yanovsky, and Yu. T. Struchkov, *Mendeleev Commun.*, 1991, 47.
8. I. V. Pisareva, F. M. Dolgushin, O. L. Tok, V. E. Konoplev, K. Yu. Suponitsky, A. I. Yanovsky, and I. T. Chizhevsky, *Organometallics*, 2001, **20**, 4216.
9. G. D. Kolomnikova, P. V. Petrovskii, P. V. Sorokin, F. M. Dolgushin, A. I. Yanovsky, and I. T. Chizhevsky, *Izv. Akad. Nauk, Ser. Khim.*, 2001, 677 [*Russ. Chem. Bull., Int. Ed.*, 2001, **50**, 706].
10. D. N. Cheredilin, F. M. Dolgushin, E. V. Balagurova, I. A. Godovikov, and I. T. Chizhevsky, *Izv. Akad. Nauk, Ser. Khim.*, 2004, 2000 [*Russ. Chem. Bull., Int. Ed.*, 2004, **53**, 2086].
11. D. N. Cheredilin, R. Kadyrov, F. M. Dolgushin, E. V. Balagurova, I. A. Godovikov, S. P. Solodovnikov, and I. T. Chizhevsky, *Inorg. Chem. Commun.*, 2005, **8**, 614.

Received September 26, 2005