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Mercury derivatives of *exo-nido*-ruthenacarborane

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We dedicate this article to Professor Stanisław Pasynkiewicz on the occasion of his 70th birthday in recognition of his outstanding contributions to organometallic chemistry

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Abstract

Symmetrical mercury derivative of *exo-nido*-ruthenacarborane was prepared by two routes: mercuration of *exo-nido*-5,6,10-[$Cl(Ph_3P)_2Ru$]-5,6,10-(μ-H)$_3$-10-H-7,8-$C_2B_9H_8$ (**1**) and interaction of $Ru(PPh_3)_3Cl_2$ with [10,10′-Hg-(7,8-$C_2B_9H_{11}$)$_2$]Cs_2. Using *o*-carboran-9-ylmercury trifluoroacetate for mercuration of **1** leads to an unsymmetrical mercury compound with 9-*o*-carboranyl and *exo-nido*-ruthenacarboranyl ligands: 5′,6′,10′-*exo-nido*-[$Cl(Ph_3P)_2Ru$]-5′,6′,10′-(μ-H)$_3$-10′-(1,2-$C_2B_{10}H_{11}$Hg-9)-7′,8′-$C_2B_9H_8$ (**4**). The same compound was prepared by the action of $Ru(PPh_3)_3Cl_2$ on [9,10′-Hg-(1,2-$C_2B_{10}H_{11}$)(7′,8′-$C_2B_9H_{11}$)]Cs (**5**). Both types of new compounds were obtained as a mixture of *cis*/*trans* isomers which were separated and characterized by elemental analysis and NMR spectra. The X-ray structure of *trans*-5′,6′,10′-*exo-nido*-[$Cl(Ph_3P)_2Ru$]-5′,6′,10′-(μ-H)$_3$-10′-(1,2-$C_2B_{10}H_{11}$Hg-9)-7′,8′-$C_2B_9H_8$ (**4b**) was determined. © 2000 Elsevier Science S.A. All rights reserved.

Keywords: Metallacarboranes; Mercuration; *exo-nido*-Ruthenacarborane; *closo*-Rhodacarborane

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1. Introduction

Electrophilic mercuration of icosahedral carboranes was shown to proceed under the action of a strong mercuration agent such as trifluoroacetate mercury in trifluoroacetic acid to give carboranyl compounds with an Hg–B σ-bond [1]. These compounds play an important role in the synthesis of organic and organometallic derivatives of carboranes [2].

Mercuration of metallacarboranes leads to the mercury derivatives of metallacarboranes, 3-Cp-3,1,2-$FeC_2B_9H_{11}$ [3], 3-Cp-3,1,2-$CoC_2B_9H_{11}$ [4], [$(1,2\text{-}C_2B_9H_{11})_2Co]^-$ [4], $(1,2\text{-}C_2B_9H_{11})_2Ni$ [5], and 3,6-Cp_2-3,6,1,2-$Co_2C_2B_8H_{10}$ [6].

2. Results and discussion

In this paper we present first data on the synthesis of mercury derivatives of *exo-nido*-metallacarboranes using the example of *exo-nido*-5,6,10-[$Cl(Ph_3P)_2Ru$]-5,6,10-(μ-H)$_3$-10-H-7,8-$C_2B_9H_8$ (**1**) [7].

Earlier bimetallic complexes based on ruthenacarborane **1** as ligand were obtained [8] that permit us to consider it as organometallic analog of 7,8-dicarba-*nido*-undecaborate anion. Therefore, we believed that the same agents used for mercuration of this anion [9] could mercurate ruthenacarborane **1**, too. Interaction of **1** with mercury acetate or trifluoroacetate in dichloromethane at 20°C leads to symmetrical mercury compound 10,10′-Hg-{*exo-nido*-5,6,10-[$Cl(Ph_3P)_2Ru$]-5,6,10-(μ-H)$_3$-7,8-$C_2B_9H_8$}$_2$ (**2**) with 46% yield (Scheme 1).

Complex **2** was obtained as a mixture of geometrical isomers of *exo-nido*-ruthenacarborane due to ligand isomerism relative to the six-coordinated ruthenium atom, similarly to osmium complexes [10]. Isomer **2a** contains the hydrogen atom HB(10) of the open frame of the *exo-nido*-carborane ligand and chloride ligand in the *cis*-position. In the other isomer (**2b**) the hydrogen atom HB(10) and chlorine atom are in the *trans*-position. Compound **2** was obtained mostly as *cis*-isomer (**2a**) with minor amounts of *trans*-isomer (**2b**); the ratio of **2a** to **2b** is equal to 5:1.

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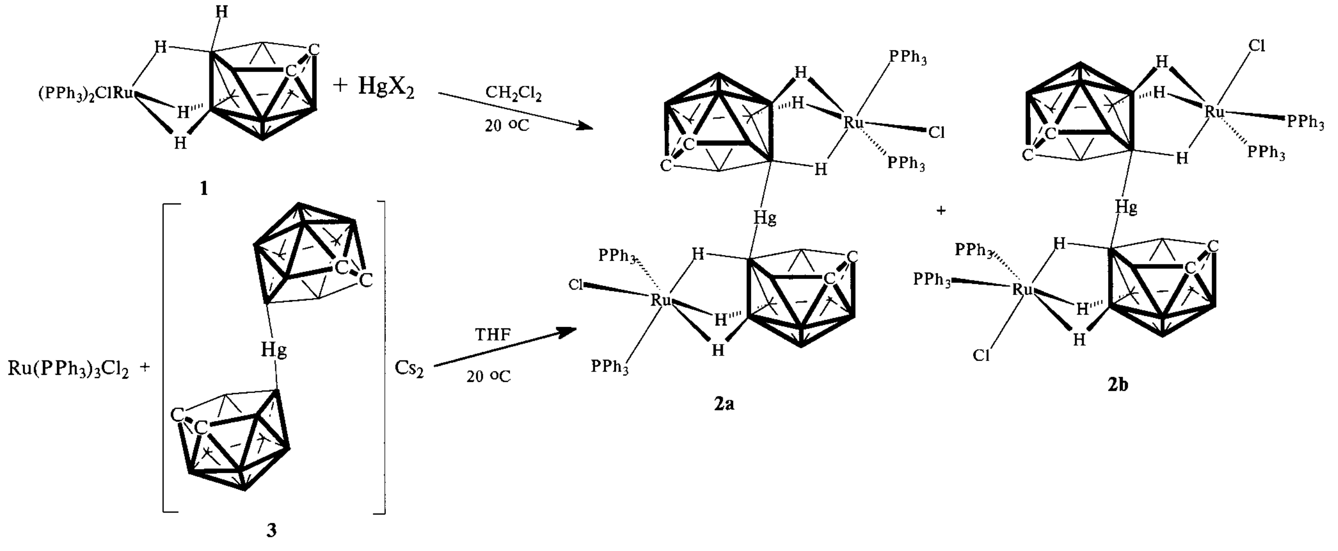
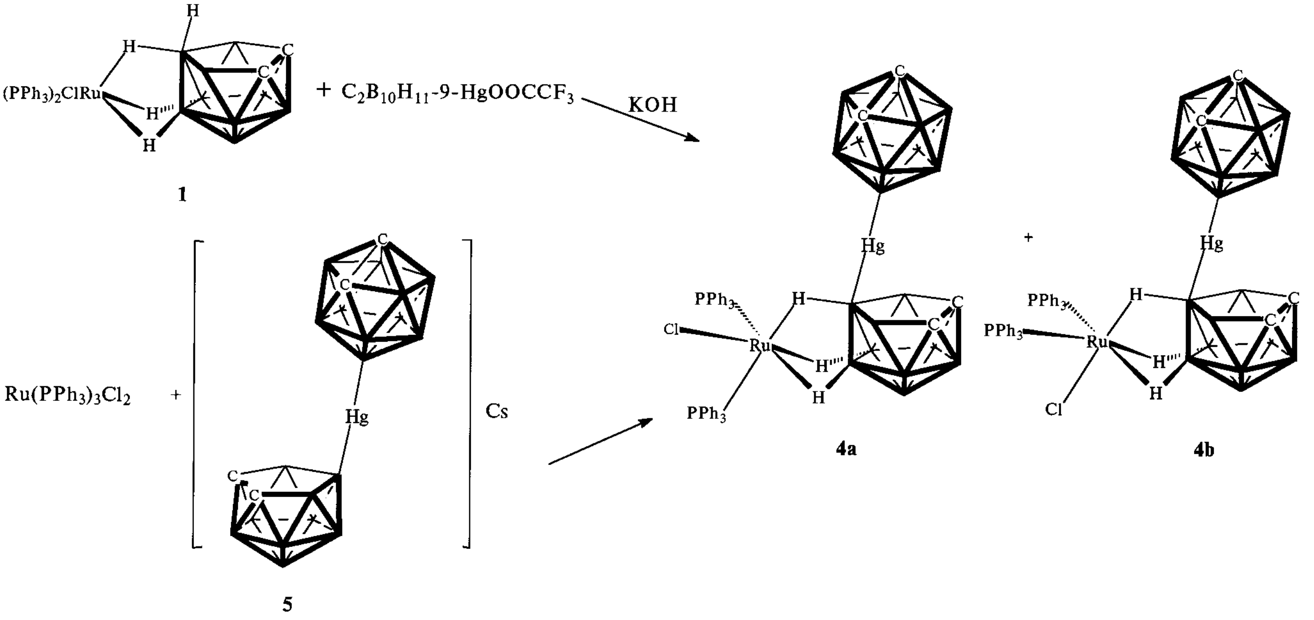


Scheme 1.



Scheme 2.

The same mixture of **2a** and **2b** was obtained under interaction of $Ru(PPh_3)_3Cl_2$ with symmetrical mercurated derivatives of 7,8-dicarba-*nido*-undecaborate anion $[10,10'\text{-}Hg\text{-}(7,8\text{-}C_2B_9H_{11})_2]Cs_2$ (**3**) [9] in a tetrahydrofuran (THF)–ether mixture (or THF) at 20°C in higher yield (89%) than in the first case. *cis*-Isomer **2a** predominates in the mixture too.

We also used $CF_3CO_2HgC_2B_{10}H_{11}$ prepared earlier [1] as the mercurating agent. Its interaction with **1** in the water–alcohol–THF mixture in the presence of alkali gives unsymmetrical mercurated ruthenacarborane 5′,6′,10′-*exo*-*nido*-$[Cl(Ph_3P)_2Ru]\text{-}5',6',10'\text{-}(\mu\text{-}H)_3\text{-}10'\text{-}(1,2\text{-}C_2B_{10}H_{11}Hg\text{-}9)\text{-}7',8'\text{-}C_2B_9H_8$ (**4**) with 40% yield. Contrary to complex **2**, compound **4** is formed predominatly as *trans*-isomer **4b**.

Compound **4** was also obtained under interaction of $Ru(PPh_3)_3Cl_2$ with $[9,10'\text{-}Hg\text{-}(1,2\text{-}C_2B_{10}H_{11})(7',8'\text{-}C_2B_9H_{11})]Cs$ (**5**) [11]. The ratio of *cis*-(**4a**) to *trans*-isomer (**4b**) equals 1:2 (Scheme 2).

Complexes **2** and **4** are crystal compounds, well soluble in dichloromethane, THF, acetone, but unsoluble in hexane and water. They are stable in the solid state, but their solutions are decomposed in air.

All isomers were isolated by column chromatography on SiO_2 and repeated crystallization from dichloromethane–hexane mixture.

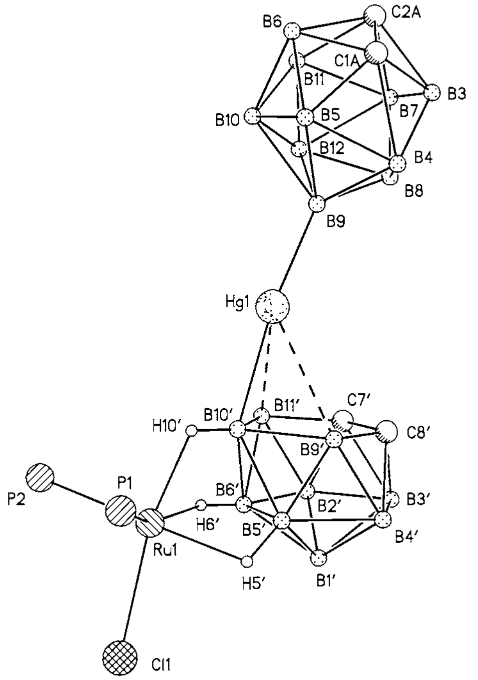


Fig. 1. General view of the molecule **4b**. The carbon atoms of the PPh_3 groups and terminal hydrogen atoms in the carborane cages are omitted. Selected interatomic distances (Å): Hg1–B9 2.00(4), Hg1–B10′ 2.27(4), Hg1···B9′ 2.63(5), Hg1···B11′ 2.76(4), Ru1–H5′ 1.89(15), Ru1–H6′ 1.92(18), Ru1–H10′ 1.77(16), Ru1···B5′ 2.33(3), Ru1···B6′ 2.45(4), Ru1···B10′ 2.33(3), Ru1–Cl1 2.393(6), Ru1–P1 2.274(7), Ru1–P2 2.314(7); selected bond angles (°): Cl1–Ru1–P1 95.2(2), Cl1–Ru1–P2 97.9(2), Cl1–Ru1–B5′ 103.4(7), P1–Ru1–P2 99.3(2), Cl1–Ru1–B6′ 104.5(8), Cl1–Ru1–B10′ 146.0(7), Ru1–H5′–B5′ 97(10), Ru1–H6′–B6′ 127(17), Ru1–H10′–B10′ 108(12), B10′–Hg1–B9 171(2).

The composition and structure of the novel complexes (**2** and **4**) were confirmed by elemental analysis, ^1H-, ^{11}B- and ^{31}P-NMR spectroscopy. There are three high-field signals in the ^1H-NMR spectra of **2a**,**b** (in CD_2Cl_2) with a 1:1:1 ratio integral intensity. Two broadened multiplet signals at -4.17 and -6.70 ppm and one broadened signal at -15.98 ppm can be assigned to protons of B–H–Ru bonds.

In the ^1H-NMR spectra of **4a**,**b** two high-field broadened multiplet signals (2:1) appear at -4.96 and -16.10 ppm. Besides, in the ^1H-NMR spectra of **2a**,**b** and **4a**,**b** there are no signals in the range of -2 to 0 ppm characteristic for *extra*-hydrogen atom at B(10) of the initial ruthenacarborane **1.** The signals of carborane ligands and phenylphospine ligands of **2a**,**b** and **4a**,**b** lie in the usual areas.

In the ^{31}P-NMR spectra there is one singlet at 48.19 ppm for **2a** and two doublets at 51.83 and 43.27 ppm for **2b.** Two doublets at 53.79, 47.02 ppm and broadened signals at 46.50 ppm appear in the ^{31}P-NMR spectra for compounds **4a**,**b**. The ^{11}B-NMR spectra of **2** and **4** contain signals at -3.5 to 0 ppm. The structure of *trans*-5′,6′,10′-*exo*-*nido*-$[Cl(Ph_3P)_2Ru]$-5′,6′,10′-$(\mu\text{-}H)_3$-10′-$(C_2B_{10}H_{11}Hg\text{-}9)$-7′,8′-$C_2B_9H_8$ (**4b**) was determined using the method of X-ray diffraction. The crystal structure for **4b** is presented in Fig. 1.

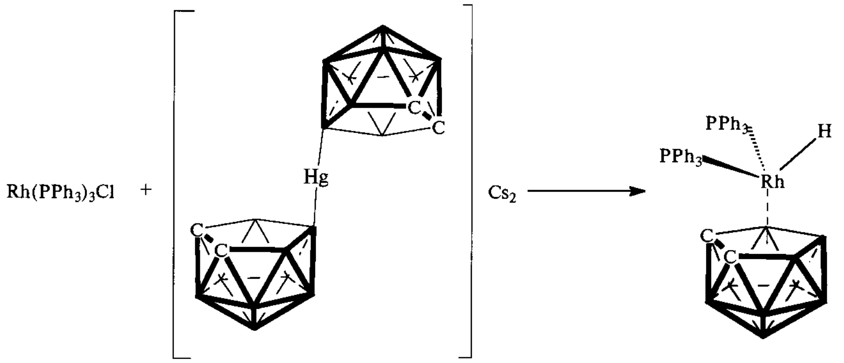
The B–Hg bond in compounds **2** and **4** is stable in HCl or $HgCl_2$ in acetone or THF at 20°C but is split under the action of Br_2 in CCl_4. Similarly to *exo*-*nido*-ruthenacarboranes substituted at carbon atoms [12], *exo*-*nido* → *closo* rearrangement of the complexes **2** and **4** was not observed under boiling in benzene.

Unfortunately the mercuration of *closo*-3,3-$(PPh_3)_2$-3-H-3-Cl-3,1,2-$RuC_2B_9H_{11}$ [13] does not proceed under the action of $Hg(OAc)_2$ in CH_2Cl_2 or acetone at 20°C, under more severe conditions (mercuration with $Hg(OAc)_2$ in AcOH) decomposition of the complex takes place.

We also studied the action of the rhodium complex $Rh(PPh_3)_3Cl$ on mercury derivatives **3** and **5**.

The interaction of $Rh(PPh_3)_3Cl$ with **3** in boiling ethanol or benzene–ethanol mixture at 20°C leads to the rupture of B–Hg bonds and formation of the known *closo*-3,3-$(PPh_3)_2$-3-H-3,1,2-$RhC_2B_9H_{11}$ (**6**) [14] in 13% yield (Scheme 3).

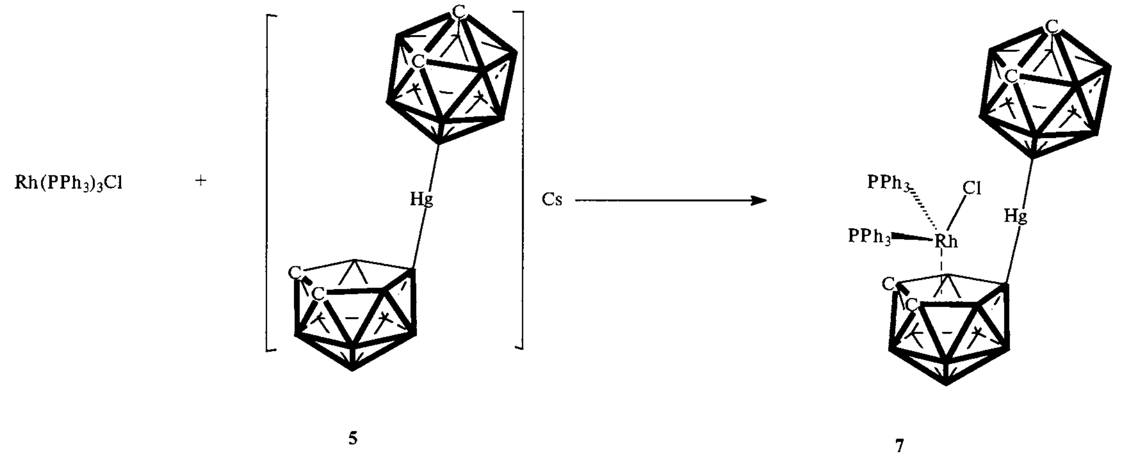
The reaction of $Rh(PPh_3)_3Cl$ with **5** in boiling ethanol, benzene–ethanol mixture or THF at 20°C gives *closo*-3,3-$(PPh_3)_2$-3-Cl-10-$(1,2\text{-}C_2B_{10}H_{11}Hg\text{-}9)$-3,1,2-Rh-$C_2B_9H_{10}]$ (**7**) (Scheme 4).



3

6

Scheme 3.



Scheme 4.

The composition and structure of complexes **6** and **7** were confirmed by elemental analysis data, ^{1}H-, ^{31}P- and ^{11}B-NMR spectroscopy.

3. Experimental

All reactions were carried out in inert atmosphere using absolute solvents prepared by standard techniques. NMR spectra were obtained on a Bruker WP 200-SY spectrometer.

3.1. Synthesis of ***2***

(i) A mixture of 0.27 g (0.34 mmol) of **1** with 0.12 g (0.34 mmol) of $Hg(OAc)_2$ in 30 ml of CH_2Cl_2 was stirred for 2 h at 20°C. After filtration solvent was removed in vacuo and the product was isolated by chromatography on an SiO_2 column with benzene as eluent to give 0.28 g (46%) of **2a**,**b**. Repeated chromatography gave 0.07 g of **2**a. ^{1}H-NMR ($CDCl_3$): δ, 8.42–6.49 (m, 60H, Ph), 5.14 (4H, $C-H_{carb}$), −4.50 (m, 1H, B–H–Ru), −6.69 (m, 1H, B–H–Ru), −16.07 (m, 1H, B–H–Ru). ^{11}B-NMR ($CDCl_3$): δ, −35.5 ($J_{B-H}=80$ Hz, 4B), −27.1 ($J_{B-H}=105$ Hz, 8B), −19.4 (6B). ^{31}P-NMR (CH_2Cl_2): δ, 51.83 (dd, $J_{P-H}=29$ Hz, $P^{1(2)}$), 43.27 (dd, $J_{P-H}=27$ Hz, $P^{2(1)}$).

(ii) A mixture of 0.73 g (0.76 mmol) $Ru(PPh_3)_3Cl_2$ and 0.28 g (0.38 mmol) of **3** was stirred in 20 ml of THF for 2 h at 20°C. Solvent was removed in vacuo and the product was isolated by chromatography on an SiO_2 column with benzene as eluent to give 1.21 g (89%) of **2a**,**b**. Anal. Calc. for $C_{76}H_{80}B_{18}Cl_2P_4HgRu_2$: C, 51.12; H, 4.52; B, 10.90; P, 6.9. Found: C, 51.42; H, 4.25; B, 10.87; P, 6.80. Repeated chromatography and crystallization from benzene–hexane mixture produced **2b**. ^{1}H-NMR ($CDCl_3$): δ, 8.28–6.66 (m, 60H, Ph), −4.15 (m, 1H, B–H–Ru), −6.55 (m, 1H, B–H–Ru), −16.16 (m, 1H, B–H–Ru). ^{31}P-NMR ($CDCl_3$): δ, 48.19 (s, $P^{1,2}$).

3.2. Synthesis of ***4***

(i) A solution of 0.27 g (0.34 mmol) of **1** and 0.19 g (0.41 mmol) of 9-carboranylmercury trifluoroacetate in a mixture of 10 ml of THF and 2 ml of water was added to a solution of 0.02 g (0.45 mmol) of KOH in a mixture of 10 ml of water and 2 ml of EtOH. The reaction mixture was stirred for 4 h at 20°C. After removing the solvent the compound was isolated by chromatography on an SiO_2 column with 1:1 CH_2Cl_2–hexane as eluent to give 0.27 g (70%) of **4a**,**b**.

(ii) A mixture of 1.9 g (1.98 mmol) $Ru(PPh_3)_3Cl_2$ with 1.73 g (22.84 mmol) of **5** in 65 ml of THF was stirred for 2 h at 20°C. Solvent was removed in vacuo and **4a**,**b** was isolated by chromatography on an SiO_2 column with 1:1 CH_2Cl_2–hexane as eluent to give 0.56 g (31%). Anal. Calc. for $C_{40}H_{52}B_{19}ClP_2HgRu$: C 42.32; H 4.58; B 18.09; P 5.46; Ru 8.90. Found: C, 42.43; H 4.41; B 18.05; P 6.06; Ru 9.66. ^{1}H-NMR (acetone-d_6–THF-d_8): δ, 11.28 (s, 1H), 7.64–7.51 (m, 30H, Ph), 5.20 (2H, $C-H_{carb}$), 4.67 (2H, $C-H_{carb}$), −4.96 (m, 2H, B–H–Ru), −16.10 (m, 1H, B–H–Ru). ^{31}P-NMR (THF): δ, 53.79 (dd, $J_{P-H}=32$ Hz, $P^{2(1)}$), 47.02 (dd, $J_{P-H}=29$ Hz , $P^{2(1)}$), 46.50 (s, $P^{1,2}$). ^{11}B-NMR (acetone-d_6–THF-d_8): δ, 13.6 (1B, B^{9}_{carb}), −1.4 ($J_{B-H}=153$ Hz, 2B), −7.96 ($J_{B-H}=158$ Hz, 3B), −12.4 ($J_{B-H}=152$ Hz, 7B), −21.5 ($J_{B-H}=100$ Hz 3B), −23.5 ($J_{B-H}=117$ Hz, 1B), −26.8 ($J_{B-H}=72$ Hz, 1B), −30.6–(−33.31) (m, 1B). **4a** and **4b** were separated after recrystallization from CH_2Cl_2–hexane. ^{31}P-NMR of **4a** (acetone-d_6–THF-d_8): δ, 53.81 (dd, $J_{P-H}=28$ Hz, $P^{1(2)}$), 47.23 (dd, $J_{P-H}=27$ Hz, $P^{2(1)}$). ^{31}P-NMR of **4b** (THF): δ, 46.09 ($P^{1,2}$, s). ^{11}B-NMR (**4b**) (THF): δ, 13.8 ($J_{B-Hg}=2191$ Hz, B-9_{closo}),

-1.2 ($J_{B-H} = 154$ Hz, 2B), -7.8 ($J_{B-H} = 155$ Hz, 3B), -12.1 ($J_{B-H} = 152$Hz, 6B), -21.4 ($J_{B-H} = 99$ Hz, 3B), -23.5 ($J_{B-H} = 119$ Hz, 1B), -24.1 (1B), -26.7 ($J_{B-H} = 84$ Hz, 1B), -31.7–(-34.7) (m, 1B). (**4b**) m.p. 174–176°C.

3.3. Reaction of $Rh(PPh_3)_3Cl$ with **3**

A total of 1.0 g (1.08 mmol) of $Ru(PPh_3)_3Cl_2$ and 0.44 g (0.60 mmol) of **3** was stirred for 3 h at 20°C in a mixture of 60 ml of benzene and 4 ml of EtOH or refluxed in EtOH. The solvent was removed in vacuo and the compound was isolated by chromatography on an SiO_2 column with benzene as eluent to give 0.11 g (13%) of **6**.

3.4. Synthesis of **7**

A total of 0.50 g (0.54 mmol) of $Rh(PPh_3)_3Cl$ and 0.50 g (0.82 mmol) of **5** in a mixture of 50 ml of benzene and 10 ml of EtOH were stirred for 4 h at 20°C. The solvent was removed in vacuo and the product was isolated by chromatography on an SiO_2 column with benzene as eluent to give 0.60 g (97%) of **7**. Interaction of 0.50 g of $Rh(PPh_3)_3Cl$ with 0.50 g of **5** in boiling EtOH or 0.30 g of $Rh(PPh_3)_3Cl$ with 0.3 g of **5** in THF at room temperature results in 0.47 g of **7** (yield 76%) or 0.33 g of **7** (89%), respectively. Anal. Calc. for $C_{40}H_{52}B_{19}ClP_2HgRh$: C 42.21; H 4.52; B 18.05; P 5.44; Rh 9.04; Hg 17.65; Cl 3.11. Found: C 42.21; H 4.37; B 18.24; P 5.12; Rh 9.0; Hg 19.3; Cl 3.5. ^{1}H-NMR (acetone-d_6): δ, 7.2–7.6 (m, 30H), 5.61 (1H, C–H_{carb}), 4.67 (1H, C–H_{carb}), 4.31 (1H, C–H_{carb}), 3.77 (1H, C–H_{carb}). ^{31}P-NMR (acetone-d_6): δ, 37.26 (d, $J_{P-Rh} = 143$ Hz). ^{11}B-NMR (acetone-d_6): δ, 14.5 ($J_{B-Hg} = 1996$ Hz, B-9$_{closo}$), -1.0 ($J_{B-H} = 139$ Hz, 2B), -7.6 ($J_{B-H} = 127$ Hz, 3B), -11.9 ($J_{B-H} = 140$ Hz, 13B).

3.5. Crystallographic data for **4b**

$C_{40}H_{56}B_{19}ClP_2HgRu \cdot 0.5CH_2Cl_2 \cdot 0.5Me_2CO$, $M = 1208.76$, monoclinic crystals, space group Cc, $a = 26.710(7)$, $b = 12.585(3)$, $c = 21.137(6)$ Å, $\beta = 128.14(2)°$, $V = 5588(3)$ Å^3, $Z = 4$, $d_{calc} = 1.437$ g cm^{-3}, μ(Mo–K$_\alpha$) = 31.98 cm^{-1}, $F(000) = 2380$. A species of extremely small red–orange monocrystal with $0.10 \times 0.15 \times 0.15$ mm dimensions was obtained by crystallization from the hexane–CH_2Cl_2 mixture. Intensities of 4208 reflections were measured on Siemens P3/PC diffractometer at 293 K (λ(Mo–K$_\alpha$) radiation, $\theta/2\theta$ scan technique, $2\theta < 48°$) and 4039 independent reflections were used in further calculations and refinement. The absorption correction was introduced using the experimental curves of azimuthal scan (14 reflections, $0 < \theta < 360°$ with an interval of 10°, $T_{min} = 0.229$ and $T_{max} = 0.950$). The structure was solved by a direct method and refined by full-matrix least-squares against F^2 in anisotropic–isotropic approximation. The analysis of the Fourier synthesis has revealed that the *closo*-carborane cage σ-bonded to the Hg(1) atom and solvate molecules of acetone and dichloromethane are disordered. Low resolution, as well as high correlation and lack of sufficient number of observed reflections, does not completely reveal a disorder of the carborane cage over two orientations. The C1A, C2A, B3–B12 atoms correspond to one cage orientation (see Fig. 1), where the C2A atomic position was assigned on the basis of the known structure of the initial compound **5** in which the mercury atom is bonded to B9 [11]. The position of the C1A atom (from five possible ones) was assigned rather arbitrarily. In the second cage orientation, only ten atoms were located and assigned as boron atoms. This disordered part of the molecule was refined isotropically with equal occupancy factors $g = 0.5$. The positions of hydrogen atoms for all Ph cycles were calculated from the geometrical point of view and were included in the final refinement using a rigid motion model. The hydrogen atoms in the non-disordered *nido*-carborane ligand were located from the difference Fourier syntheses and refined in isotropical approximation. The refinement is converged to $wR_2 = 0.1921$ and GoF = 0.819 for all 3987 independent reflections ($R_1 = 0.0619$ is calculated against F for the 2779 independent reflections with $I > 2\sigma(I)$]. The number of the refined parameters is 625. All the calculations were performed using SHELXTL PLUS 5.0 on an IBM PC/AT.

4. Supplementary material

Crystallographic data (atomic coordinates, bond lengths, bond angles and thermal parameters) for the structure reported in this paper have been deposited with the Cambridge Crystallographic Data Centre, CCDC no. 127565. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (Fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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