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Reaction of Chiral Pyrrolylphosphine with Polynuclear Carbonyl Complexes of Osmium and Rhodium

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Abstract—Reactions of labile $Os_3(CO)_{10}(NCMe)_2$ and $Rh_6(CO)_{15}(NCMe)$ clusters and dinuclear $Rh_2(CO)_4Cl_2$ complex with chiral (*S*)-[2-(4-isopropyl-4,5-dihydro-1,3-oxazol-2-yl)phenyl]di(1*H*-pyrrol-1-yl)phosphine were studied. The reaction of $Os_3(CO)_{10}(NCMe)_2$ with the chiral phosphine was diastereoselective, and it afforded only one diastereoisomer with *ee* 100%. All isolated compounds were completely characterized by NMR and mass spectra and X-ray diffraction data.

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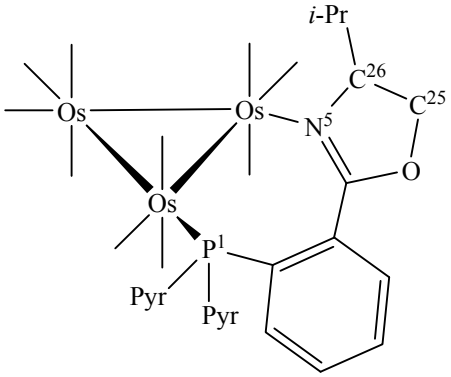
Search for new catalysts for asymmetric synthesis and studies on their properties constitute one of the most promising fields in the development of organometallic chemistry. Increased interest in efficient asymmetric is determined by the fact that more than 70% of drugs now in use and almost all perfumes are chiral compounds, many of which can be obtained as enantiomerically pure substances via catalytic reactions. Most known catalysts of asymmetric synthesis are polynuclear coordination compounds, and asymmetric induction originates exclusively from chirality of ligands present in the coordination sphere. However, in the recent years strong attention of organic chemists and specialists in the field of catalysis is attracted by polynuclear transition metal complexes (clusters) whose chirality is determined not only by the ligand nature but also by asymmetric structure of the metal framework. An effective method for the design of chiral clusters is based on introduction of heterobidentate ligands, in particular functionalized phosphines, into the coordination sphere [1–3] to obtain chelate or bridged structures. A new and promising class of such ligands includes chiral pyrrolylphosphines [4–13]; first examples of their coordination at polynuclear complexes were recently reported [12, 14]. Therefore, studies on coordination modes of pyrrolylphosphines in polynuclear clusters, properties and reactivity of the coordination compounds thus formed, and methods for isolation of their optically pure forms seem to be urgent and practically important.

Reaction of (*S*)-[2-(4-isopropyl-4,5-dihydro-1,3-oxazol-2-yl)phenyl]di(1*H*-pyrrol-1-yl)phosphine (I) with $Os_3(CO)_{10}(MeCN)_2$. Labile $Os_3(CO)_{10}(MeCN)_2$ cluster reacted with ligand **I** to give $Os_3(CO)_{10}(\mu_2,\kappa^2$-PyrPhOx) (**II**) and $Os_3(CO)_{10}(1,1;\kappa^2$-PyrPhOx) (**III**). Cluster **II** was formed in 45% yield when the reaction was performed in methylene chloride, and cluster **III** was the major product in hexane (yield 24%).

We failed to obtain crystals of cluster **II** suitable for X-ray analysis, for this compound turned out to be unstable: it underwent slow transformation into thermodynamically more stable chelate **III**. The structure of **II** was determined on the basis of its IR and NMR spectra. Comparison of the IR spectral pattern of cluster **II** in the region typical of stretching vibrations of carbonyl ligands with published led us to presume bridging mode of coordination of the ligand at the metal atoms. The frequencies and relative intensities of the carbonyl absorption bands of compound **II** are very consistent with those reported previously for triangular osmium clusters containing heterobidentate phosphines as bridging ligands [15–17]. The ^{31}P NMR spectrum of **II** contained one signal with satellites

corresponding to coupling with ^{187}Os ($^{1}J_{P,Os}$ = 297 Hz); this means that the ligand is coordinated to the metal ion through the phosphorus atom. Signals in the ^{1}H NMR spectrum of **II** were assigned on the basis of the ^{1}H–^{1}H COSY data. The COSY spectrum displayed cross peaks involving different protons in the ligand. In such a way we identified signals from four protons in the benzene ring [δ, ppm: 7.60 m (2H), 7.31 m (1H), 7.09 d.d.d (1H)], eight protons in the pyrrole rings [δ, ppm: 7.09 m (2H), 6.80 m (2H), 6.42 m (4H)], three protons in the dihydrooxazoole ring [δ, ppm: 4.34 d.d (1H), 3.94 d.t (1H), 3.63 d.d (1H)], and seven protons in the isopropyl group [δ, ppm: 2.65 m (1H), 1.00 d (3H), 0.88 d (3H)].

Considerable displacement of signals from protons in the dihydrooxazole ring in the spectrum of complex **II** relative to those in the spectrum of the free ligand [14] unambiguously indicates bidentate coordination of the ligand through the phosphorus atom an nitrogen atom in the dihydrooxazole fragment with formation of seven-membered metal-containing ring Os–P–C–C–C–N–Os.

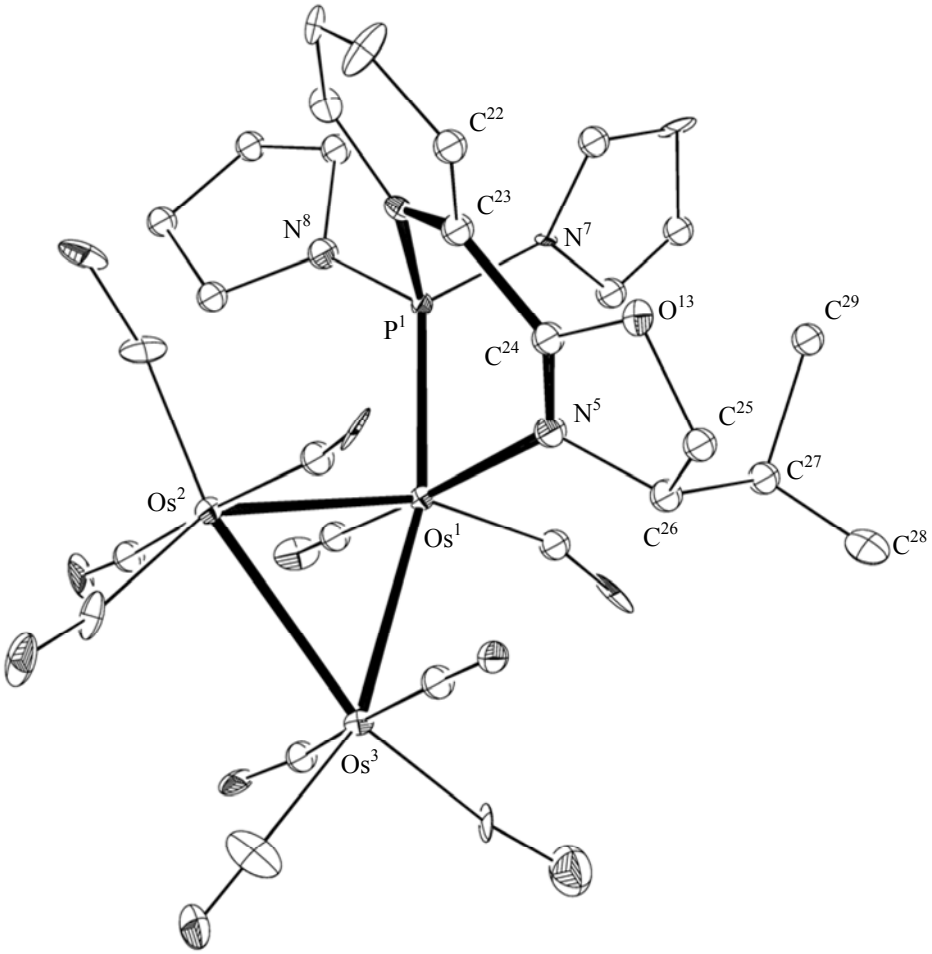


II

The formation of such strained dimetallocycle is very untypical. Only one trinuclear osmium cluster containing an analogous structural fragment has been reported [18]. The formation of seven-membered dimetallocycle in the complex $Os_3(CO)_8(\mu\text{-}NO_2)(\mu\text{-}H)(\mu\text{-}PN)$ {where PN is 1-[(*S*)-2-(diphenylphosphino)ferrocenyl]ethanamine} is likely to be determined by rigid structure of the ferrocenyl spacer connecting the phosphorus and nitrogen coordination centers. The formation of seven-membered metal-containing heteroring in cluster **II** is not energetically favorable, as compared to more stable five- or six-membered rings; therefore, moderate heating of complex **II** or column chromatography using a nonpolar solvent as eluent promotes its irreversible transformation into compound **III** having a six-membered chelate ring. Thus cluster **II** may be regarded as kinetically-controlled product of coordination of ligand **I** at the Os_3 entity, while compound **III** is thermodynamically controlled product. Compound **III** can be obtained in a moderate yield by carrying out the above reaction in hexane.

The quality of single crystals of cluster **III** grown from octane was insufficient to obtain X-ray diffraction data with a high accuracy, and the diffraction pattern was poorly informative; nevertheless, we succeeded in localizing heavy atoms and proposed a structure in agreement with the spectral data. Three osmium atoms in cluster **III** form a triangular metal framework which is surrounded by ten carbonyl ligands. The heteroligand is coordinated at two osmium atoms in chelating mode through the phosphorus atom and nitrogen atom in the dihydrooxazole ring. The phosphorus and nitrogen coordination centers in the ligand occupy, respectively, equatorial and axial positions in the metal triangle (see figure). Analogous orientation of heterobidentate (P, N) phosphine ligand was found in $Os_3(CO)_{10}$\{*N*-[2-(diphenylphosphino)benzylidene]-2-(pyridin-2-yl)ethanamine\} [19] which was the only example of trinuclear osmium clusters with P,N-chelating ligand.

The IR and NMR spectra of compound **III** confirm the structure determined for its crystal. The frequencies and relative intensities of IR bands in the region of carbonyl ligand stretching vibrations are very consistent with those reported previously for triangular osmium clusters containing heterobidentate chelating phosphine ligands [16, 19]. Participation of the phosphorus atom in **I** in the coordination to osmium in cluster **III** follows from the downfield shift of the phosphorus signal in the ^{31}P NMR spectrum to δ_P 80.5 ppm and from the presence of satellites due to coupling with ^{187}Os. In the ^{1}H NMR spectrum of **III** at room temperature we observed broadening of signals from protons in the pyrrole substituents (δ 7.5–5.0 ppm) due to restricted rotation of the pyrrole rings about the P−N bond, the latter having a partial double character [20]. Analogous slow (on the NMR time scale) rotation of pyrrole groups in phosphine ligands was observed previously in coordinated trispyrrolylphosphine ligand [21]. Lowering the temperature to −50°C completely inhibits this dynamic process, and the ^{1}H NMR spectrum recorded at 223 K contained well resolved signals from all nonequivalent protons in the pyrrole rings (see Experimental). Change of the chemical shifts and mutual arrangement and shape of signals of protons in the dihydrooxazole fragment in **III**, as compared to free ligand **I** [multiplet signals at δ



ORTEP drawing of cluster **III**.

4.28 (1H) and 3.94 ppm (2H)] indicates coordination of the heteroligand through the nitrogen atom in the dihydrooxazole ring. Signals from protons in the phenylene spacer group and dihydrooxazole ring, as well as in the isopropyl group, showed no temperature dependence; this means that the part of the ligand involved in the formation of chelate ring is firmly fixed in space.

Coordination of a heterobidentate ligand to a triangular cluster gives rise to planar chirality [2] if one donor center of the ligand occupies axial position while the other occupies equatorial position in the cluster triangle. If the heterobidentate ligand is also chiral, a couple of diastereoisomers with different conformations of the planar chiral fragment may be formed M_3(1,1-*P*,*N*). However, coordination of chiral compound **I** gives only one of the two possible diastereoisomers, as follows from the presence of only one signal in the ^{31}P NMR spectrum and one set of signals of the coordinated ligand in the ^{1}H NMR spectrum of cluster **III**. Therefore, the structure of **III** in crystal determined by X-ray analysis (see figure) may be regarded as a thermodynamically more favorable isomer with S_LS_{pl} configuration of asymmetric elements (the subscript "L" stands for the ligand, and the subscript "pl" refers to the chiral Os_3 plane. In fact, the isopropyl group in the dihydrooxazole ring in molecule **III** is oriented toward the opposite side of the Os_3 framework, i.e., its interaction with the metal and carbonyl ligand is minimal. Insofar as no other isomers were formed in the examined reactions (in a detectable amount), it may be assured that cluster **III** is formed with 100% diastereo-selectivity. These data correlate well with those obtained previously while studying replacement of carbonyl ligands by heterobidentate chiral phosphines in tetranuclear carbonyl hydride clusters [12, 14, 22, 23].

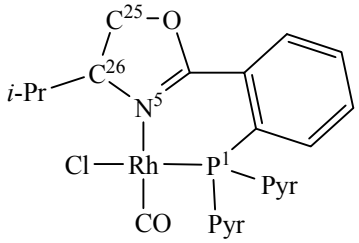
In all cases, only one of the possible diastereoisomers was formed, indicating unusualy high asymmetric induction in reactions of that sort.

It should be noted that dihydrooxazole–phosphine ligands in mononuclear complexes are often hemilabile [8], which may considerably affect their catalytic activity. On the other hand, the phosphine ligand in compound **III** displayed no hemilability in the temperature range from 25 to −50°C. Saturation of a solution of **III** with carbon(II) oxide was not accompanied by decoordination of any functionality of the ligand.

Reaction of phosphine I with the $Rh_6(CO)_{15}$·(MeCN) cluster and $Rh_2(CO)_4Cl_2$ complex. The labile $Rh_6(CO)_{15}$(MeCN) cluster reacted with compound **I** in methylene chloride with formation of mononuclear complex Rh(CO)Cl(1,1-*S*-PyrPhOx) (**IV**) as the major product (yield 23%). The reaction involves decomposition of the initial hexanuclear cluster and is accompanied by formation of a considerable amount of decomposition products (as an insoluble material) and change of the metal oxidation state from Rh(0) to Rh(I). Presumably, decomposition of the cluster is promoted by strong steric strains appearing as a result of coordination of ligand **I** at the hexanuclear metal framework. Furthermore, the coordinating center is oxidized as a result of oxidative addition of solvent molecule and coordination of chloride ion; the latter is a ligand in the coordination sphere of the mononuclear product. This reaction does not provide a convenient procedure for the synthesis of complex **IV**. The reaction of phosphine **I** with dinuclear $Rh_2(CO)_4Cl_2$ complex where the central metal ion has an oxidation number of +1 gives compound **IV** in almost quantitative yield. We failed to obtain single crystals of **IV** suitable for X-ray analysis, and its structure was determined on the basis of the IR and NMR spectra.

Coordination of the heteroligand through the phosphorus atom is confirmed by the presence in the ^{31}P NMR spectrum of **IV** of a signal at δ_P 97.9 ppm (it was displaced downfield relative to the corresponding signal of the free ligand) with a coupling constant $^1J_{P,Rh}$ of 214 Hz, which is typical of such complexes [24–27]. The ^1H NMR data for compound **IV** indicated conservation of the ligand structure upon coordination to rhodium. Coordination of the dihydrooxazole ring to the metal through the nitrogen atom is confirmed by an appreciable downfield shift of signal from the $C^{26}H$ proton nearest to the coordination center (δ 5.42 ppm against 4.28 ppm for the free ligand). The $C^{25}H_2$ signal shifts less significantly (from δ 3.94 to 4.42 ppm). The positions and multiplicities of signals from protons in he benzene and pyrrole rings and isopropyl group almost do not differ from those observed for compounds **II** and **III**.

The IR spectrum of **IV** contained only one band in the carbonyl region (at 2020 cm^{-1}), which is consistent with the formation of square–planar complex [Rh(CO)Cl(1,1-P,N)] where the bidentate phosphine–dihydrooxazole ligand is coordinated to the central metal ion through the phosphorus and nitrogen atoms [24–27]. The most probable configuration of complex **IV** is that where the carbonyl ligand is oriented *cis* with respect to the coordinated phosphorus atom; just this arrangement is typical of all so far known complexes [Rh(CO)Cl(1,1-P,N)] possessing a coordinated dihydrooxazole fragment [24–27]. Thus the spectral data indicated that compound **IV** is a square–planar mononuclear rhodium complex.



IV

The phosphine ligand coordinated to Rh(I) displays no hemilability toward CO. Saturation of a solution of complex **IV**with carbon(II) oxide does not lead to any variation in the ^1H and ^{31}P NMR spectra.

EXPERIMENTAL

The IR spectra were recorded on a Perkin–Elmer FT-IR Spectrum Bx II spectrometer. The ^1H, ^1H–^1H COSY, and ^{31}P–{^1H} NMR spectra were measured on a Bruker DPX 300 instrument from solutions in $CDCl_3$; the chemical shifts were determined relative to the residual solvent signal (^1H) or 85% H_3PO_4 (^{31}P). Thin-layer chromatography was performed on aluminum plates coated with a 0.5-mm layer of silica gel 60 (Merck). Silica gel with a grain size of 5–40 μm was used for preparative column chromatography.

Chloroform, acetonitrile, methanol, saturated hydrocarbons (hexane, heptane, and octane), and petroleum ether were commercial products (from *Vekton*) and were used without additional purification. Me-

thylene chloride was treated with a 10% aqueous solution of K_2CO_3, dried over $CaCl_2$, and distilled over P_2O_5. The initial clusters $Os_3(CO)_{10}(MeCN)_2$ [28], $Rh_6(CO)_{15}(MeCN)$ [29], and $Rh_2(CO)_4Cl_2$ [30] and (*S*)-[2-(4-isopropyl-4,5-dihydro-1,3-oxazol-2-yl)phenyl]-di-(1*H*-pyrrol-1-yl)phosphine (**I**) [14] were synthesized according to known methods.

$Os_3(CO)_{10}(\mu^2,\kappa^2$-*S*-PyrPhOx) (II). A solution of 78 mg of $Os_3(CO)_{10}(MeCN)_2$ in 25 ml of methylene chloride was concentrated in a stream of argon to a volume of ~15–20 ml, and 28 mg of anhydrous compound **I** was added under stirring. The mixture was kept overnight under argon, and it changed from yellow to orange. The solvent was distilled off, the oily residue was dissolved in 4 ml of methylene chloride–hexane (1 : 4, by volume), the solution was applied to a chromatographic column, and the product was isolated by slow elution with methylene chloride–hexane (1 : 1, by volume), a broad orange zone being washed off. Yield 43 mg (45%). IR spectrum (hexane), ν(C=O), cm^{-1}: 2096 m, 2068 w, 2028 m, 2012 v.s, 1956 w, 1948 w. ^1H NMR spectrum (293 K), δ, ppm (*J*, Hz): 7.60 m (2H, H_{arom}), 7.31 m (1H, H_{arom}), 7.09 d.d.d (1H, H_{arom}, J_{HH} = 12.5, 12.4, 1.4), 7.09 m (2H, pyrrole), 6.80 m (2H, pyrrole), 6.42 m (4H, pyrrole), 4.34 d.d (1H, 5-H, J_{HH} = 9.0, 4.0), 3.94 d. t (1H, 4-H, J_{HH} = 9.0, 4.0), 3.63 d.d (1H, 5-H, J_{HH} = 9.0), 2.65 m (1H, C*H*Me_2), 1.00 d and 0.88 d (3H each, CH*Me*$_2$, J_{HH} = 6.6). $^{31}P-\{^1H\}$ NMR spectrum (293 K): δ_P 66.1 ppm, m ($^1J_{P,Os}$ = 297 Hz). Found, %: C 35.74; H 3.40; N 3.11. $C_{34}H_{31}N_3O_{11}\cdot Os_3P_1\cdot C_6H_{14}$. Calculated, %: C 35.71; H 3.37; N 3.12.

$Os_3(CO)_{10}(1,1;\kappa^2$-PyrPhOx) (III). The complex $Os_3(CO)_{10}(MeCN)_2$, 95 mg, was dispersed in 50 ml of hexane preliminarily degassed by passing argon over a period of 20 min. Compound **I**, 38 mg, was added under argon to the suspension, and the mixture was stirred for 5 days at room temperature. The progress of the reaction was monitored by TLC, following the disappearance of the initial cluster. When the reaction was complete, the solvent was distilled off, the oily residue was treated with octane (2×4 ml), and the extract was applied to a chromatographic column which was slowly eluted with methylene chloride–hexane (1 : 4, by volume); a broad orange zone was washed off. Yield 30 mg (24%). IR spectrum (hexane), ν(CO), cm^{-1}: 2092 m, 2044 s, 2016 s, 1996 v.s, 1988 s, 1976 w, 1968 w, 1960 w, 1928 w. ^1H NMR spectrum, δ, ppm (*J*, Hz): at 293 K: 8.05 m (1H, H_{arom}), 7.57 m (2H, H_{arom}), 6.22 m (1H, H_{arom}), 6.42 m (4H, pyrrole), 7.50–5.00 br (4H, pyrrole), 4.52 d (1H, 5-H, J_{HH} = 7.0), 4.42 d.d (1H, 5-H, J_{HH} = 8.4, 7.0), 3.98 d (1H, 4-H, J_{HH} = 7.0), 2.43 m (1H, C*H*Me_2), 0.90–0.85 d (3H, CH_3, CH*Me*$_2$, J_{HH} = 7.3), –0.02 d (3H, CH*Me*$_2$, J_{HH} 7.3); at 223 K: 8.02 m (1H, H_{arom}), 7.65 m (1H, pyrrole), 7.59 m (2H, H_{arom}), 7.18 m (1H, pyrrole), 6.53 s (1H, pyrrole), 6.50 s (1H, pyrrole), 6.39 s (1H, pyrrole), 6.27 s (1H, pyrrole), 6.10 d.d (1H, H_{arom}, J_{HH} = 9, 1.0), 5.97 s (1H, pyrrole), 5.46 s (1H, pyrrole), 4.52 d (1H, 5-H, J_{HH} = 8.4), 4.38 d.d (1H, 5-H, J_{HH} = 8.4, 7.0), 3.89 d (1H, 4-H, J_{HH} = 7.0), 2.34 m (1H, C*H*Me_2), 0.85 d (3H, CH*Me*$_2$, J_{HH} = 6.2), –0.08 d (3H, CH*Me*$_2$, J_{HH} = 6.2). $^{31}P-\{^1H\}$ NMR spectrum (293 K): δ_P 80.5 ppm, m ($^1J_{P,Os}$ = 275 Hz). Found, %: C 32.44; H 2.55; N 3.36. $C_{34}H_{31}N_3O_{11}Os_3P_1$. Calculated, %: C 32.43; H 2.48; N 3.34. Single crystals of **III** for X-ray analysis were obtained by slow evaporation of a solution of **III** in octane at room temperature.

Reaction of compound I with $Rh_6(CO)_{15}(MeCN)$. Compound **I**, 20 mg, was added under stirring to a solution of 50 mg of $Rh_6(CO)_{15}(MeCN)$ in 20 ml of methylene chloride. The mixture turned turbid, and a fine black solid separated. Thin-layer chromatography monitoring showed consumption of compound **I** and accumulation of complex **IV** (a yellow spot). Subsequent addition of compound **I** increased the amount of **IV**. The mixture was filtered from the black precipitate, the filtrate was evaporated to dryness, and the yellow–brown residue was dissolved in 2 ml of a methylene chloride–hexane–acetonitrile mixture (1:2:0.1, by volume) and applied to a chromatographic column which was eluted with methylene chloride–hexane–acetonitrile (1 : 2 : 0.1, by volume) to isolate (in order of elution) initial $Rh_6(CO)_{15}(MeCN)$ cluster (10 mg, brown zone) and complex **IV** (14 mg, narrow yellow band).

Reaction of compound I with $Rh_2(CO)_4Cl_2$. Compound **I**, 55 mg, was added under stirring to a solution of 31 mg of $Rh_2(CO)_4Cl_2$ in 10 ml of methylene chloride. The originally orange solution quickly turned yellow. The mixture was stirred for 10 min and evaporated, and the residue was dissolved in 4 ml of methylene chloride–hexane (1 : 1, by volume) and applied to a chromatographic column which was slowly eluted with methylene chloride–hexane–acetonitrile (1 : 2 : 0.1, by volume). Complex **IV** (narrow yellow band) was additionally recrystallized from the eluent with addition of diethyl ether. Yield 70 mg (86%). IR spectrum (CH_2Cl_2): ν(CO) 2020 cm^{-1}, v.s. ^1H NMR spectrum (293 K), δ, ppm (*J*, Hz): 8.05 d.d (1H, H_{arom}, J_{HH} = 6.7, 5.0), 7.68 t (1H, H_{arom}, J_{HH} =

7.6), 7.60 t (1H, H_{arom}, J_{HH} = 7.5), 6.49 d.d (1H, H_{arom}, J_{HH} = 11.2, 7.8), 7.02 m (2H, pyrrole), 6.58 m (2H, pyrrole), 6.38 m (4H, pyrrole), 5.42 m (1H, 4-H), 4.42 m (2H, 5-H), 2.40 m (1H, C*H*Me_2), 0.84 d (3H, CH*Me*$_2$, J_{HH} = 7.0), –0.29 d (3H, CH*Me*$_2$, J_{HH} = 6.8). $^{31}P–\{^1H\}$ NMR spectrum (293 K): δ_P 97.9 ppm, d ($^1J_{P,Rh}$ = 214 Hz). Found, %: C 49.40; H 4.52; N 10.10. $C_{21}H_{22}ClN_3O_2PRh \cdot CH_3CN$. Calculated, %: 49.44; H 4.51; N 10.03.

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