Journal of Organometallic Chemistry, 397 (1990) 245–253
Elsevier Sequoia S.A., Lausanne
JOM 21007

Complexation of rhodium(I) with 5-hydro-3,8-*R*,*R*-1,6-dioxa-4,9-diaza-5-phosphaspiro[4.4]nonane

E.E. Nifantyev *, K.N. Gavrilov, G.I. Timofeeva, A.T. Teleshev, S.N. Krasnokutsky, E.Y. Zhorov, V.A. Pavlov and E.I. Klabunovsky

Division of Organic Chemistry, V.I. Lenin Moscow State Pedagogical Institute, M. Pirogovaskaya 1, Moscow, 119882 (U.S.S.R.)

(Received April 20th, 1990)

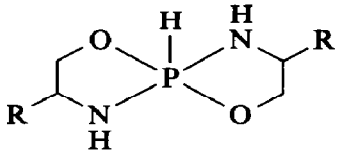
Abstract

The interaction of hydrospirophosphoranes with square-planar d^8 rhodium(I) complexes was studied. The structures of the complexes formed are discussed on the basis of IR, ^{31}P, ^{15}N and ^{13}C NMR and X-ray electron spectral data.

Introduction

Rhodium(I) complexes with "head-footed" *P*,*N*-bidentate ligands combining the properties of hard and soft bases have found wide applications in both organometallic chemistry and homogeneous catalysis [1–5]. Exchange reactions of a metal complex with derivatives of phosphorus(III) with remote secondary and tertiary amine groups are usually employed for the preparation of such compounds. For example, the interaction of $[Rh(CO)_2Cl]_2$ with $Ph_2PCH_2NEt_2$ leads to the dinuclear rhodium complex $[(CO)Rh(\mu\text{-}CO)(\mu\text{-}Cl)(\mu\text{-}Ph_2PCH_2NEt_2)_2RhCl]$ [6]. Another widely used procedure for the preparation of rhodium(I) species is based on employing hydrophosphoranes. It is assumed that the ability of the latter to coordinate metals is associated with their tautomeric transition into the phosphite forms, which are in fact the true ligands [7,8].

In this work we propose a novel procedure for the preparation of rhodium complexes with *P*,*N*-bidentate amidophosphitoamine ligands based on reactions of metal species with 5-hydro-3,8-*R*,*R*-1,6-dioxa-4,9-diaza-5-phosphaspiro[4.4]nonanes (hydrospirophosphoranes (HSP)):



(R = H (Ia), Et (Ib))

0022-328X/90/$03.50

These compounds are available [9] and have been thoroughly investigated [10]. Much less is known about their coordination ability. We anticipated that complexation of HSP with rhodium(I) would afford amidophosphitoamine complexes with a remote primary amino group. Such complexes have not yet been reported.

Results and discussion

The interaction of HSP with Ia and Ib with $[Rh(CO)_2Cl]_2$ can be described as follows:

$$n(\text{Ia, b}) + \frac{n}{2}[Rh(CO)_2Cl]_2 \xrightarrow{-n\,CO} [(CO)RhCl(H_2NCHRCH_2OP\langle O, NH, R\rangle)]_n$$

(IIa, b)

The reaction is performed in CH_2Cl_2 or toluene at a molar ratio L/Rh = 1 at 20 °C. It is accompanied by intensive evolution of gaseous carbon monoxide, a colour change from light yellow to darkbrown and the precipitation of the orange-brown product. The reaction was followed by thin-layer chromatography and IR, ^{31}P NMR and ^{15}N NMR spectroscopy. The complexation of HSP was shown to be slower than that of common phosphorus(III) ligands. The reaction rate depends on the nature of the solvent. In particular, if the reaction is carried out in toluene, the ^{31}P NMR signal from the starting HSP Ia is observed after heating the solution for 30 min at 60 °C. If the more polar methylene chloride is used instead, the signal disappears after 30 min at 20 °C. A new product appears, characterized by a low-field doublet: IIa – δ_P 131.3 ppm, ^1J(P, Rh) 244.3 Hz; IIb – δ_P 134.8, 130.3 ppm, ^1J(P, Rh) 245.7, 243.9 Hz, two diastereomers [7]. These parameters correspond to the ^{31}P NMR signals from the model rhodium complexes IIIa and IIIb with simple P-monodentate amidophosphites which were prepared independently:

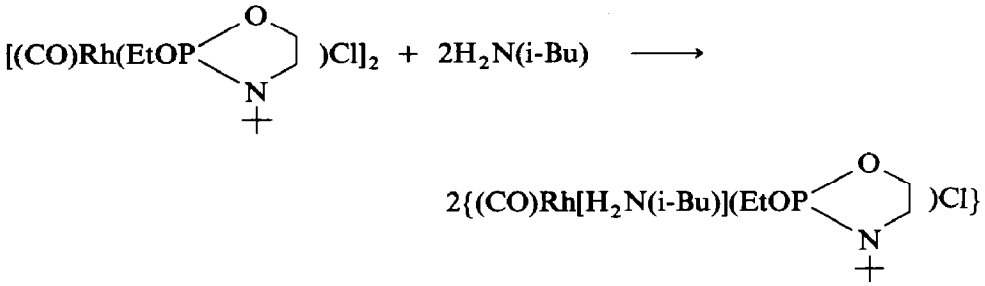
$$[Rh(CO)_2Cl]_2 + 2EtOP\langle O, NR\rangle \xrightarrow{-2CO} 2[(CO)Rh(EtOP\langle O, NR\rangle)Cl]_2$$

(IIIa, b)

(R = Et (IIIa): δ_p 132.0 ppm, ^1J(P, Rh) 260.0 Hz; ^{t}Bu (IIIb): δ_p 122.8 ppm, ^1J(P, Rh) 261.6 Hz)

The similarity among the ^{31}P NMR spectral features of the compounds under consideration becomes more pronounced on the introduction of primary amines

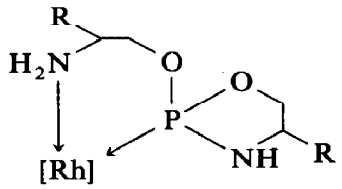
into the coordination sphere of IIIb:



(IV)

(Complex IV: δ_P 129.5 ppm, ^1J(P, Rh) 238.3 Hz)

The spectral data obtained indicate that the reaction of rhodium(I) with HSP involves cleavage of one of the phospholane rings to give a *P,N*-bidentate ligated species in which the amidophosphite part of the ligand is coordinated through phosphorus, while the exocyclic substituent is coordinated through the amino group:



The IR and ^{15}N NMR spectral data support this assumption. The IR spectra are characterized by the disappearance of the characteristic bands from free HSP, at 2376–2364 cm^{-1} (ν(P–H)) in the case of Ia, for example, and the appearance of a broad band at 3245 cm^{-1} (CH_2Cl_2) ascribed to a species coordinated with rhodium primary amino group.

The ^{15}N NMR study was carried out in the case of $[Rh(CO)_2Cl]_2$ and Ia′ enriched to 96.4% with ^{15}N. On addition of the latter to a solution of $[Rh(CO)_2Cl]_2$ in CD_2Cl_2 at −20 °C, and with a monotonic increase in the temperature to 20 °C over a period of 40 min, one can observe a gradual loss of the original signal from HSP (δ_N −332.3 ppm, ^1J(N, P) 30.7 Hz) and the development of a broad doublet from the coordinated oxaphospholane ring at δ_N −312.2 ppm, together with a broad singlet from the coordinated amino group at δ −368.5 ppm with 19 Hz at half-height. It should be pointed out that the literature data for ^1J(N, Rh) of 14–18 Hz [11,12] and ^{15}N NMR chemical shifts on noncoordinated primary amines [13] allow us to ascribe a signal with δ_N −368.5 ppm to primary amino group coordinated to rhodium. It should also be pointed out that the product has a tendency to aggregate in methylene chloride solution, thus decreasing the separation in the ^{15}N NMR spectra.

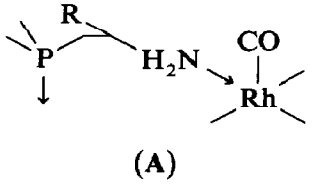
Isolated compounds IIa,b are fine, amorphous orange-brown powders. They are poorly soluble in common organic solvents but not in dimethylsulfoxide (DMSO). The complexes are chromatographically mobile in a thin silica gel layer when a

highly polar eluent is used (^{i}PrOH/aqueous NH_3), in contrast to the complexes of rhodium(I) with *P*-monodentate amidophosphites [14].

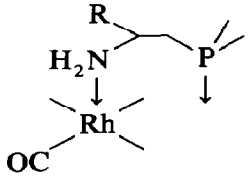
The composition and structure of complexes IIa,b were established on the basis of ^{31}P-, ^{13}C- and ^{15}N-NMR spectroscopy, X-ray electron diffraction, IR spectroscopy, mass spectroscopy using both the electron impact and fast-atom bombardment methods and ultracentrifugation, and were additionally supported by elemental analysis.

A mass spectroscopy investigation of IIa,b using the electron impact technique revealed the presence of molecular ions with m/z 316 and 374, respectively, corresponding to the fragment [Rh(CO)Cl(H_2NCHRCH$_2$O P<O, NH, R>)]. A similar result was obtained by using the fast-atom bombardment technique. A molecular ion with *m/z* 316 was observed in the case of IIa.

Molecular mass measurements under milder conditions by ultracentrifugation (sedimentative equilibria) in DMSO at 25 °C indicate the polynuclear nature of complexes IIa,b: [Rh(CO)Cl(H_2NCHRCH$_2$O P<O, NH, R>)]$_n$ where $n = 4$ (IIa, R = H, m.w. 1240), $n = 6$ (IIb, R = Et, m.w. 2240). Polynuclearity of the compounds reflects complex inter-relations between the central rhodium metal and its ligand environment. In fact, the IR spectra of solid complexes, in addition to the ν(N–H) band at 3245 cm^{-1} and the ν(POC) doublet at 1032–1004 cm^{-1} (which correspond to the coordinated amino group and endo- and exocyclic POC-groups of the ligand, respectively), contain two strong ν(CO) bands at 2080 and 2000 cm^{-1}. These probably arise from different orientations of the exocyclic [H_2NCH(R)CH$_2$O] moiety at phosphorus with respect to rhodium in terms of the polynuclear structure. The higher frequencies refer to the axial carbonyl function (structure **A**):



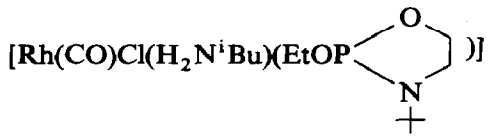
(**A**)



(**B**)

The validity of this assignment is supported by a shift in ν(CO) to lower frequencies, 2032 and 1964 cm^{-1}, respectively, in the case of complex IIa″ enriched with ^{13}CO to 76.4%.

Evidence for different orientations of the carbonyl ligands also comes from the ^{13}C NMR spectrum of a freshly prepared solution of IIa″ in DMSO. It has a doublet of doublets, δ_C 187.5 ppm, ^{1}J(C, Rh) 70.8 Hz, ^{2}J(C, P) 21.0 Hz, and a doublet, δ_C 182.4 ppm, ^{1}J(C, Rh) 80.6 Hz,which correspond to structures **B** and **A**. In the latter case the value of ^{2}J(C, P) is close to zero. It should be noted that in the case of model compound IV:



^1J(C, Rh) is equal to 81.9 Hz. A different coordination of the exocyclic [$H_2NCH(R)CH_2O$] moiety is manifested in the ^{31}P NMR spectra. In particular, two doublets are seen at δ_P 135.5 ppm, ^1J(P, Rh) 244.1 Hz and 133.3 ppm, ^1J(P, Rh) 216.6 Hz in the case of IIa in DMSO. Note that a solution of HSP Ia and $[Rh(CO)_2Cl]_2$ in CH_2Cl_2 has the only doublet in the ^{31}P NMR spectra (see above). Such a comparison, together with all the facts described above, point to the fact that initially the complexation of HSP and $[Rh(CO)_2Cl]_2$ leads to a product in which the exocyclic amino group is coordinated to rhodium in only one way, as in structure **A**, for example. Further aggregation (irreversible precipitation) takes place owing to recombination of the remote amino group, the carbonyl ligand and the central atom (the formation of structure B, in particular). The driving force for this recombination may be the instability of the axial carbonyl, the formation of hydrogen bonds [$OCH_2CH(R)NH_2 \cdots ClRh$] [7] (the presence of a strong and broad (Rh–Cl) IR band in IIa,b supports this) an the tendency of the system to from weak intermetallic contacts. Evidence for Rh–Rh interactions was provided by X-ray electron spectroscopy. The energy of the Rh $3d_{5/2}$ bond in IIa, b is equal to 309.1 and 309.2 eV, respectively, and is somewhat greater than expected taking into account the binding of rhodium with the donor amino group (for comparison, the model compound IIIa [(CO)Rh(EtO P<O, NEt>)Cl]$_2$ is characterized by E(Rh $3d_{5/2}$) of 309.2 eV). The aggregation or formation of a polynuclear rhodium species occurs during the interaction of HSP with $[Rh(CO)_2Cl]_2$, and the products are no longer soluble in common organic solvents (see above). The polynuclear nature of the complexes is preserved in DMSO solutions, but there is probably some structural rearrangement. The dynamics of the ^{15}N NMR spectra of IIa′ containing the *P,N*-ligand enriched with ^{15}N illustrates this proposal. The ^{15}N NMR spectrum of the complex shortly after dissolution (ca. 5 min) is given by a series of signals (Table 1) among which the high-field resonances stand out (δ_N −368.2 and −381.8 ppm) and are ascribed to the coordinated amino group in structures **A** and **B**. Unexpectedly, we observed a signal from the non-coordinated amino group (δ_N −367.1 ppm). This became broader with time and after ca. 60 min disappeared, together with a concomitant doublet at δ_N −325.6 ppm. Concluding this part of our report, it should be emphasized that the existence of complex interrelations between the ligands and the central atom in complexes II is in the first place due to coordinative behaviour of the remove exocyclic amino group and, possibly, to the nature of such amidophosphite ligands. One manner of ligand formation is the reaction of HSP as a tautomeric form of phosphorus(III) [15]:

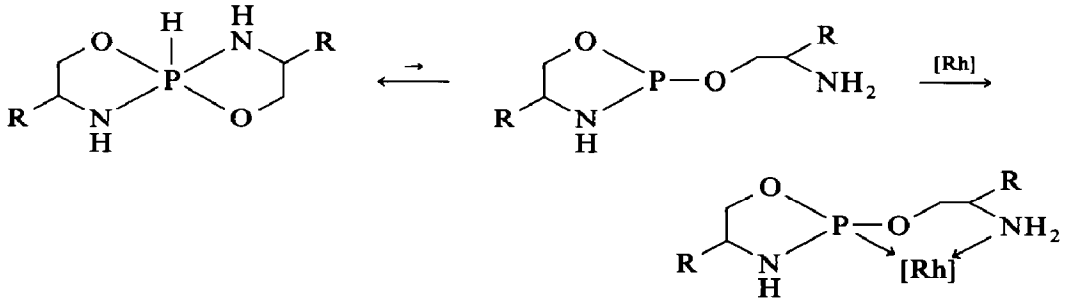


Table 1

^{15}N NMR spectral data for solutions of IIa′ in DMSO-d_6

Signal multiplicity	δ_N [a]	^1J(N, P) [b]	^1J(N, Rh)	^2J(N, P)	^2J(N, Rh)	^1J(N, H)	Assignment
q m [c]	−309.1	22.0	–	–	4.4	– 84.8	$H\overset{\vert}{\underset{\vert}{N}}(CH_2)_2O\overset{\vert}{\underset{\vert}{P}}\rightarrow Rh$
d	−325.6	14.6	–	–	0	not determined [d]	$H\overset{\vert}{\underset{\vert}{N}}(CH_2)_2O\overset{\vert}{\underset{\vert}{P}}\rightarrow Rh$
d q [c]	−338.2	28.0	–	–	0	– 83.0	$H\overset{\vert}{\underset{\vert}{N}}(CH_2)_2O\overset{\vert}{\underset{\vert}{P}}\rightarrow Rh$
s	−367.1	–	–	–	–	not determined [d]	$H_2N(CH_2)_2O-$
q m [c]	−368.2	–	10.2	4.4	–	– 73.0; 67.0	$Rh\leftarrow NH_2(CH_2)_2O-$
d t [c]	−381.8	–	11.7	0	–	– 72.0; 67.0	$Rh\leftarrow NH_2(CH_2)_2O-$

[a] δ_N in ppm. [b] J in Hz. [c] Without {H}. [d] Not observed because of instability of the compound.

The trivalent form of HSP Ia was not, however, detected by spectral means [16,17]. We did not observe it in methylene chloride or toluene by ^{1}H, ^{15}N, ^{13}C and ^{31}P-NMR spectroscopy (in the latter case in the temperature range -80 to $+80\,^{\circ}C$) or IR spectroscopy.

It should be added that HSP Ia,b do not react with BF_3 in toluene, but addition of a calculated amount of alkyl amine ($HNEt_2$, NEt_3) at Rh/N = 1 to the methylene chloride solution of starting $[Rh(CO)_2Cl]_2$ completely inhibits the reaction. At the same time the ordinary amidophosphite $EtOP(O)(CH_2)_2NEt$ reacts with the system $[Rh(CO)_2Cl]_2$/alkyl amine instantaneously and quantitatively. Note that the HSP studied do not react with $acacRh(CO)_2$, even after several hours mixing in CH_2Cl_2 at 25 °C, while the latter complex of Rh^{I} reacts very rapidly with organophosphorus(III) ligands [14]. The data described cast doubt on the reaction of Rh^{I} with HSP via phosphorus(III) and point to an alternative mechanism for HSP in which the properties of both phosphines [18] and hydrophosphoryl compounds [19] are displayed.

Experimental

IR spectra were recorded on Specord 75IR and Specord M80 instruments in KBr disks or in Nujol and CH_2Cl_2 between CsI plates. ^{1}H NMR spectra were run on a Bruker AM-400 instrument at 400 MHz with HMDS as a standard. ^{13}C NMR spectra were run on a Bruker AM-300 instrument at 75.5 MHz versus TMS. ^{15}N NMR data were obtained on a Bruker WP200 instrument at 20.3 MHz versus 1 *M* $H^{15}NO_3$ in D_2O. ^{31}P NMR spectra were run on a Bruker WP80 instrument at 32.4 MHz versus 85% H_3PO_4 in D_2O. Mass spectra were obtained on a MAT-311 instrument using a FAB50TC device for direct sampling, with xenon as reagent gas, an energy of 8 keV, and a glycerol matrix. The sedimentation equilibria were studied using a MOM 31-80 ultracentrifuge at 25 °C. X-ray electron spectroscopy data were collected on a MAC-2 Riber spectrometer calibrated with Ag (lines at 901.5 and 367.9 eV); the samples were charged at 284.6 eV. The precision in determining the maximum of an individual spectral line was 0.1 eV.

Thin-layer chromatography of IIa,b was carried out by using Silufol UV-254 plates and a 3:1 mixture of i-PrOH and 25% aqueous NH_3. The plates were developed under I_2.

5-Hydro-3,8-*R*,*R*-1,6-dioxa-4,9-diazaphosphaspiro[4.4]nonanes Ia, b were prepared as described previously [9] from monoethanolamine (Ia) or (*R*)-2-aminobutanol-1 (Ib) by reaction with hexamethyltriaminophosphine (at a molar ratio of aminoalcohol to $P(NMe_2)_3$ of 2:1) under an argon atmosphere at 100 °C and subsequent short-time heating at 130 °C until the evolution of $HNMe_2$ ceased. The products were recrystallized from hexane.

HSP Ia was obtained from ^{15}N-enriched monoethanolamine. The latter was prepared from $^{15}NH_3$ (96.4% enrichment) and aqueous ethene oxide.

Compounds Ia and Ib were prepared for the first time.

5-Hydro-1,6-dioxa-4,9-diazaphosphaspiro[4.4]nonane (Ia')

White crystals. Yield 96.4%, m.p. 110–111 °C. IR cm^{-1}: ν(N–H) 3476, ν(P–H) 2376, 2364 (CH_2Cl_2). ^{31}P NMR: δ_P −54.9 ppm, ^{1}J(P, H) 725.4 Hz (CH_2Cl_2). Lit. [17]: m.p. 110–111 °C; IR cm^{-1}: ν(N–H) 3474, ν(P–H) 2370 (benzene).

5-Hydro-1,6-dioxa-4,9-diaza-(^{15}N)-phosphaspiro[4.4]nonane (Ia')

White crystals. Yield 96%, m.p. 110–111°C. IR cm^{-1}: ν(N–H) 3468, ν(P–H) 2380, 2364 (CH_2Cl_2). ^{31}P NMR: δ_P −52.8 ppm, ^1J(P, H) 744.7 Hz, ^1J(P, N) 25.3 Hz. ^{15}N NMR: δ_N −332.3 ppm, ^1J(N, P) 30.7 Hz, ^1J(N, H) 90.3 Hz, ^2J(N, H) 7.3 Hz. Found: C, 32.1; H, 7.0; P, 20.4. $C_4H_{11}O_2N_2P$ calcd.: C, 32.2; H, 7.4; P, 20.6%.

5-Hydro-3,8-diethyl-1,6-dioxa-4,9-diaza-5-phosphaspiro[4.4]nonane (Ib)

White crystals. Yield 93%, m.p. 132–133°C. $[\alpha]_D^{20}$ −15.2° (*c* 1.05, $CHCl_3$), IR cm^{-1}: ν(N–H) 3460, ν(P–H) 2350 (broad) (CH_2Cl_2). ^{31}P NMR: δ_P −55.7, −56.7, −57.7 ppm, ^1J(P, H) 732.4 Hz (toluene). Found: C, 46.5; H, 9.5; P, 15.3. $C_8H_{19}O_2N_2P$ calcd.: C, 46.6; H, 9.3; P, 15.0%.

Poly-[chloro(2-β-aminoalkoxy-1,3,2-oxaphospholanecarbonylrhodium(I)] complexes IIa, IIa', IIb were prepared as follows: to a solution of $[Rh(CO)_2Cl]_2$ (10^{-3} mol) in 15 ml absolute methylene chloride kept under argon at 20°C a solution of HSP (10^{-3} mol) in 15 ml CH_2Cl_2 was added on intense stirring. The solution changed colour from bright yellow to dark brown. The major part of the CH_2Cl_2 was removed in vacuo at 25°C (10 mmHg) and 20 ml absolute ether added. A fine orange-brown precipitate was separated by centrifugation, washed with ether and dried in vacuo at 25°C (1 mmHg).

Poly-[chloro(2-β-aminoethoxy-1,3,2-oxaphospholane)carbonylrhodium] (IIa)

Red-brown powder. Yield 86%, m.p. 208–227°C with dec. R_f 0.65, IR cm^{-1}: ν(N–H) 3245, ν(CO) 2080, 1996, ν(PO–C) 1032, 1004 (KBr disc), ν(Rh–Cl) 312 broad (Nujol, CsI); ν(CO) 2064, 1984 (DMSO); ν(N–H) 3245, ν(CO) 2085, 2000, ν(PO–C) 1032, 1004 (CH_2Cl_2, KBr). ^{31}P NMR: δ_P 131.3 ppm, ^1J(P, Rh) 244.3 Hz (CH_2Cl_2); δ_P 135.5 ppm, ^1J(P, Rh) 244.1 Hz; δ_P 133.3 ppm, ^1J(P, Rh) 261.6 Hz (DMSO). ^{13}C NMR: δ_C 187.5 ppm (q), ^1J(C, Rh) 70.8 Hz, ^2J(C, P) 21.0 Hz; δ_C 182.4 ppm (d), ^1J(C, Rh) 80.6 Hz (DMSO). M.w. 1240 (DMSO). Found: C, 18.7; H, 3.7; P, 9.5. $C_5H_{11}ClO_3N_2PRh$ calcd.: C, 19.0; H, 3.2; P, 9.8%.

Poly-[chloro(2-β-amino[^{15}N]ethoxy-1,3,2-oxaza[^{15}N]phospholanecarbonylrhodium] (IIa')

Red-brown powder. Yield 86%, m.p. 208–227°C with dec. R_f 0.65. IR cm^{-1}: ν(N–H) 3232, ν(CO) 2084, 2000, ν(PO–C) 1032, 1006 (KBr disc). ^{15}N NMR (see Table 1). Found: C, 19.0; H, 3.6; P, 9.6. $C_5H_{11}ClO_3\,{}^{15}N_2PRh$ calcd.: C, 19.0; H, 3.2; P, 9.8%.

Poly-[chloro(2-β-aminobutoxy-4-ethyl-1,3,2-oxazaphospholane)carbonylrhodium] (IIb)

Orange-brown powder. Yield 89%, m.p. 230–255°C with dec. R_f 0.55. IR cm^{-1}: ν(N–H) 3240, ν(CO) 2080, 2000, ν(PO–C) 1036 (broad) (KBr disc); ν(Rh–Cl) 320 (broad) (Nujol, CsI). ^{31}P NMR: δ_P 134.8 ppm, ^1J(P, Rh) 245.7 Hz, δ_P 133.3 ppm, ^1J(P, Rh) 243.9 Hz (CH_2Cl_2); δ_P 137.5 ppm, ^1J(P, Rh) 240 Hz, δ_P 131.9 ppm, ^1J(P, Rh) 256 Hz (DMSO). M.w. 2240 (DMSO). Found: C, 29.4; H, 4.8; P, 8.2. $C_9H_{19}ClO_3N_2PRh$ calcd.: C, 29.0; H, 5.1; P, 8.3%.

References

1 D. Bondoux, J. Tkatchenko, D. Houalla, R. Wolf, J. Riess and B.F. Mentzen, J. Chem. Soc., Chem. Comm., (1978) 1022.
2 D. Bondoux, D. Houalla, B. Mentzen, J. Riess, J. Tkatchenko and R. Wolf, Proc. Symp. Rhodium Homogeneous Catal., (1978) 1.
3 D. Bondoux, D. Houalla, C. Prodat, J. Riess, J. Tkatchenko and R. Wolf, Fundam. Res. Homogeneous Catal., 3 (1978) 969.
4 M.C. Bonnet and J. Tkatchenko, Now. J. Chim., 7 (1983) 601.
5 E.E. Nifantyev, A.T. Teleshev and T.A. Shikovets, Zh. Obsch. Khim., 56 (1986) 298.
6 R. Tuppin, P. Dagnac and Poilblanc, J. Organomet. Chem., 319 (1987) 247.
7 J. Riess, C. Pradat, O. Bondoux, B. Mentzen, J. Tkatchenko and D. Houalla, J. Am. Chem. Soc., 101 (1979) 2234.
8 E.G. Burns, S. Chu, P. de Meester and M. Lattman, Organometallics, 5 (1986) 2383.
9 T. Reetz and J.F. Powers (Monsanto Co.), U.S. Patent 3, 172,903. (1965).
10 B.A. Arbuzov and N.A. Polezhaeva, Usp. Khim., 43 (1974) 933.
11 R. Meis, D. Stufkens and K. Vrieze, J. Organomet. Chem., 164 (1979) 353.
12 K. Bose and E. Abbott, Inorg. Chem., 19 (1977) 3190.
13 J. Mason, Chem. Rev., 81 (1981) 205.
14 E.E. Nifantyev, A.T. Teleshev, T.A. Shikovets, A.R. Bekker, A.N. Chernega, M.Yu. Antipin and Yu.T. Struchkov, J. Organomet. Chem., 336 (1987) 237.
15 R. Burgada, Bull. Soc. Chim. Fr., 1–2 (1975) 407.
16 M. Sanchez, J. Ferekn, J. Brazier, A. Munoz and R. Wolf, Roczniki Chemii, 45 (1971) 131.
17 N.P. Grechkin, R.R. Shagidulin and L.N. Grishina, Dokl. Akad. Nauk SSSR, 161 (1965) 115.
18 R. Schunn, Inorg. Chem., 12 (1973) 1573.
19 M. Bennett and T. Mitchell, J. Organomet. Chem., 70 (1974) C30.
20 J.A. McLeverty and G. Wilkinson, Inorg. Synth., 8 (1966) 211.