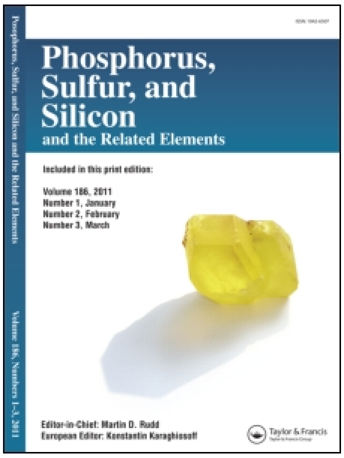

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SYNTHESIS, STRUCTURE AND CATALYTIC PROPERTIES OF NEUTRAL CARBONYL COMPLEXES OF RHODIUM(I) WITH 2-AMINO-1,3,2-DIOXAPHOSPHORINANES

E. E. Nifantyev[a]; A. T. Teleshev[a]; G. M. Grishina[a]; A. A. Borisenko[ab]
[a] Chair of Organic Chemistry, V. I. Lenin Moscow State Pedagogical Institute, Moscow, USSR [b] Department of Chemistry, M. V. Lomonosov Moscow State University, Moscow, USSR

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SYNTHESIS, STRUCTURE AND CATALYTIC PROPERTIES OF NEUTRAL CARBONYL COMPLEXES OF RHODIUM(I) WITH 2-AMINO-1,3,2-DIOXAPHOSPHORINANES*

E. E. NIFANTYEV, A. T. TELESHEV, G. M. GRISHINA
and A. A. BORISENKO†

*Chair of Organic Chemistry, V. I. Lenin Moscow State Pedagogical Institute,
Moscow G-21, USSR*

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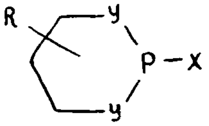
Neutral mono- and di-nuclear carbonyl 2-amino-1,3,2-dioxaphosphorinane rhodium complexes of types $RhCl(CO)L_2$ and $[RhCl(CO)L]_2$ have been synthetized. The complexing process in the $[RhCl(CO)_2]_2$-phosphorinane system has been investigated at different molar ratios of reagents by means of ^{31}P NMR spectroscopy. The dinuclear phosphorinane complexes, in contrast to mononuclear ones, exhibit activity in the catalytic hydrogenation of olefins.

Neutral carbonyl phosphorus-containing rhodium(I) complexes, e.g., trans-$RhCl(CO)L_2$ are well known as catalysts for the hydrogenation and hydroformylation of unsaturated compounds.[2,3] Alkyl-, aryl-phosphines, aminophosphines, phosphites,[2,4,5] i.e., basically symmetrical systems, have been used as organophosphorus ligands in obtaining these complexes. To obtain further insight into the functions of these rhodium catalysts, it was considered important to synthetize complexes with asymmetrical PXY_2 ligands, which will permit tracing the influence of the X substituent on the properties of both the ligand and the complex. It has to be noted that, in a symmetrical PX_3 system, analysis of this kind presents difficulties because X substituents without axial symmetry are not always orientated in the same way with respect to the phosphorus atom. Therefore, the electronic effect of three identical substituents at the phosphorus atom will not adhere to simple additivity rules.

Working with PXY_2 ligands will not always lead to unambiguously interpretable results. It is known that many trivalent phosphorus derivatives, PXY_2, are easily symmetrizable and enter into other reactions of substituents exchange.[6] This exchange is especially accelerated in the presence of acids whose proton is coordinated on the ligand.[7] Coordination of the asymmetrical ligand on the metal will, probably, also facilitate the exchange process of substituents on the phosphorus atom. Taking all this into account we have chosen 1,3,2-diheterophosphorinanes



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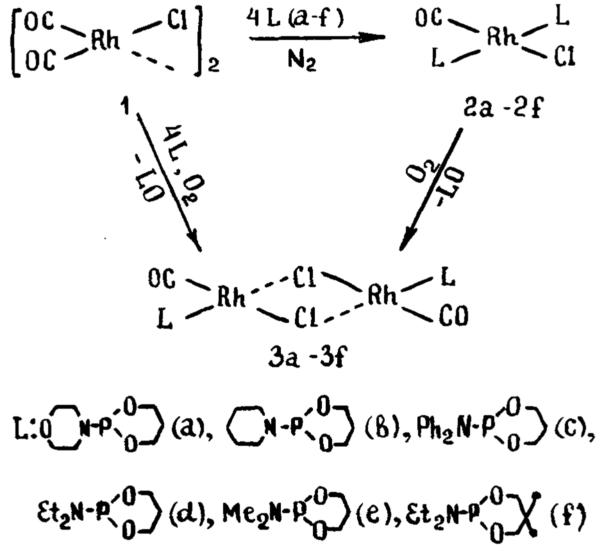
† Department of Chemistry, M. V. Lomonosov Moscow State University, Moscow V-234, USSR
* See preliminary communication.[1]

as a promising class of PXY_2 ligands. These compounds are stabilized by their cyclic structure and show no tendency to disproportionation under the conditions of our experiments. They are readily available. And finally they can exist in different geometric forms, which opens the way for investigating the influence of spatial orientation of the X substituents on the properties of the complex.

In this communication we report our data on neutral carbonyl rhodium(I) complexes with amides of 1,3-propylidenephosphorous acid (2-amino-1,3,2-dioxaphosphorinanes) as the ligands.

The principal method of obtaining such complexes consists in the interaction of μ-dichlorotetracarybonyldirhodium(I) with trivalent phosphorus derivatives.[8]

We have carried out a comprehensive study of the interaction of μ-dichlorotetracarbonyldirhodium **1** with 2-amino-1,3,2-dioxaphosphorinanes (L). It has been established that, when the reaction is conducted in an atmosphere of inert gas in anhydrous ether (but not in benzene, which has been recommended for this kind of reactions[8]) at the ratio of L/2Rh = 4 yellow, crystalline mononuclear trans-complexes, **2**, are formed in high yield. If the reaction is conducted under the above conditions, but under dry air, the mononuclear complexes **2** become contaminated with dinuclear complexes **3**, which were isolated from the reaction mixture chromatographically.*



SCHEME 1

In all the experiments conducted in air the reaction mixtures also reveal (^{31}P NMR) the products of ligands oxidation—2-amino-2-oxo-1,3,2-dioxaphosphorinanes (LO). The oxidation seems to proceed intraspherically. This is indicated by the oxidation rate of ligands situated in the coordination sphere being higher than that of free ligands.

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*In our previous communication[9] complex **3** was mistakenly designated as the cis-isomer of complex **2**.

All the complexes of types **2** and **3**, which were obtained have been characterized by conventional, physico-chemical methods (see Table I), and elementary analysis. Molecular masses have been measured by mass spectrommetry and vapour-pressure osmometry. Using these data the structures of compounds have been determined.

The structures of the mononuclear *trans*-complexes **2** follow from their ^{31}P NMR spectra: doublets at δ 124.0–132.8 ppm. ^1J(Rh—P) 172.0–186.1 Hz, and the IR

TABLE I

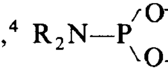
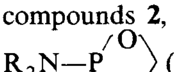
Physico-chemical properties of neutral carbonyl–rhodium(I)-complexes with 2-amino-1,3,2-dioxaphosphorinanes

					^{31}P NMR[c]		Elementary analysis General formula
Complex	Yield, %	M.p.,°C	R_f[a]	ν(CO) cm^{-1}[b]	δ, ppm	^1J(Rh—P), Hz	Found. %: C, H, Cl, P Calc., %: C, H, Cl, P
2a	90	169–171	0.44	1990	128.2 (128.5)	174.5 (176.5)	$C_{13}H_{28}ClN_2O_7P_2Rh$ 32.6; 5.3; 6.6; 10.8 32.8; 5.1; 6.5; 11.3
2b	95	165–166	0.88	1986	128.1	172.9	$C_{17}H_{32}ClN_2O_5P_2Rh$ 38.0; 5.0; 6.7; 10.9 37.5; 5.9; 6.5; 11.4
2c	77	208–209	0.75	1992	123.5	186.1	$C_{31}H_{32}ClN_2O_5P_2Rh$ 52.7; 5.0; — 8.5 52.2; 4.5; — 8.7
2d	90	165–166	0.68	(1977)	131.7 (131.9)	172.0 (173.2)	$C_{15}H_{32}ClN_2O_5P_2Rh$ 34.3; 6.8; — 10.5 34.5; 6.2; — 10.9
2e	95	115–116	0.68	1987	130.7	173.0	$C_{11}H_{24}ClN_2O_5P_2Rh$ 29.3; 4.8; — 12.5 28.4; 5.1; — 13.4
2f	90	183–185	0.64	(1985)	(132.8)	(173.4)	$C_{19}H_{40}ClN_2O_5P_2Rh$ 39.9; 6.2; — 11.2 39.7; 6.9; — 10.7
3a	87[d] 87[e]	157–159	0.86	1998	(127.5)	(243.0)	$C_{16}H_{28}Cl_2N_2O_8P_2Rh_2$ 25.4; 3.7; 9.3; 9.2 26.8; 3.9; 9.9; 8.7
3b	84 55	162–164	0.96	1994	(127.3)	(242.1)	$C_{18}H_{32}Cl_2N_2O_6P_2Rh_2$ 31.0; 4.8; — 8.9 30.4; 4.5; — 8.7
3c	76 52	196–197	0.82	1994	broad band in 120–140 region	—	$C_{32}H_{32}Cl_2N_2O_6P_2Rh_2$ — — — 7.9 — — — 7.1
3d	85 57	162–163	0.77	(1984)	(129.9)	(241.0)	$C_{16}H_{32}Cl_2N_2O_6P_2Rh_2$ 28.3; 5.1; — 9.5 28.7; 5.4; — 9.4
3e	84 54	106–107	0.72	1995	broad band in 120–140 region	—	$C_{12}H_{24}Cl_2N_2O_6P_2Rh_2$ 23.2; 3.9; — 9.8 22.8; 3.8; — 9.8
3f	85 60	113–114	0.74	(1992)	(130.5)	(240.5)	$C_{20}H_{40}Cl_2N_2O_6P_2Rh_2$ 32.8; 5.2; — 8.7 32.9; 5.5; — 8.3

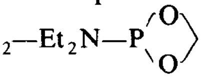
[a] *System-silufol UV-254, system A, for* **2f,3f**-*system B (for A and B (see experimental section).*
[b] In toluene, in brackets in KBr pellets.
[c] In dimethylformamide, in brackets for solutions in toluene (30°C).
[d] Method (a).
[e] Method (b) (see experimental section).

spectra: one strong absorption band ν(CO) in the region of 1986–1992 cm^{-1} (see Table I).

The dinuclear complexes **3** are stable in the crystalline state and in toluene solution. Mass spectrometric studies confirm the dimeric structures of these compounds. Thus, for complex **3** with 2-diethylamino-1,3,2-dioxaphosphorinane the heaviest masses have in the spectrum a corresponding group of peaks with m/z 660, 658, 656 that conform to the ion of the dimeric complex **3** without one carbonyl group $(M{-}CO)^+$. It should be noted that in toluene solutions we obtained lower molecular mass values when using vapour-pressure osmometry. For instance, for complex **3** with 2-diethylamino-1,3,2-dioxaphosphorinane the observed molecular mass was 630 ± 5, whilst the calculated value was 686. This seems to indicate partial dissociation of the compounds. The ^{31}P NMR spectra of complexes **3** display a doublet in the region of δ 127.3–130.5 ppm, ^1J(Rh—P) 240.5–243.0 Hz. A higher ^1J(Rh—P) value in complexes **3** than in complexes **2** is associated with the differences in the *trans*-effect of the chlorine anion and the organophosphorus ligand.[10] The IR spectra of complexes **3**, have a characteristic strong absorption band ν(CO) 1994–1998 cm^{-1} in toluene, and their indicate, according to Ref. 11, the *trans*-configuration of the compounds. A higher ν(CO) value in complexes **3**, as compared with those of compounds **2**, is associated with the difference in strength of the chlorine anion and the organophosphorus ligand *trans*-effect.

The spectral data of complexes permit one to make an assumption of the relative donor-acceptor strength of phosphorinane ligands. Thus, from the ν(CO) values of complexes **2**, 2-amino-1,3,2-dioxaphosphorinanes occupy an intermediate place between PPh_3 and $P(OPh)_3$, which indicates their distinct electron-acceptor nature. The spectrochemical series of the ligands is as follows (ν CO, cm^{-1}): $P(C_6H_{11})_3$ (1941),[4] PPh_3 (1978),[4]  $R_2N{-}P$ (1986–1990), $P(OPh)_3$ (2017).[4] The electron-acceptor nature of phosphorinanes is also confirmed by the ^1J(Rh—P) values of compounds **2**, L (^1J Rh—P, Hz): PBu_3 (115.9),[12] PEt_3 (116.1),[12] PPh_3 (124.0),[12]  $R_2N{-}P$ (162.0–186.1), $P(OMe)_3$ (193.9).[12]

The type **2** and **3** complexes described by us are not the only products of the interaction between the starting complex **1** and 2-amino-1,3,2-dioxaphosphorinanes. Other intermediate complexes also form in the reaction mixture.[11,13,14] We have, therefore, investigated the influence of the L/Rh molar ration on the course of complexation in the compound **1**-phosphorinane system. The data on the composition of, and the relationship between, the complexes formed has been obtained by ^{31}P NMR spectroscopy (see Tables II and III). Since a number of intermediate compounds can be in exchange with one another or with the solvent, the ^{31}P NMR spectra were recorded at −80°C. A characteristic spectrum is shown in Figure 1.

Let us now examine the $[RhCl(CO)_2]_2$— $Et_2N{-}P$ system (see Scheme 2, Table II).* At molar ratios L/2Rh = 0.5–1.0 two basic rhodium complexes 4 and 5 exist in the reaction mixture. Compound 5 with δ 125.9 ppm has ^1J(Rh—P) 235.5 Hz. The latter is characteristic of the equatorial phosphorinane ligand in dinuclear

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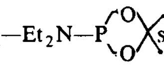
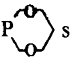
*In a similar way one can examine the $[RhCl(CO)_2]_2$— $Et_2N{-}P$ system (see Table III).

TABLE II

Chemical shifts δ (ppm), spin–spin coupling constants $^1J(\mathrm{Rh—P})$ (Hz) and the composition of individual components in the reaction mixture depending on L/Rh molar ratio for the $[(\mathrm{CO})_2\mathrm{RhCl}]_2$—$\mathrm{Et_2N}$—P system. Toluene, −80°C

Parameters of signals	Composition, %						
	a	b	c	d	e	f	g
δ	125.9	121.1	124.7	120.3	128.6	129.4	131.2
^1J	235.5	184.0	141.9	100.0	238.5	236.2	171.4
L/2Rh							
0.5[a]	68	32	—	—	—	—	—
1.0	70	30	—	—	—	—	—
1.5	40	24	5	5	19	3	4
2.0[b]	17	19	6	7	25	4	22
2.5	4	19	4	4	22	3	44
3.0	—	15	—	—	6	—	79
3.0[c]	—	8	—	—	10	—	82
2.5[d]	—	12	—	—	22	3	63
2.0	—	18	—	—	34	7	41
1.5	5	16	—	—	52	9	18
1.0	6	15	—	—	58	7	14

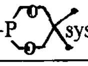
[a] In first experiments at L/Rh = 0.5–3.0 content of **1** is $5.2 \cdot 10^{-2}$ mmole in 2 ml toluene;

[b] Spectrum given in Figure 1.

[c] Spectrum taken after keeping the sample at 20°C for 1 h.

[d] In experiments with L/2Rh = 2.5–1.0 solid **1** was gradually added to solution.

TABLE III

Chemical shifts δ (ppm), spin–spin coupling constants $^1J(\mathrm{Rh—P})$ (Hz) and the composition of individual components in the reaction mixture depending on L/Rh molar ratio for the $[(\mathrm{CO})_2\mathrm{RhCl}]_2$[a]—$\mathrm{Et_2N}$—Psystem. Toluene, −80°C

Parameters of signals	Composition, %						
	a	b	c	d	e	f	g
δ	126.9	122.2	124.8	120.0	129.7	130.7	132.2
^1J	234.8	184.0	145.5	106.6	237.8	236.5	171.4
L/2Rh							
0.5	68	32	—	—	—	—	—
1.0	76	24	—	—	—	—	—
1.5	33	21	10	3	19	2	12
2.0	8	24	8	2	24	1	33
2.5	—	24	2	2	20	—	52
2.5[b]	—	6	2	—	43	—	49
3.0	—	5	—	—	13	—	82
3.7	—	4	—	—	—	—	96

[a] Rhodium content of $5.2 \cdot 10^{-2}$ mmole in 2 ml toluene.

[b] Spectrum taken after keeping the sample at 20°C for 6 h.

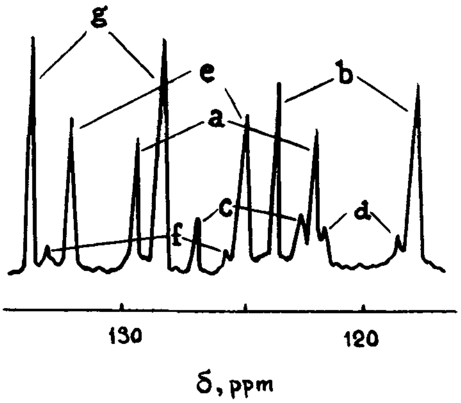
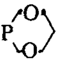
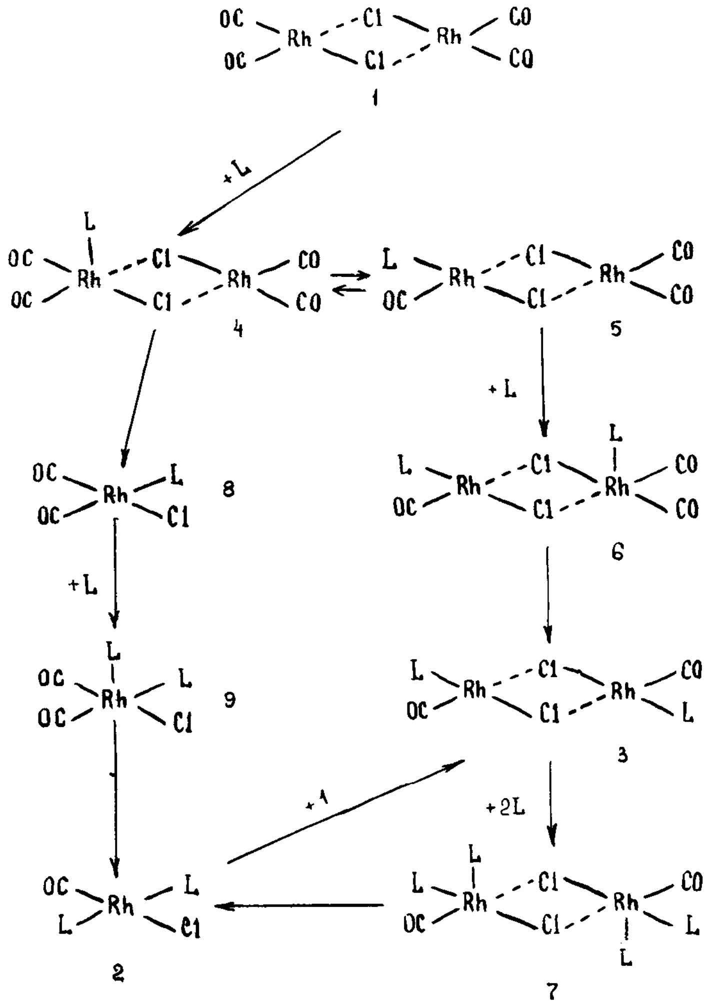


FIGURE 1 ^{31}P NMR spectrum of system [(CO)$_2$RhCl]$_2$—Et$_2$N-P in toluene at −80°C, molar ratio L/2Rh = 2 (for a–g Table II).

complexes of type **3**, ^1J(Rh—P) 238.5 Hz. It should be noted that the ^1J(Rh—P) value decreases with a decrease in sample temperature. The lower ^1J(Rh—P) values observed in compounds **5** are associated with a stronger influence of the CO group in complex **5** in comparison with the phosphorinane ligand in compound **3**. The signal at δ 121.1 ppm, ^1J(Rh—P) 184.0 Hz, seems to correspond to complex **4** with an axial location of the phosphorinane ligand. The assumption that the signal belongs to mononuclear complexes of type **8** is inconsistent with these compounds having a higher ^1J(Rh—P) value, 184.4 Hz, than that of complexes **2**, 171.4 Hz. The data presented in Ref. 15 confirm that the axial organophosphorus ligand has a smaller ^1J(Rh—P) value than the equatorial ligand. This is in agreement with the measured constants for axial phosphorinane in dinuclear complexes of type **4**, ^1J(Rh—P) 184.0 Hz, and for the equatorial ligand in complexes **5**, ^1J(Rh—P) 235.5 Hz. Compounds containing an axial phosphorinane ligand exist within the whole range of investigated ratios L/2Rh = 0.5–3.0 (see Table II), which makes it possible to assume the existence of intermediate complexes **6** and **7** in solution.

Mononuclear complexes **8**, and **9** seem to be characterized in the spectra by the signals at δ 124.7 ppm, ^1J(Rh—P) 141.9 Hz and δ 120.3 ppm, ^1J(Rh—P) 100.0 Hz. A reduced ^1J(Rh—P) value, 141.9 Hz, for equatorial phosphorinane in compounds **8**, and **9** relative to 171.4 Hz for complex **2** is associated with a stronger trans-effect of CO in comparison with the phosphorinane. The ^1J(Rh—P) constant of 100 Hz for the axial phosphorinane in compound **9**, as would be expected, is lower than ^1J(Rh—P) 141.9 Hz for the equatorial phosphorinane in complexes **9**, **8**.

Mononuclear *trans*-complexes **2** and dinuclear *trans*-complexes **3** simultaneously appear in solutions at L/2Rh = 1.5. Complexes **3** accompany the reaction products whose parameters δ 129.4 ppm and ^1J(Rh—P) 236.2 Hz are very close to their own. This is assumed to be associated with a possible dissociation of *trans*-complexes **3** and the formation of [(CO)RhCl(S)] compounds, where S is the solvent. This assumption is in agreement with the apparent lower molecular weights observed for compounds **3** in solution. This is also indicated by the ^{31}P NMR spectra of complexes **3** in solvents, manifesting a strong tendency to coordination. For in-



SCHEME 2

stance, the spectra of complex **3** in dimethylformamide exhibit a diffuse band characteristic of exchange processes (see Table I). Summing up the aforesaid it should be stressed that rhodium complexes **2** and **3** accompany each other in the course of synthesis. It is possible for compound **3** to be converted into complex **2** when there is an excess of L, The inverse process of complex **2** being converted into compound **3** by reaction with starting complex **1** is also possible (see Scheme 2). Formation of pentacoordinated intermediate rhodium complexes is connected with the associative mechanism of the reaction between the phosphorinane ligand and the initial compound **1**, which agrees with the data presented in Ref. 13.

Complexes **2** and **3** have been investigated in catalytic hydrogenation. 1-Heptene was used as the model substrate. Complexes **2** proved to be inactive in hydrogenation at 70°C and atmospheric pressure of hydrogen in toluene solution. Such

TABLE IV

Specific activity (α) of phosphorinane carbonyl rhodium complexes in 1-heptene hydrogenation (70°C, P_{H_2} = 1 atm., [Rh] = $2.5 \cdot 10^{-3}$ mole/l, [1-heptene] = 1 mole/l, reaction time 30 min)

Complex[a]	**2**	**3b**	**3a**	**3d**	**3e**	**3c**
α, mole H_2/mole Rh · min	0	1.9	1.3	1.5	1.4	1.2
trans-2-heptene,[b] %	0	24[c]	20	29	37	28
cis-2-heptene,[b] %	0	2[b]	11	14	18	44

[a] See Scheme 1.
[b] Reaction time 120 min, GLC data.
[c] 77 min.

behaviour of complexes **2** is caused by the electron-acceptor nature of phosphorinane ligands and is similar to that of complexes with bisubstituted phosphites.[4] Dinuclear complexes **3** manifest catalytic activity in the hydrogenation and isomerization of olefins (see Table IV). The activity of compounds **3** seems to be connected with their possible dissociation and the formation of coordinatively unsaturated complexes [L(CO)RhCl]. Indifference of compounds **2** to catalytic processes offers in

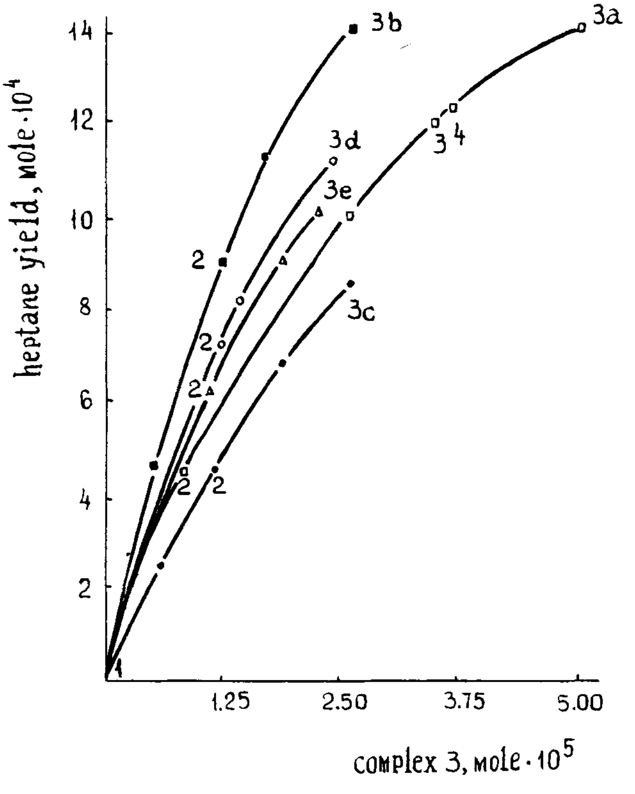


FIGURE 2 Heptane yield vs. the concentration of complexes **3a–3e**. 1—complex **2**, 2—natural mixture of complexes **2** and **3**, 3—artificial mixture of complexes **2** and **3** in the ratio of 35 : 65, 4—theoretical point for mixture of complexes **2** and **3** in the ratio of 35 : 65 (70°C, P_{H_2} = 1 atm., toluene 7.5 ml, 1-heptene = $2.5 \cdot 10^{-3}$ mole, reaction time 30 min).

a number of cases an opportunity to estimate the content of complexes **2** and **3** in reaction mixtures. Thus, the synthesis of rhodium phosphorinane complexes in an atmosphere of dry air in ether at 20°C and molar ration L/2Rh = 4 results in the formation of a mixture of complexes **2** and **3**. The available dependence of heptane specific yield on the concentration of active complex (see Figure 2) makes it possible to calculate the relative content of complexes **2** and **3**. The content (%) of complex **3** depending on L amounts, equals respectively, to:

3a (65); **3b** (69); **3c** (46); **3d** (52); **3e** (52).

The relative error calculated at comparing the practical activity of an artificial mixture of complexes **2** and **3** (see Figure 2, point 3) with the theoretically possible activity (point 4) amounts to ±5%.

EXPERIMENTAL

IR spectra of solutions in toluene were taken on a "UR-20" instrument. ^{31}P NMR spectra were obtained at a frequency of 32.2 MHz on an FT-80A, "Varian", instrument; chemical shifts δ were measured with respect to 85% H_3PO_4 as the external standard. Toluene and dimethylformamide were used as solvents at 30°C, toluene—at −80°C.

Mass spectra were taken on an AEI MS-30 instrument at ionizing voltage 70 v, temperature of the system of direct sample introduction 100°C and ionization chamber temperature 250°C. Molecular masses were also determined by vapour-pressure osmometry in toluene on a "Hitachi 115" instrument.

Thin-layer chromatography of compounds was performed on silufol UV-254 (Czechoslovakia) in benzene–dioxane, 5 : 1 (system A) and benzene–dioxane, 10 : 1 (system B); chromatograms were developed by calcination at 200–250°C.

2-Amino-1,3,2-dioxaphosphorinanes were synthetized in accordance with procedures described in Ref. 1. For 2-diethylamino-5,5-dimethyl-1,3,2-dioxaphosphorinane described in Ref. 16.

Synthesis of complexes.

Chloro-bis(2-amino-1,3,2-dioxaphosphorinane)carbonylrhodium **2**. $4 \cdot 10^{-4}$ mole 2-amino-1,3,2-dioxaphosphorinane in 10 ml ether in an atmosphere of dry deoxygenated nitrogen was added with intensive stirring to a solution containg 10^{-4} mole of the initial complex **1**[17] in 10 ml of dry ether. The color of the solution changed from yellow to light-yellow. The ether was evaporated in vacuo (10 mm merc.col.) at 20°C in an atmosphere of nitrogen. The product was purified on a chromatographic column in accordance with the following procedure. The complex suspended in benzene, was applied on adsorbent-Pitri wide-pore silica gel with 2.5% starch and eluted, first with benzene and then successively with solvent systems (B) and (A). The isolated complex, after solvent evaporation to minimal volume in vacuo (10 mm) at 20°C in the atmosphere of dry inert gas, was precipitated with dry freshly distilled hexane at liquid nitrogen temperature. The product is a yellow, crystalline compound.

Trans-μ-dichloro(bis-2-amino-1,3,2-dioxaphosphorinane)dicarbonyldirhodium **3**.

(a) $6 \cdot 10^{-4}$ mole 2-amino-1,3,2-dioxaphosphorinane in 10 ml ether was added drop by drop to $3 \cdot 10^{-4}$ mole complex **1** in 15 ml dry ether in an atmosphere of inert gas with stirring. The reaction mixture was stirred at 20°C for 30 min. The ether was distilled off to minimal volume *in vacuo*, and dry hexane was added. On slow cooling to −10°C yellow crystals of the complex precipitate from solution.

(b) Oxygen at room temperature was passed for 1.5–2 h through solution containing $3 \cdot 10^{-4}$ mole of complex **2** in 20 ml dry benzene. The course of complexing was controlled chromatographically. On the completion of reaction the solvent was evaporated to minimal volume in vacuo (10 mm) at 20°C in an atmosphere of dry inert gas. The dinuclear complex was obtained purified on a chromatographic column, adsorbent-Pitri wide-pore silica gel with 2.5% starch, and eluted, first with benzene, then successively with solvent systems (B) and (A). The movement and separation of the yellow-coloured complexes **2** and **3** were clearly seen against the white background of the adsorbent. The solvent was evaporated to 1–2 ml volume in vacuo (10 mm) at 20°C in an atmosphere of dry inert gas. The complex was precipitated from solution with freshly distilled hexane at liquid nitrogen temperature.

Catalytic hydrogenation. The hydrogenation was conducted as described in Ref. 18. The rhodium complex was dissolved in toluene, the solution was placed in a catalytic reactor, 1-heptene was added, and the mixture was shaken for 5 h at 90°C in an atmosphere of dry, oxygen free nitrogen (purification from water—4 Å molecular sieves, from oxygen—copper on kiesselgur at 150°C. The temperature was then lowered to 70°C and the reactor blown through with hydrogen (hydrogen purification similar to that of nitrogen). The onset of hydrogenation was taken to coincide with the switching-on of the rocker (700–800 roc/min). The course of hydrogenation was controlled by the amount of hydrogen absorbed in a unit of time and by the gas-liquid chromatography method. Chromatograph "Carlo Erba", detector—katharometer. Analysis was performed on a column containing 15%, ββ-dioxydipropionitrile on chromosorb P; length of column 6 m, diameter 6 mm. Separation temperature 50°C.

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