## Synthesis and reactivity of metal-containing monomers 51.\* Synthesis and molecular structure of the cluster-containing complex [Rh<sub>6</sub>(CO)<sub>14</sub>(μ,η<sup>2</sup>-PPh<sub>2</sub>CH<sub>2</sub>CH=CH<sub>2</sub>)]

S. I. Pomogailo, I. I. Chuev, G. I. Dzhardimalieva, A. V. Yarmolenko, V. D. Makhaev, S. M. Aldoshin, and A. D. Pomogailo\*

Institute of Problems of Chemical Physics, Russian Academy of Sciences, 1424432 Chernogolovka, Moscow Region, Russian Federation. Fax: +7 (096) 515 3588. E-mail:adpomog@icp.ac.ru

The reaction of  $Rh_6(CO)_{15}MeCN$  with allyldiphenylphosphine under mild conditions afforded the cluster-containing complex  $[Rh_6(CO)_{14}(\mu,\eta^2-PPh_2CH_2CH=CH_2)]$ . Its molecular structure was characterized. The resulting complex is an octahedral Rh cluster with ten terminal and four  $\mu_3$ -bridging CO ligands. The average Rh—Rh distance is 2.762(2) Å. The unsaturated ligand is additionally coordinated to the metal center (Rh(4)-C(232), 2.37(1) Å; and Rh(4)-C(233), 2.32(2) Å) to form a  $\pi$ -bond.

Key words: cluster-containing complex, molecular structure, rhodium clusters, coordination,  $\pi$ -complex, synthesis.

Recently, polymer-immobilized metal clusters attracted considerable attention of researchers because of their valuable and sometimes unique properties, such as catalytic, magnetic, optical, etc.<sup>2</sup> Previously,<sup>3</sup> we have prepared and characterized mono- and disubstituted derivatives of  $Rh_6(CO)_{16}$  (1) based on 4-vinylpyridine (4-VinPy). A desired change in the stability and reactivity of polymers based on cluster-containing monomers can be achieved by changing the nature of unsaturated ligands in these monomers. For this purpose, in the present work we studied the synthesis and structure of  $[Rh_6(CO)_{14}(\mu,\eta^2-PPh_2CH_2CH=CH_2)]$  (2). Since these compounds are potential comonomers for preparing metallopolymers by their copolymerization with traditional monomers (styrene, methyl methacrylate, or acrylonitrile), the polymer chains formed are diluted with inert comonomer units and the content of cluster fragments is low. In our studies, allyldiphenylphosphine (AllPPh<sub>2</sub>) was of interest as an unsaturated ligand in two aspects. First, AllPPh2, unlike 4-VinPy, is less reactive in copolymerization conversions due to degradation chain transfer, which makes it possible to prepare copolymers with a low content of rhodium, viz., low-percentage catalysts. Second, it is known that it is phosphoruscontaining Rh complexes (including those of polymeric nature) that are active and selective catalysts of reduction, hydroformylation, etc.4,5

## **Results and Discussion**

Synthesis of complex 2. The reaction of selective replacement of the CO group in 1 by  $AllPPh_2$  was

\* For Part 50, see Ref. 1.

performed in two stages. At the first stage, by analogy with a known procedure,<sup>6</sup> compound 1 in chloroform in the presence of acetonitrile was converted into its monosubstituted derivative containing a labile ligand, *viz.*, acetonitrile, under photochemical excitation (UV radiation,  $\lambda > 300$  nm).

$$Rh_6(CO)_{16} + MeCN \xrightarrow{hv} Rh_6(CO)_{15}NCMe + CO$$
 (1)  
3

Photoreaction (1) made it possible to prepare compound 3 in quantitative yield. The composition of 3 was confirmed by the data of elemental analysis and IR spectroscopy. The positions and relative intensities of absorption bands in the carbonyl region correspond to the spectral characteristics of the monosubstituted product based on  $Rh_6(CO)_{16}$  (v/cm<sup>-1</sup>: 2294 (CN); 2104, 2068, 2040, 2018 (t-CO); and 1792 ( $\mu$ -CO)).

It is known<sup>6,7</sup> that the acetonitrile ligand in these carbonyl clusters is readily and selectively replaced by other ligands. As expected, at the second stage of the synthesis, the reaction of AllPPh<sub>2</sub> with the compound 3 that formed proceeded under mild conditions at room temperature:

$$Rh_{6}(CO)_{15}MeCN + PPh_{2}CH_{2}CH=CH_{2} \longrightarrow$$

$$[Rh_{6}(CO)_{15}(\eta^{1}-PPh_{2}CH_{2}CH=CH_{2})] + MeCN. (2)$$

$$4$$

The course of reaction (2) was monitored by IR spectroscopy, which demonstrated that intermediate 4 was initially formed. Thus, the carbonyl region of the IR spectrum of the reaction mixture had absorption bands at 2104, 2068, 2039, and 2017 cm<sup>-1</sup> (t-CO) and 1790 cm<sup>-1</sup> ( $\mu$ -CO) corresponding to the monosubstituted derivative

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Fig. 1. Molecular structure of the cluster-containing monomer  $[Rh_6(CO)_{14}(\mu,\eta^2-PPh_2CH_2CH=CH_2)]$ .

Atom	x	у	ζ	$U_{eq}/Å$	Atom	x	У	z	U <sub>eq</sub> /Å
Rh(1)	8227(1)	2317(1)	9027(1)	36(1)	C(5)	8925(6)	614(9)	7456(15)	51(4)
Rh(2)	9512(1)	1806(1)	11520(1)	30(1)	O(5)	8742(6)	98(8)	6766(13)	85(4)
Rh(3)	8488(1)	970(1)	10844(1)	34(1)	C(5')	9968(6)	1296(10)	7808(16)	51(4)
Rh(4)	9225(1)	3136(1)	9715(1)	35(1)	O(5')	10341(4)	1177(8)	7149(11)	73(4)
Rh(5)	9239(1)	1429(1)	8723(1)	33(1)	C(6)	8318(6)	2290(11)	13637(16)	64(5)
Rh(6)	8478(1)	2652(1)	11817(1)	39(1)	O(6)	8228(6)	2065(11)	14741(13)	116(6)
C(11)	9012(6)	2648(8)	7663(14)	43(4)	C(6')	8133(6)	3773(10)	12077(19)	66(5)
0(11)	9055(4)	2896(6)	6550(10)	57(3)	O(61')	7862(15)	4370(21)	12504(54)	157(21
C(22)	7776(6)	1863(10)	10953(16)	58(4)	O(62')	7965(12)	4490(22)	12043(37)	81(10)
O(22)	7301(4)	1793(7)	1193(11)	60(3)	P	10474(1)	2123(2)	11392(4)	37(1)
C(33)	9414(6)	584(9)	10470(15)	48(4)	C(211)	10907(5)	1409(9)	10396(14)	37(3)
O(33)	9620(4)	-134(5)	10556(10)	48(3)	C(212)	11286(5)	1681(10)	9360(16)	55(4)
C(44)	9356(6)	3165(10)	1922(17)	58(4)	C(213)	11593(6)	1073(13)	8667(18)	75(6)
O(44)	9541(4)	3724(6)	12694(12)	62(3)	C(214)	11575(7)	140(11)	8923(16)	67(5)
C(1)	7807(6)	3342(10)	8707(17)	54(4)	C(215)	11194(7)	-110(9)	9915(17)	60(5)
0(1)	7537(4)	3947(7)	8523(14)	77(4)	C(216)	10854(5)	490(9)	10644(15)	46(4)
C(1')	7788(7)	1642(9)	7805(18)	59(5)	C(221)	10876(6)	2240(8)	13023(14)	39(4)
O(1')	7513(5)	1235(7)	7108(13)	78(4)	C(222)	10687(6)	2831(11)	14014(19)	64(5)
C(2)	9612(5)	1359(9)	13304(16)	46(4)	C(223)	10979(8)	2985(11)	15293(20)	74(5)
O(2)	9651(5)	1047(9)	14394(11)	83(4)	C(224)	11489(7)	2530(10)	15466(16)	62(5)
C(3)	8458(6)	332(9)	12545(14)	49(4)	C(225)	11711(6)	1922(11)	14476(18)	65(5)
O(3)	8435(6)	-24(8)	13523(12)	85(4)	C(226)	11378(6)	1775(10)	13257(17)	58(4)
$C(3^{+})$	8116(5)	70(9)	9781(15)	43(4)	C(231)	10543(6)	3207(8)	10551(17)	50(4)
0(3')	7860(4)	-462(7)	9249(13)	68(3)	C(232)	10208(6)	3302(10)	9226(18)	67(5)
C(4)	8919(6)	4301(10)	9617(14)	52(4)	C(233)	9947(7)	4026(10)	8863(19)	63(5)
O(4)	8696(6)	4946(7)	9580(16)	96(5)					

Table 1. Coordinates of nonhydrogen atoms  $(\times 10^4)$  in molecule 2

 $Rh_6(CO)_{16}$ . In addition, a band at 2294 cm<sup>-1</sup> corresponding to the CN stretching vibration of the coordinated acetonitrile molecule disappeared. The presence of

Table 2. Selected bond lengths (d) in molecule 2

Bond	d/Å	Bond	d/Å
Rh(1) - C(1')	1.87(2)	Rh(6) - C(6')	1.907(14)
Rh(1) - C(1)	1.87(2)	Rh(6)-C(22)	2.20(2)
Rh(1) - C(22)	2.23(2)	Rh(6) - C(44)	2.206(14)
Rh(1) - C(11)	2.311(14)	C(11) - O(11)	1.13(2)
Rh(1)-Rh(4)	2.731(2)	C(22) - O(22)	1.14(2)
Rh(1) - Rh(5)	2.747(2)	C(33)O(33)	1.20(2)
Rh(1)-Rh(3)	2.7571(14)	C(44)-O(44)	1.21(2)
Rh(1)-Rh(6)	2.772(2)	C(1) - O(1)	1.13(2)
Rh(2) - C(2)	1.848(14)	C(1') - O(1')	1.11(2)
Rh(2)-C(33)	2.128(14)	C(2)-O(2)	1.15(2)
Rh(2)—C(44)	2.14(2)	C(3)-O(3)	1.08(2)
Rh(2)-P	2.311(3)	C(3')-O(3')	1.13(2)
Rh(2)-Rh(4)	2.7447(14)	C(4)-O(4)	1.11(2)
Rh(2)-Rh(6)	2.763(2)	C(5)-O(5)	1.11(2)
Rh(2) - Rh(3)	2.7943(14)	C(5')O(5')	1.09(2)
Rh(2)—Rh(5)	2.803(2)	C(6)-O(6)	1.13(2)
Rh(3) - C(3)	1.892(14)	C(6')-O(61')	1.18(4)
Rh(3) - C(3')	1.916(14)	C(6')O(62')	1.16(3)
Rh(3) - C(22)	2.16(2)	P-C(211)	1.766(13)
Rh(3)—C(33)	2.277(13)	P-C(221)	1.828(14)
Rh(3)—Rh(6)	2.729(2)	PC(231)	1.845(13)
Rh(3)—Rh(5)	2.7721(14)	C(211) - C(212)	1.39(2)
Rh(4)C(4)	1.92(2)	C(211)—C(216)	1.43(2)
Rh(4)—C(44)	2.13(2)	C(212)-C(213)	1.35(2)
Rh(4)C(11)	2.152(13)	C(213)C(214)	1.45(2)
Rh(4) - C(233)	2.32(2)	C(214)C(215)	1.35(2)
Rh(4) - C(232)	2.366(14)	C(215)—C(216)	1.40(2)
Rh(4) - Rh(6)	2.763(2)	C(221) - C(222)	1.38(2)
Rh(4) - Rh(5)	2.7692(14)	C(221)-C(226)	1.39(2)
Rh(5) - C(5)	1.883(14)	C(222)C(223)	1.42(2)
Rh(5) - C(5')	1.93(2)	C(223) - C(224)	1.39(2)
Rh(5) - C(33)	2.147(14)	C(224)-C(225)	1.42(2)
Rh(5) - C(11)	2.181(12)	C(225) - C(226)	1.42(2)
Rh(6) - C(6)	1.86(2)	C(232)-C(233)	1.31(2)

Table 3. Selected bond angles ( $\omega$ ) in molecule 2

the reactive AllPPh<sub>2</sub> ligand, which exhibits bidentate properties owing to the presence of the double bond, in the coordination sphere of the carbonyl cluster resulted in the formation of the final product 2 with an additional coordination, as has been observed previously<sup>8</sup> in the synthesis of  $[Rh_6(CO)_{14}(\mu_2,\eta^2-PPh_2(CH_2)_2PPh_2)]$ .

Molecular structure of complex 2. The molecular structure of compound 2 is shown in Fig. 1. The atomic coordinates, bond lengths, and bond angles are given in Tables 1-3, respectively. Compound 3 is an octahedral Rh cluster containing ten terminal and four µ3-bridging CO ligands. The average Rh-Rh distance (2.762(2) Å) agrees with the corresponding value in the vinylpyridinesubstituted cluster Rh<sub>6</sub>(CO)<sub>15</sub>(4-VinPy)<sup>3</sup> but is smaller than that in the nonsubstituted cluster<sup>8</sup> as well as those in the triphenylphosphino- and bis(diphenylphosphinoethane)-substituted Rh<sub>6</sub>(CO)<sub>16</sub> clusters<sup>9</sup> (2.774-2.779 Å). The average Rh-C bond lengths in the terminal (1.89(2) Å) and  $\mu_3$ -bridging (2.19(2) Å) CO groups coincide to within the experimental error with the corresponding values determined previously<sup>3</sup> (1.86 and 2.18 Å, respectively). As expected, the carbonyl bond in the terminal group (1.13(2) Å) is shorter than that in the bridging group (1.17(1) Å).

The PPh<sub>2</sub>CH<sub>2</sub>CH=CH<sub>2</sub> ligand is attached to the Rh(2) atom (Rh(2)-P 2.311(3) Å). This ligand is coordinated to the cluster at one more site through the Rh(4)...C=C interaction between the unoccupied d-orbital of the Rh atom and the bonding  $\pi$ -orbital of the double bond (Rh(4)--C(232), 2.37(1); and Rh(4)-C(233), 2.32(2) Å). The charge transfer in complex 2 is insignificant, which leads to a decrease in the electron density at the Rh nucleus and, consequently, to a decrease in the Rh-Rh distance compared to that in the triphenylphosphino-substituted cluster,<sup>9</sup> as mentioned above.

It should be noted that this coordination of vinylphosphine ligands is observed in many carbonyl cluster complexes. Interestingly, this coordination bond can be readily cleaved, for example, upon heating or treatment with CO, as was observed in the case of the

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Angle	ω/deg	Angle	ω/deg	Angle	₀/deg
Rh(4) - Rh(1) - Rh(5)	60.73(4)	Rh(5) - Rh(3) - Rh(2)	60.47(4)	$\frac{1}{Rh(4)-Rh(5)-Rh(2)}$	59.02(3)
Rh(4) - Rh(1) - Rh(3)	89.99(4)	C(233) - Rh(4) - Rh(1)	144.1(4)	Rh(3) - Rh(5) - Rh(2)	60.16(4)
Rh(5) - Rh(1) - Rh(3)	60.48(4)	C(232) - Rh(4) - Rh(1)	146.8(4)	Rh(3) - Rh(6) - Rh(2)	61.17(4)
Rh(4) - Rh(1) - Rh(6)	60.27(4)	C(233) - Rh(4) - Rh(2)	118.2(4)	Rh(3) - Rh(6) - Rh(4)	89.92(4)
Rh(5) - Rh(1) - Rh(6)	90.53(4)	C(232) - Rh(4) - Rh(2)	87.9(4)	Rh(2) - Rh(6) - Rh(4)	59.57(4)
Rh(3) - Rh(1) - Rh(6)	59,14(4)	Rh(1) - Rh(4) - Rh(2)	91.33(4)	Rh(3) - Rh(6) - Rh(1)	60.16(4)
P-Rh(2)-Rh(4)	92,99(9)	C(233) - Rh(4) - Rh(6)	150.9(4)	Rh(2) - Rh(6) - Rh(1)	90.09(4)
P - Rh(2) - Rh(6)	139,92(9)	C(232) - Rh(4) - Rh(6)	142.1(4)	Rh(4) - Rh(6) - Rh(1)	59.13(4)
Rh(4) - Rh(2) - Rh(6)	60.22(4)	Rh(1) - Rh(4) - Rh(6)	60.6Ò(4)	C(211) - P - C(221)	102.8(6)
P - Rh(2) - Rh(3)	157.30(10)	Rh(2) - Rh(4) - Rh(6)	60.21(4)	C(211) - P - C(231)	105.6(6)
Rh(4) - Rh(2) - Rh(3)	88.94(4)	C(233) - Rh(4) - Rh(5)	114.9(4)	C(221) - P - C(231)	103.7(6)
Rh(6) - Rh(2) - Rh(3)	58.81(4)	C(232) - Rh(4) - Rh(5)	91.2(4)	C(211) - P - Rh(2)	117.4(4)
P - Rh(2) - Rh(5)	102.47(10)	Rh(1) - Rh(4) - Rh(5)	59.93(4)	C(221) - P - Rh(2)	118.7(4)
Rh(4) - Rh(2) - Rh(5)	59.88(4)	Rh(2) - Rh(4) - Rh(5)	61.10(4)	C(231) - P - Rh(2)	107.2(5)
Rh(6) - Rh(2) - Rh(5)	89.57(4)	Rh(6) - Rh(4) - Rh(5)	90.26(4)	C(233) - C(232) - C(231)	123.0(2)
Rh(3) - Rh(2) - Rh(5)	59.37(3)	Rh(1) - Rh(5) - Rh(4)	59.35(4)	C(233) - C(232) - Rh(4)	71.8(9)
Rh(6) - Rh(3) - Rh(1)	60.70(4)	Rh(1) - Rh(5) - Rh(3)	59.94(4)	C(231) - C(232) - Rh(4)	109.6(10)
Rh(6) - Rh(3) - Rh(5)	90.92(4)	Rh(4) - Rh(5) - Rh(3)	88.90(4)	C(232) - C(231) - P	114.1(10)
Rh(1) - Rh(3) - Rh(5)	59.58(4)	Rh(1) - Rh(5) - Rh(2)	89.76(4)	C(232) - C(233) - Rh(4)	75.8(9)
Rh(6) - Rh(3) - Rh(2)	60.01(4)	Rh(1) - Rh(3) - Rh(2)	89.74(4)		

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isomers  $[Co_{3}(\mu_{3}-CR)(CO)_{7}(\mu_$ vinylphosphine PPh<sub>2</sub>CH=CH<sub>2</sub>)].<sup>10</sup> Analogous conversions as well as other types of conversions also occur in a solution of compound 2 due, apparently, to activation of the ligand in the coordination sphere of the rhodium atom. Thus, the <sup>1</sup>H NMR spectrum of complex 2 was complicated, which made its interpretation difficult. The preliminary results on liquid-phase copolymerization of 2 with styrene in benzene in the presence of AIBN (2 wt.%) indicate that the multiple bond in the molecule of the cluster-containing monomer retains its ability to undergo cleavage. Conversions, the stability, and the effect of  $\pi$ -coordination of the phosphoalkene ligand on the course of polymerization conversions of the cluster under study will be the subject of independent investigations.

## Experimental

The IR spectra were recorded on a Specord M-80 spectrophotometer in chloroform and on a Perkin--Elmer-325 instrument in KBr pellets. The <sup>1</sup>H NMR spectra were obtained on a Bruker AC-200 spectrometer in CDCl<sub>3</sub>. TLC was performed on Silufol plates (Chemapol, Czech Republic). Column chromatography was carried out on silica gel L (40/100  $\mu$ m) (Chemapol, Czech Republic).

All syntheses were carried out in anhydrous solvents. The initial  $Rh_6(CO)_{16}$  (1) was prepared by boiling a hexane solution of  $Rh_4(CO)_{12}$ , which was synthesized according to a known procedure,  $\Pi$  for 5 h.

Allyldiphenylphosphine  $PPh_2CH_2CH=CH_2$  (AllPPh<sub>2</sub>) was prepared by the reaction of allyl bromide with lithium diphenylphosphide in THF according to a known procedure.<sup>12</sup>

Cluster Rh<sub>6</sub>(CO)<sub>15</sub>NCMe (3). Rh<sub>6</sub>(CO)<sub>16</sub> (0.80 g, 0.75 mmol) was placed into a reactor equipped with an immersed lamp (DRSh 200, a filter for  $\lambda_{irrad} > 300$  nm). Chloroform (250 mL) and acetonitrile (10 mL) were added and the reaction mixture was stirred for 1 h. Then a weak stream of argon was passed through the mixture and the lamp was switched on for 5 min after which the reaction mixture was stirred for 30 min without irradiation (to dissolve the remaining solid  $Rh_6(CO)_{16}$ ) and the lamp was switched on for 5 min once again. The above procedure was repeated two more times. The reaction mixture was filtered and concentrated on a rotary evaporator (the temperature of the bath was no higher than 25 °C) to dryness. Crystals of 3 that precipitated were washed with hexane and dried under an argon atmosphere. Compound 3 was obtained in nearly quantitative yield. The course of the reaction was monitored by TLC (a 100 : 50 : 1 hexane-chloroform-acetonitrile mixture as the eluent) and IR spectroscopy in the carbonyl region. Found (%): C, 18.1; H, 0.2; N, 1.26; Rh, 56.8.  $C_{17}H_3NRh_6O_{15}$  Calculated (%): C, 18.93; H, 0.28; N, 1.30; Rh, 57.24. IR (CHCl<sub>3</sub>), v/cm<sup>-1</sup>: 2294 (CN); 2104, 2068, 2040, 2018 (t-CO); 1792 (µ-CO).

**Complex** [Rh<sub>6</sub>(CO)<sub>14</sub>( $\mu$ , $\eta^2$ -PPh<sub>2</sub>CH<sub>2</sub>CH=CH<sub>2</sub>)] (2). A solution of PPh<sub>2</sub>CH<sub>2</sub>CH=CH<sub>2</sub> (0.198 g, 0.88 mmol) in chloroform was added dropwise to an intensively stirring solution of compound 3 (0.733 g, 0.68 mmol) in chloroform. The reaction mixture was additionally stirred for 10 min, diluted with hexane, and transferred to a chromatographic column (4×15 cm) with silica gel. A 2:3 hexane—chloroform mixture was used for chromatography. Trace amounts of 3 and the

major band (2) were obtained in that order of elution. Found (%): C, 27.51; H, 1.48; P, 2.47; Rh, 48.6.  $C_{29}H_{15}PRh_6O_{14}$ . Calculated (%): C, 28.18; H, 1.22; P, 2.51; Rh, 49.96. IR (CHCl<sub>3</sub>), v/cm<sup>-1</sup>: 2092 m, 2055 s, 2040, 2018 (t-CO), and 1790 ( $\mu$ -CO); 1622 m (C=C); 1436 w, 1485 w, 1001 w (P-C).

X-ray diffraction study of compound 2. Colorless crystals of 2 are monoclinic. The principal crystallographic parameters are as follows: M = 1235.84, a = 23.454(5) Å, b = 15.250(3) Å, c = 9.538(2) Å, Z = 4,  $d_{calc} = 2.406(2)$  g cm<sup>-3</sup>, V = 3411.5(12) Å<sup>3</sup>, C<sub>29</sub>H<sub>15</sub>O<sub>14</sub>PRh<sub>6</sub>,  $\mu = 29$  cm<sup>-1</sup>, space group  $P2_12_12_1$ .

The X-ray diffraction data were collected from a crystal of 2 of dimensions  $0.20 \times 0.10 \times 0.20 \text{ mm}^3$  on an automated fourcircle KM-4 diffractometer (Mo-K $\alpha$  radiation). A total of 1709 reflections with  $I > 2\sigma(I)$  were measured. The final values of the R factors were as follows:  $R_f = 0.030$ , wR = 0.072, S = 1.12.

The structure was solved by the direct method with the use of the SHELX-86 program package<sup>13</sup> and refined anisotropically by the full-matrix least-squares method (SHELXL-93).<sup>14</sup> The O(6) atom at the terminal CO group was disordered over two sites with occupancies of 0.55 (O(61)) and 0.56 (O(62)).

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