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Cyclophosphites from Oligomethylenephenols

E. E. Nifant'ev, A. T. Teleshev, and A. A. Zhdanov

Moscow State Pedagogical University, Moscow, Russia

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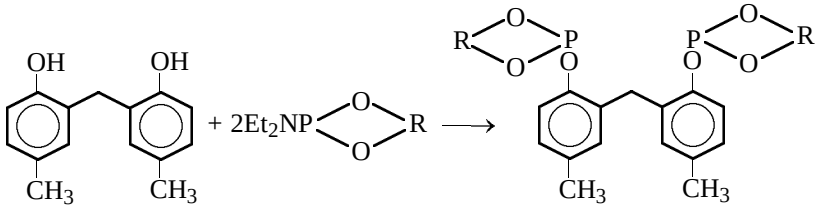
Abstract — New types of oligomethylenephenol cyclophosphites containing 1–3 phosphorus atoms are synthesized by reactions of available oligomethylenephenols with phosphorous acid amides. Contrary to their simple analogs, the resulting phosphites are stable on handling, which allowed design on their basis of complex coordination systems holding promise for metal complex catalysis.

Nowadays phosphorylated oligomethylenephenols attract both theoretical [1] and practical interest [2]. Proceeding with our previous work in this field [3], here we synthesized from available diols and triols, such as 2,2'-dihydroxy-5,5'-dimethyl-1,1'-diphenylmethane (**I**) and 4-methyl-2,6-bis(2-hydroxy-5-methylbenzyl)phenol (**II**), a new type of phosphites and studied their principal chemical properties. Contrary to diol **I**, triol **II** has the third arylene cycle, and, hence, the second methylene bridge, which imparts additional conformational lability to this compound. From published data it follows that such structures allowed synthesis of catalytic systems with enhanced activity, for example, in hydroformylation reactions [4].

It should be noted that some diphosphites derived from diphenols whose phenyl rings are connected by alkylene bridges were prepared by Pastor *et al*. [5] by means of the acid chloride procedure, but the reaction products were contaminated with amine hydrochorides.

In the present work we phosphorylated diol **I** and triol **II** with phosphoroamidites, such as propanediyl diethylphosphoramidite and 2,2-dimethylpropanediyl diethylphosphoramidite, as well as hexaethylphosphorous triamide. Using phosphorous amides enabled us to obtain purer phosphorylation products. In addition, phosphorous amides more convenient for experimentation.

Phosphorylation of diol **I** occurred under mild conditions: at 20–25°C for 20 h and at 130°C for 30–50 min.



I **IIIa, IIIb**

R = (**a**), (**b**).

The contents of diphosphites **IIIa** and **IIIb** in the postreaction mixtures were close to quantitative. The ^{31}P NMR spectra of the postreaction mixtures contained no other signals as singlets at 124 (**IIIa**) and 115 ppm (**IIIb**), characteristic of compounds having dioxaphosphorinane cycles with aromatic substituents [6–8]. We failed to distill diphosphites **IIIa** and **IIIb** in a vacuum and isolated them by reprecipitation with hexane from dioxane or ether solutions.

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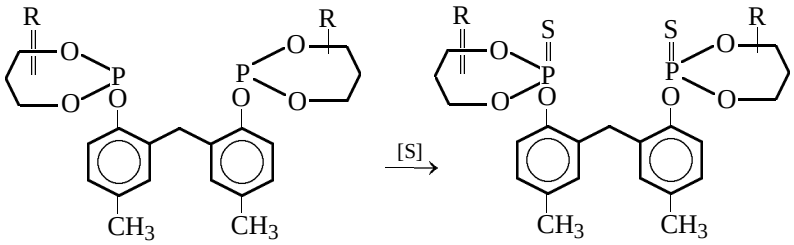
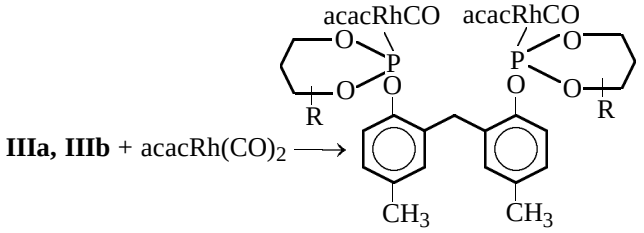


IIIa, IIIb **IVa, IVb**

Sulfurization of compounds **IIIa** and **IIIb** occurs at a temperature of no higher than 100°C, and the target products are formed in high yields. They are either oils [compound **IVa**] or amorphous powders [compound **IVb**]. The ^{1}H NMR spectra of compounds **IIIa, IIIb, IVa**, and **IVb** contain one set of signals from each proton group. The IR spectra of these products gave evidence for the absence of free hydroxy groups.

Phosphites **IIIa** and **IIIb** were studied as complex-forming agents in reaction with acacRh(CO)$_2$. The complex formation was carried out at 1:2 or 1:1 ligand-to-metal ratios. The reactions of diphosphites **IIIa** and **IIIb** with acacRh(CO)$_2$ at a 1:2 in ligand-to-metal molar ratio give monosubstituted complexes **Va** and **Vb** containing one metal atom per each phosphorus center. The reactions were accompanied by vigorous gas evolution. The P–Rh coupling constant in the ^{31}P NMR spectra of the resulting complexes was 284.1 Hz, δ_P 118.0 ppm, coordination shift $\Delta\delta_P$ –2 ppm, which is characteristic of square-planar complexes of rhodium with phosphite ligands [9].

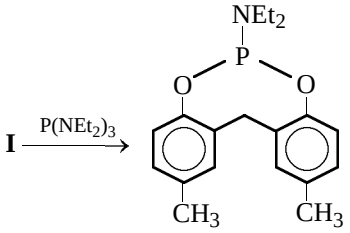


Va, Vb

The absorption band of the carbonyl group at the Rh atom in the IR spectrum (2000 cm^{-1}) confirms the composition of the complex. It was isolated by precipitation with hexane from methylene chloride solution as yellow fine crystals.

When the complex formation was carried out at a 1:1 ligand-to-metal molar ratio, the ^{31}P NMR spectrum contained a signal at δ_P 118.0 ppm (J_{PRh} 284.1 Hz) and one more signal at δ_P –15 ppm, characteristic of phosphates. The intensity ratio of these signals was 1:1. This fact suggests that the complex formation involves one phosphorus center, and the complex that forms works as a catalytic system for oxidation of the second phosphorus atom of this molecule.

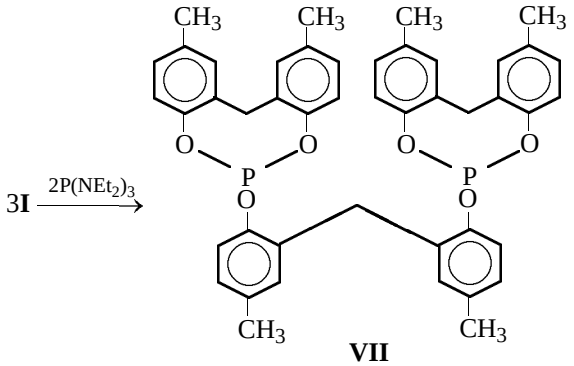
Phosphorylation of diol **I** with hexaethylphosphorous triamide at a temperature of no higher than 50°C gives, independently of the reagent ratio, a single product, 6-(diethylamino)-2,10-dimethyl-12*H*-dibenzo[*d*,*g*]-1,3,2-dioxaphosphocine (**VI**).



VI

The content of cyclic phosphoramidite **VI** in the postreaction mixture is close to quantitative. The ^{31}P NMR spectrum of the postreaction mixture contained only a singlet at δ_P 143.5 ppm, i.e. in a range characteristic of compounds containing amidophosphocane cycles with aromatic substituents [10–12].

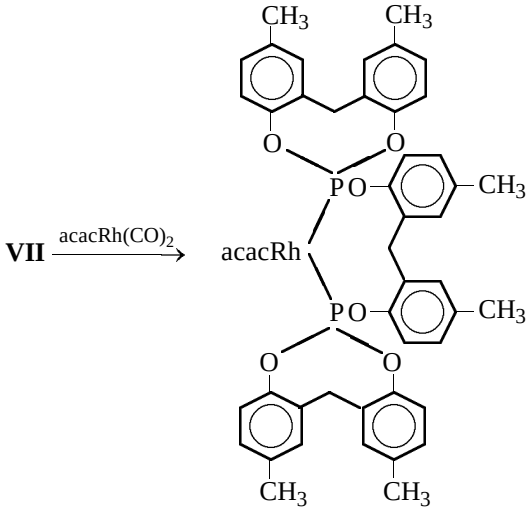
High-vacuum distillation gave compound **VI** in 60% yield. This product is a white crystalline substance. Its structure and composition were proved by physicochemical methods. Compound **VI** is very stable even in air. It does not oxidize or hydrolyze during 100 h at 20°C, and thus can be used as a phosphorylating agent. Phosphorylation of diol **I** with hexaethylphosphorous triamide at 55–80°C and a 3:2 molar ratio gave 2,2'-[2,2'-methylenebis(4-methyl-1-phenoxy)]bis[2,10-dimethyl-12*H*-dibenzo[*d*,*g*]-1,3,2-dioxaphosphocine] (**VII**). The reaction was complete in 8 h at 80°C. The ^{31}P NMR spectrum of the postreaction mixture contained only a singlet at δ_P 118.8 ppm.



VII

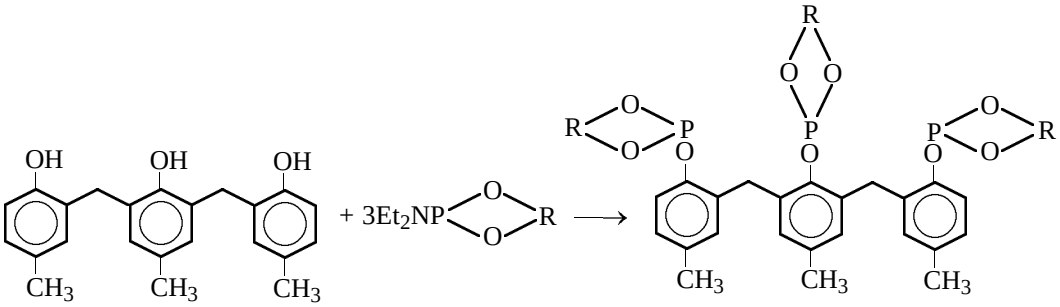
Compound **VII** is very stable. Under an inert atmosphere it may be stored up to 8 months, which is confirmed by the stability of its spectral characteristics (δ_P 118.8 ppm).

In terms of organometallic synthesis, using P(III) oligomethylenephenols as a ligand surrounding is absolutely urgent, because, being highly active in complex formation [presence of P(III)], they are also very stable (to oxidation and hydrolysis) ligands and impart stability to their complexes. The complex formations with diphosphite **VII** were carried out at 1:2 and 1:1 reagent molar ratios. It was found that, independently of the ligand-to-metal molar ratio, the reactions result in exclusive formation of chelate **VIII**. It is a yellow finely crystalline powder with a high decomposition point (174–176°C). Chelate **VIII** was isolated by reprecipitation with hexane from a methylene chloride solution. Its ^{31}P NMR spectrum contains a doublet at δ_P 125.0 ppm (J_{PRh} 302.0 Hz, $\Delta\delta$ 9.3 ppm), which is characteristic of complexes with phosphite ligands [9]. The observed characteristics suggest a chelate-type complex formation between acacRh(CO)$_2$ and compound **VII**. The IR spectrum of compound **VIII** lacked a characteristic carbonyl absorption band at 2000 cm^{-1}, providing evidence for chelation.



VIII

Phosphorylation of triol **II** with cyclic phosphorous acid amides proceeds under mild conditions at 20–25°C.

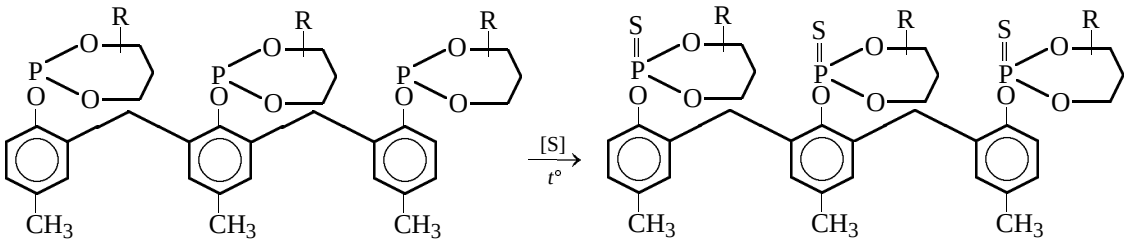


II **IXa, IXb**

R = (**a**), (**b**).

The contents of triphosphites **IXa** and **IXb** in the postreaction mixtures were close to quantitative. The ^{31}P NMR spectra of these mixtures contained two signals at 119.3 and 115.0 ppm, characteristic of compounds having dioxaphosphorinane cycles with aromatic substituents. Note that the integral intensity ratio of the signals of two terminal phosphorus centers and the internal one is 2:1.

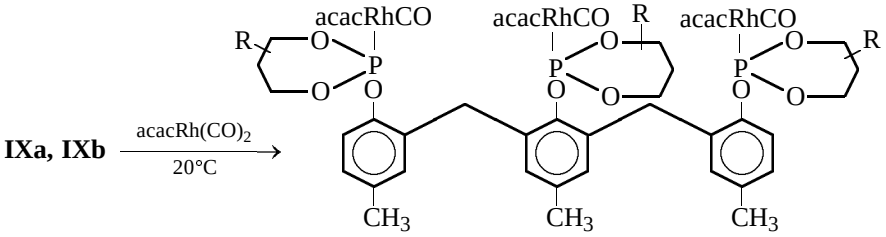
Sulfurization of phosphites **IXa IXb** gave corres-



IXa, IXb **Xa, Xb**

ponding thio derivatives **X**. It is noteworthy that in the ^{31}P NMR spectrum of compounds **Xa** and **Xb** the integral intensity ratio of the signals belonging to internal and terminal phosphorus centers remains equal to 1:2. The composition, structure, and individuality of the obtained compounds were confirmed by physicochemical methods.

The reaction Rh(acac)$(CO)_2$ with 2,6-bis(5,5-dimethyl-1,3,2-dioxaphosphorinan-2-yl)-1-[2-(5,5-dimethyl-1,3,2-dioxaphosphorinan-2-yloxy)-5-methylbenzyl]-4-methylbenzene (**IXb**) at a 1:3 ligand-to-metal molar ratio was accompanied by gas evolution. Reprecipitation with hexane from methylene chloride solution gave a complex whose ^{31}P NMR pectrum contained two doublets at δ_P 119.4 (terminal) and 123.2 ppm (internal). The integral intensity ratio of these signals was 2:1. The presence of two doublets in the ^{31}P NMR spectrum is explained by that ligands **IXa** and **IXb** contain three prosphorus centers: two terminal and one internal. The Rh–P coupling constants for the doublets belonging to terminal and internal centers are 282.9 and 281.9 Hz, respectively. The coordination shifts $\Delta\delta_P^{term}$ and $\Delta\delta_P^{int}$ are 8.7 and 8.6 ppm, which is characteristic of complexes with phosphite ligands [9]. The above characteristics suggest substitution of one of the carbonyl groups in acacRh(CO_2) by the phosphorus-containing ligand.



XIa, XIb

The IR spectrum contains a characteristic carbonyl absorption band at 1998 cm^{-1}, which implies that only one CO group is substituted. Complex **XI** is an amorphous yellow powder (decomp. point 115–118°C). The reactions of compounds **IXa** and **IXb** with acacRh$(CO)_2$ at 1:2 ligand-to-metal ratios involved no chelate formation. Therewith, the ^{31}P NMR spectrum contained a phosphate signal at δ_P –15 ppm. This is explained by the fact the resulting complex catalyzes oxidation of phosphorus centers which have not taken part in complex formation with acacRh$(CO)_2$.

Hence, we succeeded in extending the range of promising ligands for complex formation. Note that the described ligands and complexes derived from them are stable compounds.

EXPERIMENTAL

The IR spectra of compounds **IIIa, IIIb, Va, Vb**–**IXa, IXb, XIa**, and **XIb** were recorded on a Specord IR-75 spectrometer for methylene chloride solutions in NaCl cells. The ^1H NMR spectra of compounds **I, II, IIIa, IIIb, VI, VII, IXa**, and **IXb** were recorded on a Bruker WP-250 spectrometer (250 MHz) in

$CDCl_3$ against TMS. The ^{31}P NMR spectra of compounds **IIIa, IIIb, IVa, IVb, Va, Vb, VI, VII, VIII, IXa, IXb, XIa**, and **IXb** methylene chloride were recorded on a Bruker WP-80SY spectrometer (32.4 MHz) against external 85% phosphoric acid.

All manipulations were carried out under argon. Solvents were purified according to known procedures [13]. TLC was carried out on Silufol plates, eluents 1:1 ether–hexane (A), 1:1 dioxane–benzene (B), 1:1 benzene–hexane (C), 1:2 hexane–ether (D), and 2:1 benzene–hexane (E). Development by iodine vapor or silver nitrate, or by calcination.

2,2'-Dihydroxy-5,5'-dimethyl-1,1'-diphenylmethane (I). *p*-Cresol, 100 g, and 25 g of a 30% formaldehyde solution was heated at 50°C for 2 h in the presence of 2.5 ml of concentrated hydrochloric acid. Then the reaction mixture was cooled to room temperature and mixed with 150 ml of benzene. The precipitate that formed was filtered off, and the filtrate was distilled. After removal of the solvent, water, and excess *p*-cresol, 2,2'-dihydroxy-5,5'-dimethyl-1,1'-diphenylmethane was distilled at 240°C (15 mm), mp 125–126°C, R_f 0.61 (A), 0.5 (B). Yield 15%. ^1H NMR spectrum ($CDCl_3$), δ, ppm: 2.15 s (6H, CH_3), 3.76 s (2H, CH_2), 6.65 d (2H, Ar), 6.8 q (2H, Ar), 7.1 s (2H, Ar), 7.4 s (2H, OH).

2,2'-Bis(1,3,2-dioxaphosphorinan-2-yloxy)-5,5'-dimethyl-1,1'-diphenylmethane (IIIa). Diol **I**, 2 g, was treated with 3.2 g of 2-diethylamino-1,3,2-dioxaphosphorinane and heated at 130°C for 30 min (or at 20–25°C for 20 h). The evolving diethylamine was removed in a vacuum. After cooling, the reaction mixture was dissolved in dioxane or diethyl ether, and the product was precipitated with hexane. The precipitate was filtered off and dried at 40°C (15 mm). Yield 3.06 g (73%), mp 78–80°C, R_f 0.86 (B), δ_P 123.5 ppm. ^1H NMR spectrum, δ, ppm: 0.82 s, 2.22 s (6H, CH_3–Ar), 3.35 m (4H, $C^{4,6}H_a$), 3.82 s (2H, CH_2), 4.2 m (4H, $C^{4,6}H_e$), 6.85 m (4H, Ar), 7.0 s (2H, Ar). Found, %: C 56.84; H 5.71; P 14.1. $C_{21}H_{26}O_6P_2$. Calculated, %: C 57.79; H 5.96; P 14.22.

2,2'-Bis(5,5-dimethyl-1,3,2-dioxaphosphorinan-2-yloxy)-5,5'-dimethyl-1,1'-diphenylmethane (IIIb). Diol **I**, 2 g, was treated with 3.6 g of phosphamide and heated at 130°C for 30 min. The evolving diethylamine was removed in a vacuum. After cooling, the reaction mixture was dissolved in diethyl ether and filtered. The solvent was removed from the filtrate, and the residue was dried at 40°C (15 mm). Yield 3.06 g (71%), mp 82–83°C, R_f 0.49 (B), δ_P 115.3 ppm. ^1H NMR spectrum, δ, ppm: 0.82 s, 1.26 s (12H, CH_3–c), 2.22 s (6H, CH_3–Ar), 3.35 m (4H, $C^{4,6}H_a$), 3.82 s (2H, CH_2), 4.2 m (4H, $C^{4,6}H_e$), 6.85 m (4H, CH_2–Ar), 7.0 s (2H, CH_2–Ar). Found, %: C 61.18, H 6.51; P 12.60. $C_{25}H_{32}O_6P_2$. Calculated, %: C 61.22; H 6.53; P 12.65.

6-(Diethylamino)-2,10-dimethyl-12*H*-dibenzo[*d,g*]-1,3,2-dioxaphosphocine (VI). A mixture of diol **I**, 6.6 g, and 7.15 g of hexaethylphosphorous triamide was heated without solvent at 50°C in a vacuum (15 mm) for 40 min. The product was isolated by distillation, bp 215°C (5 mm), white solid, yield 60%, mp 89–91°C, R_f 0.5 (C). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 1.25 t (6H, NCH_3CH_3), 2.3 s (6H, Ar–CH_3), 3.333.5 m (4H, NCH_2CH_3), 3.4 q (2H, CH_2), 4.3 q (2H, CH_2), 6.8–7.0 m (4H, Ar), 7.1 s (2H, Ar). ^{31}P NMR spectrum (benzene): δ_P 143.5 ppm. Found, %: C 68.4; H 7.01; P 8.98. $C_{19}H_{24}NO_2P$. Calculated, %: C 69.3; H 7.29; P 9.42.

2,2'-[2,2'-Methylenebis(4-methyl-1-phenoxy)]bis-[2,10-dimethyl-12*H*-dibenzo[*d,g*]-1,3,2-dioxaphosphocine] (VII). A mixture of 5 g of diol **I**, 3.61 g of hexaethylphosphorous triamide, and 3 ml of benzene was heated at 80°C for 8 h. After cooling, white crystals precipitated. Yield 81%, mp 178–180°C, R_f 0.65 (C). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 2.22 s (18H, CH_3–Ar), 3.8 s (6H, CH_2), 6.68 s (6H, Ar), 6.7 s (6H, Ar); 6.8–6.9 q (6H, Ar). ^{31}P NMR spectrum: δ_P 118.8 ppm. Found, %: C 71.8; H 5.45; P 8.02. $C_{45}H_{42}O_6P$. Calculated, %: C 72.9; H 5.67; P 8.37.

2,2'-[2,2'-Methylenebis(4-methyl-1-phenoxy)]bis-(5,5-dimethyl-1,3,2λ^5-dioxaphosphorinane 2-sulfide) (IVb). A mixture of 0.1 g of compound **IIIb** and 0.013 g of sulfur was heated for 10 h at 80°C. After cooling, the reaction mixture was dissolved in diethyl ether, the solution was filtered, and the solvent was removed. Yield 0.071g (71%), R_f 0.68 (B). ^1H NMR spectrum, δ, ppm: 0.82 s, 1.26 s (12H, CH_3–c), 2.22 s (6H, CH_3–Ar), 3.35 m (4H, $C^{4,6}H_a$), 3.82 s (2H, CH_2), 4.2 m (4H, $C^{4,6}H_e$), 6.85 m (4H, CH_2–Ar), 7.0 s (2H, CH_2–Ar). ^{31}P NMR spectrum: δ_P 53.94 ppm. Found,%: C 53.1; H 5.8; P 10.7; S 11.1. $C_{25}H_{32}O_6P_2S_2$. Calculated,%: C 54.1, H 5.7; P 11.2; S 11.5.

2,6-Bis(1,3,2-dioxaphosphorinan-2-yl)-1-[2-(1,3,2-dioxaphosphorinan-2-yloxy)-5-methylbenzyl]-4-methylbenzene (IXa). A mixture of 2 g of triol **II**, 3.05 g of 2-(diethylamino)-1,3,2-dioxaphosphorinane, and 5 ml of benzene was kept in a vacuum for 4 h at 25°C. The solvent was then removed to leave a viscous oil, yield 3.33 g (88%), R_f 0.89 (A). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 2.24 d (9H, Ar–CH_3), 3.82 s (4H, Ar–CH_2), 3.94 m (9H, CH_{2e}, $^2J_{H_aH_e}$ 10.67 Hz), $^3J_{H_aP}$ 4.1 Hz, $^3J_{H_eP}$ 4.3 Hz), 4.15 m (9H, CH_{2a}), 6.67 d (3H, Ar), 6.85 d (2H,

Ar), 7.05 s (3H, Ar). ^{31}P NMR spectrum, δ_P, ppm: 119.3 (internal), 115.0 (terminal); internal/terminal signal ratio 1 : 2.

2,6-Bis(5,5-dimethyl-1,3,2-dioxaphosphorinan-2-yl)-1-[2-(5,5-dimethyl-1,3,2-dioxaphosphorinan-2-yloxy)-5-methylbenzyl]-4-methylbenzene (IXb). A mixture of 1 g of triol **II** and 2.28 g of 2,2-dimethylpropanediyl diethylphosphoramidite was kept in a vacuum (15 mm) for 4 h at 80°C to obtain a viscous oil, yield 1.72 g (87%), R_f 0.69 (A). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 0.82 s (9H, CH_3), 1.26 s (9H, CH_3), 2.24 d (9H, Ar–CH_3), 3.82 s (4H, Ar–CH_2), 3.94 m (9H, CH_{2e}, $^2J_{H_aH_e}$ 10.67 Hz, $^3J_{H_aP}$ 4.1 Hz, $^3J_{H_eP}$ 4.3 Hz), 4.15 m (9H, CH_{2a}), 6.67 d (3H, Ar), 6.85 d (2H, Ar), 7.05 s (3H, Ar). ^{31}P NMR spectrum, δ_P, ppm: 119.3 (internal), 115.0 (terminal); internal/terminal signal ratio 1:2. Found, %: C 60.2; H 6.72; P 11.8. $C_{38}H_{51}O_9P_3$. Calculated, %: C 61.2; H 6.85; P 12.5.

2,6-Bis(5,5-dimethyl-2-thioxo-1,3,2λ^5-dioxaphosphorinan-2-yl)-1-[2-(5,5-dimethyl-2-thioxo-1,3,2-dioxaphosphorinan-2-yloxy)-5-methylbenzyl]-4-methylbenzene (Xb). A mixture of 1 g of **IXb** and 0.129 g of sulfur was kept at 80°C for 10 h to obtain a viscous oil, yield 0.98 g (87%), R_f 0.79 (A). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 0.82 s (9H, CH_3), 1.26 s (9H, CH_3), 1.26 s (9H, CH_3), 2.24 d (9H, Ar–CH_3), 3.82 s (4H, Ar–CH_2), 3.94 m (9H, CH_{2e}, $^2J_{H_aH_e}$ 10.67 Hz, $^3J_{H_aP}$ 4.1 Hz), 4.15 m (9H, CH_{2a}), 6.67 d (3H, Ar), 6.85 d (2H, Ar); 7.05 s (3H, Ar). ^{31}P NMR spectrum, δ_P, ppm: 55.8 (internal), 53.4 (terminal), internal/terminal signal ratio 1 : 2. Found, %: C 53.2; H 5.9; P 10.8; S 10.4. $C_{38}H_{51}O_9P_3S_3$. Calculated, %: C 54.2; H 6.0; P 11.0, S 11.4.

Complex Vb. Dicarbonylrhodium acetylacetonate, 0.0929 g, in 5 ml of methylene chloride was treated with 0.08858 g of compound **IIIb**. When CO_2 no longer evolved, the solvent was removed, and the product was precipitated with hexane. Yield 0.14 g (82%), yellow powder, decomp. point 105–108°C, R_f 0.43 (A). IR spectrum: ν(CO) 1990 cm^{-1}. ^{31}P NMR spectrum: δ_P 119.3 ppm (J_{PRh} 287.0 Hz).

[2,2′-[2,2′-Methylenebis(4-methyl-1-phenoxy)]-bis[2,10-dimethyl-12*H*-dibenzo[*d,g*]-1,3,2-dioxaphosphocin-2-yl]]rhodium acetyacetonate (VIII). Compound **VII**, 0.286 g, was added to a mixture of 0.1 g of dicarbonylrhodium acetylacetonate and 5 ml of benzene. When CO no longer evolved, the solvent was removed, and the target compound was precipitated with hexane. Yield 0.3 g (84%), yellow powder, decomp. point 174–176°C. ^{31}P NMR spectrum: δ_P 125.0 ppm (J_{PRh} 302.6 Hz).

Complex XIb. To a mixture of 0.174 g of dicarbonylrhodium acetylacetonate and 5 ml of benzene, 0.168 g of compound **IXb** was added. When CO no longer evolved, the solvent was removed, and the target compound was precipitated with hexane. Yield 0.262 g (81%), yellow powder, decomp. point 115–117°C. IR spectrum: ν(CO) 1980 cm^{-1}. ^{31}P NMR spectrum, δ_P, ppm: 123.1 (internal), 119.3 (terminal) (J_{PRh} 281.9 Hz), internal/terminal signal ratio 1 : 2.

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