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Cyclo[bis(1,7-naphthylene-dialkylamidophosphites)]

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ABSTRACT: *The possibility of regioselective cyclobisphosphorylation of nonsymmetrical bisphenols was shown for the 1,7-dihydroxynaphthalene–phosphorous acid triamide system. The structure and essential chemical properties of the first nonsymmetrical phosphacyclophane were studied. New data demonstrating peculiar features of bisphenol diamidophosphites were obtained.* © 2003 Wiley Periodicals, Inc. Heteroatom Chem 14:404–412, 2003; Published online in Wiley InterScience (www.interscience.wiley.com). DOI 10.1002/hc.10143

INTRODUCTION

Recently we obtained the first cavitand systems containing two phenylene, biphenylene [1], or naphthylene residues [2] linked by phosphorus bridges. These original compounds belong to the class of cyclophanes and they were named phosphacyclophanes. They were synthesized by cyclobisphosphorylation of corresponding symmetrical bisphenols with available phosphorous acid triamides. The aim of this work was to perform the regioselective cyclobisphosphorylation of nonsymmetrical bisphenols and to study the structure and the main chemical properties of resulting compounds.

RESULTS AND DISCUSSION

We studied the reaction between phosphorous hexaalkyltriamide (**1**) and 1,7-dihydroxynaphthalene (**2**), whose molecules include α- and β-hydroxyl groups in different aromatic rings. We used two methods for the synthesis of cyclic systems.

The first method was molecular assemblage. This is a two-step technique, involving 1,7-naphthylenebisphosphorylated derivatives (**3**) and their reaction with the equimolar amount of diol **2** resulting in the formation of target products **4**. The reaction proceeded in acetonitrile at room temperature without removal of the amine formed. Intermediate bisphosphorylated naphthodiols (**3a–d**) were viscous oils. Their ^{31}P NMR spectra exhibited two singlets with similar integral intensities in the range typical for diamidophosphites (133–136 ppm); the chemical shift difference was 3 ppm on the average. Freshly prepared bisphosphites **3a–d** were treated with the equimolecular amount of **2**. Shortly after the addition of **2**, a mixture of crystalline (**4′**) and oily (**4″**) compounds differing in physicochemical and structural properties began to precipitate. Crystalline compounds **4′** were isolated with yields of 48–50%. These products resulted from regioselective cyclobisphosphorylation, during which two

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amidophosphite bridges linked the α and β positions of two naphthalene nuclei (for properties and structure of **4′** see Scheme 1). Oily products **4″** were isomeric systems formed via the α-, α- and β-, β-linking of naphthalene nuclei (Scheme 1).

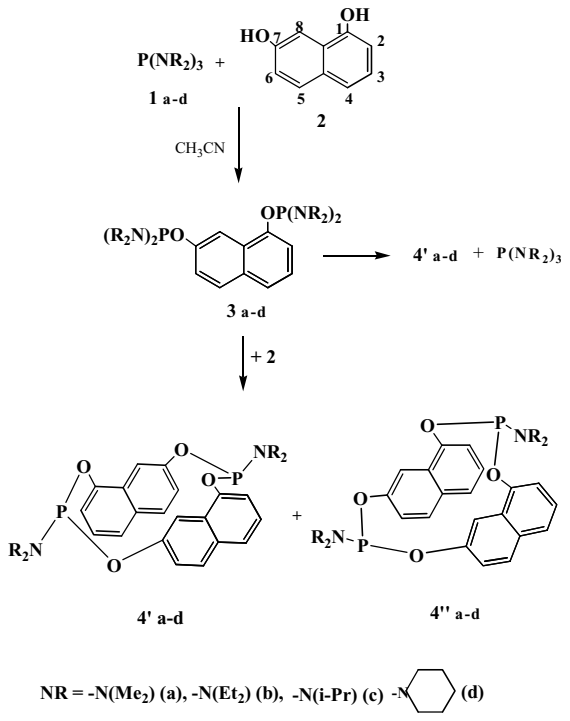
We noted that intermediate 1,7-naphthylenebis(dialkylamido)phosphites **3a–d** were unstable compounds. When they were left in acetonitrile, a precipitate composed of 80% cyclophosphite **4′** was formed and phosphorous triamides **1** appeared in the solution. The spontaneous dismutation of compounds **3** could be used as an alternative synthetic method (Scheme 1).

The dismutation comes to the end in 4–5 weeks; its rate decreases continually because of decreasing reagent concentrations. It is important that the slow reaction ensures regioselectivity because the slow approach of naphthalene nuclei creates better conditions for their mutual symmetric orientation. We emphasize that the dismutation of phosphorous acid derivatives is well known, but no evidence is available in the literature for its purposeful use in the regioselective synthesis. Thus, the reaction performed is of principal importance.

The structure and spatial organization of synthesized cyclobis-1,7-naphthylenedialkylamidophosphites **4′** were studied using NMR spectroscopy, X-ray diffraction analysis, and molecular mass determination. It was found that the ^{31}P NMR spectra of all products exhibited a resonance in the range typical for monoamidophosphites, but the spectra slightly differed in character. Two narrow singlets with $\delta = 135.05$ and 135.59 ppm and similar integral intensities were observed in the spectrum of cyclophosphite containing the N—Me substituent at the phosphorus atom (**4′a**); a narrow singlet was found in the spectrum of **4′c** containing the N—iPr substituent, and broadened signals were observed for cyclophosphites with the N—Et (**4′b**) and piperidyl (**4′d**) substituents. We believe that the observed differences depend on the peculiar features of conformational transitions in these systems.

The X-ray diffraction analysis of compound **4′a** shows that it is a cycloamidophosphite in the 1,7,1,7-isomeric form (Fig. 1, Table 1). The molecule is totally symmetric and has a benched structure. It represents an antiisomer with the chair–bath conformation of bridging fragments. The O^1PO^2 angle is 93.8°; the angle between the aromatic planes is 11° and the distance between two nearest hydrogen atoms directed inward of the cycle is 2.14 Å.

Oily products **4″a–d** were separated from the total precipitate by repeated washing with benzene followed by evaporation of the solution and drying of the residue in vacuo. They represent low-melting amorphous compounds. Their ^{31}P NMR spectra exhibited two singlets at $\delta = 139.1$ and 139.4 (**4″a**) or 140.0 and 140.9 ppm (**4″b**) with similar integral intensities and a broadened signal at 143.0 or 135.3 ppm (**4″c** and **4″d**, respectively). In our opinion, this points to the inequivalence of phosphorus atoms and the dissymmetry of the molecule. Unfortunately, only a minor part of **4′** passed into the benzene solution



SCHEME 1

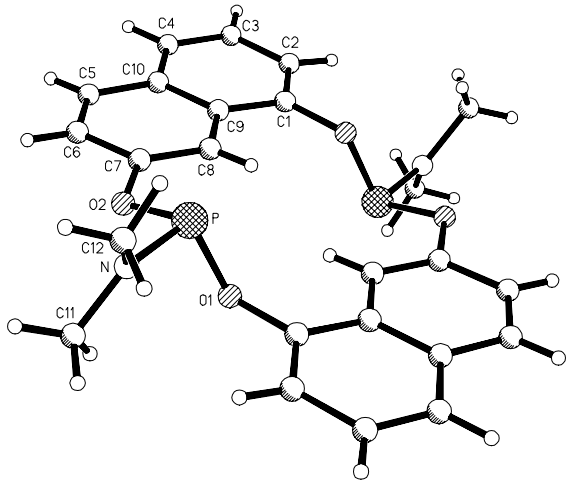


FIGURE 1 Molecular structure of **4′a**.

TABLE 1 Bond Lengths (Å) and Angles (degrees) for **4′a**

P—N	1.644(2)	NP O^2	97.29(9)
P—O^2	1.651(2)	NP O^1	103.37(9)
P—O^1	1.668(2)	O^2PO^1	93.77(8)
O^1—$C^{1'}$	1.391(2)	C^7O^2P	122.92(12)
O^2—C^7	1.381(2)	$C^{1'}O^1P$	117.35(12)
N—C^{12}	1.4403	$C^2C^1O^{1'}$	121.14(19)
N—C^{11}	1.456(3)	$O^{1'}C^1C^9$	118.17(16)
C^1—C^2	1.366(3)		
C^1—C^9	1.421(3)		
C^2—C^3	1.399(4)		
C^3—C^4	1.355(3)		
C^4—C^{10}	1.414(3)		
C^5—C^6	1.358(3)		
C^5—C^{10}	1.415(3)		
C^6—C^7	1.400(3)		
C^7—C^8	1.362(3)		
C^8—C^9	1.411(3)		
C^9—C^{10}	1.411(3)		

Symmetry transformations used to generate equivalent atoms: $-x+1, -y+1, -z+1$.

during the separation of **4″** from **4′**, which could be observed only in the ^{1}H NMR spectra. This gave no way to completely characterize the **4″** isomers.

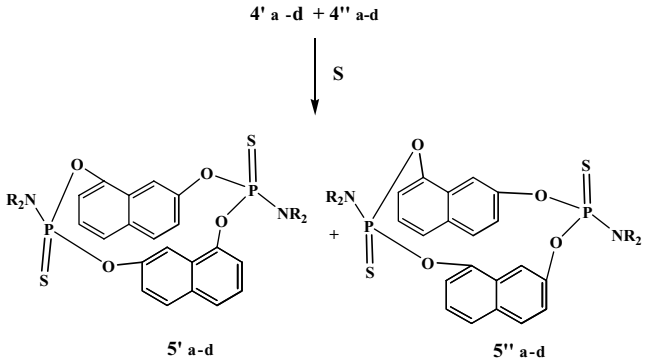
In this work, we continued to study the main chemical properties of this cyclophosphite type. Our interest was focused on the reactions proceeding at the trivalent phosphorus atom (sulfurization, oxidation, and complexation). We used the mixture of isomers **4′a–d** and **4″a–d** for sulfurization (Scheme 2). Oxidation and complexation were studied with pure 1,7,1,7-isomers of **4′a,b**.

Sulfurization proceeded easily at room temperature. The reaction products were separated by column chromatography. Thionophosphates **5′a–d** were high-melting crystalline compounds. Their ^{31}P NMR spectra exhibited narrow singlets in the thionophosphate range. The difference in the spectral characteristics of cyclophosphites (**4′a–d**) and -thionophosphates (**5′a–d**) can be related to the increase in molecular rigidity during sulfurization, and hence the loss in tendency for conformer transitions. The ^{1}H NMR spectra of compounds **5′a–d** completely corresponded to the structure indicated. In addition, the X-ray diffraction analysis of thionophosphate **5′b** showed that the configuration of its molecule hardly differs from that of cyclobisphosphite (Fig. 2, Table 2). The O^1PO^2 angle is 97.7°, compared to 100° in diarylamidothionophosphate [3]. The distance between planes in the molecule increases by 0.5 Å, and the distance between two nearest hydrogen atoms directed inward of the cycle decreases as compared to the cyclobisphosphite structure.

Compounds **5″** isolated by column chromatography were amorphous powders. Their melting points were lower than those of **5′** isomers. Their ^{31}P NMR spectra exhibited two narrow singlets with similar integral intensities in the range typical for thionophosphates, indicating the structural inequivalence of the phosphorus atoms. A broadening of signals from all groups of protons is observed in the ^{1}H NMR spectra of compounds **5″**. The analysis of molecular models showed that the molecule of this isomer could have several conformer forms, and their mutual transition determines, in our opinion, the broadening of signals in the ^{1}H NMR spectra.

Cycloamidophosphites **4′a,b** were oxidized with $CO(NH_2)_2 \cdot H_2O_2$ in methylene chloride at room temperature (Scheme 3).

Note that the oxidation proceeded more slowly than that of other naphthodiol cyclophosphites [2].



SCHEME 2

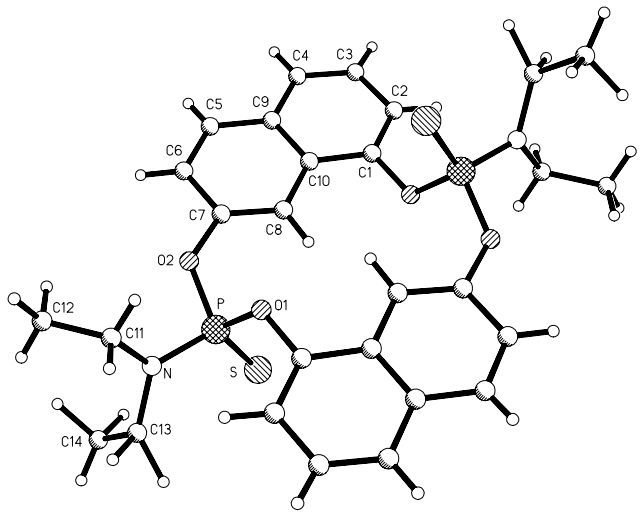


FIGURE 2 Molecular structure of **5′b**.

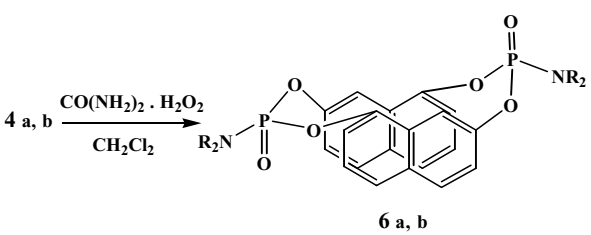
TABLE 2 Bond Lengths (Å) and Angles (degrees) for **5′b**

P—N	1.615(2)	NP O^2	101.70(11)
P—S	1.910(1)	NP O^1	108.05(11)
P—O^2	1.597(2)	O^2—P—S	116.88(8)
P—O^1	1.616(2)	N—P—S	115.83(9)
O^1—$C^{1'}$	1.405(3)	O^1—P—S	114.52(7)
O^2—C^7	1.395(3)	O^2PO^1	97.69(10)
N—C^{13}	1.462(4)	C^7O^2P	126.89(15)
N—C^{11}	1.473(3)	$C^{1'}O^1P$	120.13(15)
C^1—C^2	1.360(4)	$C^2C^1O^{1'}$	121.1(2)
C^1—C^{10}	1.417(3)	$O^{1'}C^1C^{10}$	117.0(2)
C^2—C^3	1.409(4)		
C^3—C^4	1.351(4)		
C^4—C^9	1.414(4)		
C^5—C^6	1.355(4)		
C^5—C^9	1.405(4)		
C^6—C^7	1.405(3)		
C^7—C^8	1.360(3)		

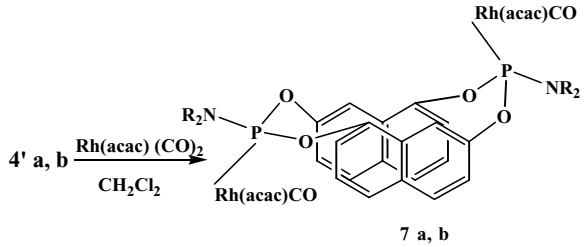
Symmetry transformations used to generate equivalent atoms: $-x+1, -y+1, -z$.

This could be explained by the low concentrations of initial compounds in the solution because of their low solubility. The rate of oxidation can be increased by heating the reaction mixture to 40°C. The resulting phosphates **6a,b** are colorless crystals slightly soluble in organic solvents, which become turbid and friable under keeping. Their ^{31}P NMR spectra exhibited a narrow singlet in the range typical for phosphates, as well as the spectra of corresponding thion derivatives **5′a,b**. Signals from all groups of protons contained in the systems under study were found in the ^{1}H NMR spectra.

The complexation of cycloamidophosphites **4′a,b** with Rh(acac)$(CO)_2$ proceeded in methylene chloride at room temperature (Scheme 4). In spite of the low initial concentrations of reagents, the reaction was completed in an hour, as was evidenced by ^{31}P NMR monitoring. Pale-yellow finely crystalline complexes **7a,b** were precipitated from the reaction mixture within 24 h. Like phosphates **6a,b**, they were hardly soluble in organic solvents but stable when



SCHEME 3

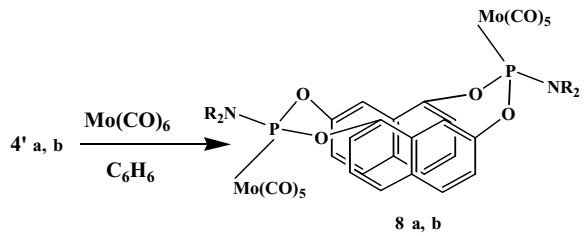


SCHEME 4

kept in the air and solution. Similar complexes of 1,5- and 2,7-naphthodiol cyclophosphites, on the contrary, were well soluble in methylene chloride and unstable in solution and as air-dry forms upon long-term storage [4]. The ^{31}P NMR spectra of complexes **7a,b** exhibited a typical doublet with $J_{\text{P-Rh}}$ 263 Hz and a coordination shift of 0.85 (**7a**) or −1.22 (**7b**).

Molybdenum complexes **8a,b** were obtained by the reaction of cycloamidophosphites **4′a,b** with 2 mol of $Mo(CO)_6$ in benzene at 90°C in a sealed ampoule in an argon atmosphere, because the reagents could decay under heating in the air (Scheme 5).

At the first step of complexation with *N*-methyl derivative **4′a**, a singlet with $\delta = 171.4$ ppm and a signal from the unreacted amidophosphite (135 ppm) were recorded in the ^{31}P NMR spectrum. Another signal with $\delta_P = 164.6$ ppm appeared in the course of reaction. When the initial cycloamidophosphite was consumed, this signal alone remained in the spectrum. The reaction product was isolated from the reaction mixture with a yield of 88% and was identified as binuclear complex of cycloamidophosphite with Mo coordinated to both phosphorus atoms, as was confirmed by ^{1}H NMR spectroscopy and X-ray diffraction analysis. The product with $\delta = 171.4$ ppm observed at the first reaction steps is probably cycloamidophosphite, with the coordination of Mo to only one phosphorus atom. It is notable that a similar situation was observed for the molybdenum complexes with phosphocavitands, and it was shown that the chemical shifts of coordinated phosphorus atoms in the ^{31}P NMR spectra



SCHEME 5

decreased when their number in the molecule increased [3]. A similar situation is also observed for ethyl derivative **4′b**; in this case, the final product has $\delta^{31}P = 163.1$ ppm and the monocoordinated product has $\delta = 164.9$ ppm. The complexes were synthesized as colorless crystals slightly soluble in organic solvents and unstable under storage and in light.

The X-ray diffraction analysis of complexes **8a,b** revealed no structural difference between the complexes with the methyl and ethyl groups at the nitrogen atom (Figs. 3–5, Tables 3 and 4). The angle between the aromatic fragments in the molecule of **8a**, as compared to the initial cyclophosphite **4′a** decreased from 11° to 0°; the fragments became almost parallel. The distance between them also decreased from 1.45 to 0.58 Å. In the crystal, all molecules are arranged in parallel, and the distance between the aromatic fragments in the methyl derivative is shorter than that in the ethyl derivative (4.3 and 5.42 Å, respectively), which explains, in our opinion, the poorer solubility and the higher melting point of **8a**, as compared to **8b**.

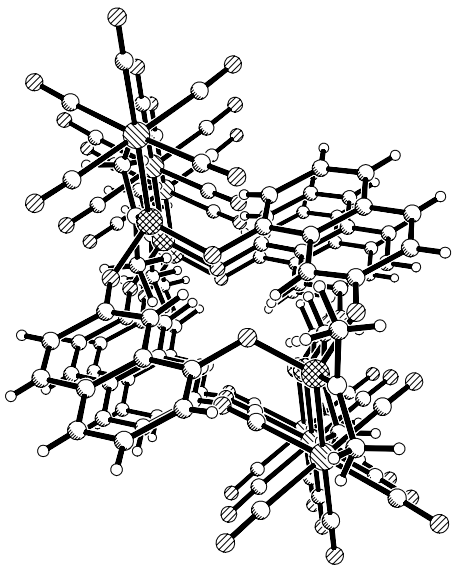


FIGURE 4 Crystal structure of **8a**.

EXPERIMENTAL

^{1}H NMR spectra were recorded on a Bruker WH-250 instrument at 250 MHz. ^{31}P NMR spectra were recorded on a Bruker WP-80 SY at 32.4 MHz (85% H_3PO_4 being used as an external standard). IR spectra of **7a,b** in methylene chloride were recorded on a Specord IR-75 spectrometer. The mass spectrum of compound **4′a** was recorded on a KRATOS KOMPACT-4 instrument (MALDI TOF MS, 250 mkJ).

Column chromatography was carried out on L 100/160 silica gel; TLC, on Silufol plates, using 5:1 (A) and 10:1 (B) benzene/dioxane, 3:1 (C) and 5:1 (D) hexane/dioxane, and 5:1 (E) chloroform/methanol systems as eluants. The detection of compounds on Silufol plates was achieved using iodine vapor treatment, calcination, and the treatment with a 1% aqueous solution of $AgNO_3$.

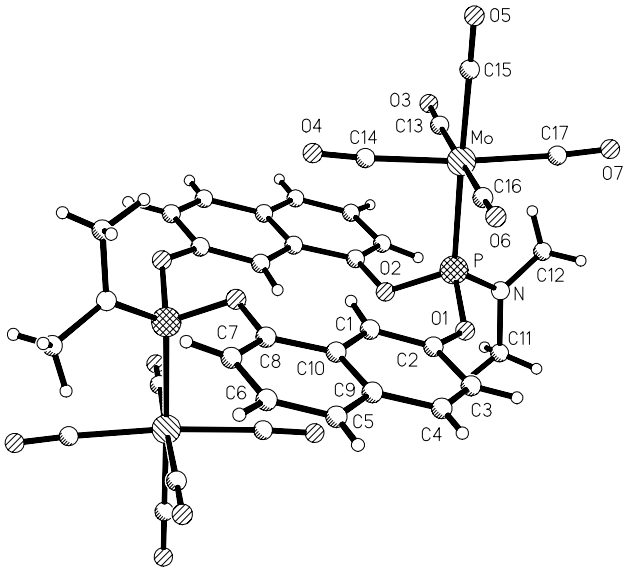


FIGURE 3 Molecular structure of **8a**.

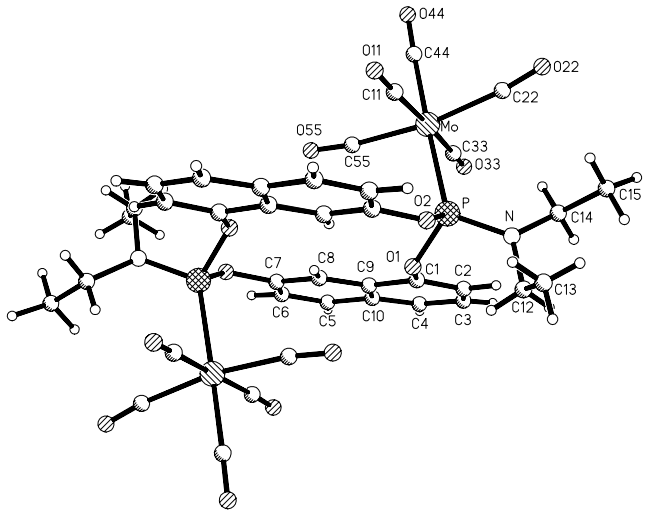


FIGURE 5 Molecular structure of **8b**.

TABLE 3 Bond Lengths (Å) and Angles (degrees) for **8a**

Mo—C^{15}	2.003(7)	O^1PN	97.8(2)
Mo—C^{17}	2.039(7)	O^2PN	104.6(2)
Mo—C^{13}	2.049(7)	O^1P O^2	95.9(2)
Mo—C^{16}	2.050(7)	C^{15}MoC^{17}	85.6(3)
Mo—C^{14}	2.056(7)	C^{15}MoC^{13}	88.8(3)
Mo—P	2.4924(2)	C^{17}MoC^{13}	93.4(3)
P—O^1	1.618(4)	C^{15}MoC^{16}	89.2(3)
P—N	1.635(5)	C^{17}MoC^{16}	87.9(3)
P—O^2	1.652(4)	C^{13}MoC^{16}	177.5(2)
O^1—C^2	1.393(7)	C^{15}MoC^{14}	87.5(3)
O^2—$C^{8'}$	1.395(6)	C^{17}MoC^{14}	172.6(3)
N—C^{11}	1.472(9)	C^{13}MoC^{14}	89.2(3)
C^1—C^2	1.357(8)	C^{16}MoC^{14}	89.2(2)
C^1—C^{10}	1.419(8)	C^{15}MoP	178.7(2)
C^2—C^3	1.406(7)	C^{17}MoP	93.13(2)
C^3—C^4	1.357(9)	C^{13}MoP	90.84(2)
C^4—C^9	1.406(9)	C^{16}MoP	91.21(2)
C^5—C^6	1.349(10)	O^1PMo	116.51(2)
C^5—C^9	1.419(9)	NPMo	121.14(2)
C^6—C^7	1.414(9)	O^2PMo	116.68(1)
C^7—C^8	1.357(8)	$C^{8'}O^2$P	121.8(3)
C^8—C^{10}	1.426(7)	C^2O^1P	128.1(3)
C^9—C^{10}	1.414(7)	C^{14}MoP	93.78(2)
N—C^{12}	1.456(9)		

Symmetry transformations used to generate equivalent atoms: $-x+1, -y+1, -z+1$.

TABLE 4 Bond Lengths (Å) and Angles (degrees) for **8b**

Mo—C^{44}	1.998(6)	O^1PN	105.3
Mo—C^{11}	2.033(4)	O^2PN	97.2
Mo—C^{55}	2.043(6)	O^1P O^2	95.6
Mo—C^{33}	2.054(4)	C^{44}MoC^{11}	88.02(2)
Mo—C^{22}	2.054(6)	C^{44}MoC^{55}	86.44(2)
Mo—P	2.512(7)	C^{11}MoC^{55}	88.36(2)
P—O^1	1.653	C^{44}MoC^{33}	89.86(2)
P—N	1.642	C^{11}MoC^{33}	175.88(2)
P—O^2	1.652(4)	C^{55}MoC^{33}	87.98(2)
O^1—C^1	1.392	C^{44}MoC^{22}	84.75(2)
O^2—$C^{7'}$	1.383	C^{11}MoC^{22}	88.86(2)
N—C^{11}	1.472(9)	C^{55}MoC^{22}	170.85(2)
C^1—C^2	1.365	C^{33}MoC^{22}	94.47(2)
C^1—C^9	1.423	C^{44}MoP	176.(1)
C^2—C^3	1.406	C^{11}MoP	89.45(2)
C^3—C^4	1.368	C^{55}MoP	95.76(2)
C^4—C^{10}	1.407	C^{33}MoP	92.8(2)
C^5—C^6	1.348	O^2PMo	116.14(1)
C^5—C^{10}	1.413	NPMo	121.78(2)
C^6—C^7	1.408	O^1PMo	116.36(2)
C^7—C^8	1.362	$C^{1'}O^2$P	128.2
C^8—C^9	1.408	C^1O^1P	121.0
C^9—C^{10}	1.419	C^{22}MoP	92.93(2)
N—C^{12}	1.484		

Symmetry transformations used to generate equivalent atoms: $-x+1, -y+1, -z+1$.

*Synthesis of Cyclo[bis(1,7-naphthylenedialkylamidophosphites)] (**4′a–d**) Using the Molecular Assemblage Technique*

Diol **2** (2 mmol) in 30 ml of acetonitrile was treated with 4 mmol of phosphotriamide **1a–d**, and the reaction mixture was stirred for 5 min (**a**), 20 min (**b**), 1 h (**c**), or 4 h (**d**); 2 mmol more of **2** was added, and the mixture was stirred at room temperature for 4 h. The next day, the supernatant was decanted; the residue was washed with acetonitrile and several times with benzene to remove isomers **4″a–d**. The residue was recrystallized from methylene chloride and dried in vacuo for 3 h (50°C, 1 mmHg).

*Synthesis of Cyclo[bis(1,7-naphthylenedialkylamidophosphites)] (**4′a–d**) Using the Dismutation Method*

Diol **2** (2 mmol) in 30 ml of acetonitrile was treated with 4 mmol of **1a–d**, and the reaction mixture kept at room temperature for 13 (**a**), 14 (**b**), 15 (**c**), or 17 (**d**) days. The supernatant was decanted; the residue was washed with acetonitrile, recrystallized from methylene chloride, and dried in vacuo for 3 h (50°C, 1 mmHg).

*Cyclo[bis(1,7-naphthylenedimethylamidophosphite)] (**4′a**).* Yield 48%, m.p. 191–192°C, R_f 0.69 (A). ^{1}H NMR ($CDCl_3$): δ 2.88 d (12H, NCH_3, $^3J_{HP}$ 9.4 Hz), 7.01 d (2H, C(2)H, $^3J_{H(2)\text{-}H(3)}$ 7.7 Hz), 7.26 dd (2H, C(6)H, $^3J_{H(6)\text{-}H(8)}$ 2.2 Hz, $^3J_{H(6)\text{-}H(5)}$ 8.3 Hz), 7.34 dd (2H, C(3)H, $^3J_{H(3)\text{-}H(4)}$ 8.3 Hz, $^3J_{H(3)\text{-}H(2)}$ 7.7 Hz), 7.58 ≠ (2H, C(4)H, $^3J_{H(4)\text{-}H(3)}$ 8.3 Hz), 7.82 d (2H, C(5)H, $^3J_{H(5)\text{-}H(6)}$ 8.3 Hz), 8.70 d (2H, C(8)H, $^4J_{HH}$ 2.2 Hz). ^{31}P NMR (CH_2Cl_2): δ 135.05, 135.59. Anal. Calcd for $C_{24}H_{24}N_2O_4P_2$: C, 61.80; H, 5.19; P, 13.28; *M*, 466.41. Found: C, 61.58; H, 5.24; P, 13.18; *M*, 466.00.

*Cyclo[bis(1,7-naphthylenediethylamidophosphite)] (**4′b**).* Yield 49%, m.p. 173–174°C, R_f 0.73 (A), 0.61 (C). ^{1}H NMR ($CDCl_3$): δ 1.23 t (12H, $CH_2\underline{CH_3}$, $^3J_{HH}$ 7.3 Hz), 3.32 m (4H, NCH_2—, $^3J_{HP}$ 9.0 Hz), 3.39 m (4H, NCH_2—, $^3J_{HP}$ 9.0 Hz), 7.10 d (2H, C(2)H, $^3J_{H(3)\text{-}H(2)}$ 7.7 Hz), 7.24 dd (2H, C(6)H, $^3J_{H(6)\text{-}H(5)}$ 8.5 Hz, $^4J_{H(6)\text{-}H(8)}$ 2.6 Hz), 7.28 dd (2H, C(3)H, $^3J_{H(3)\text{-}H(4)}$ 8.1 Hz, $^3J_{H(3)\text{-}H(2)}$ 7.7 Hz), 7.56 d (2H, C(4)H, $^3J_{H(4)\text{-}H(3)}$ 8.1 Hz), 7.80 d (2H, C(5)H, $^3J_{H(5)\text{-}H(6)}$ 8.5 Hz), 8.71 dd (2H, C(8)H, $^4J_{H(8)\text{-}H(6)}$ 2.6 Hz, $^4J_{HP}$ 1.7 Hz). ^{31}P NMR (CH_2Cl_2): δ 137.35 br.

*Cyclo[bis(1,7-naphthylenediisopropylamidophosphite)] (**4′c**).* Yield 32%, m.p. 182–183°C, R_f 0.74 (D). ^{1}H NMR ($CDCl_3$): δ 1.27 d (12H, $CH(\underline{CH_3})_2$, $^3J_{HH}$ 7.15 Hz), 1.36 d (12H, $CH(\underline{CH_3})_2$, $^3J_{HH}$ 7.15 Hz),

3.93 m (2H, NCH$(CH_3)_2$, $^3J_{HP}$ 10.44 Hz), 3.99 m (2H, NCH$(CH_3)_2$, $^3J_{HP}$ 10.44 Hz), 7.15 d (2H, C(6)H, $^3J_{H(6)-H(5)}$ 8.8 Hz, $^4J_{H(6)-H(8)}$ 2.2 Hz), 7.27 d (2H, C(3)H), 7.52 d (2H, C(2)H), 7.56 d (2H, C(4)H), 7.78 d (2H, C(5)H, $^3J_{H(5)-H(6)}$ 8.8 Hz), 8.70 dd (2H, C(8)H, $^4J_{H(8)-H(6)}$ 2.2 Hz, $^4J_{HP}$ 2.7 Hz). ^{31}P NMR (CH_2Cl_2): δ 141.6.

Cyclo[bis(1,7-naphthylenepiperidylphosphite)] (**4′d**). Yield 39%, m.p. 175–176°C, R_f 0.63 (D), 0.84 (B). ^{1}H NMR ($CDCl_3$): δ 1.62 m (12H, CH_2), 3.25 m (4H, C^aH_2, J_{HH} 13.2 Hz, $^3J_{HP}$ 8.11 Hz), 3.36 m (4H, C^eH_2, J_{HH} 13.2 Hz, $^3J_{HP}$ 6.4 Hz), 7.15 d (2H, C(2)H, $^3J_{H(2)-H(3)}$7.3 Hz), 7.25 dd (2H, C(6)H, $^3J_{H(6)-H(5)}$ 9 Hz, $^4J_{H(6)-H(8)}$ 2.1 Hz), 7.30 dd (2H, C(3)H, $^3J_{H(3)-H(2)}$ 7.7 Hz, $^3J_{H(3)-H(4)}$ 8.1 Hz), 7.56 d (2H, C(4)H, $^3J_{H(4)-H(3)}$ 8.1 Hz), 7.81 d (2H, C(5)H, $^3J_{H(5)-H(6)}$ 9.0 Hz), 8.68 dd (2H, C(8)H, $^4J_{H(8)-H(6)}$ 2.1 Hz, $^4J_{HP}$ 1.7 Hz). ^{31}P NMR (CH_2Cl_2): δ 131.99 br. Anal. Calcd for $C_{30}H_{32}N_2O_4P_2$: C, 65.93; H, 5.90; N, 5.13; P, 11.33. Found: C, 65.99; H, 5.81; N, 5.21; P, 11.21.

Synthesis of Cyclo[bis(1,7-naphthylenedialkylamidothionophosphates)] (**5′a–d, 5″a–d**)

A solution containing 1.2 mmol of the mixture of isomers **4′a–d** in 50 ml of methylene chloride was treated with 2.4 mmol of sulfur and stirred at room temperature for 2.5 h. The solution was filtered and evaporated in vacuo to 20 ml; the crystals precipitated were filtered off, washed with benzene, and recrystallized from chloroform (**5′a–d**). The filtrate was evaporated in vacuo, and column chromatography was performed on the residue; the resulting products were eluted by the hexane/dioxane (10:1) (**5′a–d**) or benzene/dioxane (10:1) system (**5″a–d**).

Cyclo[bis(1,7-naphthylenedimethylamidothionophosphate)] (**5′a**). Yield 44%, m.p. 297–298°C, R_f 0.74 (A), 0.62 (B). ^{1}H NMR ($CDCl_3$): δ 3.10 d (12H, NCH_3, $^3J_{HP}$ 12.1 Hz), 7.28 d (2H, C(2)H, $^3J_{H(2)-H(3)}$ 7.7 Hz), 7.32 dd (2H, C(6)H, $^3J_{H(6)-H(5)}$ 8.8 Hz, $^4J_{H(6)-H(8)}$ 2.1 Hz), 7.40 dd (2H, C(3)H, $^3J_{H(3)-H(2)}$ 7.7 Hz, $^3J_{H(3)-H(4)}$ 8.3 Hz), 7.68 d (2H, C(4)H, $^3J_{H(4)-H(3)}$ 8.3 Hz), 7.88 d (2H, C(5)H, $^3J_{H(5)-H(6)}$ 8.8 Hz), 8.97 dd (2H, C(8)H, $^4J_{H(8)-H(6)}$ 2.1 Hz, $^4J_{HP}$ 1.7 Hz). ^{31}P NMR (CH_2Cl_2): δ 67.7. Anal. Calcd for $C_{24}H_{24}N_2O_4P_2S_2$: C, 54.33; H, 4.56; P, 11.68; *M*, 530.54. Found: C, 54.16; H, 4.83; P, 11.41; *M*, 531.0.

Cyclo[bis(1,7-naphthylenediethylamidothionophosphate)] (**5′b**). Yield 41%, m.p. 281–282°C, R_f 0.78 (A), 0.66 (B). ^{1}H NMR ($CDCl_3$): δ 1.30 t (12H, $CH_2\underline{CH_3}$, $^3J_{HH}$ 7.1 Hz), 3.57 m (8H, NCH_2—, $^3J_{HP}$ 14.3 Hz), 7.28 dd (2H, C(6)H, $^3J_{H(6)-H(5)}$ 8.8 Hz, $^4J_{H(6)-H(8)}$ 2.2 Hz), 7.30 dd (2H, C(3)H, $^3J_{H(3)-H(2)}$ 7.7 Hz, $^3J_{H(3)-H(4)}$ 7.2 Hz), 7.39 d (2H, C(2)H, $^3J_{H(2)-H(3)}$7.7 Hz), 7.65 d (2H, C(4)H, $^3J_{H(4)-H(3)}$7.2 Hz), 7.84 d (2H, C(5)H, $^3J_{H(5)-H(6)}$ 8.8 Hz), 9.01 dd (2H, C(8)H, $^4J_{H(8)-H(6)}$ 2.2 Hz, $^4J_{HP}$ 1.7 Hz). ^{31}P NMR (CH_2Cl_2): δ 66.9. Anal. Calcd for $C_{28}H_{32}N_2O_4P_2S_2$: C, 57.33; H, 5.50; P, 10.56. Found: C, 57.40; H, 5.48; P, 10.59.

Cyclo[bis(1,7-naphthylenediisopropylamidothionophosphate)] (**5′c**). Yield 32%, m.p. 182–183°C, R_f 0.74 (hexane/dioxane, 5:1). ^{1}H NMR ($CDCl_3$): δ 1.27 d (12H, CH$(\underline{CH_3})_2$, $^3J_{HH}$ 7.15 Hz), 1.36 d (12H, CH$(\underline{CH_3})_2$, $^3J_{HH}$ 7.15 Hz), 3.93 m (2H, NCH$(CH_3)_2$, $^3J_{HP}$ 10.44 Hz), 3.99 m (2H, NCH$(CH_3)_2$, $^3J_{HP}$ 10.44 Hz), 7.15 d (2H, C(6)H, $^3J_{H(6)-H(5)}$ 8.8 Hz, $^4J_{H(6)-H(8)}$ 2.2 Hz), 7.27 d (2H, C(3)H), 7.52 d (2H, C(2)H), 7.56 d (2H, C(4)H), 7.78 d (2H, C(5)H, $^3J_{H(5)-H(6)}$ 8.8 Hz), 8.70 dd (2H, C(8)H, $^4J_{H(8)-H(6)}$ 2.2 Hz, $^4J_{HP}$ 2.7 Hz). ^{31}P NMR (CH_2Cl_2): δ 141.6. Anal. Calcd for $C_{32}H_{40}N_2O_4P_2S_2$: C, 59.80; H, 6.27; N, 4.36; P, 9.64. Found: C, 59.40; H, 6.39; N, 4.48; P, 9.75.

Cyclo[bis(1,7-naphthylenedipiperidylthionophosphate)] (**5′d**). Yield 32%, decomp. 296–300°C, R_f 0.77 (A), 0.35 (C). ^{1}H NMR ($CDCl_3$): δ 1.69 m (12H, CH_2), 3.53 m (8H, CH_2, $^3J_{HP}$ 9.81 Hz), 7.31 dd (2H, C(6)H, $^3J_{H(6)-H(5)}$ 8.9 Hz, $^4J_{H(6)-H(8)}$ 2.1 Hz), 7.39 dd (2H, C(3)H, $^3J_{H(3)-H(2)}$ 7.6 Hz, $^3J_{H(3)-H(4)}$ 7.7 Hz), 7.46 d (2H, C(2)H, $^3J_{H(2)-H(3)}$7.6 Hz), 7.67 d (2H, C(4)H, $^3J_{H(4)-H(3)}$ 7.7 Hz), 7.86 d (2H, C(5)H, $^3J_{H(5)-H(6)}$ 8.9 Hz), 8.95 dd (2H, C(8)H, $^4J_{H(8)-H(6)}$ 2.1 Hz, $^4J_{HP}$ 1.6 Hz). ^{31}P NMR (CH_2Cl_2): δ 63.8. Anal. Calcd for $C_{30}H_{32}N_2O_4P_2S_2$: C, 59.00; H, 5.28; P, 10.14. Found: C, 59.11; H, 5.19; P, 10.12.

Cyclo[bis(1,7-naphthylenedimethylamidothionophosphate)] (**5″a**). Yield 32%, m.p. 92–94°C, R_f 0.69 (A). ^{1}H NMR ($CDCl_3$): δ 2.85 br d (12H, NCH_3), 7.32 br d, 7.50 br m, 7.69 br d, 7.95 br d (12H, CH). ^{31}P NMR (CH_2Cl_2): δ 67.06, 67.78. Anal. Calcd for $C_{24}H_{24}N_2O_4P_2S_2$: C, 54.33; H, 4.56; P, 11.68. Found: C, 54.33; H, 4.56; P, 11.68.

Cyclo[bis(1,7-naphthylenediethylamidothionophosphate)] (**5″b**). Yield 36%, m.p. 73–75°C, R_f 0.72 (A). ^{1}H NMR ($CDCl_3$): δ 1.00 br t (12H, $CH_2\underline{CH_3}$), 3.39 br m (8H, NCH_2—), 7.31 br d, 7.52 br m, 7.63 br d, 7.98 br d (12H, CH). ^{31}P NMR (CH_2Cl_2): δ 66.49, 67.06.

Cyclo[bis(1,7-naphthylenediisopropylamidothionophosphate)] (**5″c**). Yield 31%, m.p. 104–105°C, R_f

0.79 (benzene/dioxane, 5:1). ^{1}H NMR ($CDCl_3$): δ 1.34 br m (24H, CH_3), 3.96 br m (2H, NCH—), 4.05 br m (2H, NCH—), 7.33 br d, 7.52 br d, 7.64 br m, 8.07 br d (12H, CH). ^{31}P NMR (CH_2Cl_2): δ 64.15, 64.69. Anal. Calcd for $C_{32}H_{40}N_2O_4P_2S_2$: C, 59.80; H, 6.27; N, 4.36; P, 9.64. Found: C, 59.89; H, 6.18; N, 4.32; P, 9.68.

*Cyclo[bis(1,7-naphthylenedipiperidylamidothionophosphate)] (**5″d**).* Yield 28%, m.p. 112–115°C, R_f 0.84 (A). ^{1}H NMR ($CDCl_3$): δ 1.39 br m (12H, CH_2), 3.40 br m (8H, NCH_2—), 7.33 br d, 7.52 br d, 7.64 br m, 8.03 br d (12H, CH). ^{31}P NMR (CH_2Cl_2): δ 64.15, 64.69.

*Synthesis of Cyclo[bis(1,7-naphthylenedialkylamidophosphates)] (**6a,b**)*

A solution containing 0.5 mmol of cyclophosphite **4a,b** in 30 ml of methylene chloride was treated with 1 mmol of complex $CO(NH_2)_2 \cdot H_2O_2$ and stirred at 40°C for 6 h. The supernatant was filtered off and evaporated in vacuo; the residue was recrystallized from chloroform and dried in vacuo for 2.5 h (70°C, 1 mmHg).

*Cyclo[bis(1,7-naphthylenedimethylamidophosphate)] (**6a**).* Yield 92%, m.p. 249–251°C, R_f 0.67 (E). ^{1}H NMR ($CDCl_3$): δ 2.87 d (12H, NCH_3, $^3J_{HP}$ 10.5 Hz), 7.34 dd (2H, C(6)H, $^3J_{H(6)\text{-}H(5)}$ 8.3 Hz, $^4J_{H(6)\text{-}H(8)}$ 2.2 Hz), 7.40 dd (2H, C(3)H, $^3J_{H(3)\text{-}H(2)}$ 7.7 Hz, $^3J_{H(3)\text{-}H(4)}$ 7.7 Hz), 7.49 d (2H, C(2)H, $^3J_{H(2)\text{-}H(3)}$ 7.7 Hz), 7.66 d (2H, C(4)H, $^3J_{H(4)\text{-}H(3)}$ 7.7 Hz), 7.86 d (2H, C(5)H, $^3J_{H(5)\text{-}H(6)}$ 8.3 Hz), 8.76 dd (2H, C(8)H, $^4J_{H(8)\text{-}H(6)}$ 2.2 Hz, $^4J_{HP}$ 1.7 Hz). ^{31}P NMR (CD_2Cl_2): δ 1.62. Anal. Calcd for $C_{24}H_{24}N_2O_6P_2$: C, 57.84; H, 4.85; P, 12.43. Found: C, 57.86; H, 4.83; P, 12.46.

*Cyclo[bis(1,7-naphthylenediethylamidophosphate)] (**6b**).* Yield 89%, m.p. 268–270°C, R_f 0.70 (E). ^{1}H NMR ($CDCl_3$): δ 1.17 t (12H, $CH_2\underline{CH_3}$, $^3J_{HH}$ 6.8 Hz), 3.31 m (8H, NCH_2—, $^3J_{HP}$ 12.3 Hz), 7.32 dd (2H, C(6)H, $^3J_{H(6)\text{-}H(5)}$ 8.9 Hz, $^4J_{H(6)\text{-}H(8)}$ 2.6 Hz), 7.37 dd (2H, C(3)H, $^3J_{H(3)\text{-}H(2)}$ 7.7 Hz, $^3J_{H(3)\text{-}H(4)}$ 8.1 Hz), 7.53 d (2H, C(2)H, $^3J_{H(2)\text{-}H(3)}$ 7.7 Hz), 7.63 d (2H, C(4)H, $^3J_{H(4)\text{-}H(3)}$ 7.1 Hz), 7.83 d (2H, C(5)H, $^3J_{H(5)\text{-}H(6)}$ 8.9 Hz), 8.81 dd (2H, C(8)H, $^4J_{H(8)\text{-}H(6)}$ 2.6 Hz, $^4J_{HP}$ 1.7 Hz). ^{31}P NMR (CD_2Cl_2): δ 1.18. Anal. Calcd for $C_{28}H_{32}N_2O_6P_2$: C, 60.65; H, 5.82; P, 11.17. Found: C, 60.71; H, 5.94; P, 11.15.

*Synthesis of μ{Cyclo[bis(arylenedialkylamidophosphite)]}-bis[acetylacetonatocarbonylrhodium(I)] (**7a,b**)*

A solution of 2.4 mmol cyclobisphosphite **4′a,b** in 35 ml of methylene chloride was added dropwise to a solution of 1.2 mmol $Rh(acac)(CO)_2$ in 5 ml of methylene chloride, and the reaction mixture was kept at room temperature for 2.5 h. The solution was evaporated to half the volume; the precipitate formed was filtered off, washed with hexane, and dried in vacuo for 2.5 h (40°C, 1 mmHg).

*μ-{Cyclo[bis(arylenedimethylamidophosphite)]}-bis[acetylacetonatocarbonylrhodium(I)] (**7a**).* Yield 96%, decomp. 236-240°C. ^{1}H NMR ($CDCl_3$): 1.96 s (6H, CH_3 acac), 2.02 s (6H,CH_3 *acac*), 3.00 d (12H, NCH_3, $^3J_{HP}$ 9.8 Hz), 5.35 s (2H, CH *acac*), 7.01 d (2H, C(2)H), 7.26 dd (2H, C(6)H), 7.34 dd (2H, C(3)H), 7.58 ≠ (2H, C(4)H), 7.82 d (2H, C(5)H), 8.70 d (2H, C(8)H). ^{31}P NMR (CH_2Cl_2): δ 135.6 d, $^3J_{PRh}$ 263 Hz. IR spectrum (ν, cm^{-1}): 1990 (CO—Rh), 1510, 1570 (*acac*). Anal. Calcd for $C_{36}H_{38}N_2O_{10}P_2Rh_2$: P, 6.69. Found: P, 6.62.

*μ-{Cyclo[bis(arylenediethylamidophosphite)]}bis-[acetylacetonatocarbonylrhodium(I)] (**7b**).* Yield 94%, decomp. 233–236°C. ^{31}P NMR (CH_2Cl_2): δ 136.0 d, $^3J_{PRh}$ 261.5 Hz. IR spectrum (ν, cm^{-1}): 1995 (CO—Rh), 1512, 1571 (*acac*). Anal. Calcd for $C_{40}H_{46}N_2O_{10}P_2Rh_2$: P, 6.31. Found: P, 6.34.

*Synthesis of μ-{Cyclo[bis(naphthylenedialkylamidophosphite)]}di[pentacarbonylmolybdenum(0)] (**8a,b**)*

$Mo(CO)_6$ (0.30 mmol) was added to a solution of 0.15 mmol cyclophosphite **4′a,b** in 2 ml of benzene, and the reaction mixture was heated in a sealed ampoule to 100°C for 24 h. The solvent was evaporated in vacuo; the residue was recrystallized from chloroform and dried in vacuo for 4 h (50°C, 1 mmHg).

*μ-{Cyclo[bis(naphthylenedimethylamidophosphite)]}di[pentacarbonylmolybdenum(0)] (**8a**).* Yield 79%, decomp. 209–210°C, R_f 0.66 (hexane/dioxane, 3:1). ^{1}H NMR ($CDCl_3$): δ 3.01 d (12H, NCH_3, $^3J_{HP}$ 11.1 Hz), 7.20 d (2H, C(2)H, $^3J_{H(2)\text{-}H(3)}$8.1 Hz), 7.30 dd (2H, C(6)H, $^3J_{H(6)\text{-}H(5)}$ 8.5 Hz, $^4J_{H(6)\text{-}H(8)}$ 1.7 Hz), 7.37 dd (2H, C(3)H, $^3J_{H(3)\text{-}H(2)}$ 8.1 Hz, $^3J_{H(3)\text{-}H(4)}$ 8.1 Hz), 7.64 d (2H, C(4)H, $^3J_{H(4)\text{-}H(3)}$ 8.1 Hz), 7.86 d (2H, C(5)H, $^3J_{H(5)\text{-}H(6)}$ 8.5 Hz), 8.98 d (2H, C(8)H, $^4J_{H(6)\text{-}H(8)}$ 1.7 Hz). ^{31}P NMR (C_6H_6): δ 164.6. Anal. Calcd for

$C_{34}H_{24}Mo_2N_2O_{14}P_2$: C, 43.52; H, 2.58; P, 6.60. Found: C, 43.71; H, 2.18; P, 6.55.

μ-{Cyclo[bis(naphthylenediethylamidophosphite)]}-di[pentacarbonylmolybdenum(0)] (**8b**). Yield 88%, decomp. 158–160°C, R_f 0.72 (hexane/dioxane, 3:1). ^{1}H NMR ($CDCl_3$): δ 1.18 t (12H, $NCH_2\underline{CH_3}$, $^3J_{HH}$ 7.0 Hz), 3.34 m (4H, NCH_2—, $^3J_{HP}$ 12.3 Hz), 3.57 m (4H, NCH_2—, $^3J_{HP}$ 12.3 Hz), 7.29 d (2H, C(6)H, $^3J_{H(6)\text{-}H(5)}$ 8.5 Hz), 7.31 d (2H, C(2)H, $^3J_{H(2)\text{-}H(3)}$ 7.7 Hz), 7.36 dd (2H, C(3)H, $^3J_{H(3)\text{-}H(2)}$ 7.7 Hz, $^3J_{H(3)\text{-}H(4)}$ 8.1 Hz), 7.62 d (2H, C(4)H, $^3J_{H(4)\text{-}H(3)}$ 8.1 Hz), 7.85 d (2H, C(5)H, $^3J_{H(5)\text{-}H(6)}$ 8.5 Hz),8.96 s (2H, C(8)H). ^{31}P NMR (C_6H_6): δ 163.23. Anal. Calcd for $C_{38}H_{32}Mo_2N_2O_{14}P_2$: C, 45.89; H, 3.24; P, 6.23. Found: C, 45.93; H, 3.28; P, 6.15.

X-ray analysis of **4′a**, **5′b**, and **8a,b** was performed on an automatic CAD-4 Enraf-Nonius diffractometer with Mo Kα radiation.

Compound **4′a**: Colorless monoclinic crystal ($C_{24}H_{24}N_2O_4P_2$, $M = 468.41$), size $0.55 \times 0.50 \times 0.20$ mm, space group $P2(1)$, $a = 8.247(2)$ Å, $b = 10.546(2)$ Å, $c = 12.965(3)$ Å. $V = 1119.8(4)$ Å^3. $Z = 2$ (1.389 Mg/cm^3). $F(000) = 492$, $\theta/2\theta$ data collection, θ range 2.49° to 24.96° in $0 \le h \le 9$, $0 \le k \le 12$, $-15 \le l \le 15$, independent reflections: 1434/1330 [R(int) = 0.0215]. Refinement method: Full-matrix least squares on F^2; data/restraints/parameters: 1330/0/194, GoF on $F^2 = 0.961$, final R indices $[I > 2\sigma(I)]$: $R_1 = 0.0265$, w$R_2 = 0.0731$, largest difference peak and hole: 0.212 and −0.181 eÅ^{-3} (CCDC 195457 [5]).

Compound **5′b**: Colorless monoclinic crystal ($C_{28}H_{32}N_2O_4P_2S_2$, $M = 586.62$), size $0.55 \times 0.28 \times 0.22$ mm, space group $P2(1)/c$, $a = 8.089(2)$ Å, $b = 14.357(3)$ Å, $c = 12.634(3)$ Å, $\alpha = 90°$, $\beta = 104.58(3)°$, $\gamma = 90°$, $V = 1420.0(6)$ Å^3, $Z = 2$ (1.372 Mg/cm^3), $F(000) = 616$, $\theta/2\theta$ data collection, θ range 2.19°–24.97° in $0 \le h \le 9$, $0 \le k \le 16$, $-14 \le l \le 14$, independent reflections: 1740/1624 [R(int) = 0.0243]. Refinement method: Full-matrix least squares on F^2; data/restraints/parameters: 1624/0/173, GoF on $F^2 = 0.930$, final R indices $[I > 2\sigma(I)]$: $R_1 = 0.0329$, w$R_2 = 0.0890$, largest difference peak and hole: 0.458 and −0.235 eÅ^{-3} (CCDC 195458 [5]).

Compound **8′a**: Colorless monoclinic crystal ($C_{34}H_{24}Mo_2N_2O_{14}P_2$, $M = 938.36$), size $0.60 \times 0.15 \times 0.08$ mm, space group $P2(1)/c$, $a = 10.792(2)$ Å, $b = 19.840(4)$ Å, $c = 8.726(3)$ Å, $\alpha = 90°$, $\beta = 100.11(3)°$, $\gamma = 90°$, $V = 1844.8(7)$ Å^3, $Z = 2$ (1.689 Mg/cm^3), $F(000) = 936$, $\theta/2\theta$ data collection, θ range 2.05°–24.96° in $-12 \le h \le 12$, $-23 \le k \le 0$, $0 \le l \le 10$, independent reflections: 1692/1585 [R(int) = 0.0136]. Refinement method: Full-matrix least squares on F^2; data/restraints/parameters: 1585/0/292, GoF on $F^2 = 1.113$, final R indices $[I > 2\sigma(I)]$: $R_1 = 0.0246$, w$R_2 = 0.0757$, largest difference peak and hole: 0.456 and −0.293 eÅ^{-3} (CCDC 195459 [5]).

Compound **8′b**: Colorless monoclinic crystal ($C_{38}H_{32}Mo_2N_2O_{14}P_2$, $M = 994.48$), size $0.70 \times 0.35 \times 0.10$ mm, space group $P2(1)/c$, $a = 9.127(2)$ Å, $b = 10.036(2)$ Å, $c = 22.405(4)$ Å, $\alpha = 90°$, $\beta = 93.49(3)°$, $\gamma = 90°$, $V = 2048.5(7)$ Å^3, $Z = 2$ (1.612 Mg/cm^3), $F(000) = 1000$, $\theta/2\theta$ data collection, θ range 1.82°–24.97° in $-10 \le h \le 10$, $0 \le k \le 11$, $0 \le l \le 2$, independent reflections: 2278/2219 [R(int) = 0.0254]. Refinement method: Full-matrix least squares on F^2; data/restraints/parameters: 2219/0/326, GoF on $F^2 = 1.029$, final R indices $[I > 2\sigma(I)]$: $R_1 = 0.0236$, w$R_2 = 0.0619$, largest difference peak and hole: 0.425 and −0.379 eÅ^{-3} (CCDC 195460 [5]).

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[5] Crystallographic data reported in this paper have been deposited with Cambridge Crystallographic Data Centre as supplementary publication. Copies of the data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge, CB2 1EZ, UK; fax: +44 1223 336033; or deposit@ccdc.cam.ac.uk).

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