Cyclic bis-amidophosphites based on 1,6-dihydroxynaphthalene

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Phosphorylation of 1,6-dihydroxynaphthalene with various triamidophosphites gives 2 : 2 cyclic adducts with the 1,6´,1´,6-bonding of the naphthalenediyl moieties through the amidophosphite bridges.

Key words: phosphorylation, 1,6-dihydroxynaphthalene, triamidophosphites, amidophosphites, naphthophosphacyclophanes, NMR spectroscopy.

One of the most important objectives of modern organophosphorus chemistry is development and studies of phosphorus-containing macrocyclic systems. The cavity size in these macrocycles is determined by the cycle size, mutual arrangement of functional groups, and other specific structural features. It should be also noted that they can serve as a basis for the development of unusual supramolecular systems, studies of specific features of molecular recognition, carrying out competing reactions in the system of identical phosphorus-containing fragments, *etc*. Besides, they can be used as molecular containers for the solution of various scientific, technical, and medicinal problems.

Among new classes of the cavity-containing systems, the most interesting are the structures with a regular combination of phosphorus fragments and aromatic blocks, for example, naphthalene ones, having a large conjugated system. Dihydroxynaphthalene derivatives are widely used as dyes, fluorescent compounds, antioxidants, and antiseptics.[1—5] Having a condensed aromatic block in the structure, they serve as a basis for the synthesis of macrocyclic systems of the cyclophane type,[6—9] whose phosphorus-containing analogs were obtained in the last decades.[10—18]

The purpose of the present work is the studies of cyclophosphorylation of nonsymmetric 1,6-dihydroxynaphthalene (**1**) with triamidophosphites. In the molecule of this compound, the hydroxy group of the first ring occupies α-position, whereas the hydroxy group of the second ring is placed at the maximal remote β-position. Such an arrangement of the hydroxy groups makes it possible to synthesize the "double-deck" phosphacyclophanes, which seem quite interesting compounds.

In this work, hexamethyltriamide (HMTA, **2a**), hexaethyltriamide (HETA, **2b**), and tripiperidide of phosphorous acid (TPP, **2c**) were used as the phosphorylating agents. These derivatives readily react with phenols already at room temperature. The reaction can be carried out in various organic solvents without removal of the liberated secondary amine.[19]

Three preparative methods that showed good results in the preceding studies were used for the synthesis of the biscyclic structures indicated: a molecular assembly (*A*), a direct cyclophosphorylation (*B*), and a dismutation of bisphosphorylated naphthodiols (*C*) (Scheme 1).

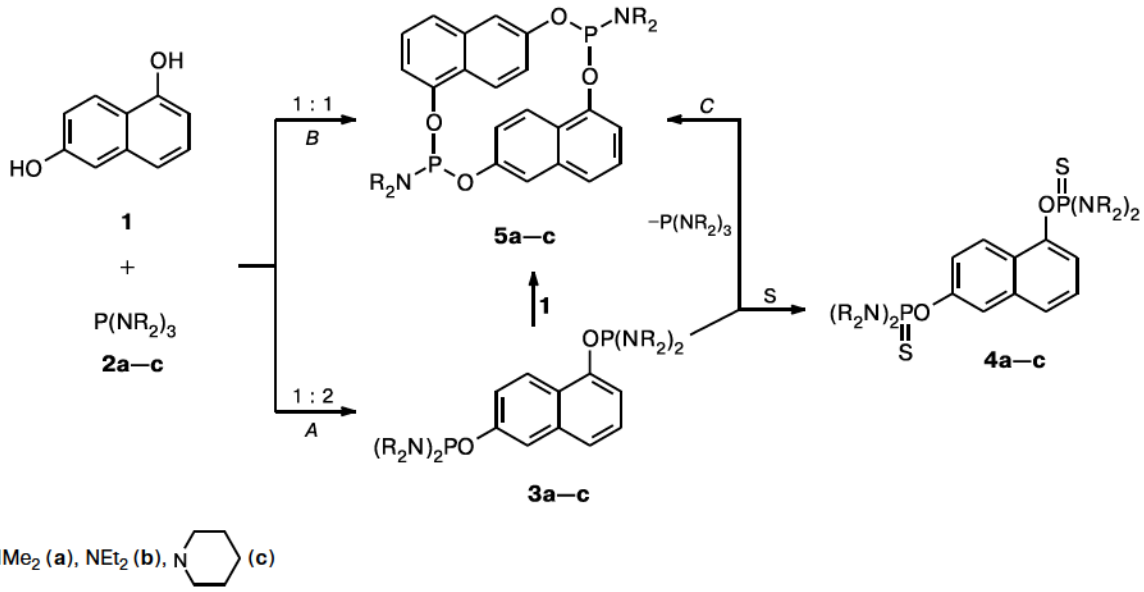
All the syntheses were performed at room temperature in anhydrous acetonitrile. Molecular assembly of cycloamidophosphites was carried out in two steps. In the first step, the bisphosphorylated derivatives **3a—c** were obtained. The process was conducted at the ratio diol—triamide 1 : 2, its course was monitored by ^{31}P NMR spectroscopy.[20]

The phosphorylation time was shown to be dependent on the nature of the starting triamide **2a—c**. The reaction reached completion within 7 min for HMTA, 40 min for TPP, and 1.5 h for HETA.

All the synthesized bisphosphorylated derivatives **3a—c** are oily compounds, unstable in solutions because of their tendency to dismutation.[21,22] For identification, they were converted to the stable thionophosphates **4a—c**. These phosphates were purified by column chromatography and obtained as crystalline compounds. The ^{31}P NMR spectra of these phosphates exhibit two singlets of equal integral intensities in the region δ 74—82 with the 0.8—2 ppm difference, which is explained by the nonsymmetric character of the 1,6-dihydroxynaphthalene molecule. The ^{1}H NMR spectra exhibited all the signals for all the groups of protons with the corresponding ratio of integral intensities. The structure of the 1,6-bis(*N*,*N*,*N*´,*N*´-tetramethyl-

Published in Russian in *Izvestiya Akademii Nauk. Seriya Khimicheskaya,* No. 9, pp. 2023—2031, September, 2013.

Scheme 1



NR_2 = NMe_2 (**a**), NEt_2 (**b**), N (**c**)

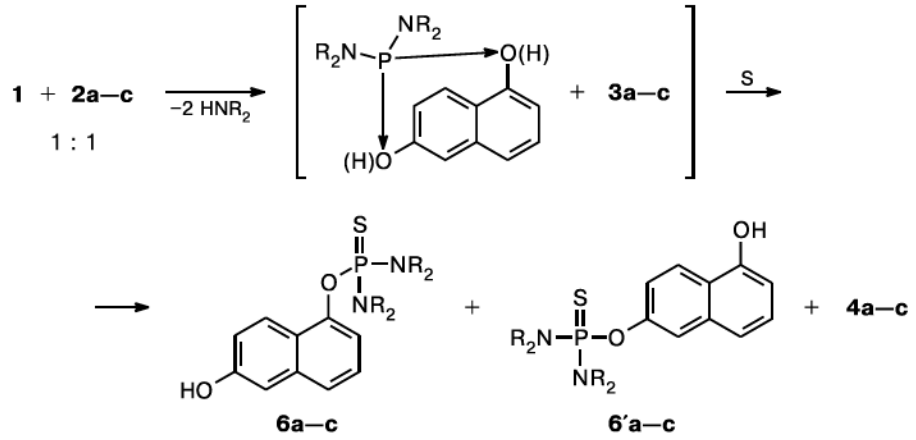
diaminothiophosphoryloxy)naphthalene molecule (**4a**) was also confirmed by X-ray diffraction analysis.[13]

The second step of molecular assembly included the cyclization of bisphosphites **3a**—**c** with one equivalent of the starting diol **1**. The cycloamidophosphites **5a**—**c** separated from the acetonitrile solution as dense oils well soluble in dichloromethane, dioxane, diethyl ether, and benzene, with the solubility decreasing in the order **5b**—**5c**—**5a**. After drying the cycloamidophosphites *in vacuo*, derivatives **5a**,**b** were obtained as brittle foams with melting points 229—230 °C and 101—102 °C, respectively, whereas piperidine derivative **5c** remained oily. Characteristics of cycloamidophosphites synthesized differ from characteristics of similar compounds based on the isomeric 1,7-dihydroxynaphthalene.[23] The structure and individuality of cyclobisamidophosphites **5a**—**c** were confirmed by their ^{31}P, ^{1}H, and ^{13}C NMR spectra and elemental analysis. Thus, their ^{31}P NMR spectra exhibited singlets in the region δ 138—140 characteristic of phosphorous acid amido diesters with aromatic substituents. In the ^{1}H NMR spectrum of cyclophosphite **5a**, two doublets were observed in the high-field region with chemical shifts δ 2.84 and 2.93 (with the ratio 1 : 1) from the methyl groups of the amide fragment with $^{3}J_{P,H}$ = 10 and 11 Hz, respectively. This difference is explained by their different arrangement with respect to the plane of the cyclophane framework. In the low-field region, a set of signals was observed at δ 7.05—8.21 from the aromatic fragments with the indicative spin-spin coupling constants $^{3}J_{H,H}$. It is interesting to note that the most downfield signals corresponded to the protons at position 8 of the naphthalene fragments, which had different chemical shifts, *i.e.* were nonequivalent. Though the difference in the chemical shifts was small (Δδ ≈ 0.3 ppm), the difference in the spin-spin coupling constants $^{3}J_{H(7)—H(8)}$ and $^{3}J_{H(7')—H(8')}$ reached 1.1 Hz. The integral curve completely agreed with the theoretical assignment.

In a direct synthesis (method *B*), the reaction of the equimolar amounts of the starting compounds was performed. The process was complete within 5 h for reagent **2a** and within 24 h for reagents **2b**,**c**. The phosphorylation of nonsymmetric 1,6-dihydroxynaphthalene was expected to be regioselective because of the different rates of phosphorylation of α- and β-hydroxy groups (Scheme 2). However, such a pattern was observed only when HETA **2b** was used: the hydroxy group at β-position was phosphorylated slightly faster than the hydroxy group at α-position. According to the ^{1}H NMR spectroscopy, the ratio of isomers was close to 2 : 1.

When HMTA **2a** was used as the phosphorylating agent, no selectivity of phosphorylation was observed at all, and the isomers **6** and **6´** were formed in the equal ratios. Such conclusions were drawn based on the ^{1}H NMR spectra of monothiophosphates **6a**—**c** and **6´a**—**c**, where α- and β-isomers are well distinguishable, while in the ^{31}P NMR spectra and upon chromatographic analysis they were almost indistinguishable. The use of TPP **2c** as the phosphorylating agent virtually gave no monophosphorylated product. In this case, the reaction mixture contained a bisphosphorylated product and the residual unphosphorylated 1,6-dihydroxynaphthalene. Apparently, such directions of the process can be explained by both the structure of diol **1** and the activity of the leaving group of phosphorylating agents **2a**—**c**. The literature data[23,24] show that no regioselectivity was observed in the phosphorylation of 1,7-dihydroxynaphthalene with compound **2b**.

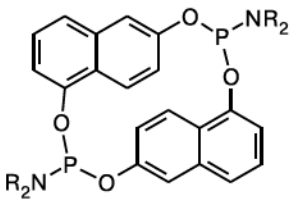
Scheme 2



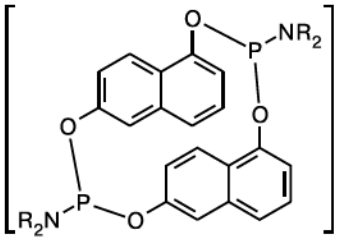
All the physicochemical characteristics of cycloamidophosphites **5a—c**, obtained by these two methods completely agreed, however, the better yields were observed in the direct synthesis (68—72%).

The dismutation synthesis of cyclobisamidophosphites **5a—c** (method *C*) using bisphosphorylated derivatives **3a—c** was considered to be complete when the signals for the starting bisdiamidophosphite **3a—c** (δ 132—134) in the ^{31}P NMR spectrum disappeared. Simultaneously, the accumulation of the signals for cycloamidophosphite **5a—c** (δ 138—141) and phosphorous acid triamide **2a—c** (δ 117—122) liberated in the course of the reaction was observed. The course of this process depended not only on the nature of substituent at the nitrogen atom, but also on the solvent used. Thus, in the case of the methyl derivative and acetonitrile as the solvent the process stopped after 40% of the starting bisphosphite **3a** was consumed. A prolonged heating the mixture at 70 °C did not improve conversion of the reagents, however, the addition of dimethylamine hydrochloride allowed the process to reach completion within 10 days. The dismutation of ethyl derivative **3b** was complete only within 17 days. The use of diethyl ether or dioxane instead of acetonitrile did not change the essence and the rate of the process. The use of dichloromethane as the solvent sharply accelerated the process, which was complete within 9 days. No dismutation of any of the derivatives takes place in hexane. Such results indicate a complicated mechanism, apparently, including various factors such as solvation, removal of the product formed from the reaction sphere, formation of supramolecular structures between the forming and the starting compounds, as well as the solvent effects.

1,6-Dihydroxynaphthalene derivatives can be formed as two structural isomers: with a sequential (1,6′,1′,6) and an in pairs (1,1′,6′,6) bonding the oxy groups in the ring, as it was observed for 1,7-dihydroxynaphthalene derivatives.[23,24]



1,6′,1′,6-Isomer



1,1′,6′,6-Isomer

Actually, irrespective of the preparation method the structures based on 1,6-dihydroxynaphthalene were formed only as one isomer, that was confirmed by the spectral data. Thus, the ^{31}P NMR spectrum of derivative **5a** recorded at 20 °C exhibited three signals in the region δ 139—140 (Δδ ≈ 0.64 ppm), that is due to the presence of conformers for cycloamidophosphite. Heating the sample to 95 °C (dioxane) resulted in the collapse of the signals to a sharp singlet and its slight downfield shift (δ 142.5). The ^{1}H and ^{13}C NMR spectra had one set of signals related to the corresponding groups of atoms. Analysis of the solutions of cycloamidophosphites by TLC using various eluents showed the presence of only one spot, indicating the formation of one product. In the alternative 1,1′,6′,6-isomer, the phosphorus atoms are chemically nonequivalent.

Calculations of the equilibrium geometry of the cycloamidophosphite molecule **5a** on the PM3 semiempiri-

cal level and *ab initio* HF(3-21G) quantum chemical calculations showed that each of two possible isomers can form two configurations (Fig. 1).

When oxy groups in the cycle are bonded in pairs (1,1′,6′,6), formation of two configurations is possible: with the naphthalene rings completely eclipsed (Fig. 1, *a*) and with those shifted with respect to each other (Fig. 1, *b*). In this case, two signals would have been observed in the ^{31}P NMR spectrum and the protons at position 8 of the naphthalene system would have been equivalent.

When oxy groups in the cycle are bonded sequentially (1,6′,1′,6), formation of two configurations is also possible: with the shifted (Fig. 1, *c*) and the perpendicularly crossed naphthalene fragments (Fig. 1, *d*). The homonuclear spatial correlation spectra (ROESY) showed that the configuration with the perpendicularly crossed naphthalene fragments is the most probable (Fig. 1, *d*), since the spectra have the cross-peaks responsible for the spatial interaction of protons H(7)—H(8′) and H(7′)—H(8). If the configuration with the shifted naphthalene rings would be in place, such peaks would come from protons H(5) and H(8). The assignment made is also confirmed by the fact that protons H(8) of the naphthalene system have different chemical shifts, since the calculations show that the distances between protons H(7)—H(8′) and H(7′)—H(8) differ by 0.05 Å.

It was also shown that the arrangement of the aromatic fragments with respect to each other did not depend on the substituent at the phosphorus atom.

The studies performed allowed us to suggest that the isolated products **5a—c** are the 1,6′,1′,6-isomers and have the configuration with the perpendicularly crossed naphthalene fragments.

Some chemical properties of the compounds obtained were studied by their sulfurization and oxidation (Scheme 3).

Sulfurization was carried out in dichloromethane and readily occurred at room temperature within 24 h. The formed thionophosphates **7a—c** after purification by column chromatography were either powdered compounds with low melting points (**7a**,**b**) or oily compounds (**7c**). It should be noted that column chromatography sharply decreased the yield of the products **7b**,**c** because of their partial destruction. In the ^{31}P NMR spectra of compounds **7a—c**, a singlet was observed in the region δ 66—74 characteristic of cyclomonoamidothionophosphates.

Oxidation was carried out with percarbamide in the solution in dichloromethane for 24 h (see Scheme 3). The obtained cyclophosphates **8a—c** after reprecipitation from dichloromethane with hexane were powders. In their ^{31}P NMR spectra, a singlet was observed in the region δ 1 characteristic of monoamidophosphates. Cycloamido-

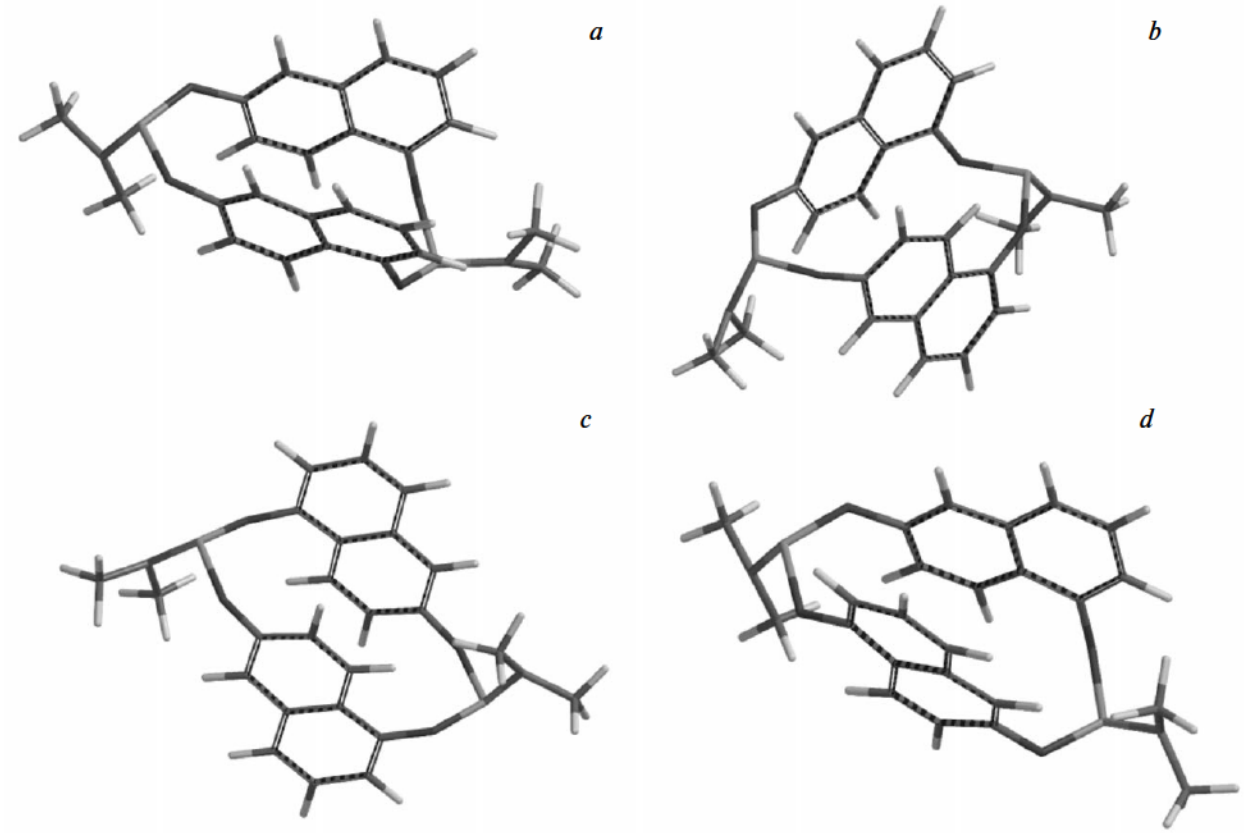
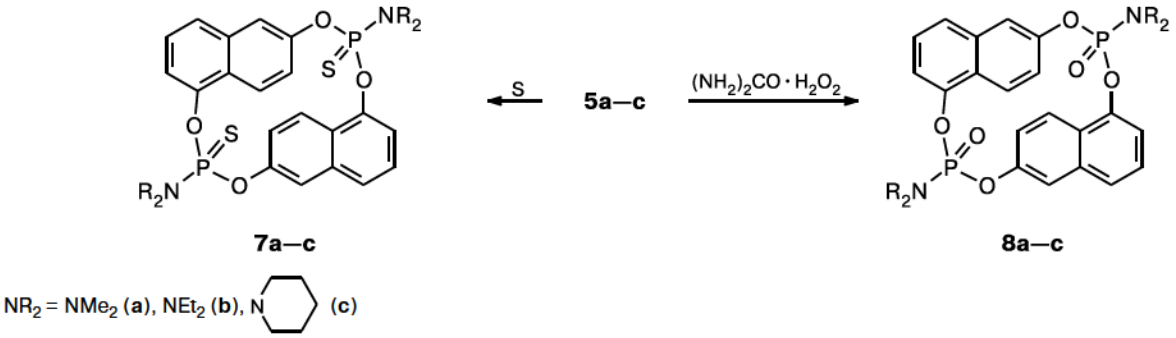


Fig. 1. Calculation of the equilibrium geometry of cyclo[bis(1,6-naphthylenedimethylamidophosphite)] molecule (**5a**) on the PM3 level.

Scheme 3



phosphates **8a—c** had lower melting points than those of cycloamidothionophosphates **7a—c**.

The structure and individuality of thionophosphates **7a—c** and phosphates **8a—c** were confirmed by TLC, ^{31}P, ^{1}H, and ^{13}C NMR spectroscopy, elemental analysis, and MALDI-TOF. It should be emphasized that in the ^{1}H NMR spectra of thionophosphates **7a—c** and phosphates **8a—c**, the signals for all the groups of protons were broadened and the region of the aromatic protons was considerably narrowed as compared to the similar cyclophosphites **5a—c**. Apparently, this effect was caused by the increase in the rigidity of the aromatic framework of the molecule because of the change in the configuration of the phosphorus fragment. Also note that in the ^{1}H NMR spectra of derivatives **7a—c**, the protons at positions 8 of different naphthalene rings H(8) and H(8′) had the same chemical shift.

Calculations of the equilibrium geometry of the cycloamidothionophosphate molecule **7a** on the PM3 semiempirical level (Fig. 2) showed that unlike in derivative **5a**, the distance between protons H(7)—H(8′) and H(7′)—H(8) is the same, that defines the pattern of the ^{1}H NMR spectrum.

The NMR spectroscopic data, low melting points (110—156 °C), and analysis of the literature data on naphthalenophanes[25,26] and naphthophosphacyclophanes[18] also indicate that the aromatic rings are placed at an angle to each other and eclipsed only partially.

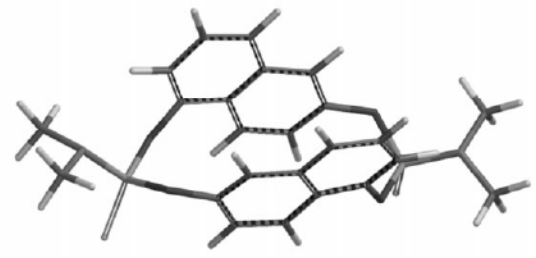


Fig. 2. Calculation of the equilibrium geometry of cyclo[bis-(1,6-naphthylenedimethylamidothionophosphate)] molecule (**7a**) on the PM3 level.

In the molecules of 1,6-dihydroxynaphthalene (**1**) and its derivatives, the protons of the part of the aromatic system containing α-oxy group form an ABC-system, whereas of that containing β-oxy groupy form an ABX-system. The ^{1}H NMR spectrum of compound **1** exhibited one triplet ($H_{B'}$), four doublets ($H_{A'}$, $H_{C''}$, H_A, H_B), and one singlet (H_X) with chemical shifts 7.23, 6.64, 7.08, 7.25, 8.10, and 7.11, respectively, the spin-spin coupling constants H_A—$H_{B'}$ and H_A—H_B were 6.4 and 9.1 Hz, respectively (Fig. 3).

In the acyclic derivative **9** (see Fig. 3) synthesized based on 1,6-dihydroxynaphthalene and tetraethyldiamidophenylphosphite, an expected downfield shift of virtually all the aromatic signals was observed because of the presence of the electron-withdrawing amidothionophosphate group. An exception was the signals for the H_A proton at position 8, which was shifted upfield. The protons H_X and $H_{A'}$ were shifted the most ($\Delta\delta = 0.52$ and 0.74 ppm, respectively). For the cyclic structure **7b**, the upfield shift of the signals (with respect to the acyclic derivative **9**) was observed only for protons H_X and $H_{A'}$ because of the greater shielding these protons as a result of their interaction with the second aromatic ring located opposite, whereas for the other protons, the signals were shifted downfield even further. This indicates that the aromatic rings are not eclipsed and arranged cross-like with respect to each other, that agrees with the simulation results (see Fig. 2). The spin-spin coupling constants $H_{A'}$—$H_{B'}$ and H_A—H_B going from compound **1** to acyclic product **9** and further to naphthophosphacyclophane **7b** virtually do not change ($\Delta J \approx 0.2$ Hz).

Note that, like in the case with cyclophosphites, the spectra also exhibited signals for a secondary amine, diethylamine or piperidine, which remained in the product even after purification by column chromatography. This is probably due to the formation of an intermolecular complex stabilized by the lone electron pair of nitrogen and the π-accepting aromatic block of the naphthalene ring. This-type complexes are described in the literature[27,28] and are stable enough systems.

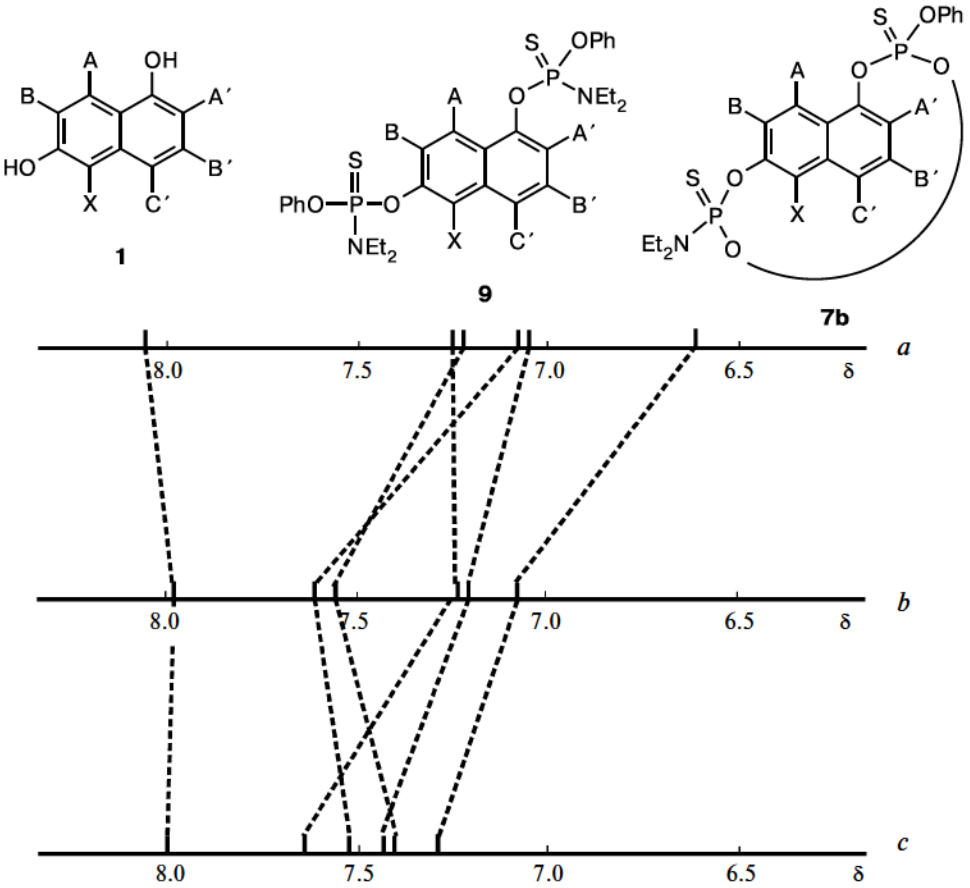


Fig. 3. Chemical shifts (δ) of the aromatic spin-spin systems in compounds **1** (*a*), **9** (*b*), and **7b** (*c*), (solvent $CDCl_3$, 400 MHz).

Experimental

^1H and ^{13}C NMR spectra of compounds **4a—c**, **5c**, **6b**, **7a—c** were recorded on a Bruker AC-200 spectrometer (200 and 80 MHz, respectively), ^1H and ^{13}C NMR spectra of compounds **5a,b**, **8a—c**, **9** and ^{31}P NMR spectra were recorded on a JEOL ECX-400 spectrometer (400, 100.5, and 161.8 MHz, respectively). In a number of cases, the $J_{P,H}$ spin-spin coupling constants were determined using the double resonance experiments. Chemical shifts (δ) for ^1H and ^{13}C are given relative to $SiMe_4$, for ^{31}P, relative to 85% aqueous phosphoric acid.

Mass spectra were obtained on a Bruker Reflex III instrument, using $CHCl_3$ as a solvent, 2,4,6-trihydroxyacetophenone as a matrix. All the syntheses were performed in anhydrous solvents under dry nitrogen. Adsorption column chromatography was carried out on silica gel L 100/250 μm, TLC on Silufol plates. Compound were visualized in the iodine vapors and charring. The full amides of phosphorous acid were obtained according to the procedures described earlier: HMTA (**2a**),[29] HETA (**2b**),[30] and TPP (**2c**).[31]

1,6-Bis(*N*,*N*,*N*′,*N*′-tetraalkyldiaminothiophosphoryloxy)-naphthalenes (4a—c). 1,6-Dihydroxynaphthalene (**1**) (0.2 g. 1.25 mmol) in MeCN (5 mL) was added to triamidophosphite **2a—c** (2.5 mmol) at room temperature and with continuous stirring. After 5 min (**a**), 1.5 h (**b**), or 40 min (**c**), sulfur (0.08 g. 2.5 mmol) was added to the reaction mixture and it was stirred for another 4 h. Then, the solution was filtered, the solvent was evaporated *in vacuo*, and the residue was subjected to chromatography on a column, eluting products with the system C_6H_{14}—dioxane, 10 : 1. The products obtained were dried *in vacuo* for 2 h (70 °C, 1 Torr).

1,6-Bis(*N*,*N*,*N*′,*N*′-tetramethyldiaminothiophosphoryloxy)-naphthalene (4a). ^1H NMR (CD_3CN), δ: 2.73 (d, 12 H, CH_3, $^3J_{P,H}$ = 11.5 Hz); 2.78 (d, 12 H, CH_3, $^3J_{P,H}$ = 12.1 Hz); 7.16 (br.d, 1 H, H(2), $^3J_{H(2),H(3)}$ = 7.7 Hz); 7.30 (dm, 1 H, H(7), $^3J_{H(7),H(8)}$ = 9.4 Hz, $^4J_{P,H}$ = 1.2 Hz); 7.46 (dd, 1 H, H(3), $^3J_{H(2),H(3)}$ = 7.7 Hz, $^3J_{H(3),H(4)}$ = 8.3 Hz); 7.51 (br.s, 1 H, H(5)); 7.66 (d, 1 H, H(4), $^3J_{H(3),H(4)}$ = 8.3 Hz); 8.10 (d, 1 H, H(8), $^3J_{H(7),H(8)}$ = 9.4 Hz). ^{31}P NMR (CH_3CN), δ: 81.5, 80.9.

1,6-Bis(*N*,*N*,*N*′,*N*′-tetraethyldiaminothiophosphoryloxy)-naphthalene (4b). ^1H NMR (CD_3CN), δ: 1.15 (t, 12 H, CH_3, ^3J = 6.6 Hz); 1.17 (t, 12 H, CH_3, ^3J = 7.2 Hz); 3.21 (m, 8 H, CH_2N, $^3J_{P,H}$ = 12.1 Hz); 3.28 (m, 8 H, CH_2N, $^3J_{P,H}$ = 14.8 Hz); 7.35 (br.d, 1 H, H(2), $^3J_{H(2),H(3)}$ = 7.7 Hz); 7.38 (d, 1 H, H(7), $^3J_{H(7),H(8)}$ = 8.0 Hz); 7.43 (dd, 1 H, H(3), $^3J_{H(2),H(3)}$ = 7.7 Hz, $^3J_{H(3),H(4)}$ = 8.3 Hz); 7.57 (d, 1 H, H(5), $^4J_{P,H}$ = 2.2 Hz); 7.60 (d, 1 H, H(4), $^3J_{H(3),H(4)}$ = 8.3 Hz); 8.10 (d, 1 H, H(8), $^3J_{H(7),H(8)}$ = = 8.8 Hz). ^{31}P NMR (CH_3CN), δ: 76.3, 75.5.

1,6-Bis(dipiperidinothiophosphoryloxy)naphthalene (4c). ^1H NMR (CD_3CN), δ: 1.58 (m, 24 H, CH_2); 3.17 (m, 8 H, $C_{ax}H_2N$, $^3J_{P,H}$ = 7.7 Hz); 3.21 (m, 8 H, $C_{eq}H_2N$, $^3J_{P,H}$ = 6.6 Hz); 7.36 (d, 1 H, H(2), $^3J_{H(2),H(3)}$ = 7.7 Hz); 7.37 (d, 1 H, H(7), $^3J_{H(7),H(8)}$ = = 9.0 Hz); 7.48 (dd, 1 H, H(3), $^3J_{H(2),H(3)}$ = 7.7 Hz, $^3J_{H(3),H(4)}$ =

= 7.2 Hz); 7.53 (d, 1 H, H(4), $^3J_{H(3),H(4)}$ = 7.2 Hz); 7.56 (d, 1 H, H(5), $^4J_{P,H}$ = 1.8 Hz); 8.07 (d, 1 H, H(8), $^3J_{H(7),H(8)}$ = 9.1 Hz). ^{31}P NMR (CH_3CN), δ: 74.3, 74.9.

Cyclo[bis(1,6-naphthylenealkylamidophosphites)] (5a,b). ***Method of molecular assembly.*** A solution of naphthodiol **1** (0.32 g, 2 mmol) in anhydrous MeCN (5 mL) was added to a solution of triamide **2a**,**b** (4 mmol) in the same solvent (6 mL) at room temperature with continuous stirring. Then, an additional portion of naphthodiol **1** (0.32 g. 2 mmol) in MeCN (5 mL) was added to the reaction mixture after 4 min for **2a** or 1.5 h for **2b**, and stirring was continued for another 3 h. A precipitate formed within 24 h was washed with MeCN and dried *in vacuo* for 2.5 h (70 °C, 1 Torr).

Method of direct synthesis. A solution of naphthodiol **1** (0.64 g, 4 mmol) in anhydrous MeCN (5 mL) was added to a solution of triamide **2a**,**b** (4 mmol) in the same solvent (5 mL). After 24 h, a solution was decanted from a precipitate formed, the precipitate was washed with MeCN and dried *in vacuo* for 2.5 h (70 °C, 1 Torr).

Cyclo[bis(1,6-naphthylenedimethylamidophosphite)] (5a). ^{1}H NMR ($CDCl_3$), δ: 2.84 (d, 6 H, CH_3, $^3J_{P,H}$ = 9.9 Hz); 2.93 (d, 6 H, CH_3, $^3J_{P,H}$ = 11.0 Hz); 7.04 (d, 2 H, H(2), H(2′), $^3J_{H(2),H(3)} = {}^3J_{H(2'),H(3')}$ = 7.7 Hz); 7.25 (d, 1 H, H(7), $^3J_{H(7),H(8)}$ = 8.8 Hz); 7.28 (d, 1 H, H(7′), $^3J_{H(7'),H(8')}$ = 9.4 Hz); 7.34 (dd, 2 H, H(3), H(3′), $^3J_{H(2),H(3)} = {}^3J_{H(2'),H(3')}$ = 7.7 Hz, $^3J_{H(3),H(4)} = {}^3J_{H(3'),H(4')}$ = 9.4 Hz); 7.44 (d, 2 H, H(4), H(4′), $^3J_{H(3),H(4)} = {}^3J_{H(3'),H(4')}$ = 9.4 Hz); 7.47 (s, 2 H, H(5), H(5′)); 8.17 (d, 1 H, H(8), $^3J_{H(7),H(8)}$ = 8.8 Hz); 8.21 (d, 1 H, H(8′), $^3J_{H(7'),H(8')}$ = 9.4 Hz). ^{13}C NMR ($CDCl_3$), δ: 34.7 (d, CH_3, $^2J_{P,C}$ = 22.3 Hz); 35.2 (d, CH_3, $^2J_{P,C}$ = 22.3 Hz); 112.1 (d, C(2), C(2′), $^3J_{P,C}$ = 14.9 Hz); 115.2 (d, C(5), C(5′), $^3J_{P,C}$ = 10.6 Hz); 120.9 (d, C(7), C(7′)); 121.9 (s, C(4), C(4′)); 124.0 (s, C(9), C(9′)); 124.3 (s, C(8), C(8′)); 126.5 (s, C(3), C(3′)); 135.9 (s, C(10), C(10′)); 149.8 (d, C(1), (1′), $^2J_{P,C}$ = 12.1 Hz); 152.0 (d, C(6), C(6′), $^3J_{P,C}$ = 11.8 Hz). ^{31}P NMR (CH_2Cl_2), δ: 139.4.

Cyclo[bis(1,6-naphthylenediethylamidophosphite)] (5b). ^{1}H NMR ($CDCl_3$), δ: 1.12 (t, 12 H, CH_3, ^3J = 7.7 Hz); 3.41 (m, 8 H, CH_2N, $^3J_{P,H}$ = 10.1 Hz); 7.06 (d, 2 H, H(2), H(2′), $^3J_{H(2),H(3)} = {}^3J_{H(2'),H(3')}$ = 8.7 Hz); 7.23 (d, 2 H, H(7), H(7′), $^3J_{H(7),H(8)} = {}^3J_{H(7'),H(8')}$ = 9.2 Hz); 7.25 (d, 2 H, H(4), H(4′), $^3J_{H(3),H(4)} = {}^3J_{H(3'),H(4')}$ = 9.2 Hz); 7.39 (dd, 2 H, H(3), H(3′), $^3J_{H(2),H(3)} = {}^3J_{H(2'),H(3')}$ = 8.7 Hz, $^3J_{H(3),H(4)} = {}^3J_{H(3'),H(4')}$ = 9.2 Hz); 7.44 (s, 2 H, H(5), H(5′)); 8.12 (d, 1 H, H(8), $^3J_{H(7),H(8)}$ = 9.2 Hz); 8.17 (d, 1 H, H(8′), $^3J_{H(7'),H(8')}$ = 9.2 Hz). ^{13}C NMR ($CDCl_3$), δ: 14.9 (s, CH_3); 38.2 (d, CH_2, $^2J_{P,C}$ = 28.1 Hz); 111.9 (d, C(2), C(2′), $^3J_{P,C}$ = 15.2 Hz); 115.2 (d, C(5), C(5′), $^3J_{P,C}$ = 10.6 Hz); 120.9 (d, C(7), C(7′)); 121.8 (s, C(4), C(4′)); 124.1 (s, C(9), C(9′)); 124.5 (s, C(8), C(8′)); 136.0 (s, C(10), C(10′)); 150.2 (d, C(1), C(1′), $^3J_{P,C}$ = 13.8 Hz); 152.3 (d, C(6), C(6′), $^3J_{P,C}$ = 14.2 Hz). ^{31}P NMR (CH_2Cl_2): δ = 140.9.

Cyclo[bis(1,6-naphthylenepiperidinophosphite)] (5c). ***Method of molecular assembly.*** A solution of 1,6-dihydroxynaphthalene **1** (0.32 g. 2 mmol) in MeCN (25 mL) was added to TPP **2c** (1.13 g, 4 mmol) dissolved in C_6H_6 (10 mL) at room temperature with continuous stirring. After 40 min, the solvents were completely evaporated from the reaction mixture, and naphthodiol **1** (0.32 g, 2 mmol) in MeCN (20 mL) was added. The solution obtained was stirred for another 2 h. After 24 h, the solution was decanted from a precipitate formed, the precipitate was washed with MeCN and dried *in vacuo* for 2 h (70 °C, 1 Torr).

Method of direct synthesis. A solution of 1,6-dihydroxynaphthalene **1** (0.64 g. 4 mmol) in MeCN (20 mL) was added to TPP **2c** (1.13 g. 4 mmol) dissolved in C_6H_6 (10 mL) at room temperature with continuous stirring. Then, the solution was stirred for 30 min, the solvents were evaporated, the reaction mixture was dissolved in MeCN (20 mL) and stirred for another 2 h. After 24 h, the solution was decanted from a precipitate formed, cyclophosphite **5c** was washed with MeCN and dried *in vacuo* for 2 h (70 °C, 1 Torr). ^{1}H NMR ($CDCl_3$), δ: 1.53 (m, 12 H, CH_2); 3.34 (m, 8 H, CH_2N, $^3J_{P,H}$ = 8.3 Hz); 7.11 (d, 2 H, H(2), H(2′), $^3J_{H(2),H(3)} = {}^3J_{H(2'),H(3')}$ = 7.7 Hz); 7.25 (d, 1 H, H(7), $^3J_{H(7),H(8)}$ = 8.8 Hz); 7.29 (d, 1 H, H(7′), $^3J_{H(7'),H(8')}$ = = 9.9 Hz); 7.31 (t, 2 H, H(3), H(3′), $^3J_{H(2),H(3)} = {}^3J_{H(2'),H(3')}$ = = 7.7 Hz); 7.37 (br.d, 2 H, H(4), H(4′)); 7.45 (s, 2 H, H(5), H(5′)); 8.17 (d, 1 H, H(8), $^3J_{H(7),H(8)}$ = 8.8 Hz); 8.21 (d, 1 H, H(8′), $^3J_{H(7'),H(8')}$ = 9.9 Hz). ^{31}P NMR (C_6H_6): δ = 135.7.

6-Hydroxy-1-(*N*,*N*,*N*′,*N*′-tetraethyldiaminothiophosphoryloxy)naphthalene (6b) and 1-hydroxy-6-(*N*,*N*,*N*′,*N*′-tetraethyldiaminothiophosphoryloxy)naphthalene (6′b). A solution of diol **1** (0.32 g. 2 mmol) in MeCN (10 mL) was added to HETA **2b** (0.5 g, 2 mmol) at room temperature with stirring. After 90 min, sulfur (0.8 g. 2.5 mmol) was added to the reaction mixture. After 24 h, the solution was filtered, the solvent was evaporated *in vacuo*, the residue was subjected to chromatography on a column, eluting a mixture of isomers with the system C_6H_6—dioxane, 10 : 1. The mixture obtained was dried *in vacuo* for 2 h (70 °C, 1 Torr). Compound **6b**: ^{1}H NMR (CD_3CN), δ: 1.15 (t, 12 H, CH_3); 3.28 (m, 8 H, CH_2N, $^3J_{P,H}$ = 7.2 Hz); 7.14 (d, 1 H, H(2)); 7.17 (t, 1 H, H(3)); 7.31 (d, 1 H, H(7), $^3J_{H(7),H(8)}$ = 8.8 Hz); 7.33 (s, 1 H, H(5)); 7.50 (d, 1 H, H(4)); 8.01 (d, 1 H, H(8), $^3J_{H(7),H(8)}$ = 8.8 Hz). ^{31}P NMR (1,4-dioxane): δ = 76.0. Compound **6′b**: ^{1}H NMR (CD_3CN), δ: 1.14 (t, 12 H, CH_3); 3.28 (m, 8 H, CH_2N, $^3J_{P,H}$ = 6.6 Hz); 6.82 (d, 1 H, H(2)); 7.20 (t, 1 H, H(3)); 7.24 (d, 1 H, H(7), $^3J_{H(7),H(8)}$ = 9.4 Hz); 7.30 (s, 1 H, H(5)); 7.50 (d, 1 H, H(4)); 8.13 (d, 1 H, H(8), $^3J_{H(7),H(8)}$ = 9.4 Hz). ^{31}P NMR (1,4-dioxane): δ = 74.5.

Cyclo[1,6-bis(naphthylenedialkylaminothionophosphates)] (7a—c). Sulfur (0.13 g. 4 mmol) was added to cyclophosphite **5a—c** (2 mmol) in CH_2Cl_2 (12 mL). The mixture was stirred at room temperature for 2 h and allowed to stand for 24 h. Then, the solution was filtered, the solvent was evaporated *in vacuo*, the residue was subjected to chromatography on a column, eluting the products obtained with the system C_6H_6—dioxane, 7 : 1 (for **7a** and **7c**), or C_6H_6—dioxane, 10 : 1 (for **7b**). The isolated cyclothionophosphates were dried *in vacuo* for 2 h (70 °C, 1 Torr).

Cyclo[bis(1,6-naphthylenedimethylaminothionophosphate)] (7a). ^{1}H NMR ($CDCl_3$), δ: 3.08 (d, 12 H, CH_3, $^3J_{P,H}$ = 11.8 Hz); 7.38 (d, 2 H, H(2), H(2′), $^3J_{H(2),H(3)} = {}^3J_{H(2'),H(3')}$ = 7.7 Hz); 7.41 (d, 2 H, H(7), H(7′), $^3J_{H(7),H(8)} = {}^3J_{H(7'),H(8')}$ = 8.3 Hz); 7.51 (t, 2 H, H(3), H(3′), $^3J_{H(2),H(3)} = {}^3J_{H(2'),H(3')}$ = 7.7 Hz); 7.54 (s, 2 H, H(5), H(5′)); 7.65 (d, 2 H, H(4), H(4′)); 8.01 (d, 2 H, H(8), H(8′), $^3J_{H(7),H(8)} = {}^3J_{H(7'),H(8')}$ = 8.2 Hz). ^{31}P NMR (CH_2Cl_2): δ = 67.8.

Cyclo[bis(1,6-naphthylenediethylaminothionophosphate)] (7b). ^{1}H NMR ($CDCl_3$), δ: 1.17 (t, 12 H, CH_3); 3.54 (m, 8 H, CH_2N, $^3J_{P,H}$ = 11.0 Hz); 7.39 (d, 2 H, H(2), H(2′)); 7.43 (d, 2 H, H(7), H(7′), $^3J_{H(7),H(8)} = {}^3J_{H(7'),H(8')}$ = 8.3 Hz); 7.49 (t, 2 H, H(3), H(3′)); 7.57 (s, 2 H, H(5), H(5′)); 7.70 (d, 2 H, H(4), H(4′)); 8.03 (d, 2 H, H(8), H(8′), $^3J_{H(7),H(8)} = {}^3J_{H(7'),H(8')}$ = = 8.3 Hz). ^{31}P NMR (CH_2Cl_2): δ = 66.4.

Cyclo[bis(1,6-naphthylenepiperidinothionophosphate)] (7c). ^{1}H NMR ($CDCl_3$), δ: 1.58 (br.d, 12 H, CH_2); 3.05 (br.m, 8 H,

Table 1. Physicochemical characteristics of compounds synthesized

Compound	Yield (%) (method)	M.p./°C	R_f (system)*	Found / Calculated (%)				Molecular formula	MS	
				C	H	N	P		Found *m/z*, M	Calculated M
4a	42	150—151	0.59 (A)	46.91 46.93	6.54 6.57	12.18 12.17	13.46 13.46	$C_{18}H_{30}N_4O_2P_2S_2$	—	—
4b	38	Oil	0.63 (A)	54.32 54.52	8.10 8.10	9.71 9.78	10.84 10.76	$C_{26}H_{46}N_4O_2P_2S_2$	—	—
4c	39	Oil	0.70 (A)	— —	— —	— —	9.96 9.98	$C_{30}H_{46}N_4O_2P_2S_2$	620.3	621
5a	68 (*A*) 70 (*B*) 46 (*C*)	229—230	0.63 (B)	61.58 61.79	5.24 5.18	6.41 6.01	13.18 13.08	$C_{24}H_{24}N_2O_4P_2$	—	—
5b	62 (*A*) 66 (*B*) 52 (*C*)	101—102	0.75 (B)	—	—	—	11.81 11.86	$C_{28}H_{32}N_2O_4P_2$	522.6	522
5c	36 (*A*) 41 (*B*)	Oil	0.69 (B)	65.88 65.93	5.98 5.90	5.20 5.13	11.27 11.33	$C_{30}H_{32}N_2O_4P_2$	—	—
6b and **6′b**	38	Oil	0.33 (A)	—	—	—	—	—	—	—
7a	79	110—111	0.91 (B)	—	—	—	11.58 11.67	$C_{24}H_{24}N_2O_4P_2S_2$	530.1	530
7b	77	138—139	0.82 (B)	57.29 57.32	5.48 5.50	4.75 4.72	10.57 10.56	$C_{28}H_{32}N_2O_4P_2S_2$	—	—
7c	28	Oil	0.51 (B)	—	—	—	10.19 10.14	$C_{30}H_{32}N_2O_4P_2S_2$	—	—
8a	94	155—156	0.69 (C)	57.83 57.83	4.75 4.75	5.62 5.62	12.46 12.43	$C_{24}H_{24}N_2O_6P_2$	—	—
8b	93	131—132	0.81 (C)	—	—	—	11.15 11.17	$C_{28}H_{32}N_2O_6P_2$	554.2	555
8c	90	133–134	0.88 (C)	62.36 62.28	5.50 5.58	4.89 4.84	10.71 10.77	$C_{30}H_{32}N_2O_6P_2$	—	—
9	78	Oil	0.79 (A)	—	—	—	—	—	—	—

* C_6H_{14}—dioxane, 3 : 1 (A); C_6H_6—dioxane, 5 : 1 (B); $CHCl_3$—EtOH, 5 : 1 (C).

CH_2N, $^3J_{P,H}$ = 6.4 Hz); 7.08 (d, 2 H, H(2), H(2′)); 7.41 (br.m, 4 H, H(5), H(5′), H(7), H(7′)); 7.59 (br.t, 2 H, H(3), H(3′)); 7.73 (d, 2 H, H(4), H(4′)); 8.06 (d, 2 H, H(8), H(8′)). ^{31}P NMR (CH_2Cl_2): δ = 64.2.

Cyclo[bis(naphthylenedialkylaminophosphates)] (8a—c). Percarbamide (0.5 g) was added to cyclophosphite **5a—c** (2 mmol) in CH_2Cl_2 (8 mL). The mixture was stirred at room temperature for 4 h and allowed to stand for 24 h. Then, the solution was cooled to −5 °C and filtered, the solvent was evaporated *in vacuo* to the minimum volume, followed by addition of hexane (20 mL). The solution was decanted from a precipitate formed, the precipitate was dissolved in CH_2Cl_2 (1 mL), C_6H_{14} (20 mL) was added. After 24 h, the solution was decanted from a precipitate formed, the precipitate was dried *in vacuo* for 2 h (70 °C, 1 Torr).

Cyclo[bis(1,6-naphthylenedimethylaminophosphate)] (8a). ^{1}H NMR ($CDCl_3$), δ: 2.82 (d, 6 H, CH_3, $^3J_{P,H}$ = 17.6 Hz); 2.85 (d, 6 H, CH_3, $^3J_{P,H}$ = 17.6 Hz); 7.42 (d, 2 H, H(2), H(2′), $^3J_{H(2),H(3)} = {}^3J_{H(2'),H(3')}$ = 7.7 Hz); 7.47 (d, 1 H, H(7), $^3J_{H(7),H(8)}$ = = 10.1 Hz); 7.50 (d, 1 H, H(7′), $^3J_{H(7'),H(8')}$ = 10.8 Hz); 7.53 (dd, 2 H, H(3), H(3′), $^3J_{H(2),H(3)} = {}^3J_{H(2'),H(3')}$ = 7.7 Hz, $^3J_{H(3),H(4)} = {}^3J_{H(3'),H(4')}$ = 8.3 Hz); 7.57 (d, 2 H, H(4), H(4′), $^3J_{H(3),H(4)} = {}^3J_{H(3'),H(4')}$ = 8.3 Hz); 7.72 (s, 2 H, H(5), H(5′)); 8.06 (d, 1 H, H(8), $^3J_{H(7),H(8)}$ = 10.1 Hz); 8.12 (d, 1 H, H(8′), $^3J_{H(7'),H(8')}$ = 10.7 Hz). ^{31}P NMR (CH_2Cl_2): δ = 1.8.

Cyclo[bis(1,6-naphthylenediethylaminophosphate)] (8b). ^{1}H NMR ($CDCl_3$), δ: 1.03 (t, 12 H, CH_3, ^3J = 6.5 Hz); 3.28 (m, 8 H, CH_2N, $^3J_{P,H}$ = 11.3 Hz); 7.37 (m, 4 H, H(2), H(2′), H(7), H(7′)); 7.53 (m, 4 H, H(3), H(3′), H(4), H(4′)); 7.72 (s, 2 H, H(5), H(5′)); 8.01 (d, 1 H, H(8), $^3J_{H(7),H(8)}$ = 10.9 Hz); 8.05 (d, 1 H, H(8′), $^3J_{H(7'),H(8')}$ = 10.5 Hz). ^{13}C NMR ($CDCl_3$), δ: 13.9 (s, CH_3); 40.1 (d, CH_2, $^2J_{P,C}$ = 32.4 Hz); 114.4 (d, C(2), C(2′)); 116.6 (d, C(5), C(5′), $^3J_{P,C}$ = 10.1 Hz); 120.7 (d, C(7), C(7′)); 121.8 (s, C(4), C(4′)); 124.1 (s, C(9), C(9′)); 124.3 (s, C(8), C(8′)); 135.6 (s, C(10), C(10′)); 147.0 (d, C(1), C(1′), $^2J_{P,C}$ = 10.2 Hz); 149.3 (d, C(6), C(6′)). ^{31}P NMR (CH_2Cl_2): δ = 1.5.

Cyclo[bis(1,6-naphthylenepiperidinophosphate)] (8c). ^{1}H NMR ($CDCl_3$), δ: 1.45 (m, 12 H, CH_2); 3.29 (m, 8 H, CH_2N, $^3J_{P,H}$ = = 9.9 Hz); 7.43 (m, 4 H, H(2), H(2′), H(7), H(7′)); 7.58 (m, 4 H, H(3), H(3′), H(4), H(4′)); 7.75 (s, 2 H, H(5), H(5′)); 8.07 (d, 1 H,

H(8), $^3J_{H(7),H(8)}$ = 9.9 Hz); 8.13 (d, 1 H, H(8′), $^3J_{H(7′),H(8′)}$ = = 10.3 Hz). ^{31}P NMR (CH_2Cl_2): δ = –0.3.

1,6-Bis[(diethylamino)phenoxythiophosphoryloxy)naphthalene (9). A solution of diol **1** (0.16 g. 1 mmol) was added to tetraethyldiamidophenylphosphite[32] (0.54 g. 2 mmol) dissolved in dioxane (4 mL) with continuous stirring, and the reaction mixture was heated at 70 °C for 3 h and kept for 24 h. Then, sulfur (0.07 g. 2 mmol) was added to the system obtained, which was allowed to stand for 3 days. After this, the solution was filtered, the solvent was evaporated *in vacuo*, the residue was subjected to chromatography on a column, eluting product **9** with the system C_6H_{14}—dioxane, 7 : 1. Thionophosphate obtained was dried *in vacuo* for 2 h (70 °C, 1 Torr). ^{1}H NMR ($CDCl_3$), δ: 1.16 (t, 12 H, CH_3, ^3J = 7.3 Hz); 3.47 (m, 8 H, CH_2N, $^3J_{P,H}$ = 8.7 Hz); 7.16 (d, 1 H, H(2), $^3J_{H(2),H(3)}$ = 7.3 Hz); 7.17—7.30 (m, 10 H, O—Ph); 7.28 (d, 1 H, H(7), $^3J_{H(7),H(8)}$ = = 9.2 Hz); 7.34 (m, 1 H, H(3)); 7.57 (d, 1 H, H(4)); 7.63 (s, 1 H, H(5)); 7.95 (d, 1 H, H(8), $^3J_{H(7),H(8)}$ = 9.2 Hz). ^{31}P NMR (CH_2Cl_2), δ: 67.1; 67.9.

This work was financially supported by the Council on Grants at the President of the Russian Federation (Program of State Support for Young Russian Scientists-Candidates of Science (Grant MK-5272.2013.3).

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Received May 24, 2013;
in revised form June 25, 2013