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Phosphorylation of Aromatic Diols with Phosphorous Acid Triamides with Bulky Substituents

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Abstract—Phosphorylation of naphthodiols with hexaisopropyl- and hexabutyltriamides of phosphorous acid was studied for the first time. The features of these reactions were considered. A comparative analysis of the properties of the synthesized naphthophosphacyclophanes and dismutation of bisamidophosphites was performed.

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Chemistry of naphthalene systems is rich and diverse. Naphthalene and its hydroxy derivatives are good acceptors for small molecules [1, 2], involved into the formation of charge-transfer complexes [3] used in the synthesis of oligo- and polymers [4–6], and also they are widely utilized in various biological research [7–10]. One of the most important aspects of the use of hydroxy-substituted naphthalenes is the synthesis of macrocyclic structures of naphthocyclophane type [11–13]. It is obvious that the introduction of the phosphorus atoms into the naphthocyclophane structure would expand greatly the scope of their functional use.

The phosphorylation of dihydroxynaphthalenes with phosphorous acid amides have been widely studied [14–16], but phosphorylation with the reagents having bulky substituents have not been studied yet. However, the presence of bulky substituent in the molecule will not only increase the solubility of the macrocyclic systems, but also increase their preorganization level in creating supramolecular assemblies. In connection with the above, phosphorous acid triamides $P(NR_2)_3$, where R = *i*-Pr, Bu, were chosen as the phosphorylating reagents; 1,6-, 2,6-, and 2,7-dihydroxynaphthalenes **I–III** with maximum spatially removed hydroxy groups were used as aromatic components. Acetonitrile and dioxane were used as solvents.

Phenols are known to react with phosphorous triamides in various organic solvents even at room temperature without the removal of the secondary amine formed in the course of the reaction [18]. Furthermore, the phosphorylation rate depends on the nature of the leaving group: the smaller is the substituent, the faster is the reaction. Yet it was shown in [19] that when the molecule of phosphorous acid triamide contained amide groups of different reactivity and volume, the first leaving group would be the diisopropylamide moiety.

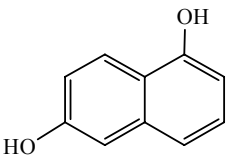
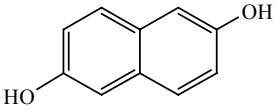
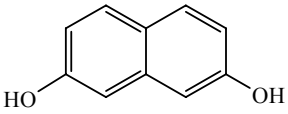
Phosphorylation was performed at room temperature at 1 : 2 ratio of the reagents. The reaction products were bisphosphorylated compounds **V–VII**. When acetonitrile was used as the solvent, the reaction rate was higher than in the case of dioxane, which was similar to the results obtained for the other triamidophosphites [15–17]. The reaction progress was monitored by ^{31}P NMR. The reaction was considered to be completed when the signal of phosphorous acid triamide [δ_P 138.2 (**IVa**), 121.3 ppm (**IVb**)] disappeared, and the signal of aryl-substituted diamidoesters of phosphorous acid appeared (δ_P 130–132 ppm).

In the case of hexaisopropyltriamidophosphite **IVa** the phosphorylation of all dihydroxynaphthalenes proceeded in acetonitrile for 2 h (Table 1).

Phosphorylation of dihydroxynaphthalenes with hexabutyltriamidophosphite **IVb** in acetonitrile occurred more slowly, which can be attributed to steric hindrances of dibutylamide leaving group (Table 1).

Unlike the derivatives **Va**, **VIa**, **VIIa**, the bisphosphorylated compounds with butyl substituents

Table 1. Time (h) of phosphorylation of dihydroxynaphthlenes **I–III** with phosphorous acid triamides **IVa, IVb** in acetonitrile

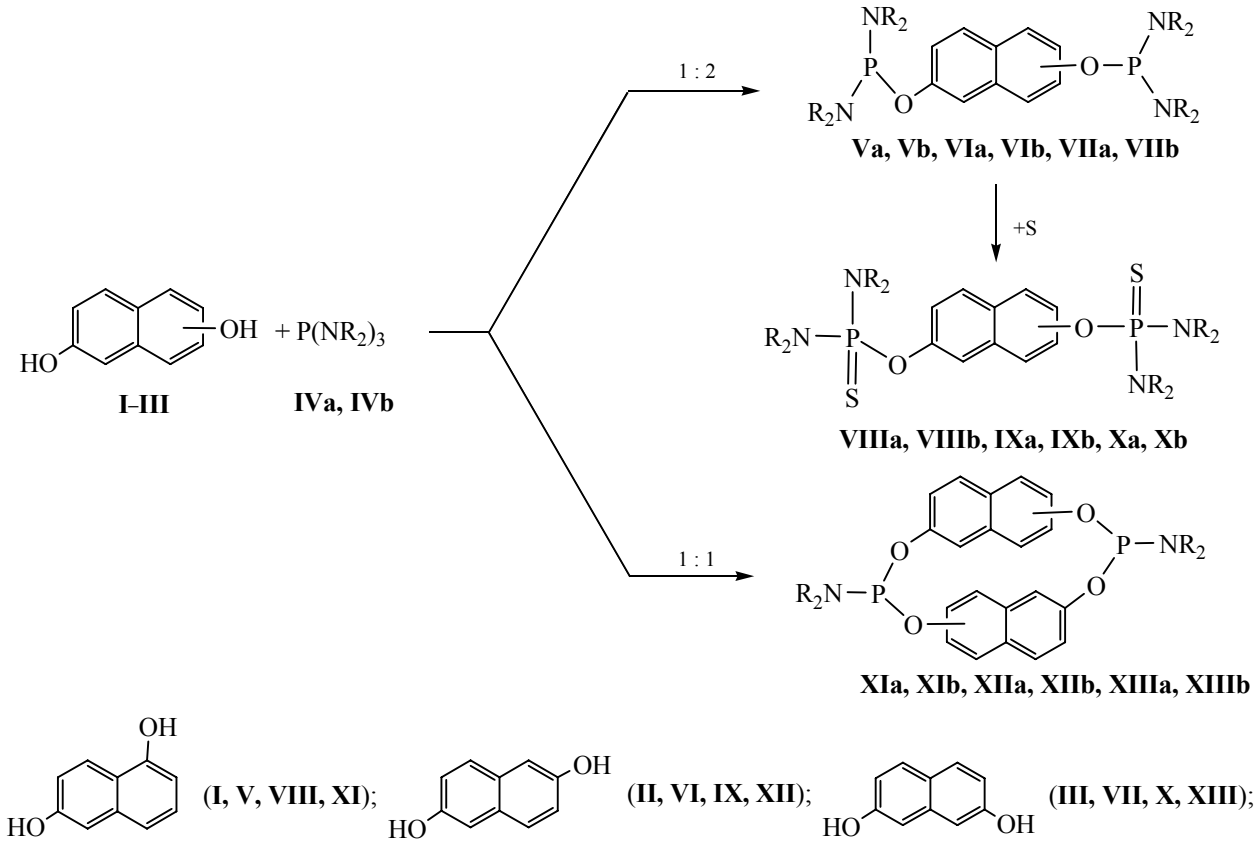
Dihydroxynaphthalene	$-CH(CH_3)_2$ (**a**)	$-CH_2CH_2CH_2CH_3$ (**b**)
	2.5	64
	2	52
	2	52

Vb, **VIb**, **VIIb** do not undergo dismutation, which is characteristic of all the diamidoarylated phosphorous acid esters [20, 21]. Note that the rate of dismutation of **Va**, **VIa**, **VIIa** is not high.

Bisamidophosphites **V–VII** were isolated by column chromatography as thiophosphates **VIII–X**. Thioamidophosphates **VIII–X** are viscous, readily soluble oils. Only derivatives **VIIb** and **Xa** were isolated in pure form.

Phosphorylation in acetonitrile at a 1 : 1 ratio of the reagents afforded naphthophosphacyclophanes **XI–XIII**. Unlike reactions with other triamides, in the case of phosphite **IVa** no precipitate formation was observed. The derivatives **XIa**, **XIIa**, **XIIIa** are readily soluble in methylene chloride, dioxane, benzene, diethyl ether and acetonitrile. ^{31}P NMR spectra of naphthophosphacyclophanes **XIa**, **XIIa**, **XIIIa** contain characteristic signals (Scheme 1, Table 2).

Scheme 1.



R = *i*-Pr (**a**); *s*-Bu (**b**).

The phosphorylation rate in acetonitrile decreases sharply when going from phosphite **IVa** to **IVb**: the reaction proceeds for over 6 days. The formed cycloamidophosphites **XIb**, **XIIb**, **XIIIb** precipitated in small quantities due to high solubility in acetonitrile. The rate of phosphorylation of dihydroxynaphthalenes with phosphite **IVb** increases with an increase in reaction temperature up to 60°C. In this case the reaction occurred within 3 h; within 20 min after the reaction start the precipitation was observed (Scheme 2).

When dioxane was used, in the case of phosphite **IVb** bisphosphorylated derivatives **V–VII** formed instead of the cyclic compounds. This distinguishes phosphite **IVb** from other phosphorous acid triamides, which reacted with dihydroxynaphthalenes in dioxane to form cyclic products [22, 23].

Since we failed to isolate cycloamidophosphites **XI–XIII** in a pure form, they were converted into the stable thiophosphates **XIV–XVI**.

In all cases sulfurization proceeded in methylene chloride for 1 day. In the ^{31}P NMR spectra there were the signals in the range of 62–64 (R = *i*-Pr) or 67–69 ppm (R = *sec*-Bu) characteristic of the aryl-substituted monoamidoesters of thiophosphoric acid. Cycloamidothiophosphates **XIV–XVI** were isolated by column chromatography. They are oily readily soluble compounds. It should be noted that the column chromatography reduces dramatically the yield of the products obtained, but it is the only method that allows to isolate amidothiophosphates **XIV–XVI** in a pure form.

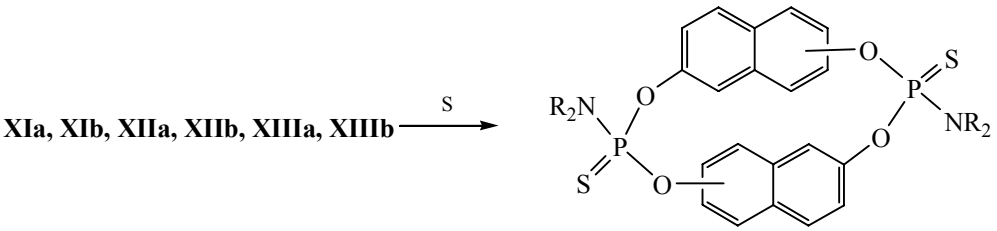
Table 2. ^{31}P NMR parameters (δ_P, ppm, CH_3CN) of diamidophosphites **V–VII** and cycloamidophosphites **XI–XIII**

Comp. no.	$-CH(CH_3)_2$ (**a**)	$-CH_2CH_2CH_2CH_3$ (**b**)
V	132.4, 130.2	131.8, 130.3
VI	130.8	132.9
VII	131.6	131.5
XI	139.9	141.4
XII	140.0	142.2
XIII	140.2	141.5

When cycloamidothiophosphates **XIV–XVI** were chromatographed, also monophosphorylated derivatives (e.g. **XVIIb**) were isolated. This proves our assumption that the cyclophosphorylation of dihydroxynaphthalenes proceeds via intermediate formation of monophosphorylated derivatives followed by their cyclization [24–26] (Scheme 3).

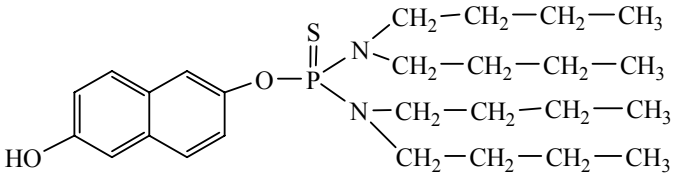
^{1}H NMR spectra of **XIV–XVI** contain the signals of dibutyl- or diisopropylamine along with the signals of the target products. The analyzis of the spectra shows that the cyclothiophosphate–dibutylamine ratio in some cases equals 1 : 1, while in the case of the corresponding isopropyl derivatives the amine content is much less (no more than 10%). We believe that the formation occurs of stable intermolecular associates capable of independent existence even under the con-

Scheme 2.



XIVa, XIVb, XVa, XVb, XVIa, XVIb

Scheme 3.



XVIIb

ditions of the harsh purification method like column chromatography.

Thus, we can conclude that the phosphorylation of dihydroxynaphthalenes with hexaisopropylttriamidophosphite obeys the previously found regularities. The special features of the phosphorylation of dihydroxynaphthalenes with hexabutyltriamidophosphite are as follows: The formation of cyclophosphites **XIb**, **XIIb**, **XIIIb** is possible only under heating to 50–60°C, which has not been observed when using triamides with other substituents (methyl, ethyl, piperidyl, isopropyl, etc.); cyclophosphites **XIb**, **XIIb**, **XIIIb** and cyclothiophosphates **XIVb**, **XVb**, **XVIb** are more soluble in comparison with their counterparts where R = CH_3, C_2H_5, C_5H_{10} (piperidyl); bisphosphorylated derivatives **Vb**, **VIb**, **VIIb** do not undergo dismutation; yields of the target products **XIb**, **XIIb**, **XIIIb** and **XIVb**, **XVb**, **XVIb** are extremely low.

EXPERIMENTAL

^1H, ^{13}C ($CDCl_3$) and ^{31}P NMR spectra were obtained on a JEOL ECX-400 spectrometer, internal reference TMS (^1H, ^{13}C), external reference 85% phosphoric acid (^{31}P). Mass spectra (MALDI-TOF) were registered on a Bruker Ultra Flex (λ 337 nm, matrix trihydroxyantracene).

All syntheses involving trivalent phosphorus compounds were carried out in a dry nitrogen atmosphere. Column chromatography was performed on a silica gel L 100–250 μ; TLC was performed on Silufol UV-254 plates eluting with hexane–acetone, 5 : 1 (A), benzene–dioxane, 5 : 1 (B). Detection was carried out by calcinations.

Phosphorous acid hexaisopropyltriamide **IVa** was synthesized by the method described in [19].

Phosphorous acid hexabutyltriamide (IVb). A solution of 5 mL (0.057 mol) of freshly distilled PCl_3 in 20 mL of anhydrous benzene was slowly added to a mixture of 58 mL (0.344 mol) of freshly distilled dibutylamine and 100 mL of anhydrous benzene with vigorous stirring and cooling (ice + water) under dry argon. The reaction mixture was stirred for 2 h and left overnight. Then, the precipitated dibutylamine hydrochloride was filtered off and washed with benzene. The filtrate was evaporated, and the residue was distilled in a vacuum. Yield 70%, bp 148–150°C (1 mm Hg). ^1H NMR spectrum, δ, ppm: 0.87 t (18H, CH_3, $^3J_{HH}$ 7.3 Hz), 1.21–1.25 m (12H, CH_2), 1.38–1.45 m (12H, CH_2), 2.74–2.77 m (12H, CH_2, $^3J_{PH}$ 15.6 Hz). ^{13}C NMR spectrum, δ_C, ppm: 14.2 s (CH_3), 20.8 s (CH_2), 31.0 s (CH_2), 46.0 d (CH_2, $^2J_{PC}$ 19.3 Hz). ^{31}P NMR spectrum, δ_P, ppm: 121.2 (without solvent), 119.4 ($CDCl_3$).

Bis(tetraalkylamidothiophosphatoxy)naphthalines (VIII–X). A mixture of 1.25 mmol of dihydroxynaphthalene **I–III** in 5 mL of acetonitrile was added to 2.5 mmol of phosphoric acid triamide **IV** at room temperature under stirring. After 2 h (method *a*) or 2 days (method *b*), 0.8 g (2.5 mmol) of sulfur was added, and the reaction mixture was left standing for 1 day. Then the precipitate was filtered off, and the solvent was distilled off in a vacuum. The residue was chromatographed on a column eluting with a mixture dioxane–hexane, 10 : 1. The resulting products were dried in a vacuum for 2 h (70°C, 1 Torr).

2,6-Bis(tetrabutyldiamidothiophosphatoxy)naphthalene (VIIIb). Yield 30%, oily substance. ^1H NMR spectrum, δ, ppm (*J*, Hz): 0.92 t (24H, CH_3, $^3J_{HH}$ 6.9), 1.31 septet (16H, CH_2, $^3J_{HH}$ 7.3), 1.59 quintet (16H, CH_2, $^3J_{HH}$ 7.3), 3.12 d. t (8H, CH_2–N, $^2J_{HH}$ 13.8, $^3J_{PH}$ 13.3), 3.15 d. t (8H, CH_2–N, $^2J_{HH}$ 14.2, $^3J_{PH}$ 12.5), 7.23 d.d (2H, $CH^{3,7}$, $^3J_{HH}$ 9.3, $^4J_{PH}$ 1.3), 7.49 s (2H, $CH^{1,5}$), 7.70 d (2H, $CH^{4,8}$, $^3J_{HH}$ 9.3). ^{13}C NMR spectrum, δ_C, ppm (*J*, Hz): 14.0 (CH_3), 20.5 (CH_2), 30.8 (CH_2), 46.6 d (CH_2–N, $^2J_{PC}$ 15.3), 117.5 ($C^{1,5}H$), 122.0 ($C^{3,7}H$), 128.8 ($C^{4,8}H$), 131.0 ($C^{9,10}H$), 148.5 d ($C^{2,6}O$, $^2J_{PC}$ 6.2). ^{31}P NMR spectrum (CH_2Cl_2): δ_P 77.6 ppm.

2,7-Bis(tetraisopropyldiamidothiophosphatoxy)-naphthalene (Xa). Yield 47%, mp 202–203°C, R_f 0.63 (A). ^1H NMR spectrum, δ, ppm (*J*, Hz): 1.29 d (24H, CH_3, $^3J_{HH}$ 6.6), 1.41 d (24H, CH_3, $^3J_{HH}$ 6.6), 3.84 m (4H, CH–N, $^3J_{PH}$ 13.2), 3.92 m (4H, CH–N, $^3J_{PH}$ 13.7), 7.51 d.d (2H, $CH^{3,6}$, $^3J_{HH}$ 9.4, $^4J_{HH}$ 2.2), 7.72 d (2H, $CH^{4,5}$, $^3J_{HH}$ 9.3), 7.85 s (2H, $CH^{1,8}$). ^{31}P NMR spectrum (CH_2Cl_2): δ_P 66.5 ppm. Found, %: C 59.58; H 9.07; N 8.16; P 9.06. $C_{34}H_{62}N_4O_2P_2S_2$. Calculated, %: C 59.62; H 9.12; N 8.18; P 9.04.

Cyclo[bis(naphthylenediisopropylamidothiophosphates)] (XIVa, XVa, XVIa). To a solution of 2 mmol of dihydroxynaphthalene **I–III** in 4 mL of anhydrous acetonitrile was added 2 mmol of triamide **IVa** at room temperature under stirring. The mixture was left standing for 1 day. The precipitate was filtered off and washed with acetonitrile. Then 2 mmol of dry sulfur was added to a solution of the precipitate in 3 mL of methylene chloride. After 1 day, the solvent was evaporated. The residue was chromatographed eluting

with a benzene–dioxane mixture, 15 : 1. The obtained amidothiophosphates were dried in a vacuum (1 mm Hg, 70°C).

Cyclo[bis(1,6-naphthylenediisopropylamidothiophosphate)] (XIVa). Yield 35%, oily substance, R_f 0.70 (B). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.31 d (6H, CH_3, $^3J_{HH}$ 5.9), 1.36 d (6H, CH_3, $^3J_{HH}$ 6.6), 1.39 d (12H, CH_3, $^3J_{HH}$ 6.2), 4.91 m (4H, CH–N, $^3J_{PH}$ 5.9), 6.77 d (2H, $CH^{2,2'}$, $^3J_{HH}$ 7.3), 7.13 d.d (2H, $CH^{7,7'}$, $^3J_{HH}$ 9.1, 10.2, $^4J_{HH}$ 2.6, $^4J_{PH}$ 1.8), 7.31 t (1H, CH^3, $^3J_{HH}$ 7.3), 7.35 t (1H, $CH^{3'}$), 7.37 d (1H, CH^4), 7.49 d (1H, $CH^{4'}$), 7.60 d (2H, CH^5, $^4J_{HH}$ 2.6), 8.08 d (1H, CH^8, $^3J_{HH}$ 10.2), 8.17 d (1H, $CH^{8'}$, $^3J_{HH}$ 9.1). ^{31}P NMR spectrum (C_6H_6): δ_P 64.8 ppm.

Cyclo[bis(2,6-naphthylenediisopropylamidothiophosphate)] (XVa). Yield 38%, oily substance, R_f 0.72 (B). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.34 d (12H, CH_3, $^3J_{HH}$ 6.1), 1.39 d (12H, CH_3, $^3J_{HH}$ 6.1), 4.76 m (2H, CH–N, $^3J_{PH}$ 9.9, $^3J_{HH}$ 3.9), 4.87 m (2H, CH–N, $^3J_{PH}$ 9.9, $^3J_{HH}$ 3.3), 7.12 d (4H, $C^{3,7}H$, *J* 9.6), 7.58 s (4H, $C^{1,5}H$), 7.67 d (4H, $C^{4,8}H$, *J* 9.6). ^{31}P NMR spectrum (C_6H_6): δ_P 63.8 ppm. Found, %: C 59.68; H 6.17; N 4.39; P 9.66. $C_{32}H_{40}N_2O_4P_2S_2$. Calculated, %: C 59.80; H 6.27; N 4.36; P 9.64.

Cyclo[bis(2,7-naphthylenediisopropylamidothiophosphate)] (XVIa). Yield 36%, mp 53–55°C, R_f 0.85 (B). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.39 d (12H, CH_3, $^3J_{HH}$ 6.6), 1.42 d (12H, CH_3, $^3J_{HH}$ 6.6), 4.05 d (4H, CH–N, $^3J_{PH}$ 10.5), 7.48 d (4H, $C^{3,6}H$, *J* 9.1), 7.63 s (4H, $C^{1,8}H$), 7.76 d (4H, $C^{4,5}H$, *J* 9.1). ^{31}P NMR spectrum (CH_2Cl_2): δ_P 61.7 ppm. Mass spectrum, *m*/*z*: 642.19 (calculated *M* 643).

Cyclo[bis(naphthylenedibutylamidothiophosphate)] (XIVb, XVb, XVIb). A mixture of 2 mmol of dihydroxynaphthalene **I–III** in 4 mL of anhydrous acetonitrile was added to 2 mmol of triamide **IVb** with stirring at room temperature. The mixture was heated for 3 h at 60°C. After cooling the precipitate was filtered off and washed with acetonitrile. Then 2 mmol of dry sulfur was added to a solution of the precipitate in 3 mL of methylene chloride. After 1 day the solvent was evaporated. The residue was chromatographed eluting with a benzene–dioxane mixture, 15 : 1. The obtained amidothiophosphates were dried in a vacuum (1 mm Hg, 70°C).

Cyclo[bis(1,6-naphthylenedibutylamidothiophosphate)] (XIVb). Yield 25%, oily substance, R_f 0.81 (B). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 0.89 t (12H, CH_3, $^3J_{HH}$ 6.9), 1.19–1.31 m (8H, CH_2, $^3J_{HH}$ 6.9), 1.50–1.61 m (8H, CH_2), 3.37–3.48 m (8H, CH_2–N, $^3J_{PH}$ 11.2), 7.25 br.d (2H, CH^2), 7.32 d (2H, CH^7), 7.39 s (2H, CH^5), 7.56 t (2H, CH^3, $^3J_{HH}$ 6.9), 7.68 d (2H, CH^4, $^3J_{HH}$ 6.9), 8.15 d (1H, CH^8, $^3J_{HH}$ 6.9), 8.17 d (1H, CH^8, *J* 6.9). ^{13}C NMR spectrum, δ_C, ppm (*J*, Hz): 13.9 (4C, CH_3), 20.2 (4C, CH_2), 30.7 (4C, CH_2), 46.7 d (4C, CH_2–N, $^2J_{PC}$ 10.7), 115.1 d (2C, C^2H, *J* 8.8), 117.6 d (2C, C^5H, $^3J_{PC}$ 9.5), 121.4 (2C, C^7H), 124.0 (2C, C^4H), 126.5 (2C, C^9), 128.9 (2C, C^8H), 130.9 (2C, C^3H), 135.5 (2C, C^{10}), 149.8 (2C, C^1O), 152.0 (2C, C^6O). ^{31}P NMR spectrum (CH_2Cl_2): δ_P 67.7 ppm.

Cyclo[bis(2,6-naphthylenedibutylamidothiophosphate)] (XVb). Yield 38%, oily substance, R_f 0.72 (B). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 0.84 t (12H, CH_3, $^3J_{HH}$ 7.3), 1.20–1.29 m (8H, CH_2), 1.48–1.56 m (8H, CH_2), 3.36 m (8H, CH_2–N, $^3J_{PH}$ 9.5), 7.40 d (4H, $C^{3,7}H$), 7.67 s (4H, $C^{1,5}H$), 7.90 d (4H, $C^{4,8}H$, $^3J_{HH}$ 8.8). ^{31}P NMR spectrum (CH_2Cl_2): δ_P 68.4 ppm. Mass spectrum, *m*/*z*: 698.25. Found P, %: 8.79. $C_{36}H_{48}N_2O_4P_2S_2$. Calculated P, %: 8.86. (calculated *M* 699).

Cyclo[bis(2,7-naphthylenedibutylamidothiophosphate)] (XVIb). Yield 20%, oily substance, R_f 0.83 (B). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 0.83 t (12H, CH_3, $^3J_{HH}$ 7.3), 1.19–1.28 m (8H, CH_2), 1.41–1.51 m (8H, CH_2), 3.10–3.22 m (8H, CH_2–N, $^3J_{PH}$ 10.8), 7.12 d (4H, $C^{3,6}H$, *J* 8.7), 7.31 s (4H, $C^{1,8}H$), 7.62 d (4H, $C^{4,5}H$, $^3J_{HH}$ 8.7). ^{31}P NMR spectrum (CH_2Cl_2): δ_P 69.1 ppm.

2-(Tetrabutyldiamidothiphosphatoxy)-6-hydroxynaphthalene (XVIIb). Yield 22%, oily substance, R_f 0.52 (B). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 0.91 t (12H, CH_3, $^3J_{HH}$ 6.8), 1.21–1.30 m (8H, CH_2), 1.51–1.59 m (8H, CH_2), 3.14 m (8H, CH_2–N, $^3J_{PH}$ 13.5), 7.05 d (1H, CH^7, $^3J_{HH}$ 9.6), 7.07 s (1H, CH^5), 7.16 d (1H, CH^3, $^3J_{HH}$ 9.3), 7.40 d (1H, CH^1, $^4J_{PH}$ 1.9), 7.52 d (1H, CH^8, $^3J_{HH}$ 9.4), 7.57 d (1H, CH^4, $^3J_{HH}$ 9.6), 7.90 br.s (1H, OH). ^{13}C NMR spectrum, δ_C, ppm (*J*, Hz): 14.0 (CH_3), 20.5 (CH_2), 30.8 (CH_2), 46.6 d (CH_2–N, $^2J_{PC}$ 15.3), 109.5 (C^5H), 117.7 (C^1H), 118.7 (C^7H), 121.9 (C^3H), 127.5 (C^8H), 128.9 (C^4H), 131.9 ($C^{9,10}H$), 148.5 d (C^2O, $^2J_{PC}$ 6.8), 153.7 (C^6OH). ^{31}P NMR spectrum (CH_2Cl_2): δ_P 77.7 ppm. Found, %: C 65.89; H 9.00; N 5.96; P 6.37. $C_{26}H_{43}N_2O_2PS$. Calculated, %: C 65.24; H 9.05; N 5.85; P 6.47.

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