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Directed Synthesis of Tertiary Phosphine Chalcogenides with Pyridine and Hydroxy Functions

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Abstract—Secondary phosphine chalcogenides react with pyridine-2-, pyridine-3-, and pyridine-4-carbaldehydes under mild noncatalytic conditions (22–43°C, 1–8.5 h) to form in 81–98% yield functional tertiary phosphine chalcogenides containing pyridine and hydroxy functions.

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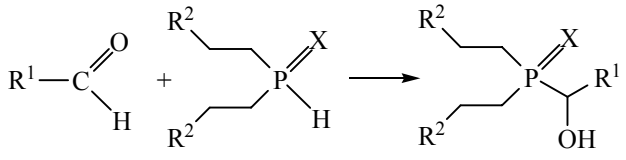
Tertiary phosphine chalcogenides with pyridine fragments are well known as effective ligands for metal complex compounds [1–10]. For example, chiral optically active PYDIPHOS is used in asymmetric metal complex catalysis [3, 9], rhodium complexes of bis(2-pyridyl)phenyl- and tris(2-pyridyl)phosphine oxides are effective in hydrogenation of unsaturated compounds [5], and peroxopolyoxotungstates containing tertiary pyridylethylphosphine oxides as ligands are promising bifunctional homogeneous metal complexes for oxidative catalysis [8]. Among the derivatives of tertiary pyridylphosphine chalcogenides biologically active compounds possessing antibacterial [11, 12], antitumor, and hepatotoxic activity [13, 14] are known. Besides, tertiary pyridylphosphine chalcogenides show the properties of effective flotoreagents [15], are used as building blocks in organic synthesis [16–19], in particular, for preparation of bipyridyls [16, 18].

The growing attention of researchers is also attracted by tertiary phosphine chalcogenides and their derivatives with hydroxy groups [20–25] used for the design of metal complex catalysts for the phase transfer reactions [20]. Besides, on the basis of hydroxyphosphine chalcogenides phosphorus-containing dendrimers [21, 22, 25], acetal methacrylates [23], and precursors of drugs with antiviral activity have been synthesized [24].

At the same time, the data on tertiary phosphine chalcogenides containing both the pyridyl and hydroxy fragments are, apparently limited to [26–29] where the reactions of secondary pyridylphosphine chalcogenides with some aliphatic [26, 29] and aromatic [27] aldehydes, as well as with imidazolylcarbaldehydes [28] are reported.

In the present work we suggest an alternative approach to the directed synthesis of the aforementioned functional pyridylphosphine chalcogenides based on the reaction of secondary phosphine chalcogenides with aldehydes of the pyridine series.

The experiment has shown that pyridinecarbaldehydes **I–III** react with bis(2-phenylethyl) phosphine oxide, -phosphine sulfide, -phosphine selenide **IV–VI**, and bis[2-(2-pyridyl)ethyl]phosphine chalcogenides **VII–IX**, as well as with bis[2-(4-pyridyl) ethyl]phosphine oxide **X** under mild conditions (22–43°C, 1–8.5 h) to form tertiary phosphine chalcogenides **XI–XXIV** in almost quantitative yield.



I–III **IV–X** **XI–XXIV**

The initial phosphine chalcogenides **IV–X** are readily prepared by the reaction of red phosphorus with styrene or vinylpyridines in superbasic systems [19, 30, 31].

The relative reactivity of the secondary pyridylphosphine chalcogenides compared to their aromatic analogs was estimated by the example of the reaction

Reaction of pyridinecarbaldehydes **I–III** with secondary phosphine chalcogenides **IV–X**[a]

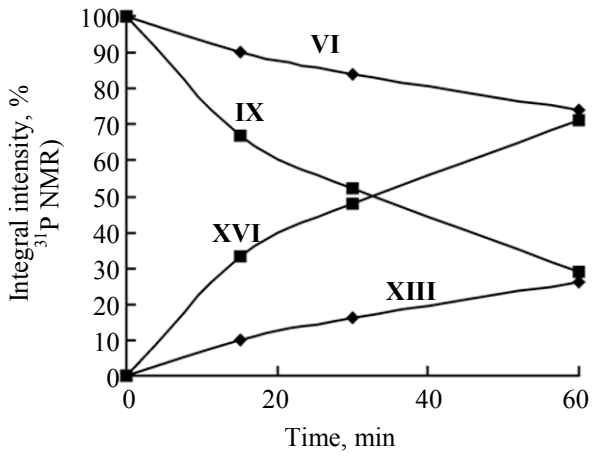
Aldehyde	R^1	$R^2_2(CH_2)_2P(X)H$	R^2	X	Solvent (3 ml)	Time, h	Adduct	Yield,[b] %
I	2-Py	**IV**	Ph	O	Benzene	1.5	**XI**	99
I	2-Py	**V**	Ph	S	Benzene	4.5	**XII**	98
I	2-Py	**VI**	Ph	Se	Benzene	2[c]	**XIII**	95
I	2-Py	**VII**	2-Py	O	Benzene	1.0	**XIV**	98
I	2-Py	**VIII**	2-Py	S	Ethanol	1.5	**XV**	95
I	2-Py	**IX**	2-Py	Se	Benzene	2	**XVI**	95
II	3-Py	**VIII**	2-Py	S	Ethanol	2.5	**XVII**	88
II	3-Py	**IX**	2-Py	Se	Chloroform	2.5	**XVIII**	89
III	4-Py	**IV**	Ph	O	Benzene	2	**XIX**	92
III	4-Py	**V**	Ph	S	Benzene	5.5	**XX**	95
III	4-Py	**VI**	Ph	Se	Benzene	3.5	**XXI**	95
III	4-Py	**VIII**	2-Py	S	Ethanol	2	**XXII**	95
III	4-Py	**IX**	2-Py	Se	Benzene	2.2	**XXIII**	98
I	2-Py	**X**	4-Py	O	Ethanol	8.5	**XXIV**	81

[a] All experiments were run at 22–23°C in an inert atmosphere (argon); in all experiments 1 mmol of phosphine chalcogenide **IV–X** and 1.1 mmol of pyridinecarbaldehyde **I–III** were taken. [b] Preparative yield. [c] The reaction temperature 43°C.

of phosphine selenides **VI** and **IX** with pyridyl-2-carbaldehyde (**I**) by the method of concurrent reactions (22–23°C, chloroform, molar ratio of the reagents 1 : 1 : 2.2). The process was monitored by the decrease of the integral intensity of the signals of the initial phosphine selenides **VI** and **IX** (2.35 and 4.57 ppm) in the ^{31}P NMR spectra and the synchronous increase of the integral intensity of the signals of the formed tertiary hydroxyphosphine selenides **XIII** and **XVI** (51.08 and 52.57 ppm, respectively). The more active in the reaction with pyridyl-2-carbaldehyde was found to be bis[2-(2-pyridyl)ethyl]phosphine selenide **IX**: after 60 min the content of the tertiary pyridylethylphosphine selenide **XVI** in the reaction mixture was 70% and only 26% of phenylethylphosphine selenide **XIII** (see the figure).

A higher reactivity of the secondary phosphine selenide with the pyridylethyl substituent at the phosphorus atom **IX** as compared to that with the phenylethyl substituent **VI** is, apparently, due to the fact that the pyridine fragment, as a base, facilitates deprotonation of the secondary phosphine selenide **IX**, thus increasing the rate of the reaction. It can be also mentioned that a higher reactivity of bis[2-(2-pyridyl)ethyl]phosphine oxide as compared to bis(2-phenylethyl)phosphine oxide was reported by the example of the reaction of the secondary phosphine oxides with 2,2,2-trichloroacetaldehyde [29].

The reactivity of pyridinecarbaldehydes in the studied reaction decreases in the order: **I** > **III** > **II**. Thus, at room temperature, the reaction of pyridylphosphine sulfide **V** with pyridyl-2-carbaldehyde (**I**) in ethanol completed in 1.5 h, with pyridyl-3-carbal-



The dynamics of formation of tertiary phosphine selenides **XIII, XVI** and conversion of secondary phosphine selenides **VI, IX** in the reaction with pyridyl-2-carbaldehyde (**I**) (22–23°C, chloroform).

dehyde (**II**), in 2.5 h, and with pyridyl-4-carbaldehyde (**III**), in 2 h (see the table).

In the ^{1}H and ^{13}C NMR spectra of the obtained tertiary phosphine chalcogenides **XI–XXIV** the most characteristic are the signals of the CH group of the OCHP=X moiety: a doublet at 4.91-5.18 ppm with the geminal spin–spin coupling constant ^{31}P–^{1}H equal 1.9–11.3 Hz (^{1}H NMR) for compounds **XI, XII**, **XIV, XIX, XX, XXII, XXIV** and a singlet for compounds **XIII, XV–XVIII, XXI, XXIII**; characteristic in the ^{13}C NMR spectra is a doublet at 70–73 ppm with direct constant $^{1}J_{PC}$ 71–80 Hz for phosphine oxides **XI, XIV, XIX, XXIV**, $^{1}J_{PC}$ 53–56 Hz for phosphine sulfides **XII, XV, XVII, XX, XXII**, and $^{1}J_{PC}$ 44–48 Hz for phosphine selenides **XIII, XVI, XVIII, XXI, XXII**.

The chemical shifts of nitrogen in the ^{15}N NMR spectra of the two pyridylethyl substituents at the phosphorus atom in the tertiary phosphine chalcogenides **XIV–XVI, XXIV** synthesized on the basis of pyridyl-2-carbaldehyde, are –(72–76) ppm. At the same time, for phosphine chalcogenides **XVII, XVIII, XXII, XXIII** prepared on the basis of pyridyl-3- and pyridyl-4-carbaldehydes, the nitrogen signals in the ^{15}N NMR spectra for one of the two rings of the pyridylethyl substituent at the phosphorus atom are observed at –(71–74) ppm, and for the second one, at –(88–90) ppm. Considerable upfield shift of the signals of nitrogen for compounds **XVII, XVIII, XXII, XXIII** may be indicative of the formation of a hydrogen bond N···HO with participation of the pyridylethyl substituent at the phosphorus atom.

The nonequivalence of the signals of two phenylethyl or two pyridylethyl groups at phosphorus in the ^{1}H, ^{13}C, and ^{15}N NMR spectra of phosphine chalcogenides **XI–XXIV** is caused by the presence of a chiral carbon atom in the OCHP=X fragment.

In the IR spectra of tertiary phosphine oxides **XI, XIV, XIX, XXIV** the stretching vibrations of the P=O bond are observed as a strong band in the range of 1141–1148 cm^{-1}. The stretching vibrations of the P=S bond in the IR spectra of phosphine sulfides **XII, XV, XVII, XX, XXII** and the P=Se bond of phosphine selenides **XIII, XVI, XVIII, XXI, XXIII** are substantially shifted to lower frequencies and appear as bands of moderate intensity, for sulfides at 610–614 cm^{-1}, and for selenides as a band at 474–484 cm^{-1}, respectively.

Therefore, the developed atom-economic, ecologically safe method of directed synthesis of tertiary phosphine chalcogenides with pyridine and hydroxy groups opens the way to new reagents for organic synthesis, precursors of optically active amphiphilic ligands for enantioselective processes, and potential precursors in the synthesis of drugs.

EXPERIMENTAL

IR spectra were taken on a Bruker IFS-25 spectrometer from layer or KBr pellets. ^{1}H, ^{13}C, ^{15}N, ^{31}P, and ^{77}Se NMR spectra were registered on Bruker DPX-400 and Bruker AV-400 spectrometers (400, 100, 40, 162, and 76 MHz, respectively) in $CDCl_3$ solution (if not stated otherwise) relative to TMS (^{1}H, ^{13}C), CH_3NO_2 (^{15}N), H_3PO_4 (^{31}P) and Me_2Se (^{77}Se). Chemical shifts in the ^{15}N NMR spectra were determined with the accuracy to 0.1 ppm using the 2D HMBC ^{15}N–^{1}H technique. All experiments were run in an inert atmosphere (argon).

Synthesis of tertiary α-hydroxyphosphine chalcogenides XI–XXIV *(general procedure)*. The solution of phosphine chalcogenide **IV–X** (1.0 mmol) and pyridinecarbaldehyde **I–III** (1.1 mmol) in 3 ml of the solvent was stirred in an argon atmosphere at 22–43°C for 1–8.5 h. The reaction was monitored by the decrease of intensity of the signals of the initial secondary phosphine chalcogenides **IV–X** in the range of 2–30 ppm and the increase of intensity of the signals of the formed tertiary phosphine chalcogenides in the range of 50–60 ppm in the ^{31}P NMR spectra. After the reaction was completed, the solvent was removed at a reduced pressure, the excess aldehyde was removed by washing the residue with small portions of ether (3× 0.3 ml), the ether was decanted. The residue was dried at 1 mm Hg to obtain compounds **XI–XXIV**.

[Bis(2-phenylethyl)phosphoryl](pyridin-2-yl)-methanol (XI). Yield 0.36 g (99%), light-beige powder, mp 108–109°C. IR (KBr, cm^{-1}): 1095 δ(COH), 1143 ν(P=O). ^{1}H NMR spectrum, δ_H, ppm: 1.82 m, 2.19–2.36 m, 2.67 m, 3.10 m (8H, PhCH_2CH_2P), 5.18 d (1H, PCH, $^{2}J_{PH}$ 8.1 Hz), 5.53 br.s (1H, OH), 7.00 s (1H, H_o), 7.01 s (1H, H_o), 7.18–7.36 m (9H, H^5, Py; H_m, H_p), 7.75 m (2H, H^{3,4}, Py), 8.58 d (1H, H^6, Py, $^{3}J_{HH}$ 5.2 Hz). ^{13}C NMR spectrum, δ_C, ppm: 26.05 d (CH_2P, $^{1}J_{PC}$ 60.2 Hz), 27.19 d (PhCH_2, $^{2}J_{PC}$ 3.4 Hz), 27.35 d (PhCH_2, $^{2}J_{PC}$ 3.4 Hz), 28.23 d (CH_2P, $^{1}J_{PC}$ 60.9 Hz), 70.62 d (PCH, $^{1}J_{PC}$ 80.4 Hz), 122.40 (C^3, Py), 123.12 (C^5, Py), 126.23, 126.34 (C_p), 127.94, 128.18 (C_o), 128.50, 128.58 (C_m), 137.00 (C^4, Py), 141.20 d (C_i, $^{3}J_{PC}$ 14.9 Hz), 147.99 (C^6, Py),

154.49 (C^2, Py). ^{31}P NMR spectrum, δ_P, ppm: 48.6. ^{15}N NMR, δ_N, ppm: –83.8 (2-Py). Found, %: C 72.69; H 6.63; N 3.88; P 8.31. $C_{22}H_{24}NO_2P$. Calculated, %: C 72.31; H 6.62; N 3.83; P 8.48.

[Bis(2-phenylethyl)phosphorothioyl](pyridin-2-yl)methanol (XII). Yield 0.373 g (98%), yellow oil. IR (thin layer, cm^{-1}): 1098 δ(COH), 610, sh 624 ν(P=S). ^1H NMR spectrum, δ_H, ppm: 2.03 m, 2.17 m, 2.33 m, 2.45 m, 2.74 m, 2.99 m, 3.08 m (8H, PhCH_2CH_2P), 5.14 d (1H, PCH, $^2J_{PH}$ 1.9 Hz), 5.71 br.s (1H, OH), 7.07 s (1H, H_o), 7.09 s (1H, H_o), 7.19–7.36 m (9H, H^5, Py; H_m, H_p), 7.78 d.d (1H, H^4, Py, $^3J_{HH}$ 8.1 Hz, $^3J_{HH}$ 7.2 Hz), 7.89 d (1H, H^3, Py, $^3J_{HH}$ 7.7 Hz), 8.59 d (1H, H^6, Py, $^3J_{HH}$ 4.4 Hz). ^{13}C NMR spectrum, δ_C, ppm: 27.95 d (CH_2P, $^1J_{PC}$ 42.2 Hz), 28.13 ($PhCH_2$), 30.47 d (CH_2P, $^1J_{PC}$ 46.1 Hz), 72.95 d (PCH, $^1J_{PC}$ 49.9 Hz), 123.08 (C^3, Py), 123.56 (C^5, Py), 126.30, 126.36 (C_p), 128.12, 128.33 (C_o), 128.51, 128.56 (C_m), 136.70 (C^4, Py), 140.87 (C_i, $^3J_{CP}$ 15.1 Hz), 147.69 (C^6, Py), 153.69 (C^2, Py). ^{31}P NMR spectrum, δ_P, ppm: 57.5. Found, %: C 69.57; H 6.35; N 3.70; P 7.92; S 8.57. $C_{22}H_{24}$NOPS. Calculated, %: C 69.27; H 6.34; N 3.67; P 8.12; S 8.41.

[Bis(2-phenylethyl)phosphoroselenoyl](pyridin-2-yl)methanol (XIII). Yield 0.407 g (95%), yellow oil. IR (thin layer, cm^{-1}): 1098 δ(COH), 479, sh 511 ν(P=Se). ^1H NMR spectrum, δ_H, ppm: 2.18 m, 2.36 m, 2.53 m, 2.76 m, 2.92 m, 3.06 m (8H, PhCH_2CH_2P), 5.19 s (1H, PCH), 5.32 br.s (1H, OH), 7.08 s (1H, H_o), 7.09 s (1H, H_o), 7.18–7.37 m (9H, H^5, Py; H_m, H_p), 7.77 d.d (1H, H^4, Py, $^3J_{HH}$ 8.4 Hz, $^3J_{HH}$ 7.8 Hz), 7.78 d.d (1H, H^4, Py, $^3J_{HH}$ 8.4 Hz, $^3J_{HH}$ 7.8 Hz), 7.95 d (1H, H^3, Py, $^3J_{HH}$ 7.8 Hz), 8.58 d (1H, H^6, Py, $^3J_{HH}$ 4.5 Hz). ^{13}C NMR spectrum, δ_C, ppm: 27.59 d (CH_2P, $^1J_{PC}$ 37.9 Hz), 29.01, 29.07 ($PhCH_2$), 30.00 d (CH_2P, $^1J_{PC}$ 39.8 Hz), 72.39 d (PCH, $^1J_{PC}$ 45.1 Hz), 123.20 (C^3, Py), 123.62 (C^5, Py), 126.35, 126.39 (C_p), 128.17, 128.35 (C_o), 128.53, 128.56 (C_m), 136.54 (C^4, Py), 140.67 d (C_i, $^3J_{PC}$ 14.7 Hz), 147.87 (C^6, Py), 153.62 (C^2, Py). ^{31}P NMR spectrum, δ_P, ppm: 51.7 ($^1J_{P,Se}$ 697.45 Hz). Found, %: C 61.30; H 5.68; N 3.34; P 7.05; Se 18.49. $C_{22}H_{24}$NOPSe. Calculated, %: C 61.68; H 5.65; N 3.27; P 7.23; Se 18.43.

[Bis(2-pyridin-2-ylethyl)phosphoryl](pyridin-2-yl)methanol (XIV). Yield 0.359 g (98%), yellow oil. IR (thin layer, cm^{-1}): 1089 δ(COH), 1140 ν(P=O). ^1H NMR spectrum, δ_H, ppm: 1.89 m, 2.06 m, 2.28 m, 2.43 m, 2.50 m, 2.78 m (8H, PyCH_2CH_2P), 5.08 d (1H, PCH, $^1J_{PH}$ 7.3 Hz), 6.89 d (1H, H^3, Py, $^3J_{HH}$ 7.7 Hz), 6.96 m (1H, H^5, Py), 7.02 m (1H, H^5, Py), 7.12 d (1H, H^3, Py, $^3J_{HH}$ 8.1 Hz), 7.15 m (1H, $H^{5'}$), 7.409 d.d (1H, H^4, Py, $^3J_{HH}$ 8.9 Hz, $^3J_{HH}$ 7.7 Hz), 7.413 d.d (1H, H^4, Py, $^3J_{HH}$ 9.1 Hz, $^3J_{HH}$ 7.7 Hz), 7.50 d.d (1H, H^4, Py, $^3J_{HH}$ 8.6 Hz, $^3J_{HH}$ 7.6 Hz), 7.504 d.d (1H, H^4, Py, $^3J_{HH}$ 9.1 Hz, $^3J_{HH}$ 7.4 Hz), 7.58–7.62 m (2H, $H^{3',4'}$), 8.34 d (1H, H^6, Py, $^2J_{HH}$ 4.6 Hz), 8.41 d (1H, H^6, Py, $^2J_{HH}$ 4.6 Hz), 8.47 d (1H, $H^{6'}$, $^3J_{HH}$ 4.8 Hz). ^{13}C NMR spectrum, δ_C, ppm: 23.53 d (CH_2P, $^1J_{PC}$ 61.9 Hz), 25.78 d (CH_2P, $^1J_{PC}$ 61.9 Hz), 29.14, 29.15 ($PyCH_2$), 73.35 d (PCH, $^1J_{PC}$ 70.9 Hz), 121.26, 121.40 (C^5, Py), 122.20 ($C^{3'}$), 122.58 (C^3, Py), 122.79 ($C^{5'}$), 122.85 (C^3, Py), 136.41 ($C^{4'}$), 136.61, 136.72 (C^4, Py), 148.10 ($C^{6'}$), 148.95, 149.32 (C^6, Py), 154.89 ($C^{2'}$), 160.25 d (C^2, Py, $^3J_{PC}$ 12.5 Hz). ^{31}P NMR spectrum, δ_P, ppm: 51.9. ^{15}N NMR, δ_N, ppm: –73.2, –75.2 (2-Py); –83.8 (2-Py'). Found, %: C 64.99; H 5.98; N 11.19; P 7.98. $C_{20}H_{22}N_3O_2P$. Calculated, %: C 65.39; H 6.04; N 11.44; P 8.43.

[Bis(2-pyridin-2-ylethyl)phosphorothioyl](pyridin-2-yl)methanol (XV). Yield 0.363 g (95%), yellow oil. IR (thin layer, cm^{-1}): 1088 δ(COH), 612, sh 629 ν(P=S). ^1H NMR spectrum, δ_H, ppm: 2.18 m, 2.21 m, 2.34 m, 2.63 m, 2.93 m, 3.21 m (8H, PyCH_2CH_2P), 5.15 s (1H, PCH), 7.03 d (1H, H^3, Py, $^3J_{HH}$ 7.7 Hz), 7.08 d.d (1H, H^5, 2-Py, $^3J_{HH}$ 6.7 Hz, $^3J_{HH}$ 5.3 Hz), 7.13 d.d (1H, H^5, Py, $^3J_{HH}$ 6.7 Hz, $^3J_{HH}$ 5.4 Hz), 7.21 d (1H, H^3, Py, $^3J_{HH}$ 7.8 Hz), 7.27 m (1H, $H^{4'}$, Py), 7.54 d.d (1H, H^4, Py, $^3J_{HH}$ 8.1 Hz, $^3J_{HH}$ 7.6 Hz), 7.60 d.d (1H, H^4, Py, $^3J_{HH}$ 8.1 Hz, $^3J_{HH}$ 7.5 Hz), 7.73 d.d (1H, $H^{4'}$, Py, $^3J_{HH}$ 7.7 Hz, $^3J_{HH}$ 7.2 Hz), 7.78 d (1H, $H^{3'}$, $^3J_{HH}$ 7.7 Hz), 8.46 d (1H, H^6, Py, $^3J_{HH}$ 4.5 Hz), 8.49 d (1H, H^6, Py, $^3J_{HH}$ 4.5 Hz), 8.53 d (1H, $H^{6'}$, $^3J_{HH}$ 4.5 Hz). ^{13}C NMR spectrum, δ_C, ppm: 24.80 d (CH_2P, $^1J_{PC}$ 47.7 Hz), 25.68 d (CH_2P, $^1J_{PC}$ 49.3 Hz), 28.84, 30.11 ($PyCH_2$), 72.76 d (PCH, $^1J_{PC}$ 52.8 Hz), 122.89, 122.99 (C^5, Py), 124.36 ($C^{3'}$), 124.51, 124.56 (C^3, Py), 124.80 ($C^{5'}$), 137.84 ($C^{4'}$), 137.94, 138.06 (C^4, Py), 149.59 ($C^{6'}$), 150.64 (C^6, Py), 155.59 ($C^{2'}$), 161.70 d (C^2, Py, $^3J_{PC}$ 14.6 Hz), 161.82 d (C^2, Py, $^3J_{PC}$ 14.6 Hz). ^{31}P NMR spectrum, δ_P, ppm: 59.8. ^{15}N NMR, δ_N, ppm: –73.1, –73.9 (2-Py); –81.9 (2-Py'). Found, %: C 62.52; H 5.83; N 10.85; P 7.89; S 8.31. $C_{20}H_{22}N_3OPS$. Calculated, %: C 62.65; H 5.78; N 10.96; P 8.08; S 8.36.

[Bis(2-pyridin-2-ylethyl)phosphoroselenoyl]-(pyridin-2-yl)methanol (XVI). Yield 0.408 g (95%), yellow oil. IR (thin layer, cm^{-1}): 1098 δ(COH), 484 ν(P=Se). ^1H NMR spectrum, δ_H, ppm: 2.49 m, 2.57 m, 2.81 m, 2.87 m, 3.10 m, 3.34 m (8H, PyCH_2CH_2P), 5.34 s (1H, PCH), 5.88 br.s (1H, OH), 7.19 d (1H, H^3, Py, $^3J_{HH}$ 7.2 Hz), 7.25 m (1H, H^5, Py), 7.37 d (1H, H^3,

Py, $^3J_{HH}$ 7.6 Hz), 7.40 d.d (1H, H^5, Py, $^3J_{HH}$ 8.0 Hz, $^3J_{HH}$ 5.2 Hz), 7.48 m (1H, $H^{5'}$), 7.67 d.d (1H, H^4, Py, $^3J_{HH}$ 8.0 Hz, $^3J_{HH}$ 7.5 Hz), 7.72 d.d (1H, H^4, Py, $^3J_{HH}$ 8.0 Hz, $^3J_{HH}$ 7.5 Hz), 7.85 d.d (1H, $H^{4'}$, $^3J_{HH}$ 8.0 Hz, $^3J_{HH}$ 7.5 Hz), 7.98 d (1H, $H^{3'}$, $^3J_{HH}$ 8.0 Hz), 8.61 d (1H, H^6, Py, $^3J_{HH}$ 5.0 Hz), 8.63 d (1H, H^6, Py, $^3J_{HH}$ 4.7 Hz), 8.53 d (1H, $H^{6'}$, $^3J_{HH}$ 4.4 Hz). ^{13}C NMR spectrum, δ_C, ppm: 25.22 d (CH_2P, $^1J_{PC}$ 40.3 Hz), 27.19 d (CH_2P, $^1J_{PC}$ 41.7 Hz), 31.06, 31.14 ($PyCH_2$), 73.08 d (PCH, $^1J_{PC}$ 45.2 Hz), 121.40, 121.50 (C^5, Py), 122.92, 123.11 (C^3, Py), 123.21, 123.38 ($C^{3',5'}$), 136.28 ($C^{4'}$), 136.42, 136.55 (C^4, Py), 148.13 ($C^{6'}$), 149.11, 149.15 (C^6, Py), 153.98 ($C^{2'}$), 159.89 d (C^2, Py, $^3J_{PC}$ 5.3 Hz), 160.04 d (C^2, Py, $^3J_{PC}$ 5.3 Hz). ^{31}P NMR spectrum, δ_P, ppm: 53.3. ^{15}N NMR spectrum ($CDCl_3$ + DMSO-d_6), δ_N, ppm: –71.4, –72.3 (2-Py); –77.7 (2-Py'). ^{77}Se NMR spectrum ($CDCl_3$ + DMSO-d_6): δ_{Se}, ppm: –420.3 ($^1J_{P,Se}$ 707.8 Hz). Found, %: C 55.87; H 5.09; N 9.63; P 6.89; Se 18.29. $C_{20}H_{22}N_3OPSe$. Calculated, %: C 55.82; H 5.15; N 9.76; P 7.20; S 18.35.

[Bis(2-pyridine-2-ylethyl)phosphorothioyl]-(pyridin-3-yl)methanol (XVII). Yield 0.337 g (88%), white powder, mp 128–129°C. IR (KBr, cm^{-1}): 1089 δ(COH), 613 ν(P=S). ^{1}H NMR spectrum, δ_H, ppm: 2.04 m, 2.17 m, 2.54 m, 2.76 m, 2.92 m, 3.22 m, 3.54 m (8H, PyCH_2CH_2P), 5.01 s (1H, PCH), 7.01 d (1H, H^3, Py, $^3J_{HH}$ 8.0 Hz), 7.07 d.d (1H, H^5, 2-Py, $^3J_{HH}$ 7.5 Hz, $^3J_{HH}$ 5.3 Hz), 7.19 d.d (1H, H^5, 2-Py, $^3J_{HH}$ 7.7 Hz, $^3J_{HH}$ 5.4 Hz), 7.25 m (2H, H^3, 2-Py; 1H, H^5, 3-Py), 7.52 d.d (1H, H^4, 2-Py, $^3J_{HH}$ 8.0 Hz, $^3J_{HH}$ 7.5 Hz), 7.65 d.d (1H, H^4, 2-Py, $^3J_{HH}$ 8.1 Hz, $^3J_{HH}$ 7.7 Hz), 7.95 d (1H, H^4, 3-Py, $^3J_{HH}$ 8.1 Hz), 8.43 d (1H, H^6, 2-Py, $^3J_{HH}$ 5.3 Hz), 8.44 d (1H, H^6, 2-Py, $^3J_{HH}$ 5.4 Hz), 8.51 d (1H, H^6, 3-Py, $^3J_{HH}$ 4.6 Hz), 8.82 s (1H, H^2, 3-Py), 9.10 br.s (1H, OH). ^{13}C NMR spectrum, δ_C, ppm: 24.84 d (CH_2P, $^1J_{PC}$ 45.7 Hz), 25.31 d (CH_2P, $^1J_{PC}$ 47.2 Hz), 28.67, 30.14 ($PyCH_2$), 71.98 d (PCH, $^1J_{PC}$ 53.8 Hz), 121.62, 121.91 (C^5, 2-Py), 122.75 (C^5, 3-Py), 123.09, 124.08 (C^3, 2-Py), 131.53 (C^3, 3-Py), 134.91 d (C^4, 3-Py, $^3J_{PC}$ 3.7 Hz), 136.73, 137.69 (C^4, 2-Py), 147.56 d (C^2, 3-Py, $^3J_{PC}$ 4.4 Hz), 148.10 (C^6, 2-Py), 149.06 d (C^6, 3-Py, $^3J_{PC}$ 2.9 Hz), 149.21 (C^6, 2-Py), 159.68 d (C^2, 2-Py, $^2J_{PC}$ 5.2 Hz), 160.12 d (C^2, 2-Py, $^2J_{PC}$ 14.0 Hz). ^{31}P NMR spectrum, δ_P, ppm: 57.8. ^{15}N NMR spectrum, δ_N, ppm: –73.5, –89.9 (2-Py); –72.4 (3-Py). Found, %: C 62.59; H 5.76; N 10.94; P 7.93; S 8.29. $C_{20}H_{22}N_3OPS$. Calculated, %: C 62.65; H 5.78; N 10.96; P 8.08; S 8.36.

[Bis(2-pyridin-2-ylethyl)phosphoroselenoyl]-(pyridin-3-yl)methanol (XVIII). Yield 0.382 g (89%), white powder, mp 113–115°C. IR (KBr, cm^{-1}): 1087 δ(COH), 479 ν(P=Se). ^{1}H NMR spectrum, δ_H, ppm: 2.13 m, 2.21 m, 2.54 m, 2.75 m, 2.93 m, 3.21 m, 3.55 m (8H, PyCH_2CH_2P), 5.07 s (1H, PCH), 7.02 d (1H, H^3, Py, $^3J_{HH}$ 8.0 Hz), 7.06 d.d (1H, H^5, 2-Py, $^3J_{HH}$ 7.7 Hz, $^3J_{HH}$ 5.0 Hz), 7.18 d.d (1H, H^5, 2-Py, $^3J_{HH}$ 7.7 Hz, $^3J_{HH}$ 5.2 Hz), 7.21–7.26 m (2H, H^3, 2-Py; 1H, H^5, 3-Py), 7.51 d.d (1H, H^4, 2-Py, $^3J_{HH}$ 8.0 Hz, $^3J_{HH}$ 7.7 Hz), 7.65 d.d (1H, H^4, 2-Py, $^3J_{HH}$ 8.0 Hz, $^3J_{HH}$ 7.7 Hz), 7.95 d (1H, H^4, 3-Py, $^3J_{HH}$ 8.0 Hz), 8.41 d (1H, H^6, 2-Py, $^3J_{HH}$ 5.2 Hz), 8.43 d (1H, H^6, 2-Py, $^3J_{HH}$ 5.0 Hz), 8.51 d (1H, H^6, 3-Py, $^3J_{HH}$ 4.9 Hz), 8.84 br.s (1H, H^2, 3-Py). ^{13}C NMR spectrum, δ_C, ppm: 24.15 d (CH_2P, $^1J_{PC}$ 41.3 Hz), 24.89 d (CH_2P, $^1J_{PC}$ 40.9 Hz), 29.60, 31.02 ($PyCH_2$), 71.70 d (PCH, $^1J_{PC}$ 45.0 Hz), 121.60, 121.81 (C^5, 2-Py), 122.63 (C^5, 3-Py), 123.09, 124.07 (C^3, 2-Py), 131.15 (C^3, 3-Py), 135.02 d (C^4, 3-Py, $^3J_{PC}$ 3.7 Hz), 136.66, 137.87 (C^4, 2-Py), 148.24 (C^2, 3-Py; C^6, 2-Py), 149.21 (C^6, 3-Py; C^6, 2-Py), 159.40 (C^2, 2-Py, $^2J_{PC}$ 5.9 Hz), 159.82 d (C^2, 2-Py, $^2J_{PC}$ 13.6 Hz). ^{31}P NMR spectrum, δ_P, ppm: 54.9. ^{15}N NMR spectrum, δ_N, ppm: –72.4, –89.1 (2-Py); –71.3 (3-Py). ^{77}Se NMR spectrum, δ_{Se}, ppm: –419.3 ($^1J_{P,Se}$ 708.8 Hz). Found, %: C 55.71; H 5.11; N 9.72; P 6.89; Se 18.29. $C_{20}H_{22}N_3OPSe$. Calculated, %: C 55.82; H 5.15; N 9.76; P 7.20; Se 18.35.

[Bis(2-phenylethyl)phosphoryl](pyridin-4-yl)-methanol (XIX). Yield 0.336 g (92%), white powder, mp 152–153°C. IR (KBr, cm^{-1}): 1092 δ(COH), 1140 ν(P=O). ^{1}H NMR spectrum, δ_H, ppm: 1.90 m, 2.05 m, 2.24 m, 2.74 m, 2.95 m (PhCH_2CH_2P), 5.06 d (1H, PCH, $^2J_{PH}$ 11.3 Hz), 6.30 br.s (1H, OH), 7.03–7.26 m (10H, Ph), 7.42 d (2H, $H^{3,5}$, Py, $^3J_{HH}$ 4.7 Hz), 8.46 d (2H, $H^{2,6}$, Py, $^3J_{HH}$ 4.7 Hz). ^{13}C NMR spectrum, δ_C, ppm: 25.51 d (CH_2P, $^1J_{PC}$ 60.5 Hz), 26.89 ($PhCH_2$), 27.27 d (CH_2P, $^1J_{PC}$ 59.7 Hz), 69.65 d (PCH, $^1J_{PC}$ 75.9 Hz), 121.49 ($C^{3,5}$, Py), 126.21, 126.28 (C_p), 127.61, 127.68 (C_o), 128.36, 128.41 (C_m), 140.38 (C_i, $^3J_{PC}$ 12.4 Hz), 140.50 d (C_i, $^3J_{PC}$ 12.6 Hz), 147.54 (C^4, Py), 147.87 ($C^{2,6}$, Py). ^{31}P NMR spectrum, δ_P, ppm: 50.3. Found, %: C 72.29; H 6.80; N 3.82; P 8.39. $C_{22}H_{24}NO_2P$. Calculated, %: C 72.31; H 6.62; N 3.83; P 8.48.

[Bis(2-phenylethyl)phosphorothioyl](pyridin-4-yl)methanol (XX). Yield 0.362 g (95%), white powder, mp 139–140°C. IR (KBr, cm^{-1}): 1096 δ(COH), 606, sh 625 ν(P=S). ^{1}H NMR spectrum, δ_H, ppm: 2.05 m, 2.14 m, 2.24 m, 2.82 m, 2.93 m (8H, PhCH_2CH_2P), 4.98 d (1H, PCH, $^2J_{PH}$ 6.2 Hz), 6.20 br.s (1H, OH), 7.12–7.29 m (10H, Ph), 7.49 d (2H, $H^{3,5}$, Py,

$^3J_{HH}$ 4.7 Hz), 8.46 d (2H, $H^{2,6}$, Py, $^3J_{HH}$ 5.2 Hz). ^{13}C NMR spectrum, δ_C, ppm: 28.00 ($PhCH_2$), 28.11 d (CH_2P, $^1J_{PC}$ 45.9 Hz), 28.12 ($PhCH_2$), 29.64 d (CH_2P, $^1J_{PC}$ 46.1 Hz), 72.36 d (PCH, $^1J_{PC}$ 56.1 Hz), 122.35 ($C^{3,5}$, Py), 126.45 (C_p), 128.05, 128.19 (C_o), 128.57 (C_m), 140.36 d (C_i, $^3J_{PC}$ 5.0 Hz), 140.50 d (C_i, $^3J_{PC}$ 5.0 Hz), 147.64 ($C^{2,6}$, Py), 147.93 (C^4, Py). ^{31}P NMR spectrum, δ_P, ppm: 58.5. Found, %: C 69.35; H 6.40; N 3.72; P 8.07; S 8.44. $C_{22}H_{24}NOPS$. Calculated, %: C 69.33; H 6.34; N 3.67; P 8.12; S 8.41.

[Bis(2-phenylethyl)phosphoroselenoyl](pyridin-4-yl)methanol (XXI). Yield 0.407 g (95%), white powder, mp 146–148°C. IR (KBr, cm^{-1}): 1098 δ(COH), 479 ν(P=Se). ^1H NMR spectrum, δ_H, ppm: 2.06 m, 2.21 m, 2.38 m, 2.74 m, 2.88 m, 2.93 m (8H, PhCH_2CH_2P), 5.04 d (1H, PCH, $^2J_{PH}$ 4.2 Hz), 6.59 br.s (1H, OH), 7.06–7.22 m (10H, Ph), 7.49 br.s (2H, $H^{3,5}$, Py), 8.36 br.s (2H, $H^{2,6}$, Py). ^{13}C NMR spectrum, δ_C, ppm: 27.64 d (CH_2P, $^1J_{PC}$ 37.0 Hz), 28.96 ($PhCH_2$), 29.09 d (CH_2P, $^1J_{PC}$ 38.8 Hz), 71.65 d (PCH, $^1J_{PC}$ 48.0 Hz), 122.31 ($C^{3,5}$, Py), 126.40 (C_p), 127.85, 128.00 (C_o), 128.75, 128.78 (C_m), 140.18 d (C_i, $^3J_{PC}$ 5.7 Hz), 140.04 d (C_i, $^3J_{PC}$ 5.7 Hz), 147.10 (C^4, Py), 147.84 ($C^{2,6}$, Py). ^{31}P NMR spectrum, δ_P, ppm: 53.7. Found, %: C 61.49; H 5.67; N 3.24; P 7.15; Se 18.19. $C_{22}H_{24}NOPSe$. Calculated, %: 61.68; H 5.65; N 3.27; P 7.23; Se 18.43.

[Bis(2-pyridin-2-ylethyl)phosphorothioyl](pyridin-4-yl)methanol (XXII). Yield 0.364 g (95%), white powder, mp 86–87°C. IR (KBr, cm^{-1}): 1083 δ(COH), 614 sh 622 ν(P=S). ^1H NMR spectrum, δ_H, ppm: 2.02 m, 2.13 m, 2.39 m, 2.73 m, 2.89 m, 3.24 m, 3.52 m, 3.65 m (8H, PyCH_2CH_2P), 4.94 d (1H, PCH, $^2J_{PH}$ 2.5 Hz), 7.01 d (1H, H^3, 2-Py, $^3J_{HH}$ 7.0 Hz), 7.08 d.d (1H, H^5, 2-Py, $^3J_{HH}$ 7.6 Hz, $^3J_{HH}$ 6.7 Hz), 7.22 d.d (1H, H^5, 2-Py, $^3J_{HH}$ 7.6 Hz, $^3J_{HH}$ 6.3 Hz), 7.29 d (1H, H^3, 2-Py, $^3J_{HH}$ 8.0 Hz), 7.53 d.d (1H, H^4, 2-Py, $^3J_{HH}$ 8.0 Hz, $^3J_{HH}$ 7.6 Hz), 7.60 d (2H, $H^{2,6}$, 4-Py, $^3J_{HH}$ 4.0 Hz), 7.69 d.d (1H, H^4, 2-Py, $^3J_{HH}$ 8.0 Hz, $^3J_{HH}$ 7.6 Hz), 8.45 d (1H, H^6, 2-Py, $^3J_{HH}$ 4.5 Hz), 8.48 d (1H, H^6, 2-Py, $^3J_{HH}$ 4.5 Hz), 8.56 d (2H, $H^{3,5}$, 4-Py, $^3J_{HH}$ 4.2 Hz). ^{13}C NMR spectrum, δ_C, ppm: 24.80 d (CH_2P, $^1J_{PC}$ 47.7 Hz), 25.68 d (CH_2P, $^1J_{PC}$ 49.3 Hz), 28.84, 30.11 ($PyCH_2$), 72.76 d (PCH, $^1J_{PC}$ 52.8 Hz), 121.62, 123.09 (C^5, 2-Py), 122.14, 124.08 (C^3, 2-Py; $C^{3,5}$, 4-Py), 136.64, 137.66 (C^4, 2-Py), 144.85 (C^4, 4-Py), 148.21, 149.22 (C^6, 2-Py), 149.02 ($C^{2,6}$, 4-Py), 159.39 d (C^2, 2-Py, $^3J_{PC}$ 5.9 Hz), 159.77 d (C^2, 2-Py, $^3J_{PC}$ 13.9 Hz). ^{31}P NMR spectrum, δ_P, ppm: 60.4. ^{15}N NMR, δ_N, ppm: –72.7, –88.6 (2-Py); –75.2 (4-Py). Found, %: C 62.49; H 5.81; N 10.85; P 7.91; S 8.29. $C_{20}H_{22}N_3OPS$. Calculated, %: C 62.65; H 5.78; N 10.96; P 8.08; S 8.36.

[Bis(2-pyridin-2-ylethyl)phosphoroselenoyl](pyridin-4-yl)methanol (XXIII). Yield 0.421 g (98%), white powder, mp 69–70°C. IR (KBr, cm^{-1}): 1098 δ(COH), 484 ν(P=Se). ^1H NMR spectrum, δ_H, ppm: 2.07 m, 2.45 m, 2.67 m, 2.85 m, 2.94 m, 3.17 m, 3.60 m (8H, PyCH_2CH_2P), 5.04 s (1H, PCH), 7.04 d (1H, H^3, 2-Py, $^3J_{HH}$ 7.4 Hz), 7.11 d.d (1H, H^5, 2-Py, $^3J_{HH}$ 7.2 Hz, $^3J_{HH}$ 5.3 Hz), 7.23 d.d (1H, H^5, 2-Py, $^3J_{HH}$ 7.6 Hz, $^3J_{HH}$ 5.6 Hz), 7.32 d (1H, H^3, 2-Py, $^3J_{HH}$ 7.9 Hz), 7.56 d.d (1H, H^4, 2-Py, $^3J_{HH}$ 8.3 Hz, $^3J_{HH}$ 7.8 Hz), 7.66 d (2H, $H^{3,5}$, 4-Py, $^3J_{HH}$ 4.2 Hz), 7.72 d.d (1H, H^4, 2-Py, $^3J_{HH}$ 8.3 Hz, $^3J_{HH}$ 7.8 Hz), 8.46 d (1H, H^6, 2-Py, $^3J_{HH}$ 4.6 Hz), 8.50 d (1H, H^6, 2-Py, $^3J_{HH}$ 4.6 Hz), 8.59 d (2H, $H^{2,6}$, 4-Py, $^3J_{HH}$ 6.0 Hz). ^{13}C NMR spectrum, δ_C, ppm: 24.22 d (CH_2P, $^1J_{PC}$ 40.7 Hz), 25.08 d (CH_2P, $^1J_{PC}$ 42.5 Hz), 28.84, 30.11 ($PyCH_2$), 72.45 d (PCH, $^1J_{PC}$ 43.7 Hz), 121.33, 123.12 (C^5, 2-Py), 121.95 (C^3, 2-Py), 122.25, 124.22 ($C^{3,5}$, 4-Py), 136.69, 137.78 (C^4, 2-Py), 144.91 (C^4, 4-Py), 147.62, 149.32 ($C^{2,6}$, 4-Py), 149.26 (C^6, 2-Py), 159.61 (C^2, 2-Py), 160.05 d (C^2, 2-Py, $^3J_{PC}$ 13.5 Hz). ^{31}P NMR spectrum, δ_P, ppm: 54.3. ^{15}N NMR spectrum, δ_N, ppm: –72.6, –89.9 (2-Py); –74.2 (4-Py). ^{77}Se NMR spectrum, δ_{Se}, ppm: -413.7 ($^1J_{P,Se}$ 707.6 Hz). Found, %: C 55.69; H 5.13; N 9.69; P 6.99; Se 18.26. $C_{20}H_{22}\cdot N_3OPSe$. Calculated, %: C 55.82; H 5.15; N 9.76; P 7.20; Se 18.35.

[**Bis(2-pyridin-4-ylethyl)phosphoryl](pyridin-2-yl)methanol (XXIV).** Yield 0.340 g (81%), yellow oil. IR (thin layer, cm^{-1}): 1097 δ(COH), 1142 ν(P=O). ^1H NMR spectrum, δ_H, ppm: 1.85 m, 2.16–2.22 m, 2.38 m, 2.64 m, 3.10 m (8H, PyCH_2CH_2P), 5.18 d (1H, PCH, $^2J_{PH}$ 8.0 Hz), 5.82 br.s (1H, OH), 6.92 d (2H, $H^{2,6}$, 4-Py, $^3J_{HH}$ 5.6 Hz), 7.19 d (2H, $H^{2,6}$, 4-Py, $^3J_{HH}$ 5.6 Hz), 7.35 d.d (1H, H^5, 2-Py, $^3J_{HH}$ 7.8 Hz, $^3J_{HH}$ 5.6 Hz), 7.70 d (1H, H^3, 2-Py, $^3J_{HH}$ 7.9 Hz), 7.78 d.d (1H, H^4, 2-Py, $^3J_{HH}$ 7.9 Hz, $^3J_{HH}$ 7.6 Hz), 7.79 d.d (1H, H^4, 2-Py, $^3J_{HH}$ 7.9 Hz, $^3J_{HH}$ 7.6 Hz), 8.44 d (2H, $H^{3,5}$, 4-Py, $^3J_{HH}$ 5.6 Hz), 8.53 d (2H, $H^{3,5}$, 4-Py, $^3J_{HH}$ 5.6 Hz), 8.56 d (1H, H^6, 2-Py, $^3J_{HH}$ 4.8 Hz). ^{13}C NMR spectrum, δ_C, ppm: 24.64 d (CH_2P, $^1J_{PC}$ 60.9 Hz), 26.57 d (4-$PyCH_2$, $^1J_{PC}$ 3.1 Hz), 26.78 d (4-$PyCH_2$, $^1J_{PC}$ 3.3 Hz), 27.26 d (CH_2P, $^1J_{PC}$ 62.2 Hz), 70.27 d (PCH, $^1J_{PC}$ 76.6 Hz), 122.55 (C^3, 2-Py), 123.30 ($C^{3,5}$, 4-Py), 123.57 (C^5, 2-Py; $C^{3,5}$, 4-Py), 137.30 (C^4, 2-Py), 148.16 (C^6, 2-Py), 149.90 ($C^{2,6}$, 4-Py), 150.01 ($C^{2,6}$, 4-Py), 150.01 (C^4, 4-Py), 153.75 (C^2, 2-Py). ^{31}P NMR spectrum, δ_P, ppm: 49.9. ^{15}N NMR, δ_N, ppm: –75.6,

–76.1 (4-Py); –85.6 (2-Py). Found, %: C 65.43; H 5.99; N 11.46; P 8.33. $C_{20}H_{22}N_3O_2P$. Calculated, %: C 65.39; H 6.04; N 11.44; P 8.43.

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