Chemistry of Heterocyclic Compounds, Vol. 44, No. 11, 2008

REACTION OF IMIDAZOLE SERIES ALDEYDES WITH BIS[2-(2-PYRIDYL)ETHYL]-PHOSPHINE CHALCOGENIDES: SYNTHESIS OF POLYFUNCTIONAL HETEROCYCLIC SYSTEMS

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The nucleophilic addition of bis[2-(2-pyridyl)ethyl]phosphine sulfide and bis[2-(2-pyridyl)-ethyl]phosphine selenide to 2-formyl-1-organylimidazoles and benzimidazoles occurs efficiently without catalysis at room temperature to give functionalized heterocyclic compounds containing imidazole, benzimidazole, and pyridine rings and also chalcogenophosphoryl and hydroxyl groups.

Keywords: bis[2-(2-pyridyl)ethyl]phosphine chalcogenides, 1-ethyl(vinyl)-2-{bis[2-(2-pyridyl)ethyl]-chalcogenophosphorylhydroxymethyl}imidazoles and -benzimidazoles, 2-formyl-1-organylbenzimidazoles, 2-formyl-1-organylimidazoles, hydrochalcogenophosphorylation.

Polyheterocyclic systems constructed using nitrogen heterocycles continue to be intensively studied as biologically active compounds, efficient polydentate ligands for designing metall complexes, and are widely used as reactive building blocks for organic synthesis. Thus triazolyl-, thiadiazolyl-, and oxadiazolylpyridines are known as amplifiers of fleomycin [1], some ketolides with imidazolylpyridine fragments are active antibiotics [2], functional imidazolylpyridines with fluorophenyl [3] or sulfinyl [4] groups show antiviral activity, and also affect processes occurring involving kinase [3, 5]. Metal-complex catalysts [6-10], ionic liquids which are successfully used, in particular, in the Heck reaction [9], and special materials [6, 7] can be produced based on imidazolylpyridines.

Functional heterocyclc systems have special value. Thus imidazoles and benzimidazoles containing chalcogenophosphoryl and hydroxyl functions are efficient ligands for directing the synthesis of promising biologically active metal complexes [11-14] and also as models for studying several physicochemical aspects [12-14]. One of the convenient routes for the synthesis of functional heterocyclic groups can be the reaction of the secondary pyridylphosphine chalcogenides **1, 2** with 2-formyl-1-organylimidazoles **3, 4** and –benzimidazoles **5, 6**.

The aim of this work was direct synthesis of novel heterocyclic systems containing imidazole and pyridine nuclei and also hydroxyl and chalcogenophosphoryl functions by a study of the reaction of the aldehydes **3-6** with the available bis[2-(2-pyridyl)ethyl]phosphine sulfide (**1**) [15] and the previously unreported

* Dedicated to Professor A. Pozharskii on his 70th jubilee

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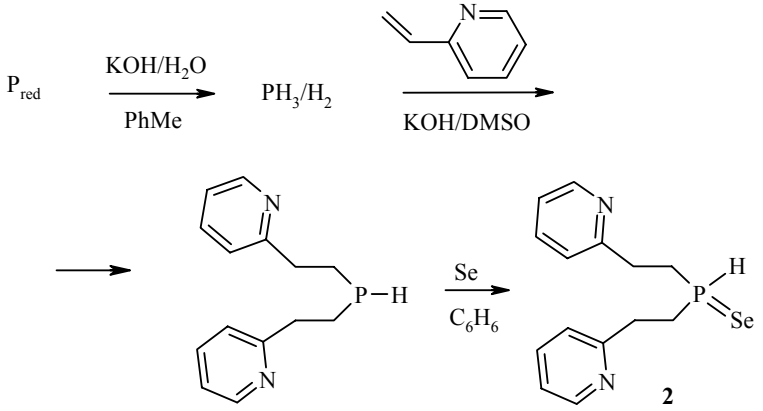
Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 11, pp. 1669-1675, November, 2008. Original article submitted July 31, 2008.

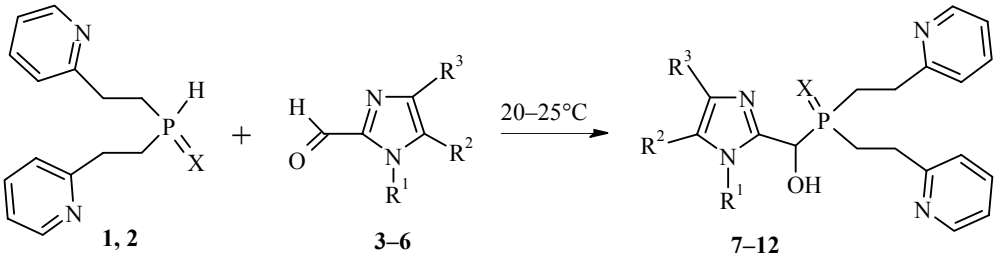
bis[2-(2-pyridyl)ethyl]phosphine selenide (**2**). The latter was prepared by us from red phosphorus and 2-vinylpyridine by a scheme involving the generation of phosphine through the degradation of the red phosphorus macromolecule with aqueous potassium hydroxide and addition of the phosphine to 2-vinylpyridine in the system KOH–DMSO to give bis[2-(2-pyridyl)ethyl]phosphine (which is readily oxidized by elemental selenium to the corresponding phosphine selenide **2**).



The experiment showed that hydrochalcogenophosphorylation of aldehydes **3-6** by the secondary pyridylphosphine chalcogenides **1, 2** takes place virtually quantitatively (^{31}P NMR data) under noncatalyzed conditions at room temperature (3 h, ethanol or THF) to give the 1-ethyl(vinyl)-2-{bis[2-(2-pyridyl)-ethyl]thiophosphorylhydroxymethyl}imidazoles **7, 8**, 1-ethyl-2-{bis[2-(2-pyridyl)ethyl]thiophosphorylhydroxy-methyl} benzimidazole (**9**), 1-ethyl(vinyl)-2-{bis[2-(2-pyridyl)ethyl]selenophosphorylhydroxymethyl}imidazoles **10, 11**, and 2-{bis[2-(2-pyridyl)ethyl]selenophosphorylhydroxymethyl}-1-vinylbenzimidazole (**12**).

Compounds **7-9** are stable at room temperature while the selenophosphorylimidazoles **10-12** undergo a reaction losing metallic selenium under these conditions. This process is markedly slower at lower temperature (0-2ºC).

The most characteristic peaks in the ^{1}H and ^{13}C NMR spectra of compounds **7-12** are the signals for the CH groups of the OCHP=X fragment. The geminal ^{31}P–C–^{1}H spin-spin coupling in the α-hydroxyphosphine chalcogenides is small and the splitting of the proton resonance signal is observed only in the ^{1}H NMR spectra of chalcogenophosphorylimidazoles **7, 9, 12**; the analogous spectra of compounds **8, 10, 11** showing only a narrow singlet in the range 5.3-5.6 ppm. The ^{13}C NMR spectra of compounds **7-12** show a characteristic doublet at 67-68 ppm ($^{1}J_{PC}$ ~ 57-59 Hz for the thiophosphorylimidazoles **7-9** and $^{1}J_{PC}$ = 49-52 Hz for the selenophosphorylimidazoles **10-12**). The nonequivalence of signals for the two pyridine rings in the ^{1}H and ^{13}C NMR spectra of compounds **7-12** is due to the presence of a chiral carbon atom in the OCHP=X fragment.



1, 7–9 X = S; **2, 10–12** X = Se; **3, 7, 10** R^1 = Et, R2 = R^3 = H;
4, 8, 11 R^1 = Vinyl, R^2 = R^3 = H; **5, 9** R^1 = Et, $R^2 + R^3 = (CH)_4$;
6, 12 R^1 = Vinyl, $R^2 + R^3 = (CH)_4$

The absorption band in the region 3108-3134 cm^{-1} of the IR spectra corresponds to the stretching vibration of the associated OH groups.

Hence hydrochalcogenophosphorylation of azole series aldehydes by secondary pyridylphosphine chalcogenides is a simple, atom efficient method for the synthesis of novel polyfunctional, chiral heterocyclic compounds with imidazole and pyridine nuclei. These highly reactive building blocks for organic synthesis are precursors of optically active, amphiphilic ligands for enantioselective processes and also precursors in the synthesis of medicinal preparations having, for example, antihypoxic, and cardio- and hepatoprotective properties [16-18].

EXPERIMENTAL

The ^{1}H, ^{13}C, ^{15}N, ^{31}P, and ^{77}Se NMR spectra were obtained on a Bruker DPX 400 instrument (400.13, 101.62, 40.56, 161.98, and 76.31 MHz respectively) using $CDCl_3$ relative to HMDS (for ^{1}H and ^{13}C NMR spectra), $MeNO_2$ (^{15}N NMR spectrum), H_3PO_4 (^{31}P NMR spectrum), and Me_2S (^{77}Se NMR spectrum). IR spectra were recorded on a Bruker IFS 25 spectrometer as a thin film. Experiments were performed in an argon atmosphere. The synthesis of the 2-formyl-1-organylimidazoles **3-6** has been described in [19].

Bis[2-(2-Pyridyl)ethyl]phosphine Selenide (2). A suspension consisting of KOH (25 g), H_2O (6 ml), and DMSO (55 ml) was purged with argon and saturated with the phosphine hydrogen mixture generated in a separate flask by the addition of 50% aqueous KOH solution (200 g) to a mixture of red phosphorus (20 g) in toluene (70 ml) at 70-75ºC. The suspension obtained was heated to 55-57ºC and a solution of the 2-vinyl-pyridine (10.5 g, 100 mmol) stabilized by hydroquinone (0.02 g) in DMSO (15 ml) was added dropwise with continuous passage of the phosphine hydrogen mixture. The reaction mixture was stirred for 30 min at 55-57ºC, purged with argon, stirred for a further 30 min at this temperature, and cooled. The product was diluted with water (100 ml) and extracted with ether (5×60 ml). The ether extract was washed with water (2×30 ml) and passed through a layer of calcined K_2CO_3. The ether was evaporated and the residue was distilled at 1 mm Hg to give the bis[2-(2-pyridyl)ethyl]phosphine (9.32 g, 76%) (^{31}P NMR spectrum, δ = -69.6 ppm). A solution of this phosphine (3.91 g, 16 mmol) in benzene (40 ml) was added with stirring to a suspension of metallic selenium (1.26 g, 16 mmol) in benzene (30 ml) over 5 min. The reaction temperature was raised to 35ºC and the mixture was stirred at this temperature for a further 15 min. Solvent was evaporated *in vacuo* and the residue was washed with a small amount of hexane (2×0.9 ml) and dried *in vacuo* to give the phosphine selenide **2** (4.93 g, 95%) with mp 40-41ºC (hexane). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.39, 2.75 (4H, m, CH_2P); 3.99 (4H, m, $PyCH_2$); 6.16 (1H, d, $^{1}J_{PH}$ = 439.8, PH); 7.12 (2H, m, H-3, Py); 7.22 (2H, d, $^{3}J_{HH}$ = 7.7, H-5, Py); 7.59 (2H, dt, $^{3}J_{HH}$ = 7.8, $^{4}J_{HH}$ = 1.7, H-4, Py); 8.49 (2H, d, $^{3}J_{HH}$ = 4.9, H-6, Py). ^{13}C NMR spectrum, δ, ppm (*J*, Hz): 27.31 (d, $^{1}J_{PC}$ = 45.3, CH_2P); 30.92 ($Py\underline{C}H_2$); 121.16 (C-5, Py); 122.63 (C-3, Py); 136.06 (C-4, Py); 148.64 (C-6, Py); 158.23 (d, $^{3}J_{PC}$ = 11.8, C-2, Py). ^{15}N NMR spectrum, δ, ppm: –72.8. ^{77}Se NMR spectrum, δ, ppm (*J*, Hz): –415.7 ($^{1}J_{P,Se}$ = 704.3). ^{31}P NMR spectrum ($CDCl_3$), δ, ppm: 5.01. Found, %: C 51.96; H 5.29; N 8.64; P 9.49; Se 24.39. $C_{14}H_{17}N_2PSe$. Calculated, %: C 52.02; H 5.30; N 8.67; P 9.58; Se 24.43.

Preparation of 1-Organyl-2-{bis[2-pyridyl)ethyl]chalcogenophosphorylhydroxymethyl}imidazoles (7-12) (**General Method**). A mixture of the aldehyde **3-6** (1.1 mmol) and pyridylphosphine chalcogenide **1, 2** (1.0 mmol) in ethanol (3 ml in the case of sulfide **1**) or in THF (2 ml in the case of the selenide **2**) was purged with argon and stirred at room temperature for 3 h. Solvent was removed *in vacuo* and the residue was washed with small portions of ethanol (3×0.3 ml) and dried *in vacuo* to give compounds **7-12** as a viscous oil, soluble in chloroform, acetone, and DMSO.

1-Ethyl-2-{bis[2-(2-pyridyl)ethyl]thiophosphorylhydroxymethyl}imidazole (7). Yield 84%. IR spectrum, ν, cm^{-1}: 3134 (OH), 573 (P=S). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.39 (3H, t, $^{3}J_{HH}$ = 7.3, CH_3); 2.38, 2.54, 2.72 (4H, m, CH_2P); 2.99, 3.21 (4H, m, $PyCH_2$); 4.21 (2H, q, $^{3}J_{HH}$ = 7.3, NCH_2); 5.31 (1H, d,

$^{2}J_{HP}$ = 1.8, PCH); 5.92 (1H, br. s, OH); 6.93, 7.03 (2H, two s, H-4,5, imidazole ring); 7.05–7.24 (4H, m, H-5,3, Py); 7.55 (2H, m, H-4, Py); 8.44 (2H, m, H-6, Py). NMR spectrum ^{13}C, δ, ppm (*J*, Hz): 15.46 (CH_3); 25.96 (d, $^{1}J_{PC}$ = 48.3, CH_2P); 27.25 (d, $^{1}J_{PC}$ = 48.3, CH_2P); 29.55 (Py<u>C</u>H_2); 29.84 (Py<u>C</u>H_2); 41.98 (NCH_2); 67.40 (d, $^{1}J_{PC}$ = 56.7, PCH); 119.70 (C-5, imidazole ring); 121.10, 121.25 (C-5, Py); 122.80, 122.96 (C-3, Py); 126.57 (C-4, imidazole ring); 136.33, 136.41 (C-4, Py); 142.55 (C-2, imidazole ring); 148.56 (C-6, Py); 159.99 (d, $^{3}J_{PC}$ = 13.9, C-2, Py); 159.72 (d, $^{3}J_{PC}$ = 11.6, C-2, Py). ^{31}P NMR spectrum, δ, ppm: 59.34. Found, %: C 59.73; H 6.29; N 13.84; P 7.61; S 8.13. $C_{20}H_{25}N_4OPS$. Calculated, %: C 59.98; H 6.29; N 13.99; P 7.73; S 8.13.

2-{Bis[2-(2-pyridyl)ethyl]thiophosphorylhydroxymethyl}-1-vinylimidazole (8). Yield 86%. IR spectrum, ν, cm^{-1}: 3112 (OH), 576 (P=S). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.35, 2.55, 2.71 (4H, m, CH_2P); 3.04, 3.20, 3.30 (4H, m, $PyCH_2$); 4.88 (1H, d, $^{3}J_{HH}$ = 8.4, =CH_2, *cis*); 5.20 (1H, d, $^{3}J_{HH}$ = 15.4, =CH_2, *trans*); 5.31 (1H, s, PCH); 6.24 (1H, br. s, OH); 7.01–7.27, 7.45–7.59 (9H, m, H-4,5, imidazole ring; CH=, H-5,3,4, Py); 8.43 (2H, m, H-6, Py). ^{13}C NMR spectrum, δ, ppm (*J*, Hz): 26.93 (d, $^{1}J_{PC}$ = 47.5, CH_2P); 27.64 (d, $^{1}J_{PC}$ = 47.5, CH_2P); 29.89 (Py<u>C</u>H_2); 30.30 (Py<u>C</u>H_2); 68.37 (d, $^{1}J_{PC}$ = 58.7, PCH); 103.38 (=CH_2); 117.37 (C-5, imidazole ring); 121.64, 121.80 (C-5, Py); 123.34, 123.53 (C-3, Py); 128.67 (CH=); 130.49 (C-4, imidazole ring); 136.88, 137.07 (C-4, Py); 143.02 (C-2, imidazole ring); 148.89, 148.99 (C-6, Py); 160.16 (d, $^{3}J_{PC}$ = 13.6, C-2, Py); 160.38 (d, $^{3}J_{PC}$ = 11.9, C-2, Py). ^{31}P NMR spectrum, δ, ppm: 57.89. Found, %: C 60.41; H 5.79; N 14.02; P 7.55; S 7.97. $C_{20}H_{23}N_4OPS$. Calculated, %: C 60.29; H 5.82; N 14.06; P 7.77; S 8.05.

1-Ethyl-2-{bis[2-(2-pyridyl)ethyl]thiophosphorylhydroxymethyl}benzimidazole (9). Yield 82%. IR spectrum, ν, cm^{-1}: 3112 (OH), 564 (P=S). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.29 (3H, t, $^{3}J_{HH}$ = 7.2, CH_3); 2.39, 2.57, 2.76 (4H, m, CH_2P); 2.96, 3.18, 3.22 (4H, m, $PyCH_2$); 4.32, 4.50 (2H, m, NCH_2); 5.44 (1H, d, $^{2}J_{HP}$ = 3.3, PCH); 6.99–7.21, 7.27–7.52 (9H, m, H-7,8,9, benzimidazole ring; H-5,3,4, Py); 7.60 (1H, d, $^{3}J_{HH}$ = 7.8, H-6, benzimidazole ring); 8.36 (1H, d, $^{3}J_{HH}$ = 4.2, H-6, Py); 8.40 (1H, d, $^{3}J_{HH}$ = 4.4, H-6, Py). ^{13}C NMR spectrum, δ, ppm (*J*, Hz): 14.57 (CH_3); 26.24 (d, $^{1}J_{PC}$ = 47.1, CH_2P); 27.48 (d, $^{1}J_{PC}$ = 47.5, CH_2P); 29.60 (Py<u>C</u>H_2); 29.94 (Py<u>C</u>H_2); 39.89 (NCH_2); 68.19 (d, $^{1}J_{PC}$ = 57.9, PCH); 109.81 (C-6, benzimidazole ring); 119.04 (C-9, benzimidazole ring); 121.13, 121.34 (C-5, Py); 122.06 (C-7, benzimidazole ring); 122.66 (C-8, benzimidazole ring); 122.89, 123.09 (C-3, Py); 134.54 (C-5, benzimidazole ring); 136.40, 136.61 (C-4, Py); 141.36 (C-4, benzimidazole ring); 148.44 (d, $^{3}J_{PC}$ = 2.8, C-6, Py); 149.30 (C-2, benzimidazole ring); 159.07 (d, $^{3}J_{PC}$ = 12.1, C-2, Py); 159.99 (d, $^{3}J_{PC}$ = 14.3, C-2, Py). ^{31}P NMR spectrum, δ, ppm: 58.07. Found, %: C 63.86; H 6.01; N 12.39; P 6.79; S 7.08. $C_{24}H_{27}N_4OPS$. Calculated, %: C 63.98; H 6.04; N 12.44; P 6.87; S 7.12.

1-Ethyl-2-{bis[2-(2-pyridyl)ethyl]selenophosphorylhydroxymethyl}imidazole (10). Yield 79%. IR spectrum, ν, cm^{-1}: 3127 (OH), 470 (P=Se). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.42 (3H, t, $^{3}J_{HH}$ = 7.3, CH_3); 2.24, 2.49, 2.81 (4H, m, CH_2P); 2.96, 3.12, 3.30 (4H, m, $PyCH_2$); 4.21 (2H, q, $^{3}J_{HH}$ = 7.3, NCH_2), 5.36 (1H, s, PCH); 6.94, 7.05 (2H, two s, H-4,5, imidazole ring); 7.09–7.69 (6H, m, H-5,3,4, Py); 8.47 (2H, H-6, Py). ^{13}C NMR spectrum, δ, ppm (*J*, Hz): 15.75 (CH_3); 25.62 (d, $^{1}J_{PC}$ = 40.5, CH_2P); 26.80 (d, $^{1}J_{PC}$ = 40.5, CH_2P); 30.58 (Py<u>C</u>H_2); 31.05 (Py<u>C</u>H_2); 42.15 (NCH_2); 67.35 (d, $^{1}J_{PC}$ = 52.3, PCH); 119.73 (C-5, imidazole ring); 121.22, 121.44 (C-5, Py); 122.99, 123.22 (C-3, Py); 127.34 (C-4, imidazole ring); 136.39, 136.63 (C-4, Py); 142.42 (C-2, imidazole ring); 148.64, 148.77 (C-6, Py); 159.76 (d, $^{3}J_{PC}$ = 11.8, C-2, Py); 160.06 (d, $^{3}J_{PC}$ = 14.3, C-2, Py). ^{31}P NMR spectrum, δ, ppm: 51.48. Found, %: C 53.56; H 5.69; N 12.49; P 6.42; Se 17.57. $C_{20}H_{25}N_4OPSe$. Calculated, %: C 53.69; H 5.63; N 12.52; P 6.92; Se 17.65.

2-{Bis[2-(2-pyridyl)ethyl]selenophosphorylhydroxymethyl}-1-vinylimidazole (11). Yield 81%. IR spectrum, ν, cm^{-1}: 3108 (OH), 475 (P=Se). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.41, 2.68, 2.84 (4H, m, CH_2P); 3.06, 3.18, 3.38 (4H, m, $PyCH_2$); 4.86 (1H, dd, $^{3}J_{HH}$ = 7.3, $^{2}J_{HH}$ = 1.3, =CH_2, *cis*); 5.17 (1H, dd, $^{3}J_{HH}$ = 15.4, $^{2}J_{HH}$ = 1.3, =CH_2, *trans*); 5.33 (1H, s, PCH); 6.23 (1H, br. s, OH); 6.98, 6.99 (2H, two s, H-4,5, imidazole ring); 7.05–7.26, 7.43–7.54 (7H, m, H-5, Py; CH=; H-3,4, Py); 8.41 (2H, H-6, Py). ^{13}C NMR spectrum, δ, ppm (*J*, Hz): 26.52 (d, $^{1}J_{PC}$ = 40.3, CH_2P); 27.14 (d, $^{1}J_{PC}$ = 40.3, CH_2P); 30.80 (Py<u>C</u>H_2); 31.28 (Py<u>C</u>H_2); 67.74 (d, $^{1}J_{PC}$ = 49.5, PCH); 103.59 (=CH_2); 117.37 (C-5, imidazole ring); 121.64, 121.80 (C-5, Py); 123.34, 123.54 (C-3, Py); 128.67 (CH=); 130.49 (C-4, imidazole ring); 136.87, 137.07 (C-4, Py); 143.02 (C-2,

imidazole ring); 148.89, 148.98 (C-6, Py);159.95 (d, $^3J_{PC}$ = 11.9, C-2, Py); 160.19 (d, $^3J_{PC}$ = 13.7, C-2, Py). ^{31}P NMR spectrum, δ, ppm: 50.38. Found, %: C 53.97; H 5.23; N 12.53; P 6.86; Se 17.69. $C_{20}H_{23}N_4OPSe$. Calculated, %: C 53.94; H 5.21; N 12.58; P 6.95; Se 17.73.

2-{Bis[2-(2-pyridyl)ethyl]selenophosphorylhydroxymethyl}-1-vinylbenzimidazole (12). Yield 78%. IR spectrum, ν, cm^{-1}: 3112 (OH), 470 (P=Se). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.45, 2.82, 2.86 (4H, m, CH_2P); 2.96, 3.23, 3.31 (4H, m, $PyCH_2$); 5.27 (1H, d, $^3J_{HH}$ = 8.2, $=CH_2$, *cis*); 5.58 (1H, d, $^3J_{HH}$ = 15.1, $=CH_2$, *trans*); 5.59 (1H, d, $^2J_{HP}$ = 2.6, PCH); 6.99–7.22, 7.47–7.59 (10H, m, H-7,8,9, benzimidazole ring; CH=; H-5,3,4, Py); 7.67 (1H, d, $^3J_{HH}$ = 7.7, H-6, benzimidazole ring); 8.39 (2H, m, H-6, Py). ^{13}C NMR spectrum, δ, ppm (*J*, Hz): 25.82 (d, $^1J_{PC}$ = 40.5, CH_2P); 26.86 (d, $^1J_{PC}$ = 40.5, CH_2P); 30.39 ($PyCH_2$); 31.11 ($PyCH_2$); 67.28 (d, $^1J_{PC}$ = 51.6, PCH); 109.71 ($=CH_2$); 111.31 (C-6, benzimidazole ring); 119.82 (C-9, benzimidazole ring); 121.25, 121.45 (C-5, Py); 122.76 (C-7, benzimidazole ring); 123.16 (C-8, benzimidazole ring); 123.40, 123.54 (C-3, Py); 129.98 (=CH); 133.74 (C-5, benzimidazole ring); 136.43, 136.99 (C-4, Py); 142.39 (C-4, benzimidazole ring); 148.36, 148.67 (C-6, Py); 149.26 (C-2, benzimidazole ring); 159.55 (d, $^3J_{PC}$ = 14.7, C-2, Py); 159.93 (d, $^3J_{PC}$ = 11.4, C-2, Py). ^{31}P NMR spectrum, δ, ppm: 51.09. Found, C 58.13; H 5.07; N 11.29; P 6.18; Se 15.86. $C_{24}H_{25}N_4OPSe$. Calculated, %: C 58.18; H 5.08; N 11.31; P 6.25; Se 15.94.

This work was carried out with the financial support of the Russian Fund for Basic Research (grant No. 07-03-00562a).

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