Russian Journal of Organic Chemistry, Vol. 41, No. 11, 2005, pp. 1610–1614. Translated from Zhurnal Organicheskoi Khimii, Vol. 41, No. 11, 2005, pp. 1644–1648.
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Reaction of Phosphoric (Phosphonic) Acids Amides with Chloromethyliso(thio)cyanatophosphonates (-phosphinates). Synthesis of 1,3,4-Oxaza(thiaza)phospholines

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Received June 25, 2005

Abstract—Addition of phosphoric (phosphonic) acids amides to chloromethyliso(thio)cyanatophosphonates (-phosphinates) gave rise to diphosphorylated ureas or thioureas that under effect of bases underwent cyclization into 1,3,4-oxaza(thiaza)phospholines.

In the last decade a considerable attention of researchers has been drawn to development of preparation methods for cyclic phosphorus derivatives containing endocyclic P–C bonds [1]. One approach to the synthesis of this type phosphacyclanes is based on intramolecular transformations of polyfunctional organophosphorus compounds. Haloalkylphosphonates(phosphinates) can be successfully used in these reactions. M.I. Kabachnik and T.A. Mastryukova *et al*. carried out a series of investigations on the synthesis of ring structures proceeding from ω-haloalkylphosphonates. The intramolecular cyclization occurs involving haloalkyl and thiophosphoryl or imidophosphoryl groups affording aza(thia)phosphacyclanes of various composition and structure [2–7]. We formerly studied the addition of primarily and secondary amines to chloromethyliso(thio)cyanatophosphonates (-phosphinates) and demonstrated that the arising chloromethylphosphonylated (-phosphinylated) thioureas under treatment with a base underwent intramolecular heterocyclization involving the halomethyl and thiocarbonyl groups to form 1,3,4-thiazaphospholines [8]. The chloromethylphosphonylated (-phosphinylated) ureas containing mobile protons at the bonding and terminal nitrogen atoms undergo intramolecular cyclization along two pathways. Depending on the character of substituents at the phosphorus and the terminal nitrogen the cyclic skeleton of the molecule is built up either through a nucleophilic attack of an oxygen atom on the carbon of the chloromethyl group providing unsaturated phosphacyclanes, 1,3,4-oxazaphospholines, or via an attack of the terminal nitrogen, and in this case form saturated ring structures, 1,3,4-diazaphospholidines. We found besides examples where the reaction proceeded along both routes. [8, 9].

In extension of this research we investigated the addition of amidophosphates (-phosphonates) to chloromethyliso(thio)cyanatophosphonates (-phosphinates) aiming at the synthesis of unsymmetrical diphosphorylated (thio)ureas and at the study of the possibility of their cyclization into new five-membered phosphorus-containing heterocycles with an endocylic P–C bond.

We established that diethylphosphoric acid amides **Ia** and **Ib** added to chloromethyliso(thio)cyanatophosphonates (-phosphinates) **IIa** and **IIb** at room temperature without catalyst to afford in a high yield diphosphorylated ureas **IIIa–IIId**. The structure of compounds **IIIa–IIId** was confirmed by IR, ^{1}H and ^{31}P NMR spectra, and the composition was proved by elemental analyses. In the ^{31}P NMR spectra of the diphosphorylated ureas two singlet peaks are observed corresponding to two non-equivalent phosphorus atoms. The chemical shifts of phosphorus nuclei of the phosphonate fragment have values in the range 2.2–4.0 ppm, phosphonate signals in compounds **IIIa** and **IIIc** appear at 13.8–13.9 ppm, and phosphinate peaks of compounds **IIIb** and **IIId** are observed at 26.0–27.0 ppm. The strong absorption bands in the IR spectra in the regions 1245–1295 and 1675–1695 cm^{-1} belong respectively to the phosphoryl and carbonyl groups. At treating the diphosphorylated ureas **IIIa–IIId** with triethylamine the formation of the base hydrochloride was observed, and oxazaphospholines **IVb–IVd** were isolated as product. Compound **IVc** is a crystalline substance, and compounds **IVb** and **IVd** were obtained as viscous fluids.

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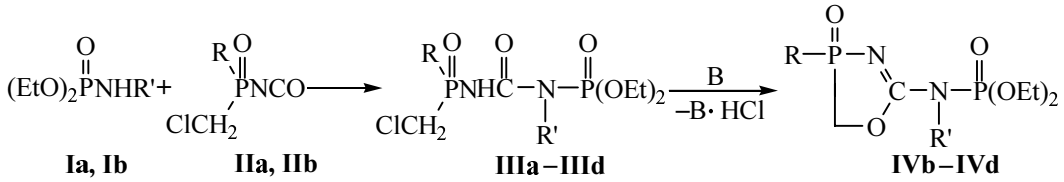


Ia, Ib **IIa, IIb** **IIIa–IIId** **IVb–IVd**

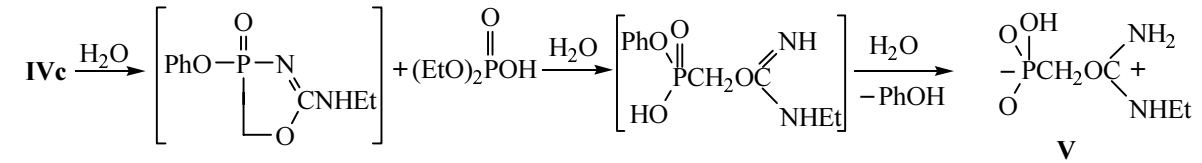
I, R = Me (**a**), Et (**b**); **II**, R = PhO (**a**), $ClCH_2$ (**b**); **III, IV**, R = PhO, R' = Me (**a**), Et (**c**); R = $ClCH_2$, R' = Me (**b**), Et (**d**).

The theoretical study of phosphorylated ureas cyclization performed by *ab initio* and semiempirical methods revealed that the process involved the primary formation of a complex of the base (triethylamine) and the urea resulting in a weakening of the N–H bond or even in formation of an ion pair $(X)^- Et_3NH^+$. The subsequent attack of the oxygen atom on the carbon of the chloromethyl group occurred with elimination of the chloride anion and with building up of the cyclic skeleton of the molecule [9]. A similar pattern apparently corresponds also to the cyclization of the diphosphorylated ureas under study.

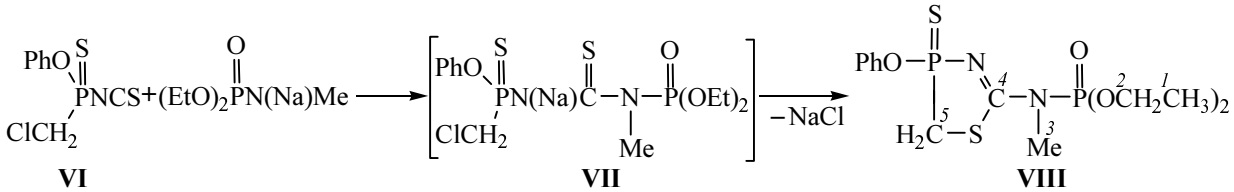
The chemical shift of the endocyclic phosphorus atom in the spectrun of oxazaphospholine **IVc** is δ_P 46.9 ppm characteristic of this type compounds, and the peak of the exocyclic phosphorus appears at δ_P –1.8 ppm. Its IR spectrum contains an absorption band of the exocyclic C=N bond (1600 cm^{-1}) but lacks the bands of the carbonyl group (1695) and of NH bond (3130 cm^{-1}) characteristic of urea **IIIc**. Compounds **IVb–IVd** suffer in air a fast hydrolysis. In particular, the hydrolysis of phospholine **IVc** involves the cleavage of the exo- and endocyclic P–N bonds, elimination of a phenoxy group, and formation of *N*-ethylisouroniomethyl(hydroxy)phosphonate (**V**). In the IR spectrum of compound **V** appear absorption bands at 1230–1240 (PO_2^-), 1565–1580, 2600–2780 (NH_2^+) cm^{-1} but the IR and ^1H NMR spectra show the absence of phenoxy groups. The chemical shift of the phosphorus atom is 10.0 ppm. Besides the phosphonate **V** we isolated from the reaction mixture the diethylphosphoric acid (δ_P 0 ppm).

Unlike isocyanates **IIa** and **IIb** the chloromethylisothiocyanatothiophosphonates did not react with phosphoric acids amides even at long heating. However replacing the amide of diethylphosphoric acid **Ia** by its sodium salt resulted in a readily occurring addition at the room temperature. The reaction apparently proceeds through a stage of formation of a sodium derivative of diphosphorylated thiourea **VII** that quickly transforms into a thiazaphospholine **VIII**. The process involves an intramolecular alkylation of the thione sulfur atom with a chloromethyl group and is accompanied with sodium chloride formation. The structure of compound **VIII** is confirmed by the data of IR, ^1H, ^{13}C, and ^{31}P NMR spectroscopy.

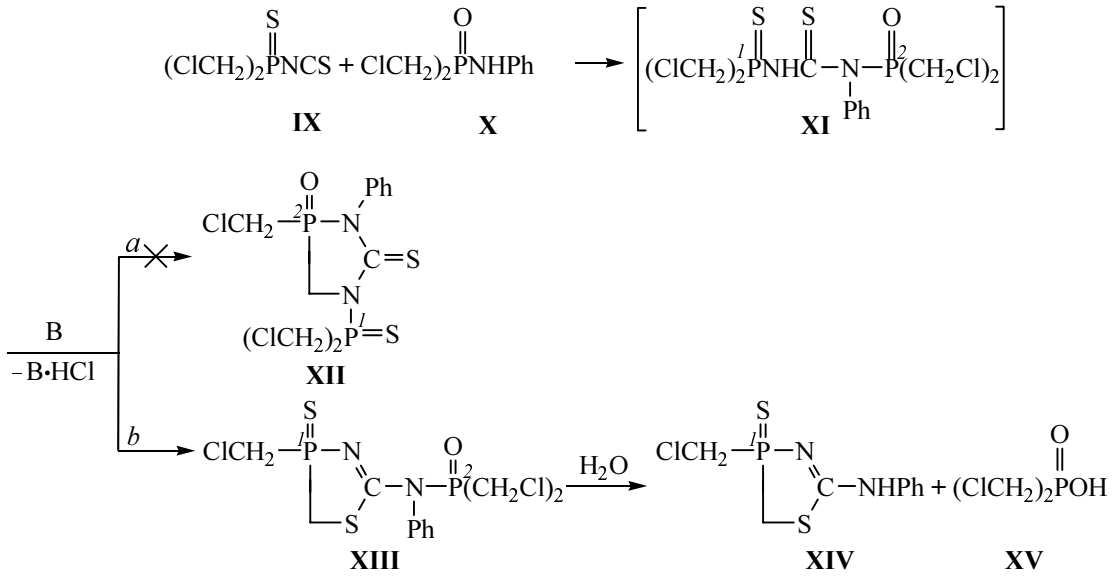
Alongside the phosphoric acids amides we used in reactions with phosphorylated isocyanates **IIa** and **IIb** and with isothiocyanatothiophosphonate **VI** also chloromethylphosphonic (-phosphinic) acids amides. The reactivity of these amides is essentially different. The reaction of isothiocyanatothiophosphinate **IX** with *O,N*-diphenylchloromethylamidophosphonate did not occur even at prolonged heating. At the same time the replacement of the phenoxy group by the chloromethyl one, i.e., at bringing into the process bis(chloromethyl)phenylaminophosphinate **X** the reaction with isothiocyanatothiophosphinate **IX** proceeded at room temperature within a week. It should be noted that the cyclization of the forming diphosphorylated thiourea **XI** may presumably occur along two pathways involving: 1) the alkylation of the nitrogen from the secondary amino group by the chloromethyl group at the phosphinate atom P^2 (path *a*) afford-



V



VI **VII** **VIII**



ing a saturated heterocycle **XII**; 2) the alkylation of sulfur atom in the thiocarbonyl group by the chloromethyl group attached to the atom P^1 (path *b*) leading to an unsaturated ring structure, 1,3,4-thiazaphospholine **XIII**.

The theoretical analysis of the energy changes characteristic of formation of diazaphospholidines and thiazaphospholines via intramolecular transformations of the phosphorylated thioureas showed that the intramolecular alkylation of the sulfur atom in the thiocarbonyl group resulting in compounds with an unsaturated heterocyclic skeleton is a thermodynamically preferred exothermal process [9]. Actually, in the example under consideration the cyclization takes exclusively the path *b* and furnishes thiazaphospholine **XIII**. In the ^{31}P NMR spectrum of crude product **XIII** two singlets are observed at δ_P 118.5 and 29.0 ppm corresponding respectively to the endo- and exocyclic phosphorus atoms. The IR spectrum of compound **XIII** contains the absorption band of the endocyclic C=N bond (1570 cm^{-1}). Thiazaphospholine **XIII** was subjected to purification from the impurity of triethylamine hydrochloride (detected by spectral data) by the column chromatography. However due to the hydrolytic cleavage of the exocyclic P–N bond the purification provided 2-phenylamino-4-thioxo-4-chloromethyl-1,3,4-thiazaphospholine (**XIV**). The spectral characteristics and the melting point of compound **XIV** were consistent with the data published for this compound [10].

EXPERIMENTAL

IR spectra were recorded on a spectrophotometer UR-20 in the range 400-3600 cm^{-1} in thin film or mulls of compounds in the mineral oil. ^1H NMR spectra were registered on spectrometers Bruker WM-250 (250.132 MHz) and Varian T-60 (60 MHz), internal reference TMS. ^{13}C and ^{31}P NMR spectra were taken on an Fourier NMR spectrometer Bruker MSL-400 at operating frequencies 100.62 and 162.98 MHz, respectively.

***N*-[*O*-Phenyl(chloromethyl)phosphonyl-*N*'-methyl-*N*'-(*O,O*-diethyl)phosphoryl]urea (IIIa).** To a solution of 1.05 g (6.29 mmol) of amidophosphate **Ia** in 5 ml of anhydrous benzene was added 1.45 g (6.29 mmol) of isocyanate **IIa**. The reaction mixture was left standing for 30 days, then the solvent was removed, and the residue was recrystallized from benzene. Yield 1.7 g (71%), mp 52°C. IR spectrum (KBr), ν, cm^{-1}: 1030 (POEt), 1160, 1220 (POPh), 1270, 1280 (P=O), 1600 (Ph), 1695 (C=O), 3140 (NH). ^1H NMR spectrum (CCl_4), δ, ppm (*J*, Hz): 1.27 m (6H, $C\underline{H}_3CH_2O$), 2.85 d (3H, CH_3N, $^3J_{PNCH}$ 7.0), 3.93 m (6H, OCH_2, $ClCH_2$), 7.23 m (5H, Ph), 9.63 br.s (1H, NH). ^{31}P NMR spectrum (acetone): δ 13.8, 2.2 ppm. Found, %: C 39.39; H 5.15; Cl 9.53; N 7.13; P 15.54. $C_{13}H_{21}ClN_2O_6P_2$. Calculated, %: C 39.15; H 5.32; Cl 8.89; N 7.03; P 15.59.

***N*-Bis(chloromethyl)phosphinyl-*N*'-methyl-*N*'-[*O,O*-(diethyl)phosphoryl]urea (IIIb).** Yield 63%, mp 87–88°C. IR spectrum (KBr), ν, cm^{-1}: 1030 (POEt), 1245, 1260 (P=O), 1675 (C=O), 3160 (NH). ^1H NMR spectrum (CCl_4), δ, ppm (*J*, Hz): 1.4 t (6H, $C\underline{H}_3CH_2O$, $^3J_{HCCH}$ 7), 2.93 d (3H, CH_3N, $^3J_{PNCH}$ 7), 4.17 m (8H, OCH_2, $ClCH_2$), 8.93 br.s (1H, NH). ^{31}P NMR spectrum (acetone): δ 26.0, 3.0 ppm. Found, %: C 26.59; H 5.00; Cl 20.05; N 8.04; P 17.68. $C_8H_{18}Cl_2N_2O_5P_2$. Calculated, %: C 27.06; H 5.12; Cl 10.90; N 7.89; P 17.44.

***N*-[*O*-phenyl(chloromethyl)phosphonyl]-*N*'-ethyl-*N*'-[(*O,O*-diethyl)phosphoryl]urea (IIIc)**. Yield 89%, n_D^{20} 1.4966. IR spectrum (KBr), ν, cm^{-1}: 1060 (POEt), 1170, 1200 (POPh), 1270, 1295 (P=O), 1590 (Ph), 1695 (C=O), 3130 (NH). ^{1}H NMR spectrum (CCl_4), δ, ppm: 1.17 m (9H, C$\underline{H}_3$CH$_2$O, C$\underline{H}_3$CH$_2$N), 3.4 m (2H, CH$_2$N), 4.03 m (5H, OCH$_2$, ClCH$_2$), 7.2 m (5H, Ph), 9.63 br.s (1H, NH). ^{31}P NMR spectrum (acetone): δ 13.9, 2.4 ppm. Found, %: Cl 8.57; P 14.78. $C_{14}H_{23}ClN_2O_6P_2$. Calculated, %: Cl 8.59; P 15.01.

***N*-Bis(chloromethyl)phosphinyl-*N*'-ethyl-*N*'-[(*O,O*-diethyl)phosphoryl]urea (IIId).** Yield 63%, mp 49–51°C. IR spectrum (KBr), ν, cm^{-1}: 1023 (POEt), 1238, 1252 (P=O), 1683 (C=O), 3169 (NH). ^{1}H NMR spectrum (CCl_4), δ, ppm: 1.3 t (9H, C$\underline{H}_3$CH$_2$), 3.4 m (2H, CH$_2$N), 4.1 m (8H, OCH$_2$, ClCH$_2$), 9.4 br.s (1H, NH). ^{31}P NMR spectrum (acetone): δ 27.0, 4.0 ppm. Found, %: C 29.33; H 5.35; Cl 20.32; N 7.30; P 16.75. $C_9H_{20}Cl_2N_2O_5P_2$. Calculated, %: C 29.28; H 5.49; Cl 19.21; N 7.59; P 16.78.

Diethoxy[*N*-(4-oxo-4-chloromethyl-Δ^{2}1,3,4-oxazaphospholin-2-yl)-*N*-methyl]amidophosphate (IVb). To a solution of 0.5 g (1.41 mmol) of diphosphorylated urea **IIIb** in 5 ml of anhydrous benzene under an atmosphere of dry argon was added dropwise at stirring 0.2 g (1.98 mmol) of triethylamine. In 20 days the triethylamine hydrochloride was filtered off, and the solvent was evaporated in a vacuum to obtain a residue as a viscous transparent substance. Yield 0.4 g (89%). ^{1}H NMR spectrum (C_6D_6), δ , ppm (*J*, Hz): 1.14 m (6H, C$\underline{H}_3$CH$_2$), 3.35 d (3H, CH$_3$N, $^3J_{PNCH}$ 7.7), 3.58 d (2H, ClCH$_2$, $^2J_{PCH}$ 9.4), 4.24 m (6H, CH$_2$O, CH$_2$P). ^{31}P NMR spectrum: δ 58.68, –0.49 ppm. Found, %: P 19.15. $C_8H_{17}ClN_2O_5P_2$. Calculated, %: P 19.47.

Diethoxy[*N*-(4-oxo-4-phenoxy-Δ^{2}1,3,4-oxazaphospholin-2-yl)-*N*-ethyl]amidophosphate (IVc). Yield 88%, mp 62–64°C. IR spectrum (KBr), ν, cm^{-1}: 1026 (POEt), 1200, 1217 (POPh), 1267, 1282 (P=O), 1587 (Ph), 1600 (C=N). ^{1}H NMR spectrum (C_6D_6), δ, ppm: 0.97 m (9H, C$\underline{H}_3$CH$_2$), 3.82 m (8H, NCH$_2$, OCH$_2$, CH$_2$P), 7.0 m (5H, Ph). ^{31}P NMR spectrum (acetone): δ 46.9, –1.8 ppm. Found, %: P 16.11. $C_{14}H_{22}N_2O_6P_2$. Calculated, %: P 16.49.

Diethoxy[*N*-(4-oxo-4-chloromethyl-Δ^{2}1,3,4-oxazaphospholin-2-yl)-*N*-ethyl]amidophosphate (IVd). Yield 92%. ^{1}H NMR spectrum (C_6D_6), δ, ppm (*J*, Hz): 1.38 m (9H, C$\underline{H}_3$CH$_2$), 2.66 m (2H, NCH$_2$), 3.63 d (2H, ClCH$_2$, $^2J_{PCH}$ 9.4), 4.07 m (6H, CH$_2$O, CH$_2$P). ^{31}P NMR spectrum (acetone): δ 59.5, –0.5 ppm. Found, %: P 18.21. $C_9H_{19}ClN_2O_5P_2$. Calculated, %: P 18.65.

***N*-Ethylisouroniomethyl(hydroxy)phosphonate (V).** To a solution of 0.38 g (1 mmol) of phosphonate **IVc** in 5 ml of anhydrous benzene was added 0.2 g (10 mmol) of water, and the mixture was heated for 1 h at 80°C. The separated precipitate was filtered off and washed with benzene. Yield of compound **V** 0.13 g (87%), mp 191–193°C. ^{1}H NMR spectrum (D_2O), δ, ppm: 1.21 m (3H, C$\underline{H}_3$CH$_2$), 3.2 m (2H, CH$_2$N) 4.3 m (2H, CH$_2$P). ^{31}P NMR spectrum (acetone): δ 10.58 ppm. Found, %: C 26.49; H 6.07; N 15.14; P 17.48. $C_4H_{11}N_2O_4P$. Calculated, %: C 26.38; H 6.10; N 15.38; P 17.00.

Diethoxy[*N*-(4-thioxo-4-phenoxy-Δ^{2}1,3,4-thiazaphospholin-2-yl)-*N*-methyl]amidophosphate (VIII). To a solution of 0.12 g (5.18 mmol) of NaH in 50 ml of anhydrous ether under an atmosphere of dry argon was added dropwise at stirring 0.7 g (5.18 mmol) of amidophosphate **IIa**. The reaction mixture was stirred for 4.5 h at 30°C, then 1.36 g (5.18 mmol) of isothiocyanatophosphonate **VI** was added dropwise. In 4 days NaCl was separated, and solvent was removed. The residue was subjected to chromatography on Al_2O_3 (neutral, II grade Brockmann activity; eluent chloroform). Yield 1.53 g (93%), n_D^{20} 1.5750. IR spectrum (KBr), ν, cm^{-1}: 695 (P=S), 1030 (POEt), 1165, 1200 (POPh), 1280 (P=O), 1530 (C=N), 1585 (Ph). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm (*J*, Hz): 1.34 t (3H, C$\underline{H}_3$CH$_2$, $^3J_{HCCH}$ 6.3), 1.36 t (3H, CH$_3$CH$_2$, $^3J_{HH}$ 6.3), 3.29 d (3H, CH$_3$N, $^3J_{PNCH}$ 7.0), 3.64 m (2H, CH$_2$P), 4.15 m (4H, OCH$_2$), 7.28 m (5H, Ph). ^{13}C NMR spectrum ($CDCl_3$), ppm (*J*, Hz): 15.62 (C^1), 15.75 (C^1), 64.39 (C^2, $^2J_{PC}$ 5.5), 64.22 (C^2, $^2J_{PC}$ 5.5), 36.66 (C^3, $^1J_{CH}$ 149, $^2J_{PC}$ 12), 171.0 (C^4, $^2J_{PC}$ 13), 35.27 (C^5, $^2J_{PC}$ 56), 150.55 (C$_i$, $^2J_{PC}$ 10), 121.23 (C$_O$, $^3J_{PC}$ 5), 129.21 (C$_m$), 124.84 (C$_p$). ^{31}P NMR spectrum (acetone): δ 112.38, –0.81 ppm. Found, %: P 15.38. $C_{13}H_{20}N_2O_4P_2S_2$. Calculated, %: P 15.71.

[*N*-(4-Thioxo-4-chloromethyl-Δ^{2}1,3,4-thiazaphospholin-2-yl)-*N*-phenyl]amidobis(chloromethyl)phosphinate (XIV). To a mixture of 0.54 g (2.27 mmol) of amidophosphinate **X** and 0.23 g (2.27 mmol) of triethylamine in 10 ml of anhydrous benzene was added 0.50 g (2.27 mmol) of isothiocyanate **IX**. In 10 days the triethylamine hydrochloride was removed, the solvent was evaporated, and the viscous residue was kept in a vacuum (0.01 mm Hg) to the constant weight. We obtained 0.85 g (88%) of compound **XIII**. IR spectrum (KBr), ν, cm^{-1}: 690 (P=S), 1270 (P=O), 1570 (C=N), 1605 (Ph).

Found ^{31}P NMR spectrum (acetone): δ 108.5, 29.0 ppm. As a result of chromatography of compound **XIII** on Al_2O_3 (neutral, II grade Brockmann activity; eluent chloroform) 0.3 g (61%) of phospholine **XIV** was isolated, mp 164°C. IR spectrum (KBr), ν, cm^{-1}: 680 (P=S), 760 (P–C–Cl), 985 (P–N), 1550 (C=N), 1600 (C_6H_5), 3040, 3130, 3225, 3250 (NH). ^{1}H NMR spectrum [$(CD_3)_2CO$], δ, ppm (*J*, Hz): 3.35 d.d, 3.98 d.d (2H, SCH_2P, $^2J_{HCH}$ 13.1, $^2J_{PCH}$ 10.0 and 6.5, respectively), 4.06 d and 4.13 d (2H, PCH_2Cl, $^2J_{PCH}$ 0 and 2.1, respectively), 6.85–7.60 m (5H, C_6H_5); 9.69 br.s (NH). ^{31}P NMR spectrum (acetone): δ 109.4 ppm. Found, %: C 38.82; H 3.37; N 10.16; P 10.48. $C_9H_{10}ClN_2PS_2$. Calculated, %: C 39.06; H 3.65; N 10.12; P 11.19.

The study was carried out under a financial support of the Russian Foundation for Basic Research (grant 03-03-33064).

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