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# Reaction of Dibromoalkanes with Silyl Phosphites. Synthesis and Properties of Mono- and Diphosphonoalkanes

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**Abstract** — Reactions of dibromoethane and dibromopropane with silyl phosphites are studied. Mono- and diphosphonoalkanes of various structures were prepared, and their chemical properties were studied.
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Over the past years we have developed procedures for synthesis of chloro(bromo)methyl(thio)phosphonylated(phosphinytaled) ureas, thioureas, urethanes, and thiourethanes by the addition of amines, alcohols, and thiols to corresponding halomethyl iso(thio)cyanatophosphonates(phosphinates). In the presence of bases, the products undergo intramolecular transformations to form cyclic compounds of various compositions and structures [1–4]. To find out if this approach is feasible for preparing larger cycles, specifically sixmembered, we set ourselves the task to introduce to the phosphorus atom a 2-haloalkyl group instead of halomethyl. According to published data, haloalkylphosphonic acid esters are most commonly synthesized by reactions of dihaloalkanes with trialkyl phosphites [5–12]. At present much interest is attached to silyl esters of P(III) acids. On the one hand, this interest is associated with the fact that silyl phosphites(phosphonites) can be easily and effectively prepared by silylation of available P(III) acids with compounds (including those produced industrially) with a Si–N bond (hexamethyldisilazane, heptamethyldisilazane, trimethylsilyldiethylamine, etc.) [13–17]. On the other hand, silyl esters of P(III) acids are highly reactive toward various electrophilic agents and have found wide application in organic synthesis [18, 19]. In particular, reactions of dihaloalkanes with silyl phenylphosphonites were used as the basis for the procedures for synthesis of vinylphosphoryl derivatives [17] and phosphorus-containing aminocarboxylic acids [20]. With this in mind, we made an attempt to develop synthetic procedures for α- and γ-bromoalkylphosphonates and diphosphonoalkanes, relying on reactions of dibromoalkanes with silyl phosphites.

The reactions of equimolar amounts of dibromoethane **Ia** or dibromopropane **Ib** with tris(trimethylsilyl) phosphite (**II**) occur under heating to form silyl phosphonates **IIIa**, **IIIb**. The latter were chlorinated with $PCl_5$ to obtain bromoalkylphosphonic dichlorides **IVa**, **IVb** that are basic starting materials for synthesis of linear and cyclic compounds with a P–C bond.

$$\underset{\textbf{Ia, Ib}}{Br(CH_2)_nBr} + \underset{\textbf{II}}{(Me_3SiO)_3P} \longrightarrow \underset{\textbf{IIIa, IIIb}}{Br(CH_2)_nP(O)(OSiMe_3)_2} \xrightarrow{PCl_5} \underset{\textbf{IVa, IVb}}{Br(CH_2)_nP(O)Cl_2},$$

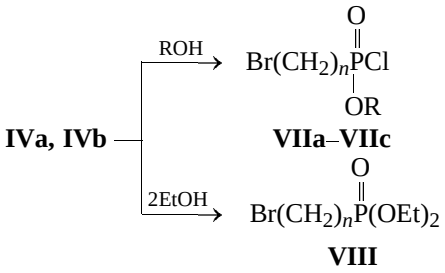
$n$ = 2 (**Ia, IIIa, IVa**), 3 (**Ib, IIIb, IVb**).

(Bromoethyl)phosphonic dichloride **IVa** was also prepared in comparable yield by the reaction of dibromoethane with diethyl trimethylsilyl phosphite. As follows from the $^{31}P$ spectrum, the latter reaction involves intermediate formation of phosphonates **V** and **VI** ($\delta_P$ 25.09, 14.35 ppm) in roughly equal amounts. Treatment of the reaction mixture with $PCl_5$, too, gave phosphonic dichloride **IVa**.

$$\textbf{Ia} + (EtO)_2POSiMe_3 \longrightarrow \underset{\textbf{V}}{Br(CH_2)_2P(O)(OEt)SiMe_3}$$

$$+ \underset{\textbf{VI}}{Br(CH_2)_2P(O)(OEt)_2} \xrightarrow{PCl_5} \textbf{IVa}.$$

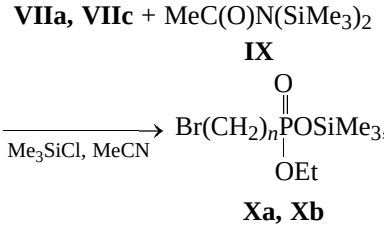
The (bromoalkyl)phosphonic dichlorides react with alcohols to give, depending on the reactant molar ration, partial or full alkoxylation products **VIIa**–**VIIc**, **VIII**.




**VII**, $n$ = 2, R = Et (**a**), Ph (**b**); $n$ = 3, R = Et (**c**).

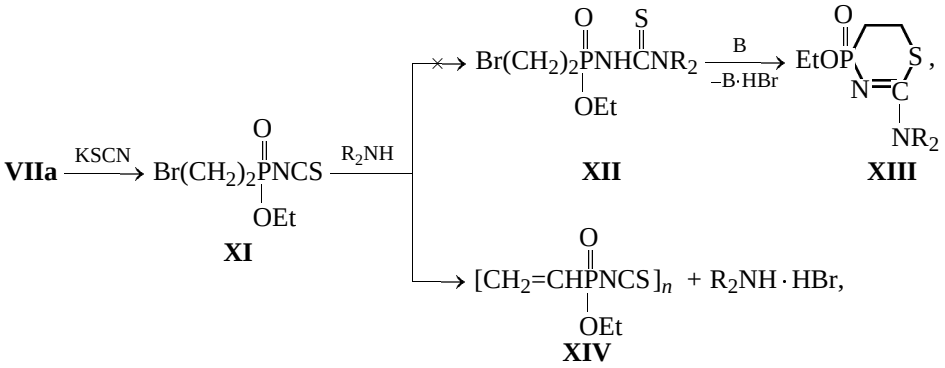
The example of phosphonochloridates **VIIa**, **VIIc** was used to show that our previous procedure involving bis(trimethylsilyl)acetamide [21] allows introduction of a siloxy group to phosphorus to obtained mixed bromoalkylphosphonates **Xa**, **Xb**.

The reaction of compound **VIIa** with potassium



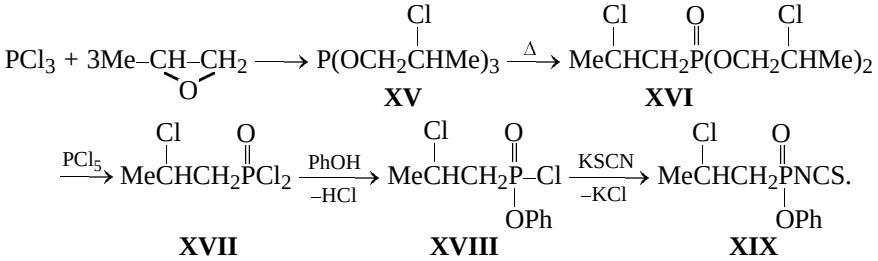

**X**, $n$ = 2, R = Et (**a**); $n$ = 3, R = Et (**b**).

thiocyanate gave phosphonoisothiocyanatidate **XI**. However, we failed to add diethyl- or dimethylamine by the isothiocyanato group to prepare phosphorylated thiourea **XII**. It was found that the major process in these conditions was dehydrobromination yielding an unstable vinylphosphonoisothiocyanatidate **XIV** (that probably undergoes fast isomerization), as evidenced by the isolation of dialkylamine hydrobromides.

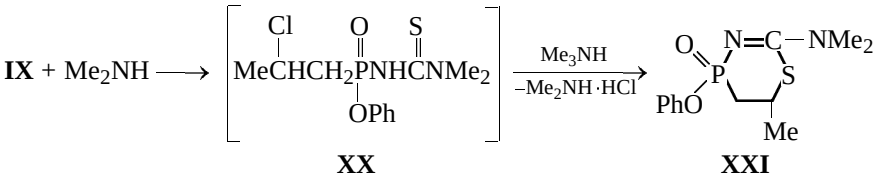



R = Me, Et.

To suppress dehydrohalogenation, we decided to obtain an analog of compound **XI**, phosphonoisothiocyanatidate **XIX** in which the halogen and phosphorus atoms are intervened by an ethylene bridge. Taking account of the data in [22], we reacted $PCl_3$ with propene oxide to obtain tris(2-chloropropyl) phosphite (**XV**) that isomerized into bis(2-chloropropyl) (2-chloropropyl)phosphonate (**XVI**) under prolonged heating. Treatment of the latter with $PCl_5$ gave the base reaction product, viz. (2-chloropropyl)phosphonic dichloride (**XVII**).



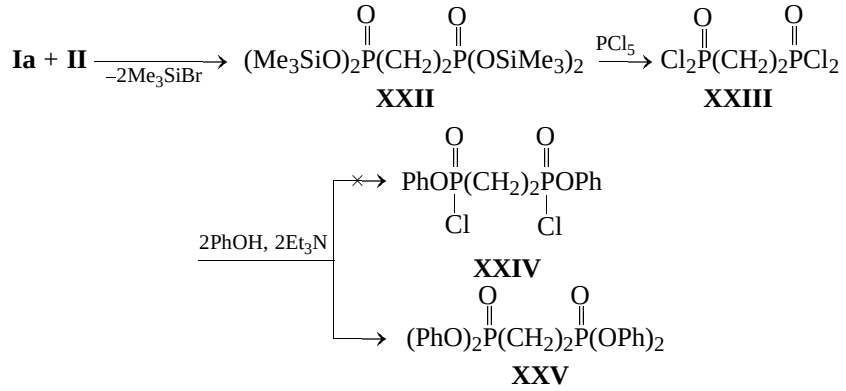
The reaction of equimolar amounts of phosphonic dichloride **XVII** and phenol afforded phosphonochloridate **XVIII** whose prolonged heating with potassium thiocyanate yielded phosphonoisothiocyanatidate **XIX**. According to the $^{31}P$ NMR spectrum, compounds **XVIII** and **XIX**, that have two chiral centers, are mixtures of two diastereomers in nearly 1 : 1 ratios. Compound **XIX** was found to decompose on vacuum distillation, and, therefore, it was further used without isolation.




**XX** **XXI**

Excess dimethylamine was bubbled through a benzene solution of phosphonoisothiocyanatidate **XIX**. The crystals that formed were a mixture of cycle **XXI** and dimethylamine hydrochloride. The cyclization probably involves intermediate formation of phosphorylated thiourea **XX** that fast cyclizes into final compound **XXI**. Since the reaction was performed in the presence of excess dimethylamine, we consider the most probable the following cyclization pathway: First a complex between base (dimethylamine) and thiourea is formed, which is accompanied by weakening of the N–H bond and, probably, ion pair formation. The subsequent nucleophilic attack of the thiourea sulfur atom on the β-carbon atom of the chloropropyl group is accompanied by chlorine expulsion from the latter and ring formation. Triple crystallization of the precipitate from benzene provided individual phospohacyclane **XXI** as a single diastereomer ($\delta_P$ 15.74 ppm). In the $^1H$ NMR spectrum of compound **XXI**, the methyl group at carbon appears as a doublet of doublets (δ 1.36 ppm, $^3J_{HH}$ 6.8, $^4J_{HP}$ 2.7 Hz). The composition and structure of compound **XXI** were also proved by its electron impact mass spectrum and elemental analysis. The mass spectrum of phosphacyclane **XXI** contains a molecular ion peak at *m/z* 284. According to X-ray diffraction data, the molecular conformation of **XXI** is *half-chair*: The four-atomic fragment $S^1C^2N^3P^4$ is planar [within 0.02(1) Å], and $C^5$ and $C^6$ deviate from this plane by 0.440(7) and –0.380(6) Å, respectively, i.e. locate on different sides of this fragment. The chiral atoms $P^4$ and $C^6$ have an *R* configuration. Note that compound **XXI** crystallizes in a $P2_1$ space group, the molecule is chiral, and we could establish its absolute configuration in crystal. In whole crystals of compound **XXI** are a conglomerate, i.e. crystallization is accompanied by spontaneous enantiomeric resolution. Thus, by cyclizing a thiourea containing a 2-chloropropylphosphinoyl we could prepare a substituted 5,6-dihydro-1,3,4λ$^5$-thiazaphosphinine, the first representative of P,N,S-containing unsaturated six-membered heterocycles of this type.

The reaction of dibromoethane with a double molar excess of silyl phosphite **II** resulted in isolation of diphosphonate **XXII** whose treatment with $PCl_5$ gave compound **XXIII**.




**XXII** **XXIII** **XXIV** **XXV**

It should be noted that we failed to effect partial phenoxylation of bisphosphonate **XXIII** with phenol in the presence of base (reagent ratio 1:2:2) to prepare dichloride **XXIV**. The major reaction product was tetraphenyl diphosphonate **XXV**. A mixed diphosphonate in which each phosphorus atom is bound with an ethoxy group and a chlorine atom (compound **XXVII**), was prepared by the action of $PCl_5$ on diphosphonate **XXVI** [23]. The reaction of diphosphonate **XXVII** with a double molar excess of methylamine, performed with the aim to prepare new P,N-containing heterocycles, failed to provide target azadiphospholanes **XXVIII**. According to the $^{31}P$ NMR spectrum, a complex mixture of products formed in this case. Mixed diphosphonate **XXIX** was obtained by the reaction of compound **XXVII** with excess methylamine.

## EXPERIEMNTAL

The IR spectrum was measured on a UR-20 instrument in the range 400–3600 $cm^{-1}$ in mineral oil. The $^1H$ NMR spectra were measured on a Bruker WM-250 spectrometer (250.132 MHz), internal reference TMS. The $^{31}P$ NMR spectra were obtained on a Bruker MSL-400 Fourier spectrometer (100.62 MHz).

The electron impact mass spectra were obtained on a Finnigan-MAT TRACE-MS instrument at 70 eV, ion source temperature 200°C. Direct sample admission into the ion source was applied. The inlet probe ampule was heated from 35 to 150°C at steps of 35 deg $min^{-1}$. The mass spectra were treated using the XCALIBUR program. The X-ray diffraction analysis of compound XXI was performed on an Enraf–Nonius CAD-4 diffractometer at 20°C ($\lambda Mo K_\alpha$ radiation, $\omega/2\theta$ scanning, $2q_{max} < 27.8°$).

Phosphite **XV** and phosphonate **XVI** were prepared by known procedures [22, 24].

**Bis(trimethylsilyl) (2-bromoethyl)phosphonate (IIIa).** A mixture of 45 g of silyl phosphite **II** and 61 g of dibromoethane was heated for 2 h at 160°C and then fractionated in a vacuum to obtain 42 g (37%) of compound **IIIa**, bp 78–79°C (0.08 mm Hg), $n_D^{20}$ 1.4437. $^{31}P$ NMR spectrum, $\delta_P$, ppm: 7.28. Found, %: P 9.39; Si 16.96. $C_8H_{22}BrO_3PSi_2$. Calculated, %: P 9.30; Si 16.81.

**Bis(trimethylsilyl) 3-(bromopropyl)phosphonate (IIIb).** A mixture of 15.0 g silyl phosphite **II** and 40 g of dibromopropane was heated for 2 h at 160°C, bromotrimethylsilane and excess dibromopropane were removed by distillation. The residue was fractionated in a vacuum to obtain 9.5 g (55%) of compound **IIIb**, bp 115°C (0.1 mm Hg), $n_D^{20}$ 1.4508. $^{31}P$ NMR spectrum, $\delta_P$, ppm: 11.58. Found, %: P 8.42; Si 15.76. $C_9H_{24}BrO_3PSi_2$. Calculated, %: P 8.93; Si 16.13.

**(2-Bromoethyl)phosphonic dichloride (IVa).** *a*. Phosphorus pentachloride, 42.0 g, was added in portions to a mixture of 33.3 g of phosphonate **IIIb** and 0.3 g of Cu2Cl2, heated to 100°C. Vacuum fractionation gave 12.8 g (57%) of compound **IVa**, bp 68°C (0.06 mm Hg). $^{31}P$ NMR spectrum, $\delta_P$, ppm: 44.21 [5].

*b*. A mixture of 21.0 g of diethyl trimethylsilyl phosphite and 75.2 g of dibromoethane was heated for 1 h at 150°C. Volatile compounds were removed, the residue was treated with 41.6 g of $PCl_5$, and the mixture was allowed to stand for 2 h. Fractionation in a vacuum gave 12.2 g (54%) of compound **IVa**, bp 68–70°C (0.06 mm Hg). $^{31}P$ NMR spectrum, $\delta_P$, ppm: 44.21.

**(3-Bromopropyl)phosphonic dichloride (IVb).** Phosphorus pentachloride, 22.5 g, was added in portions with heating to 18 g of phosphonate **IIIb**. After 5 h, chlorotrimethylsilane and $PCl_5$ were removed by distillation, and the residue was fractionated in a vacuum to obtain 10.5 g (87%) of compound **IVb**, bp 86°C (0.1 mm Hg). $^{31}P$ NMR spectrum, $\delta_P$, ppm: 48.76. Found P, %: 12.71. $C_3H_6BrCl_2OP$. Calculated P, %: 12.92.

**Ethyl (2-bromoethyl)phosphonochloridate (VIIa).** A solution of 0.92 g of ethanol in 10 ml of chloroform was added to 4.5 g of dichloride **IVa** in 15 ml of chloroform. The reaction mixture was allowed to stand for 12 h and then fractionated to obtain 1.8 g (38%) of compound **VIIa**, bp 75°C (0.1 mm Hg). $^{31}P$ NMR spectrum, $\delta_P$, ppm: 36.34. Found, %: Br 34.44; Cl 15.31; P 12.48. $C_3H_6BrCl_2OP$. Calculated, %: Br 33.97; Cl 15.07; P 13.16.

**Phenyl (2-bromoethyl)phosphonochloridate (VIIb).** A mixture of 4.5 g of dichloride **IVa** and 1.9 g of phenol was heated for 15 h at 160°C and then fractionated to obtain 2.2 g (40%) of compound **VIIb**, bp 132–135°C (0.1 mm Hg). $^{31}P$ NMR spectrum, $\delta_P$, ppm: 33.54. Found Cl, %: 12.08. $C_8H_9BrClO_2P$. Calculated Cl, %: 12.58.

**Ethyl (3-bromopropyl)phosphonochloridate (VIIc).** A solution of 1.5 g of ethanol and 3.03 g of triethylamine in 10 ml of diethyl ether was added to a solution of 7.2 g of dichloride **IVb** in 30 ml of diethyl ether at –30°C. After 20 h triethylamine hydrochloride was separated, the solvent was removed, and the residue was fractionated in a vacuum to obtain 2.4 g (32%) of compound **VIIc**, bp 104°C (0.08 mm Hg),

$n_D^{20}$ 1.4872. $^{31}$P NMR spectrum, $\delta_P$, ppm: 42.22. Found P, %: 12.80. $C_5H_{11}BrClO_2P$. Calculated P, %: 12.42.

**Diethyl (3-bromopropyl)phosphonate (VIII).** Phosphonic dichloride **IVb**, 3.6 g, was added to a mixture of 1.4 g of ethanol and 3.0 g of triethylamine in 30 ml of ether. After 2 h triethylamine hydrochloride was separated, the solvent was removed, and the residue was fractionated in a vacuum to obtain 1.7 g (44%) of phosphonate **VIII**, bp 87°C (0.06 mm Hg), $n_D^{20}$ 1.4632. $^{31}$P NMR spectrum, $\delta_P$, ppm: 30.22. Found, %: Br 31.36; P 12.24. $C_7H_{16}BrO_3P$. Calculated, %: Br 30.80; P 11.97.

**Ethyl trimethylsilyl (2-bromoethyl)phosphonate (Xa).** Silylamide **IX**, 6.0 g, was slowly added to 7.0 g of chloride **VIIa** heated to 100°C. The mixture was allowed to stand for 20 h at 20°C and fractionated in a vacuum to obtain 4.2 g (49.4 %) of compound **Xa**, bp 85°C (0.1 mm Hg), $n_D^{20}$ 1.4413. $^{31}$P NMR spectrum, $\delta_P$, ppm: 17.26. Found, %: P 10.89; Si 9.63. $C_7H_{18}BrO_3PSi$. Calculated, %: P 10.72; Si 9.68.

**Ethyl trimethylsilyl (3-bromopropyl)phosphonate (Xb).** A solution of 5 g of chloride **VIIc** and 4.0 g of silylamide **IX** in 30 ml of diethyl ether was allowed to stand for 48 h at 20°C and then fractionated in a vacuum to obtain 3.4 g (56%) of compound **Xb**, bp 92°C (0.06 mm Hg). $^{31}$P NMR spectrum, $\delta_P$, ppm: 21.13. Found P, %: 8.87. $C_8H_{20}BrO_3$· PSi. Calculated P, %: 9.12.

**Ethyl (2-bromoethyl)phosphonoisothiocyanatidate (XI).** A mixture of 5.6 g of chloride **VIIa** and 2.3 g of potassium thiocyanate was allowed to stand for 24 h at 20°C. Potassium chloride was filtered off, the solvent was removed, and the residue was fractionated in a vacuum to obtain 1.3 g (21%) of compound **XI**, bp 119–121°C (0.1 mm Hg). $^{31}$P NMR spectrum, $\delta_P$, ppm: 12.82. Found, %: Br 31.36; P 11.77. $C_5H_9BrNO_2PS$. Calculated, %: Br 31.00; P 12.01.

**Reaction of isothiocyanate XI with dialkylamines.** *a*. Excess dimethylamine was passed through a benzene solution of 2.58 g of phosphonoisothiocyanatidate **XI**, and the reaction mixture was allowed to stand for 24 h at 20°C. Dimethylamine hydrobromide, 0.89 g (71%), was separated [25], mp 140–142°C. Found, %: C 19.89; H 7.12; Br 63.68; N 11.84. $C_2H_8BrN$. Calculated, %: C 19.04; H 6.34; Br 63.49; N 11.11. The $^{31}$P NMR spectrum of the residue showed two signals at $\delta_P$ 18.89 and 29.69 ppm.

*b*. The reaction of 2.58 g of phosphonoisothiocyanatidate **XI** and 0.73 g of diethylamine gave 1.02 g (66%) of diethylamine hydrobromide, mp 218–220°C [25]. Found, %: Br 51.88; N 8.81. $C_4H_{12}BrN$. Calculated, %: Br 51.95; N 9.09. $^{31}$P NMR spectrum, $\delta_P$, ppm: 18.33, 26.29.

**(2-Chloropropyl)phosphonic dichloride (XVII).** Phosphorus pentachloride, 49.5 g, was added with stirring to 37 g of compound **XVI** heated to 140°C, in the presence of a catalytic amount (0.3 g) of CuCl. The reaction mixture was heated for 3 h at 140°C, volatile compounds were removed, and the residue was fractionated in a vacuum to obtain 10 g (43%) of compound **XVII**, bp 70–74°C (0.06 mm Hg). $^{31}$P NMR spectrum, $\delta_P$, ppm: 44.22. Found, %: Cl 54.12; P 15.54. $C_3H_6Cl_3OP$. Calculated, %: Cl 54.47; P 15.86.

**Phenyl (2-chloropropyl)phosphonochloridate (XVIII).** A mixture of 7.7 g of dichloride **XVII** and 3.7 g of phenol was heated for 18 h at 160°C. Fractionation gave 4 g (40%) of compound **XVIII**, bp 105–110°C (0.06 mm Hg). $^{31}$P NMR spectrum, $\delta_P$, ppm: 32.07, 33.08. Found, %: Cl 28.04; P 12.90. $C_9H_{11}Cl_2O_2P$. Calculated, %: Cl 28.06; P 12.25.

**2-(Dimethylamino)-6-methyl-4-oxo-4-phenoxy-5,6-dihydro-1,3,4λ$^5$-thiazaphosphinine (XXI).** A mixture of chloride **XVIII** and 0.77 g of potassium thiocyanate in 5 ml of anhydrous acetonitrile was heated for 10 h at 80°C. Potassium chloride, 0.58 g (100%), was separated from the solution of compound **XIX** ($\delta_P$ 8.03, 9.86 ppm) that formed, acetonitrile was removed, the residue was dissolved in 5 ml of anhydrous benzene, and excess dimethylamine was passed through the solution. After 24 h compound **XXI** was filtered off, thrice recrystallized from benzene, and dried in a vacuum. Yield 1.2 g (53%), mp 200–202°C. IR spectrum (KBr), $\nu$, cm$^{-1}$: 1580 (C=N), 1250 (P=O). $^1$H NMR spectrum ($CDCl_3$–$C_6D_6$), $\delta$, ppm ($J$, Hz): 1.36 d.d (3H, $CH_3C$, $^3J_{HCCH}$ 6.8, $^4J_{HCCP}$ 2.7), 1.71 m and 2.28 m (2H, $H^A$, $H^B$, $CH_2$), 3.07 br.s (3H, $CH_3N$), 3.60 m (1H, CHS), 7.0–7.3 m (5H, $C_6H_5$). $^{31}$P NMR spectrum (DMSO-$d_6$), $\delta_P$, ppm: 15.03. Found, %: C 51.15; H 6.00; N 10.13; P 10.86; S 10.86. $C_{12}H_{17}N_2O_2PS$. Calculated, %: C 50.70; H 5.98; N 9.85; P 10.91; S 11.26.

**Ethylenebis(trimethylsilyl phosphonate) (XXII).** A mixture of 9.4 g of dibromoethane and 29.8 g of silyl phosphite **II** was heated for 2 h at 160°C, volatile products were removed, and the residue was fractionated in a vacuum to obtain 12.5 g (52%) of compound **XXII**, bp 147–151°C (0.08 mm Hg), solidifies at room temperature. $^{31}$P NMR spectrum, $\delta_P$, ppm: 11.55. Found, %: P 12.73; Si 23.08. $C_{14}H_{40}O_6P_2Si_4$. Calculated, %: P 12.97; Si 23.43.

**Ethylenebis(phosphonic dichloride) (XXIII).** Phosphorus pentachloride, 20.9 g, was added in portions to 12 g of diphosphonate **XXII** at 120°C. Volatile compounds (chlorotrimethylsilane, $PCl_3$) were removed, and the residue was washed with ether to obtain 6.1 g (91%) of compound **XXIII**, mp 110°C [26].

**Ethylenebis(phenyl phosphonate) (XXV).** A solution of 5.28 g of diphosphonate **XXIII**, 3.76 g of phenol, and 4.0 g of triethylamine in 80 ml of methylene chloride was refluxed for 1 h. Triethylamine hydrochloride was separated, the solvent was removed, and the residue was dried in a vacuum to obtain 3.9 g (79%) of compound **XXV**, mp 152–156°C. $^{31}$P NMR spectrum, $\delta_P$, ppm: 21.89. Found P, %: 12.41. $C_{26}H_{24}O_6P_2$. Calculated P, %: 12.55 [27].

**Ethylenebis(ethyl *N*-methylphosphonamidate) (XXIX).** Excess methylamine was passed through a solution of 5.6 g of chloride **XXVII** in 30 ml of methylene chloride. After 4 h methylamine hydrochloride was separated, and the solvent was removed to obtain 4.3 g (79%) of compound **XXIX**, mp 118–121°C. $^{31}$P NMR spectrum, $\delta_P$, ppm: 35.58. Found, %: N 10.28; P 22.73. $C_8H_{16}N_2O_4P_2$. Calculated, %: N 10.29; P 22.79.

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