Russian Journal of General Chemistry, Vol. 73, No. 2, 2003, pp. 197–201. Translated from Zhurnal Obshchei Khimii, Vol. 73, No. 2, 2003, pp. 216–221.

New Methods of Synthesis of Phosphoryl and Thiophosphoryl Halides from Organylchlorophosphonium Hexachlorophosphorates

V. G. Rozinov, M. Yu. Dmitrichenko, and R. G. Sultangareev

Institute of Petrochemical and Coal Chemical Synthesis, Irkutsk State University, Irkutsk, Russia
Favorskii Irkutsk Institute of Chemistry, Siberian Division, Russian Academy of Sciences, Irkutsk, Russia

Received May 8, 2001

Abstract — A new method of synthesis of phosphonic and thiophosphonic chlorides is offered, based on treatment of acyclic and cyclic organylchlorophosphonium hexachlorophosphorates with dimethylformamide, dimethylacetamide, or dimethylthioacetamide. The method involves no formation of phosphoryl chloride and thiophosphoryl chloride, which facilitates isolation of the target products. The reactions of alkenylchlorophosphonium hexachlorophosphorates with amides and thioamides results in incorporation of the PCl_6^- anion into an insoluble chloroiminium salt which is easily separated by filtration. Removal of the solvent leaves almost pure phosphonic or thiophosphonic chlorides.

Phosphonic and phosphinic chlorides and their thio analogs are convenient intermediates in organophosphorus synthesis. The most common method of formation of a phosphoryl group in organic phosphorus acid chlorides is treatment of organyl-phosphonium tetrachlorophosphorates with various oxygen-containing inorganic and organic compounds [1]. Crystalline organylchlorophosphonium hexachlorophosphorates **I** formed by reactions of phosphorus pentachloride with organic nucleophiles [2] are easily separated from soluble phosphorylation and chlorination products, due to which no further purification of the target products is usually needed. However, one cannot always gain benefit from the preparative advantages of complex salts **I**, because known methods of their conversion to organic phosphorus acid chlorides involve formation of phosphoryl chloride from the PCl_6^- anion of salts **I**. Removal of phosphoryl chloride, a very reactive compound, often leads, even in a vacuum, to contamination and partial tarring of the target chlorides, especially unsaturated.

Previously we showed that *N*-alkyl-substituted tertiary amides easily react with phosphorus pentachloride to give crystalline iminium salts containing a PCl_6^- anion [3, 4]:

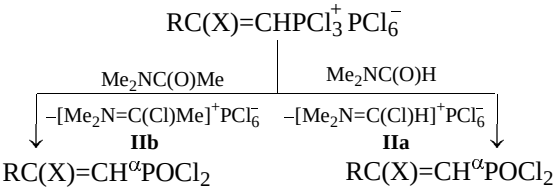
$$Alk_2NCOR + 2PCl_5 \longrightarrow [Alk_2N{=}\underset{\displaystyle Cl}{\underset{|}{C}}{-}R]^+PCl_6^- + POCl_3.$$

The PCl_6^- anion forms more stable crystalline complexes with tetraorganylammonium cations than with the organylchlorophosphonium cations of compounds **I**, which allowed us to develop a method for isolation of organyltetrachlorophosphoranes [5]:

$$R{-}PCl_3^+PCl_6^- \xrightarrow{R'_4N^+Cl^-} R{-}PCl_4 + R'_4N^+PCl_6^- .$$

It was established that in iminium salts the PCl_6^- anion forms with the organyliminium cation strong complexes insoluble in nonpolar and low-polarity solvents. With this in mind, we offer a new method for preparing unsaturated phosphonic chlorides by treatment of complex salts **I** with dimethylformamide or dimethylacetamide in benzene or chloroform. This method permits to avoid formation of phosphoryl chloride in the reaction mixture, which considerably facilitates preparative isolation of phosphonic chlorides.

A solution containing an unsaturated phosphonic chloride is readily separated from salt **IIa** or **IIb** by filtration. After removal of a volatile solvent, the phosphonic chloride is obtained in high yield.



VIII, IX, XI **V–VII, X**

R = Ph, X = H^β (**V**); R = $PhCH^\gamma{=}CH^\beta$, X = Cl (**VI**); R = EtO, X = H^β (**VII**); R = MeS, X = H^β (**VIII**); R = PrO, X = H^β (**IX**); R = PhO, X = H^β (**X**); R = Ph_2N, X = Cl (**XI**).

1070-3632/03/7302-0197 $25.00

Studying phosphorylation of ureids and thioureids with phosphorus pentachloride we found that thionic sulfur is more easily cleaved by phosphorus chlorides than carbonyl oxygen [6, 7]. This leads to different phosphorylation directions.

$$NH_2CONHCOMe \xrightarrow[-HCl,\ -POCl_3]{PCl_5} PCl_3{=}N{-}C(Cl){=}N{-}C(Cl){=}CHPCl_3^+PCl_6^-,$$

$$NH_2CSNHCOMe \xrightarrow[-HCl,\ -PSCl_3]{PCl_5} POCl_2{-}N{=}C(Cl){-}N{=}C(Cl)Me.$$

Tertiary thioamides have never been reacted with phosphorus pentachloride. We found that dimethylthioacetamide (**III**) reacts with PCl_5 like tertiary *N*,*N*-dialkylamides. That means that here, too, thionic sulfur is readily cleaved by phosphorus chloride.

$$Me_2NC(S)Me + 2PCl_5 \longrightarrow [Me_2N{=}\underset{\displaystyle Cl}{C}{-}Me]^+PCl_6^- + PSCl_3.$$

III **IV**

Hence, in the case of thio analogs of tertiary amides, the PCl_6 anion is incorporated in a benzene-insoluble iminium salt **IV**. It was expedient to extend this reaction on various alkenylphosphonium hexachlorophosphorates, aiming at preparing alkenylthiophosphonic chlorides.

Alkenylthiophosphonic chlorides are usually prepared by treatment of complex salts **I** with hydrogen, metal, or phosphorus sulfides [1]. Each of these methods has drawbacks. Hydrogen sulfide is toxic and resures thorough drying; moreover, the hydrogen chloride formed in the course of the reaction adds by the double bond of alkenylthiophosphonates. In the case of metal sulfides, the reagent, as well as substrate **I**, are insoluble in the reaction mixture, which complicates the reaction. Phosphorus sulfides are toxic and hardly available. Besides, all the above-described methods have a common drawback. These reactions involve formation of thiophosphoryl chloride from the PCl_6^- anion. The necessity of removal of $PSCl_3$ from the reaction mixture makes alkenylthiophosphonic chlorides even more difficult to isolate than alkenylphosphonic chlorides contaminated by phosphoryl chloride.

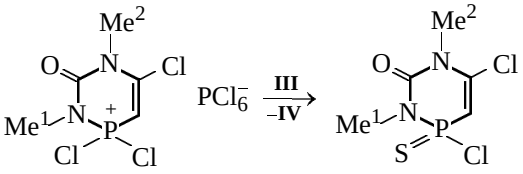
Using dimethylthioacetamide **III** permits to avoid the above-mentioned complications in the synthesis of alkenylthiophosphonic chlorides from compounds **I**. The reaction of acyclic and cyclic alkenylchlorophosphonium hexachlorophosphorates with compound **III** involves incorporation of the PCl_6 anion into an insoluble iminium salt **IV** which is easily separated by filtration. After removal of the solvent from the filtrate, an almost pure thiophosphonic chloride remain.

$$RC(X){=}CHPCl_3^+PCl_6^- \xrightarrow[-\mathbf{IV}]{\mathbf{III}} RC(X){=}CH^{\alpha}PSCl_2,$$

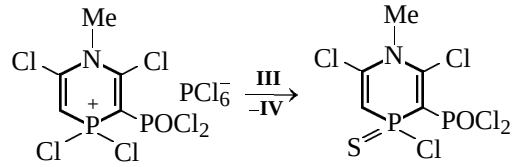
R = Ph, X = H^{β} (**XII**); R = MeS, X = H^{β} (**XIII**); R = PhO, X = H^{β} (**XIV**); R = Ph_2N, X = Cl (**XV**).

Both salt **IV** and thiophosphonic chlorides have a brown color, evidently because of the formation of small amounts of sulfur chlorides that produce darkening of the reaction mixture [8]. This effect is also characteristic of the other methods of conversion of hexachlorophosphorates **I** to thiophosphonates. It has almost no effect on the yield of the target products.

The developed method allowed preparation of P=S-containing diaza- and azaphosphinines **XVII, XIX** from cyclic organylchlorophosphonium hexachlorophosphorates **XVI, XVIII** we obtained previously [9, 10].



XVI **XVII**



XVIII **XIX**

Hexachlorophosphorate **XVIII** was used in the synthetic procedures as coarse crystals. This circumstance together with the fact that the reactivity of the dichlorophosphonium group of the heterocycle is decreased by conjugation stipulate a longer reaction time in this case compared with the other syntheses of thiophosphonic chlorides. Decreased activity of the dichlorophosphonium group toward sulfur dioxide we earlier observed with 1,4-dihydro1,4-diazaphosphinines and 1,4-oxa- and 1,4-thiaphosphininonium hexachlorophosphorates [12, 14]. Hence, dimethylthioacetamide **III** is a promising reagent for introduction of a thiocarbonyl group into organylchlorophosphoranes and chlorophosphonium hexachlorophosphorates.

The structure of the synthesized alkenylphosphonates and thiophosphonates was established by NMR

Physicochemical characteristics and ^{31}P NMR spectral parameters of phosphonic and thiophosphonic chlorides

Comp. no.	Yield, %	bp, °C (*p*, mm)	mp, °C	^{31}P NMR spectrum, δ_P, ppm	Calculated, %					
					C	H	Cl	N	P	S
V	90	183 (18)	71–72	31.9	43.44	3.17	32.13		14.03	
VI	99		94–95	24.3	42.64	2.84	37.83		11.01	
VII	69	60–61 (0.1)		34.0	25.40	3.70	37.57		16.40	
VIII	63	93–96 (3)		30.3	18.85	2.62	37.17		16.23	16.75
IX	88	94–95 (2)		33.7	29.56	4.43	34.98		15.27	
X	85	105 (0.1)		31.2	40.51	2.95	29.96		13.08	
XI	88		86–88	28.8	48.48	3.17	30.74	4.04	8.95	
XII	88	116–117 (1)		71.8	40.51	2.95	29.96		13.08	13.50
XIII	60	90–97 (1)		69.2	17.39	2.42	34.30		14.98	30.92
XIV	52	159–161 (4)		68.3	37.94	2.77	28.06		12.25	12.65
XV	72		104–106	62.7	46.34	3.03	29.38	3.86	8.55	8.83
XVII	42		120–122	53.2	24.49	2.86	28.98	11.43	12.65	13.06
XIX	20		195–197	40.5 31.8	16.42	1.09	48.56	3.83	16.96	8.76

Comp. no.	Formula	Found, %					
		C	J	Cl	N	P	S
V	$C_8H_7Cl_2OP$	43.30	3.01	31.9		14.06	
VI	$C_{10}H_8Cl_3OP$	42.65	2.82	38.05		10.90	
VII	$C_4H_7Cl_2O_2P$	25.54	3.78	37.02		15.80	
VIII	$C_3H_5Cl_2OPS$	19.50	2.94	37.51		15.54	16.88
IX	$C_5H_9Cl_2O_2P$	30.10	4.41	34.85		14.50	
X	$C_8H_7Cl_2O_2P$	40.47	2.93	29.25		12.65	
XI	$C_{14}H_{11}Cl_3NOP$	48.23	3.13	31.04	4.21	8.44	
XII	$C_8H_7Cl_2PS$	40.35	3.00	29.45		13.52	14.15
XIII	$C_3H_5Cl_2PS_2$	18.09	3.08	34.97		14.28	31.43
XIV	$C_8H_7CI_2OPS$	42.04	1.94	29.39		12.71	11.66
XV	$C_{14}H_{11}Cl_3NPS$	46.11	3.09	25.55	3.95	8.40	8.72
XVII	$C_5H_7Cl_2N_2OPS$	24.51	2.98	28.84	11.44	11.42	12.39
XIX	$C_5H_4Cl_5NOP_2S$	16.11	1.23	49.06	3.59	16.87	8.43

spectroscopy. The physicochemical characteristics and ^{31}P NMR spectral parameters are listed in the table.

The ^{31}P NMR spectrum of iminium salt **IV**, independent of the nature of hexachlorophosphorate **I** reacted with amide **III**, contains only one singlet at δ_P –295.7 ppm characteristic of the PCl_6^- anion (nitrobenzene soluton). The ^1H NMR spectrum contains three singlets at δ_P 2.98 [MeC(Cl)=], 3.80, and 3.87 (Me_2N) ppm. The nonequivalence of the alkyl groups in the dialkylamino fragment reflects a high degree of double bonding of the C=N bond. This effect we also established for other iminium salts [3]. The structure of iminium salts **II** has been thoroughly studied in [4].

EXPERIMENTAL

The ^1H and ^{31}P NMR spectra were registered on a Bruker DPX-400 spectrometer (400 and 161.98 MHz, respectively) for $CDCl_3$ solutions.

Styrylphosphonic dichloride (V). To a dispersion of 8.6 g of trichloro(styryl)phosphonium hexachlorophosphorate in 100 ml of benzene, a solution of 1.3 g of dimethylformamide in 5 ml of benzene was added in portions with stirring. Slight heat release was observed, and the crystalline became denser and heavier. After 1 h, the crystals of iminium salt **IIa** were filtered off, the benzene was removed, and the product was distilled in a vacuum. Yield 3.53 g. ^1H NMR

spectrum, δ, ppm: 6.69 d.d (1H, H_α, $^3J_{HH}$ 17.0, $^2J_{HP}$ 34.5 Hz), 7.69 d.d (1H, H_β, $^3J_{HH}$ 17.0, $^3J_{PH}$ 31.0 Hz), 7.4–7.6 m (5H, Ph).

(2-Chloro-4-phenyl-1,3-butadienyl)phosphonic dichloride (VI). To a dispesion of 33.4 g of trichloro-(2-chloro-4-phenyl-1,3-butadienyl)phosphonium hexachlorophosphorate prepared from 1-phenylbut-1-en-3-one and phosphorus pentachloride, a solution of 4.5 g of dimethylformamide in 5 ml of benzene was added dropwise with stirring. On the next day the precipitate of iminium salt **IIa** was filtered off, and the benzene was removed from the filtrate in a water-jet-pump vacuum. Crystallization of the residue from CCl_4 gave phosphonic chloride **VI** as white crystals. Yield 17.3 g. ^{1}H NMR spectrum, δ, ppm: 6.21 d (1H, H_α, $^2J_{PH}$ 24.6 Hz); 7.98 d.d (1H, H_γ, $^3J_{HH}$ 15.1 Hz, $^4J_{PH}$ 2.4 Hz), 7.2–7.5 m (6H, H_β, Ph).

(β-Ethoxyvinyl)phosphonic dichloride (VII). To a dispersion of 18.6 g of trichloro(β-ethoxyvinyl)-phosphonium hexachlorophosphorate in 30 ml of benzene, 3 g of dimethylformamide was added with stirring. After 4-h stirring, the thickened precipitate of salt **IIa** was filtered, the benzene was removed in a water-jet-pump vacuum, and compound **VII** was distilled in a vacuum. Yield 7.9 g. ^{1}H NMR spectrum, δ, ppm: 1.08 t (3H, Me, $^3J_{HH}$ 8.1 Hz), 3.86 q (2H, CH_2, $^3J_{HH}$ 8.1 Hz), 5.43 d.d (1H, H_α, $^3J_{HH}$ 13.4 Hz, $^2J_{PH}$ 23.2 Hz), 7.38 d.d (1H, H_β, $^3J_{HH}$ 13.4 Hz, $^3J_{PH}$ 13.9 Hz).

(β-Methylthiovinyl)phosphonic dichloride (VIII). To a dispersion of 3.05 g of trichloro(β-methylthiovinyl)phosphonium hexachlorophosphorate in 15 ml of benzene, a solution of 0.6 g of dimethylacetamide in 5 ml of benzene was added with stirring. The precipitate quickly thickened, and the reaction mixture became yellow. After 4 h, iminium salt **IIb** was filtered off. Yield 2.1 g (92%). Distillation of the residue gave 0.8 g of compound **VIII.** ^{1}H NMR spectrum, δ, ppm: 2.41 s (3H, Me), 5.89 d.d (1H, H_α, $^3J_{HH}$ 15.9 Hz, $^2J_{PH}$ 31.1 Hz), 7.78 d.d (1H, H_β, $^3J_{HH}$ 15.9 Hz, $^3J_{PH}$ 28.1 Hz).

(β-Propoxyvinyl)phosphonic dichloride. To a dispersion of 6.5 g of trichloro(β-propoxyvinyl)phosphonium hexachlorophosphorate in 50 ml of benzene, 1.2 g of dimethylacetamide was added in portions with vigorous stirring. The precipitate quickly thickened, which made the mixture more difficult to stir. After 2 h, the precipitate became greenish-yellow. A day later, iminium salt **IIb** was filtered off. Yield 4.2 g. Distillation of the filtrate gave 2.5 g of compound **X.** ^{1}H NMR spectrum, δ, ppm: 0.95 t (3H, Me, $^3J_{HH}$ 7.2 Hz), 1.72 m (2H, CH_2–Me, $^3J_{HH}$ 7.2 Hz, $^3J_{HH}$ 6.4 Hz), 3.86 t (2H, CH_2O, $^3J_{HH}$ 6.4 Hz), 5.28 d.d (1H, H_α, $^3J_{HH}$ 13.2 Hz, $^2J_{PH}$ 23.2 Hz), 7.36 d.d (1H, H_β, $^3J_{HH}$ 13.2 Hz, $^3J_{PH}$ 13.9 Hz).

(β-Phenoxyvinyl)phosphonic dichloride (X) was prepared analogously from 14.5 g of trichloro(β-phenoxyvinyl)phosphonium hexachlorophosphorate and 2.1 g of dimethylformamide. Yield 5.8 g. ^{1}H NMR spectrum, δ, ppm: 5.79 d.d (1H, H_α, $^3J_{HH}$ 12.9 Hz, $^2J_{PH}$ 23.2 Hz), 7.76 d.d (1H, H_β, $^3J_{PH}$ 12.9, $^3J_{PH}$ 13.2 Hz), 7.2–7.6 m (5H, Ph).

[2-Chloro-2-(*N*,*N*-diphenylamino)ethenyl]phosphonic dichloride (XI). A solution of 0.13 g of dimethylacetamide in 5 ml of benzene was quickly added with stirring to a dispersion of 0.9 g of trichloro[2-chloro-2-(*N*,*N*-diphenylamino)ethenyl]phosphonium hexachlorophosphorate. Several minutes later, the precipitate turned yellow and pasty. After 1 h, iminium salt **IIb** was filtered off. Yield 0.48 g (92%). The solvent was removed from the filtrate, and the residue was recrystallized from a 10 : 1 hexane–benzene mixture to obtain 0.45 g of compound **XI** as white crystals. ^{1}H NMR spectrum, δ, ppm: 5.24 d (1H, H_α, $^2J_{PH}$ 20.8 Hz), 7.2–7.4 m (10H, Ph).

Styrylthiophosphonic dichloride (XII). A solution of 0.3 g of dimethylthioacetamide in 5 ml of benzene was added in portions with stirring to a dispersion of trichloro(styryl)phosphonium hexachlorophosphorate. In the course of the reaction, the crystalline precipitate thickened and acquired a brown color. After 5 h, the crystals of iminium salt **IV** were filtered off, and the benzene was removed in a water-jet-pump vacuum. The residue was distilled in a vacuum to give 0.6 g of stytylthiophosphonic dichloride. ^{1}H NMR spectrum, δ, ppm: 6.96 d.d (1H, H_α, $^3J_{HH}$ 16.4 Hz, $^2J_{HH}$ 29.1 Hz), 7.66 d.d (1H, H_β, $^3J_{HH}$ 16.4 Hz, $^3J_{PH}$ 33.1 Hz), 7.4–7.5 m (5H, Ph).

(β-Methylthiovinyl)thiophosphonic dichloride (XIII). A solution of 0.5 g of dimethylthioacetamide in 5 ml of benzene was added with stirring to a dispersion of 2.2 g of trichloro(β-methylthiovinyl)phosphonium hexachlorophosphorate in 15 ml of benzene. The dispersion practically immediately thickened and darkened. After 24 h, iminium salt **IV** was filtered off. Yield 10 g (58%). The solvent was removed from the filtrate, and the residue was treated with hot hexane. Distillation of the extract gave 0.6 g of compound **XIII** as a yellow oil. ^{1}H NMR spectrum, δ, ppm: 2.44 s (3H, Me), 6.17 d.d (1H, H_α, $^3J_{HH}$ 15.4 Hz, $^2J_{PH}$ 25.6 Hz), 7.85 d.d, H_β, $^3J_{HH}$ 15.4 Hz, $^2J_{PH}$ 31.1 Hz).

(β-Phenoxyvinyl)thiophosphonic dichloride (XIV). A solution of 0.47 g of dimethylthioacetamide in 5 ml of benzene was added to a dispersion of 2.3 g

of trichloro(β-phenoxyvinyl)phosphonium hexachlorophosphorate in 15 ml of benzene. A day after, the precipitate got much denser and darker. After 24 h, the precipitate of iminium salt **IV** was filtered off, and the solvent was removed from the filtrate. The brown residue was treated with boiling hexane. It was removed, and the residue was distilled to obtain 0.6 g of compound **XIV** as a slightly yellow oil. ^{1}H NMR spectrum, ppm: 6.17 d.d (1H, H_α, $^3J_{HH}$ 12.5 Hz, $^2J_{PH}$ 16.8 Hz), 7.86 d.d (1H, H_β, $^3J_{HH}$ 12.5 Hz, $^3J_{PH}$ 14.8 Hz), 7.2–7.6 m (5H, Ph).

[2-Chloro-2-(*N*,*N*-diphenylamino)ethenyl]thiophosphonic dichloride (XV). A solution of 0.18 g of dimethylthioacetamide in 5 ml of benzene was added dropwise with stirring and cooling with ice water to a dispersion of 1.05 g of trichloro[2-chloro-2-(*N*,*N*-diphenylamino)ethenyl]phosphonium hexachlorophosphorate. A day after, the precipitate of iminium salt **IV** was filtered off, and the solvent was removed from the filtrate. The brown residue was treated with boiling hexane. After cooling, 0.45 g of compound **XV** precipitated as coarse yellow plates. ^{1}H NMR spectrum, δ, ppm: 5.61 d (1H, H_α, $^2J_{PH}$, $^2J_{PH}$ 23.4 Hz), 7.2–7.4 m (10H, Ph).

2,4-Dichloro-1,5-dimethyl-6-oxo-1,2,5,6-tetrahydro-1,5,2λ^5-diazaphosphinine 2-sulfide (XVII) was obtained in a similar way from 2.4 g of complex salt **XVI** and 0.5 g of dimethylthioacetamide. After 3 days, the light brown precipitate of iminium salt **IV** was filtered off, the benzene was removed, and the residue was recrystallized from a 10 : 1 hexane–benzene mixture to obtain 0.5 g of compound **XVII** as light yellowish prisms. ^{1}H NMR spectrum, δ, ppm: 3.34 d (3H, Me^1, $^3J_{PH}$ 10.2 Hz), 3.52 s (3H, Me_2), 5.87 d (1H, CH=, $^2J_{PH}$ 15.8 Hz).

1-Methyl-2,4,6-trichloro-3-(dichlorophosphoryl)-1,4-dihydro-1,4λ^5-azaphosphinine 4-sulfide (XIX). A solution of 0.07 g of dimethylthioacetamide in 2 ml of benzene was added in portions with stirring to a dispersion of 0.4 g of 2,4,4,6-tetrachloro-1-methyl-3-(dichlorophosphoryl)-1,4-dihydro-1,4λ^5-azaphosphinonium hexachlorophosphorate (**XVIII**) in 8 ml of benzene. Within 4 h, the reaction mixture slowly darkened. A day after, the brownish precipitate of iminium salt **IV** was filtered off. Yield 0.25 g. The solvent was removed from the filtrate, and the residue was recrystallized from a 10 : 1 hexane–benzene mixture to obtain 0.05 g of compound **XIX** as yellow crystals. Evaporation of the mother liquor gave 0.05 g of crude compound **XIX** as a brownish powder. ^{1}H NMR spectrum, δ, ppm: 4.06 s (3H, MeN), 5.93 d (1H, CH=, $^4J_{PH}$ 9.1 Hz). The P(S)Cl group appears at δ_P 40.5 ppm ($^2J_{PP}$ 46.6 Hz).

REFERENCES

1. Kormachev, V.V. and Fedoseev, M.S., *Preparativnaya khimiya fosfora* (Preparative Phosphorus Chemistry), Perm, 1992.
2. Fridland, S.V. and Malkov, Yu.K., *Reakts. Metody Issled. Org. Soedin.*, 1986, pp. 106–149.
3. Pensionerova, G.A., Rozinov, V.G., Kalabina, A.V., Donskikh, V.I., Ratovskii, G.V., and Dmitrichenko, M.Yu., *Zh. Obshch. Khim.*, 1982, vol. 52, no. 11, pp. 2491–2499.
4. Rozinov, V.G., Dmitrichenko, M.Yu., Kolbina, V.E., and Dolgushin, G.V., *Zh. Obshch. Khim.*, 1999, vol. 69, no. 9, pp. 1430–1438.
5. Rozinov, V.G., Rybkina, V.V., Kolbina, V.E., Pensionerova, G.A., and Donskikh, V.I., *Zh. Obshch. Khim.*, 1982, vol. 52, no. 9, pp. 1994–1997.
6. Dmitrichenko, M.Yu., Donskikh, V.I., Rozinov, V.G., Dolgushin, G.V., Ratovskii, G.V., Efremov, V.G., and Rybkina, V.V., *Zh. Obshch. Khim.*, 1989, vol. 59, no. 1, pp. 227–228.
7. Rozinov, V.G., Dmitrichenko, M.Yu., Dolgushin, G.V., and Donskikh, V.I., *Zh. Obshch. Khim.*, 1990, vol. 60, no. 3, pp. 706–707.
8. Buehler, C.A. and Pearson, D.E., *Survey of Organic Syntheses*, New York: Wiley, 1970, vol. 1. Translated under the title *Sintezy organicheskikh soedinenii*, 1973, vol. 1, p. 349.
9. Dmitrichenko, M.Yu., Rozinov, V.G., Donskikh, V.I., Ratovskii, G.V., Sergienko, L.M., Dolgushin, G.V., and Valeev, R.B., *Zh. Obshch. Khim.*, 1988, vol. 58, no. 10, pp. 2252–2261.
10. Dmitrichenko, M.Yu., Rozinov, V.G., Donskikh, V.I., Doilgushin, G.V., Feshin, V.P., and Kaslabina, A.V., *Zh. Obshch. Khim.*, 1987, vol. 57, no. 8, pp. 1912–1913.
11. Rozinov, V.G., Izhboldina, L.P., Donskikh, V.I., Ratovskii, G.V., Sergienko, L.M., Dolgushin, G.V., and Dmitrichenko, M.Yu., *Zh. Obshch. Khim.*, 1989, vol. 59, no. 5, pp. 997–1018.
12. Rozinov, V.G., Kolbina, V.E., and Donskikh, V.I., *Zh. Obshch. Khim.*, 1987, vol. 57, no. 4, pp. 954–956.
13. Rozinov, V.G., Kolbina, V.E., Donskikh, V.I., Ratovskii, G.V., Dolgushin, G.V., Gavrilova, G.M., Gostevskaya, V.I., and Amosova, S.V., *Zh. Obshch. Khim.*, 1990, vol. 60, no. 8, pp. 1780–1789.