

To the 85th Anniversary of birthday of late Yu.G. Gololobov

Reactions of Phosphorus Acids with *N*-*tert*-Butyl-2,2-dichloropropanimine

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Received June 27, 2015

Abstract—Reaction of *N*-*tert*-butyl-2,2-dichloropropanimine with dialkylphosphorous acids led to the formation of stable adducts, the chlorine atoms of which are inactive towards subsequent transformations. The primary salt of *O,O*-dialkyldithiophosphoric acid and *N*-*tert*-butyl-2,2-dichloropropanimine, *N*-*tert*-butyl-2,2-dichloropropaniminium *O,O*-dialkylthiophosphate, reacted with the second molecule of acid to form bis-(dialkoxythiophosphoryl) disulfide and *N*-*tert*-butyl-2-chloropropaniminium chloride as the product of reduction of the C–Cl bond of the starting imine salt. The latter reacted with another acid molecule to afford *N*-*tert*-butyl-2-(dialkoxythiophosphorylthio)propaniminium chloride via reduction-substitution of the primary iminium salt.

Keywords: *N*-*tert*-butyl-2,2-dichloropropanimine, dialkylphosphorous acid, *O,O*-dialkyldithiophosphoric acid, bis(dialkoxythiophosphoryl)disulfide, *N*-*tert*-butyl-2-chloropropaniminium chloride, *N*-*tert*-butyl-2-(dialkoxythiophosphorylthio)propaniminium chloride

DOI: 10.1134/S1070363215090145

One of the better known reactions of unsubstituted with halogen *N*-alkyl(aryl)imines of aldehydes and ketones is their interaction with hydrophosphoryl compounds, particularly with dialkylphosphites, which are used for the synthesis of phosphorus-containing amines [1–4].

The phosphorus dithioacids $R^1R^2P(S)SH$ contain two distinct functions — electrophilic and nucleophilic — due to high acidity and nucleophilicity of dithiophosphoryl triad $P(S)S$ [5]. Therefore, phosphorus dithioacids are characterized by a high reactivity towards unsaturated compounds, in particular, to imines in the absence of a catalyst [1, 2, 4–6].

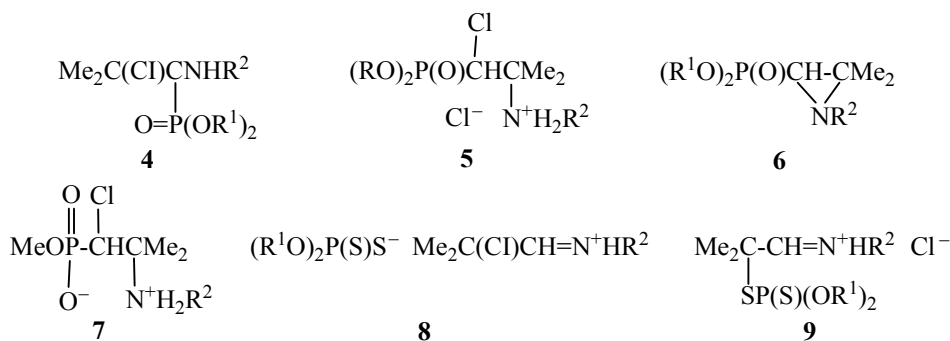
By analogy with the reactions of hydrophosphoryl compounds with imines [1, 3, 4], it was assumed that *O,O*-dialkyldithiophosphoric acid can add to the imine group of anils to form *N*-arylphenylcarbimines [6]. However, the study of this reaction involving physicochemical methods showed that the process stops at the stage of protonation of imino group to form iminium salts [7].

Among haloimines chloral imines have been first used in these reactions [8]. Since the basicity of the imine nitrogen atom is greatly reduced due to the influence of trichloromethyl group, its protonation does not occur; only adducts of the acid to the $C=N$ double bond are formed. In these adducts the chlorine atoms are inactive towards subsequent substitution processes.

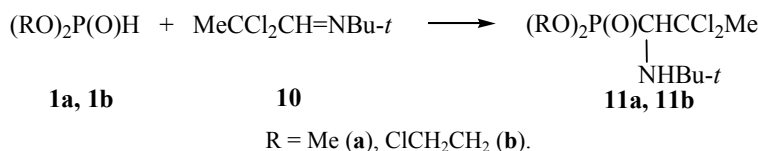
It is known that available *N*-alkyl-2-haloaldehydes [9] exhibit high reactivity due to the presence of multiple bonds like $C=N$ as well as $C-Hlg$ bond [10]. We presumed that the sole chlorine atom of the primary product of the interaction of phosphorus acids with these imines would possess high lability, in particular, they would participate in intramolecular nucleophilic substitution reactions.

Therefore we investigated the reactions of dialkylphosphorous (1) and *O,O*-dialkyldithiophosphoric (2) acids with *N*-alkyl-2-chloroaldehydes 3 $Me_2C(Cl)CH=NR$ [11–14]. It was found that due to lability of the chlorine atom in the primary addition product 4 the

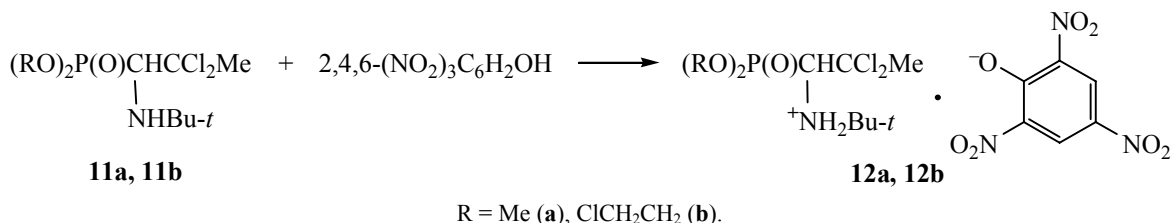
Scheme 1.



Scheme 2.



Scheme 3.



prolonged storage of the reaction mixture at room temperature led to the formation of compounds of different structure: phosphorylated chloroalkylammonium salt **5** and aziridine **6**, phosphonate betaine **7**. We also showed that the reaction of *O,O*-dialkyldithiophosphoric acids **2** with *N*-alkyl-2-methyl-2-chloropropanimines **3** proceeded in two stages: In the first stage the imine group was protonated to form an intermediate iminium salt, *N*-alkyl-2-methyl-2-chloraldiminium *O,O*-dialkyldithiophosphate **8**; in the second step the chlorine atom was replaced by dialkoxythiophosphorylthio group, and the reaction was completed to form *N*-alkyl-2-(dialkoxythiophosphorylthio)-2-methylpropaniminium chloride **9** (Scheme 1).

The reactions of acids **1** and **2** with *N-tert*-butyl-2,2-dichloropropanimine **10** has not been previously studied. We found that the reaction proceeded exothermically to give adducts **11a** and **11b** (Scheme 2).

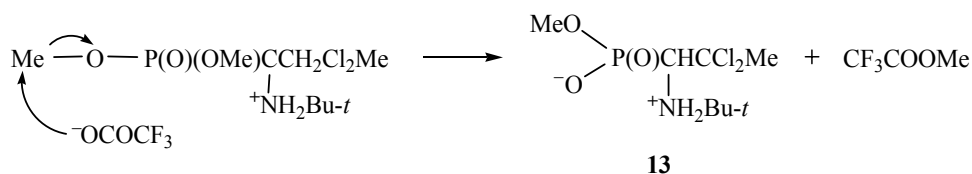
Unlike adducts **4** we previously synthesized containing the sole chlorine atom, compounds **11a** and **11b** containing dichloromethylene group were stable.

According to ^1H and ^{31}P NMR, after 15 days of keeping the reaction mixture at room temperature the conversion into other products was not detected. Composition and structure of compounds **11a** and **11b** were confirmed by elemental analysis, ^1H and ^{31}P NMR spectroscopy data as well as by converting them into the corresponding picrates **12a** and **12b** (Scheme 3).

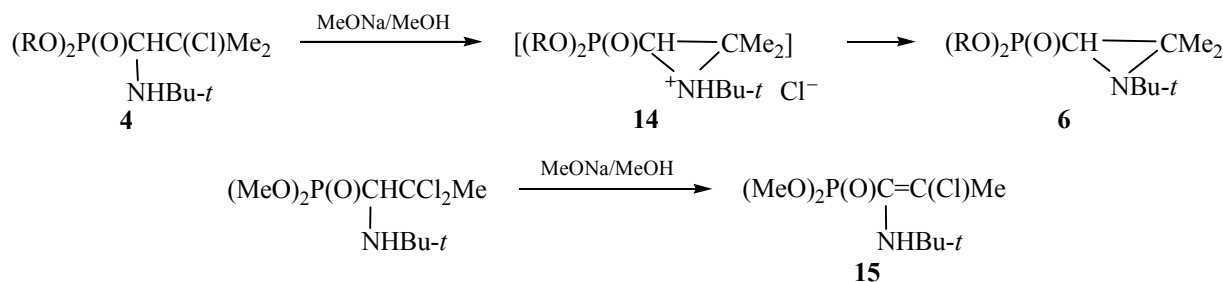
Salt **11a** was demethylated to form betaine **13** when reacting with stronger trifluoroacetic acid (Scheme 4).

It was also found that the behavior of adducts of dialkylphosphites **1** with mono- (**4**) or dichloraldimines (**9**) varied greatly with respect to the base, in particular an alcoholic solution of sodium methoxide. In the first case, labile chlorine atom underwent transformation into the phosphorylated aziridine **6** through an intermediate aziridinium salt **14**. Compound **11a** with inactive chlorine atoms cannot form an intermediate aziridinium salt and underwent dehydrochlorination to yield the corresponding enamine **15** as a mixture of two isomers (64 : 36) (Scheme 5).

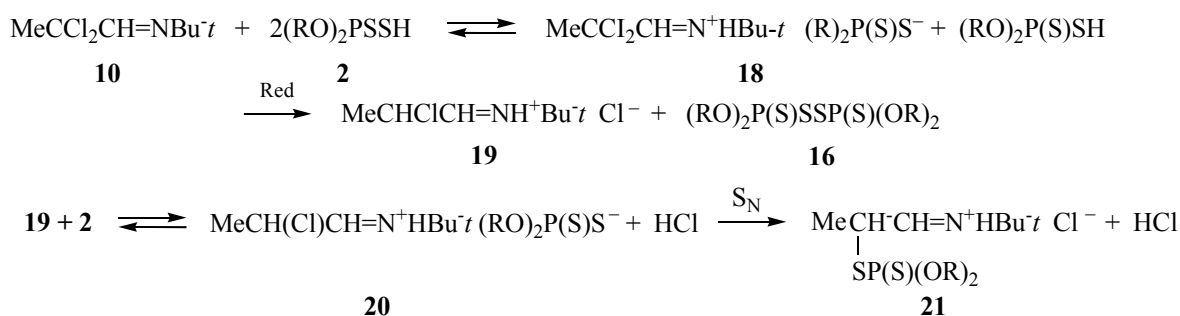
Scheme 4.



Scheme 5.

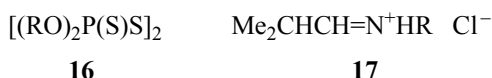


Scheme 6.



R = *i*-Pr (**a**), Et (**b**).

Recently we have discovered a new route of acid **2** reaction with chlorimine **3** when using two-fold excess of the acid: a nucleophilic substitution occurred of the chlorine atom with dialkoxythiophosphorylthio group (compound **9**) in the intermediate salt **8** as well as the reduction of iminium cation at the C–Cl bond [5]. In this case the main products were bis(dialkoxythiophosphoryl) disulfide **16** and iminium salt **17**.



We presumed that the direction of the reaction was due to the halophilic properties of the intermediate iminium salt **8** and its reduction with acid **2** excess [5]. In dichloroimine **10** the additional chlorine atom as electron acceptor should reduce the basic properties of the imine nitrogen atom by shifting the acid ↔ salt equilibrium to the left, suggesting the presence of free acid **2** in the reaction mixture and its participation in

the reduction of the primary salt **18**. In addition, the chlorine atom should increase the halophilic properties of **18**.

The main products of the reaction between compounds **2** and **10** were disulfide **16**, *N*-*tert*-butyl-2-chloropropaniminium chloride **19**, and *N*-*tert*-butyl-2-(diisopropoxythiophosphorylthio)propaniminium chloride **21** (Scheme 6).

The reaction occurred via intermediate formation of iminium salt **18**, which was involved into the reduction process with the second molecule of acid **2**. The product **19** underwent an anion exchange with acid **2** to form new dithiophosphate salt **20**. The latter was subjected to intramolecular nucleophilic substitution of the chlorine atom with dialkoxythiophosphorylthio group to afford compound **21**. When using the reagents **2** and **10** in 1 : 1 ratio, the starting imine **10** was not fully reacted since some acid was

consumed to form disulfide **16** and compound **21**. Similar reaction products were obtained when using the reagents ratio of 3 : 1. The isolated salt **19** reacted with acid **2a** to form compound **21a**.

In summary, we found that *N*-*tert*-butyl-2,2-dichloropropanimine reacted with dialkylphosphorous acids to form stable adducts with inactive chlorine atoms. Unlike adducts with the sole chlorine atom, these adducts did not participate in subsequent transformations without additional reagents. The intermediately formed *N*-*tert*-butyl-2,2-dichloropropaniminium *O,O*-dialkyldithiophosphate **18** reacted with the second molecule of acid **2** to give disulfide **16** and *N*-*tert*-butyl-2-chloropropaniminium chloride **19**, which is the iminium salt reduced at the C–Cl bond. Its interaction with another molecule of acid **2** resulted in the formation of compound **21** through substitution of the chlorine atom with dialkoxythiophosphorylthio group in salt **22**.

EXPERIMENTAL

^1H and ^{13}C NMR spectra (CDCl_3) were recorded on a Bruker AVANCE 400WB instrument operating at 400.13 and 100.61 MHz, respectively, reference TMS. ^{31}P NMR spectra were registered on a Bruker AVANCE 400WB (161.98 MHz) and Bruker MSL-400 (162 MHz) spectrometers, external reference 85% H_3PO_4 .

***O,O*-Dimethyl [1-(*tert*-butylamino)-2,2-dichloropropyl]phosphonate (11a).** A mixture of 10.02 g (0.055 mol) of *N*-*tert*-butyl-2,2-dichloropropanimine and 6.05 g (0.055 mol) of dimethylphosphorous acid was kept under argon atmosphere at room temperature for 24 h, then treated with diethyl ether. Yield 13.6 g (85%), mp 60°C (Et_2O). ^1H NMR spectrum (CDCl_3), δ , ppm (*J*, Hz): 1.29 s (9H, CMe_3), 2.30 br.s (1H, NH), 2.38 s (3H, CMe), 3.67 d (1H, PCH, $^2J_{\text{PH}}$ 20.0), 3.91 d and 3.93 d (6H, MeOP, $^3J_{\text{PH}}$ 11.0). ^{31}P NMR spectrum (CCl_4): δ_{P} 26.93 ppm. Found, %: C 36.89; H 7.02; N 4.67; P 10.53. $\text{C}_9\text{H}_{20}\text{Cl}_2\text{NO}_3\text{P}$. Calculated, %: C 37.00; H 6.90; N 4.79; P 10.60.

***O,O*-Di-(2-chloroethyl) [1-(*tert*-butylamino)-2,2-dichloropropyl]phosphonate (11b)** was prepared similarly from 5.20 g (0.025 mol) of di-(2-chloroethyl) phosphite and 5.10 g (0.028 mol) of imine **10**. Yield 8.7 g (90%). ^1H NMR spectrum (CDCl_3), δ , ppm (*J*, Hz): 1.20 s (9H, CMe_3), 2.1 br.s (1H, NH), 2.24 s (3H, CMe), 3.6 d (1H, PCH, $^2J_{\text{PH}}$ 18.0), 3.37 m (4H,

CH_2OP), 3.74 m (4H, CH_2Cl). ^{31}P NMR spectrum (CCl_4): δ_{P} 25.30 ppm. Found, %: C 33.65; H 5.46; N 3.43; P 8.03. $\text{C}_{11}\text{H}_{22}\text{Cl}_4\text{NO}_3\text{P}$. Calculated, %: C 33.96; H 5.70; N 3.61; P 7.91.

***O,O*-Dimethyl [1-(*tert*-butylamino)-2,2-dichloropropyl]phosphonate picrate (12a).** A solution of 1.14 g (0.005 mol) of picric acid in 10 mL of ethyl acetate was added in small portions to a solution of 1.46 g (0.005 mol) of compound **11a** in 15 mL of ethyl acetate. The mixture was kept at room temperature for 72 h. The resulting crystals were filtered off, recrystallized from benzene, and dried in a vacuum. Yield 21 g (81%), mp 102–103°C. ^1H NMR spectrum (acetone- d_6), δ , ppm (*J*, Hz): 1.40 s (9H, CMe_3), 2.43 s (3H, CMe), 3.92 d and 3.94 d (6H, MeOP, $^3J_{\text{PH}}$ 10.0), 4.12 d (1H, PCH, $^2J_{\text{PH}}$ 19.0), 6.04 br.s (2H, N^+H_2), 9.06 s (2H, C_6H_2). ^{31}P NMR spectrum (CHCl_3): δ_{P} 23.50 ppm. Found, %: C 34.42; H 4.57; N 10.61; P 6.01. $\text{C}_{15}\text{H}_{23}\text{Cl}_2\text{N}_4\text{O}_{10}\text{P}$. Calculated, %: C 34.56; H 4.45; N 10.75; P 5.94.

***O,O*-Di-(2-chloroethyl) [1-(*tert*-butylamino)-2,2-dichloropropyl]phosphonate picrate (12b)** was prepared similarly from 0.78 g (0.092 mol) of phosphonate **11b** and 0.46 g (0.092 mol) of picric acid. Yield 1.03 g (83%), mp 140–141°C (ethanol). ^1H NMR spectrum (acetonitrile- d), δ , ppm (*J*, Hz): 1.45 s (9H, CMe_3), 2.41 s (3H, CMe), 3.90 d (1H, PCH, $^2J_{\text{PH}}$ 19.0), 3.80 m (4H, CH_2Cl), 4.24 m (4H, CH_2OP), 7.2 br.s (2H, N^+H_2), 9.02 s (2H, C_6H_2). ^{31}P NMR spectrum (acetone- d_6): δ_{P} 21.90 ppm. Found, %: C 33.17; H 4.16; N 8.91; P 4.92. $\text{C}_{17}\text{H}_{25}\text{Cl}_4\text{N}_4\text{O}_{10}\text{P}$. Calculated, %: C 33.03; H 4.08; N 9.06; P 5.01.

***O*-Methyl [1-(*tert*-butylammonio)-2,2-dichloropropyl]phosphonate (13).** A solution of 1.14 g (0.01 mol) of trifluoroacetic acid in 10 mL of diethyl ether was added in small portions to a solution of 2.9 g (0.01 mol) of phosphonate **11a** in 20 mL of anhydrous diethyl ether. The mixture was left standing overnight and evaporated in a vacuum to yield 3.71 g (92%) of *O,O*-dimethyl [1-(*tert*-butylamino)-2,2-dichloropropyl]phosphonate trifluoroacetate as a thick syrupy liquid. ^1H NMR spectrum (acetonitrile- d), δ , ppm (*J*, Hz): 1.43 s (9H, CMe_3), 2.47 s (3H, CMe), 3.98 d and 3.96 d (6H, MeOP, $^3J_{\text{PH}}$ 11.3), 4.03 d (1H, PCH, $^2J_{\text{PH}}$ 18.7), 8.09 br.s (2H, N^+H_2). ^{31}P NMR spectrum (CHCl_3): δ_{P} 27.58 ppm. At prolonged storage of the salt obtained in diethyl ether solution crystalline *O*-methyl [1-(*tert*-butylammonio)-2,2-dichloropropyl]phosphonate **14** precipitated, mp 107–108°C. ^1H NMR spectrum

(acetone- d_6), δ , ppm (J , Hz): 1.64 s (9H, CMe₃), 2.65 s (3H, CMe), 3.72 d (3H, POME, $^3J_{PH}$ 11.2), 4.30 d (1H, PCH, $^2J_{PH}$ 14.0), 8.61 br.s (2H, N⁺H₂). ^{31}P NMR spectrum (acetone- d_6): δ_P 6.19 ppm. Found, %: C 36.29; H 6.69; N 5.65; P 11.63. C₈H₁₈Cl₂NO₃P. Calculated, %: C 36.38; H 6.87; N 5.30; P 11.73.

***O,O*-Dimethyl (1-*tert*-butyl-3,3-dimethylaziridin-2-yl)phosphonate (6a).** To a solution of 6.08 g (0.022 mol) of compound **4a** in 35 mL of methanol was added under argon 18.3 mL of 1.2 M. solution of sodium methoxide in methanol, maintaining the reaction temperature at about 3°C. The mixture was stirred for 6 h at room temperature and left overnight. NaCl was filtered off, the mother liquor was evaporated, and the residue was distilled in a vacuum. Yield 3.88 g (75%), bp 74°C (0.1 mmHg), n_D^{20} 1.4532. 1H NMR spectrum (CDCl₃), δ , ppm (J , Hz): 0.97 s (9H, CMe₃), 1.15 s (3H, MeCMe), 1.22 d (3H, MeCMe, $^4J_{PH}$ 2.3), 1.52 d (1H, PCH, $^2J_{PH}$ 22.0), 3.5 d (6H, POME, $^3J_{PH}$ 11.0). ^{31}P NMR spectrum (CHCl₃): δ_P 26.40 ppm.

***O,O*-Dimethyl [1-(*tert*-butylamino)-2-chloropropen-1-yl]phosphonate (15)** (a mixture of two isomers, 64 : 36). To 17.5 mL of 1.2 M sodium methoxide in methanol was added under argon 6.2 g (0.021 mol) of compound **11a**, maintaining the reaction temperature at about 5°C. The mixture was stirred for 6 h at room temperature, and then NaCl was filtered off. The mother liquor was evaporated, and the residue was distilled in a vacuum. Yield 3.5 g (65%), bp 67–68°C (0.08 mmHg). 1H NMR spectrum (CDCl₃), δ , ppm (J , Hz): 1.10 s and 1.17 s (9H, CMe₃), 2.29 d and 2.40 d (3H, CMe, $^4J_{PH}$ 2.9), 2.61 br.s (1H, NH), 3.71 d and 3.68 d (6H, POME, $^3J_{PH}$ 11.0). ^{31}P NMR spectrum (CHCl₃), δ_P , ppm: 18.74, 19.55. Found, %: C 42.48; H 7.39; N 5.60; P 12.16 C₉H₁₉ClNO₃P. Calculated, %: C 42.28; H 7.49; N 5.48; P 12.11.

The reaction of *O,O*-diisopropyldithiophosphoric acid 2a with *N-tert*-butyl-2,2-dichloropropanimine 10. *a.* The reagents ratio of 1 : 1. A solution of 6.13 g (0.033 mol) of dichlorimine **10** in 10 mL of CH₂Cl₂ was added dropwise to a solution of 7.2 g (0.033 mol) of acid **2a** in 25 mL of CH₂Cl₂. The reaction mixture was kept for 24 h at room temperature. According to ^{31}P NMR, the reaction mixture contained disulfide **16** (δ_P 81.45 ppm) and *N-tert*-butyl-2-diisopropoxythiophosphorylthio)propaniminium chloride **21** (δ_P 83.14 ppm) in a ratio of 65 : 35. After removing the solvent in a vacuum, the residue was treated with hexane. The resulting crystals were filtered off, washed

twice with diethyl ether, and dried to yield 1.4 g of crude *N-tert*-butyl-2-chloropropaniminium chloride **19**. 1H NMR spectrum (CDCl₃), δ , ppm (J , Hz): 1.54 s (9H, CMe₃), 1.79 d (3H, MeCH, $^3J_{HH}$ 6.8), 5.71 d.q (1H, MeCH, $^3J_{HH}$ 6.8, $^3J_{HH}$ 8.8), 8.45 d (1H, CH=N⁺, $^3J_{HH}$ 8.8), 14.1 br.s (1H, N⁺H). Found, %: C 45.47; H 8.13; Cl 38.38; N 7.43. C₇H₁₅Cl₂N. Calculated, %: C 45.67; H 8.21; Cl 38.51; N 7.61. From the hexane mother liquor 2.1 g of unreacted starting imine **10** and 2.5 g of bis(diisopropoxythiophosphoryl)disulfide **16a** with mp 91°C were isolated. 1H NMR spectrum (CDCl₃), δ , ppm (J , Hz): 1.40 d and 1.42 d (24H, Me₂CH, $^3J_{HH}$ 6.4), 4.90 d. sept (4H, CHOP, $^3J_{HH}$ 6.4, $^3J_{PH}$ 12.0). ^{31}P NMR spectrum (CDCl₃): δ_P 81.70 ppm. ^{13}C NMR spectrum (CDCl₃), δ_C , ppm (J , Hz): 23.75 d (CH₃, $^3J_{PC}$ 4.5), 23.57 d (CH₃, $^3J_{PC}$ 5.5), 74.76 d (CH, $^2J_{PC}$ 6.7).

b. The reagents ratio of 3 : 1. Similarly, from 6.4 g (0.03 mol) of acid **2a** and 1.82 g (0.01 mol) of dichlorimine **10** were obtained 2.8 g (77%) of *N-tert*-butyl-2-(diisopropoxythiophosphorylthio)propaniminium chloride **21a** as a viscous oil. 1H NMR spectrum (CDCl₃), δ , ppm (J , Hz): 1.27 d (12H, Me₂CH, $^3J_{HH}$ 6.4), 1.58 s (9H, CMe₃), 1.71 d (3H, MeCH, $^3J_{HH}$ 6.4), 4.78 d. sept (2H, CHOP, $^3J_{HH}$ 6.4, $^3J_{HH}$ 12.8), 5.07 m (1H, MeCH, $^3J_{HH}$ 6.4, $^3J_{HH}$ 7.2, $^3J_{PH}$ 12.4), 8.2 d (1H, CH=N, $^3J_{HH}$ 7.2), 16.2 br.s (1H, N⁺H). ^{31}P NMR spectrum (CCl₄): δ_P 83.15 ppm. Found, %: C 43.40; H 8.21; N 4.01; P 8.38. C₁₃H₂₉ClNO₂PS₂. Calculated, %: C 43.14; H 8.08; N 3.87; P 8.56. Disulfide **16a** (1.8 g, 85%, mp 91°C) was isolated from the mother hexane solution.

***N-tert*-Butyl-2-(diethoxythiophosphorylthio)propaniminium chloride (21b).** A solution of 1.0 g (0.0055 mol) of imine **10** in 5 mL of CH₂Cl₂ was added dropwise to a solution of 3.06 g (0.0164 mol) of acid **2b** in 15 mL of CH₂Cl₂, maintaining the reaction temperature at about –10°C. The mixture was kept at room temperature for 48 h. According to ^{31}P NMR, the reaction mixture contained compounds **16c** (δ_P 84.25 ppm) and **21b** (δ_P 86.05 ppm). After removing the solvent in a vacuum, the residue was washed three times with hexane to give 1.4 g (77%) of salt **21b** as a viscous oil. 1H NMR spectrum (CDCl₃), δ , ppm (J , Hz): 1.20 t and 1.18 t (6H, CH₃CH₂, $^3J_{HH}$ 7.2), 1.40 s (9H, CMe₃), 1.53 d (3H, CHCH₃, $^3J_{HH}$ 7.2), 4.84 d. quint and 4.81 d. quint (1H, CHCH₃, $^3J_{HH}$ = $^3J_{PH}$ = 7.2, $^3J_{HH}$ 7.8), 4.05 quint and 4.06 quint (4H, POCH₂, $^3J_{HH}$ = $^3J_{PH}$ = 7.2), 8.25 d. d (1H, CH=N, $^3J_{HH}$ = 7.8, $^3J_{HH}$ = 13.6), 15.19 br.s (1H, N⁺H). ^{31}P NMR spectrum (CCl₄): δ_P 86.05

ppm. Found, %: C 39.81; H 7.78; N 4.28; P 9.49. $C_{11}H_{25}ClNO_2PS_2$. Calculated, %: C 39.57; H 7.55; N 4.19; P 9.58.

The reaction of *N*-tert-butyl-2-chloropropaniminium chloride **19 with *O,O*-diisopropyldithiophosphoric acid **2a**.** A solution of 1.26 g (0.0059 mol) of acid **2a** in 5 mL of CH_2Cl_2 was added dropwise to a solution of 1 g (0.0059 mol) of *N*-tert-butyl-2-chloropropaniminium chloride **19** in 10 mL of CH_2Cl_2 at 0°C. The reaction mixture was kept in a vacuum (90 mmHg) to remove HCl liberated during the reaction, and then kept for 24 h at room temperature. After removal of the solvent, 1.45 g (75%) of crude *N*-tert-butyl-2-(diisopropoxythiophosphorylthio)propaniminium chloride **21a** was obtained.

ACKNOWLEDGMENTS

This work was supported by the Ministry of Education and Science (Research Work no. 1629) in the frame of the basic part of governmental contract (no. 56/2015).

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