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

Reaction of *O*,*O*-Dialkyldithiophosphoric Acids with *N*-Alkyl-2-haloaldimines

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Abstract—Synthetic results of reactions of *O*,*O*-dialkyldithiophosphoric acids with *N*-alkyl-2-haloaldimines fundamentally depend on the nature of the halogen: in the case of Cl-substituted imines the reaction afforded 2-(dialkoxythiophosphorylthio)iminium chlorides, with bromoimines bis(dialkoxythiophosphoryl) disulfide and unsubstituted iminium bromide were obtained. According to dynamic ^{31}P NMR spectroscopy data the reason for this difference is the presence or absence of a free acid in the reaction medium.

Keywords: *O*,*O*-dialkyldithiophosphoric acids, *N*-alkyl-2-haloaldimines, bis(dialkoxythiophosphoryl)disulfide, unsubstituted iminium bromide, 2-(dialkoxythiophosphorylthio)iminium chloride

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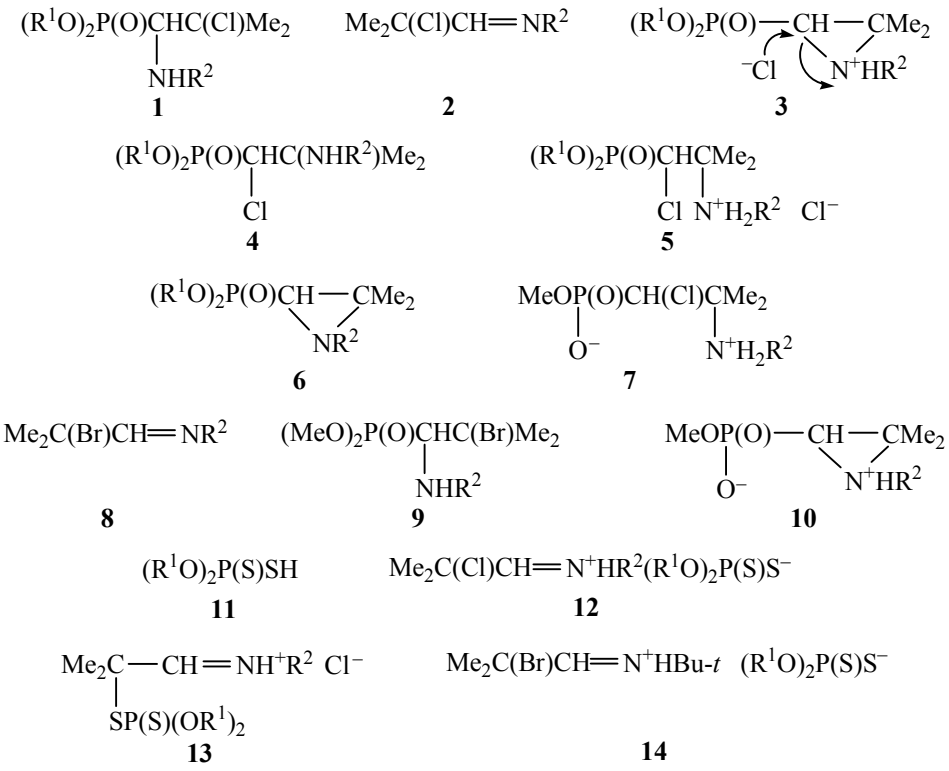
One of the basic methods for the synthesis of *N*,*P*-containing organic compounds having diverse biological activities and complexing properties is reactions of P(III) and P(IV) acids with *N*-alkyl(aryl)aldimines [1, 2]. The reaction of *N*-alkylaldimines with *O*,*O*-dialkyldithiophosphoric acids have been initially regarded as addition of the latter to the imine group [3]. However, it was found later that the reaction stops at the stage of protonation of the imine nitrogen atom to form iminium salts [4]. In the case of chloral imines, where the basicity of the imine nitrogen is low, their protonation did not occur, so only the product formed of the addition to the C=N bond [5]. In the products obtained the chlorine atoms were inactive towards subsequent substitution processes.

We have previously shown that a single chlorine atom in the initial adducts of dialkylphosphorous acids **1** to *N*-alkyl-2-chloroaldimine **2** is labile, and prolonged keeping of the reaction mixture at room temperature resulted in the formation of the products of diverse structures [6–8]. Thus, intermediate aziridinium salt **3** underwent isomerization of 2-chloroalkyl moiety at P(IV) atom to give 1-chloroalkyl derivative **4**. In addition, phosphorylated ammonium salt **5** and aziridine **6** were isolated as well as phosphonate betaine **7**. The reaction of dimethylphosphoric acid with bromoimine **8**, in contrast, occurred via transformation of the intermediate adduct **9** into aziridinium phosphonate **10**.

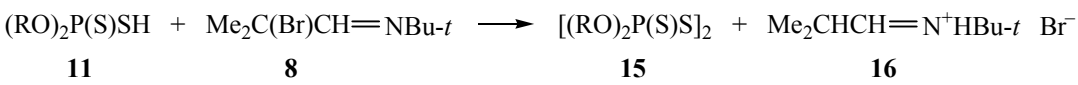
Also we have previously established [9] that the reaction of *O*,*O*-dialkyldithiophosphoric acids **11** with *N*-alkyl-2-methyl-2-chloropropanimines **2** proceeded in two stages. The first step consisted in the protonation of the imine nitrogen atom and the formation of the intermediate iminium salt, *N*-alkyl-2-methyl-2-chloropropaniminium dialkyldithiophosphate **12**, where the single chlorine atom was as labile as in the adducts of dialkylphosphorous acid **1**. In the second stage the chlorine atom was replaced by (dialkoxythiophosphorylthio) group to form *N*-alkyl-2-(dialkoxythiophosphorylthio)-2-methylpropaniminium chlorides **13** [9] (Scheme 1).

The reaction of *O*,*O*-dialkyldithiophosphoric acids **11** with *N*-alkyl-2-bromo-2-methylpropanimines **8** has not been previously described in the literature. Based on the fact that the primary adducts of dialkylphosphorous acids **1** and **9** to chloro- (**2**) and bromoimines (**8**) were transformed into diverse compounds **5**–**7**, **10**, we assumed that in the case of the reactions of imines **8** with dithio acids **11** a difference in further

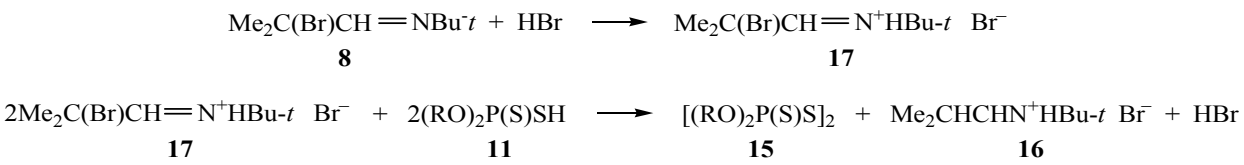
Scheme 1.



Scheme 2.



Scheme 3.



transformations of the intermediate iminium salt **14** might be observed.

Indeed, it was found that the major products of the reaction between compounds **8** and **11** at a ratios of 1 : 1 and 1 : 2 were bis(dialkoxythiophosphoryl) disulfide **15** and *N*-*tert*-butyl-2-methylpropaniminium bromide **16**. When using reagents ratio of 1 : 1 a half of the starting imine **8** did not react, and it was completely consumed at a ratio of 1 : 2 (Scheme 2).

According to ^{1}H and ^{31}P NMR spectroscopy data, the formation of the intermediate *N*-*tert*-butyl-2-bromo-2-methylpropaniminium *O*,*O*-diisopropyldithiophophate **14** occurred even at –90°C (δ_P = 107.7 ppm, δ_{N^+H} 13.0 ppm). At a temperature above –80°C it was transformed into the final salt **16** and disulfide **15**. Compound **16** was a reduced starting imine salt. To prove the existence of the stage involving reaction of acid **11** with *N*-*tert*-butyl-2-bromo-2-methylpropaniminium cation included in the intermediate salt **14** we synthesized *N*-*tert*-butyl-2-bromo-2-methylpropaniminium bromide **17**, stable salt of this cation. It was found that the reaction at a ratio 1 : 1 of these reagents yielded bis(dialkoxythiophosphoryl) disulfide **15** and *N*-*tert*-butyl-2-methylpropaniminium bromide **16** (Scheme 3).

Table 1. ^{31}P NMR study of the reaction between acid **11a** and imine **2b**

T, °C	Time, min	δ_P of **12a**, ppm	Content of **12a**, %	δ_P of **13a**, ppm	Content of **13a**, %
–60	2	107.5	100.00	–	0.00
–50	23	107.1	100.00	–	0.00
–30	54	105.1	99.94	84.5	0.06
–20	73	101.5	99.92	84.6	0.08
–10	88	100.5	97.73	84.6	2.27
0	109	99.2	91.99	84.6	8.01
10	130	97.6	75.94	84.9	24.06
20	143	95.9	54.88	85.0	45.12
20	182	98.8	20.33	85.2	79.67
25	222	98.8	10.75	85.3	89.25
25	310	98.7	4.34	85.4	95.66
25	420	–	0.00	85.4	100.00

Hence the reactions of *O*,*O*-dialkyldithiophosphoric acids with *N*-alkyl-2-halo-2-methylpropanimines fundamentally depend on the nature of the halogen and result in the formation of 2-(dialkoxythiophosphorylthio)iminium chlorides **13** at Hlg = Cl or bis-(dialkoxythiophosphoryl)disulfide **15** and iminium bromide **16** at Hlg = Br.

To determine the cause of the dependence of the reaction result on the halogen nature, we applied dynamic ^{31}P NMR spectroscopy for studying the reaction of acid **1a** with *N*-*tert*-butyl-2-methyl-chloropropanimine **2a**. Thus, in the ^{31}P NMR spectrum of the reaction mixture two signals corresponding to the phosphorus atoms of the final iminium salt **13a** and intermediate salt **12a** were observed. There was an increase in content of the reaction product **13a** and a decrease in the amount of intermediate **12a** as the temperature and the reaction time increased (Table 1).

Likewise, the reaction of acid **11a** with *N*-*tert*-butyl-2-bromo-2-methylpropanimine **8a** was studied (Table 2). Comparison of the data presented in Tables 1 and 2 shows that within 3 min after mixing the reagents the content of salt **14a** is only 12.8% unlike chloroimine salt **12a** (100%). The signal at 81.0 ppm refers obviously to the phosphorus atom of the starting acid, since in the ^{1}H NMR spectrum the signal of SH at 3.5 ppm was detected. After 37 min (–85°C) the content of salt **14a** in the reaction mixture reaches a maximum (71%) and then quite rapidly decreases to 3.3% due to disulfide **15** formation. Although acid **11a** was consumed in the reaction with salt **14a**, the signal intensity in the range of 81.0–82.1 ppm increased since the resulting disulfide **15a** contained two phosphorus atoms.

Hence, depending on the nature of the halogen atom the position of the acid ↔ salt equilibrium is different. In the case of chloroimine the equilibrium is almost completely shifted to the right: the system has no free acid **11a**, and salt **12a** is transformed into the reaction product **13a** as a result of the nucleophilic substitution of the chlorine atom with dialkoxythiophosphorylthio group. In the case of bromoimine the equilibrium is shifted to the left: the system has much free acid, which is involved into reducing the cation of the intermediate salt **14a** with disulfide **15a** formation. It should also be noted that although the reaction mixture contains a significant amount of salt **14a**, the formation of the substitution product was not found. This is obviously due to the fact that the rate of the reduction process exceeds significantly the rate of S_N process.

The reducing properties of *O*,*O*-dialkyldithiophosphoric acids have been described in [10–14]. Thus, they reduce azobenzene to benzidine and aniline [10–12], sulfoxides to dialkylsulfides [13], pyridine oxide to pyridine [14], and 4,4'-diphenoquinone to 4,4-bis-(2,6-di-*tert*-butylphenol) [15]; in so doing acids **11** are converted into disulfides **15**. In these examples the

Table 2. ^{31}P NMR study of the reaction between acid **11a** and imine **8a**

T, °C	Time, min	δ_P of **14a**, ppm	Content of **14a**, %	δ_P of **11a** and **15a**	Content of the reaction products,[a] %
–90	3	107.7	12.8	81.0	87.2
–90	11	107.7	58.8	81.0	38.8
–90	31	107.7	70.0	81.0	27.0
–85	37	107.7	71.0	81.0	25.5
–80	44	107.8	68.5	81.1	28.0
–70	56	107.8	52.0	81.2	46.0
–70	63	107.8	51.0	81.2	47.1
–60	69	107.9	36.5	81.4	62.0
–50	79	108.1	24.4	81.5	75.0
–40	97	108.4	13.3	81.6	86.7
–30	104	108.5	11.0	81.7	89.0
–15	109	108.6	7.9	81.9	92.1
0	123	108.8	3.3	82.1	96.7

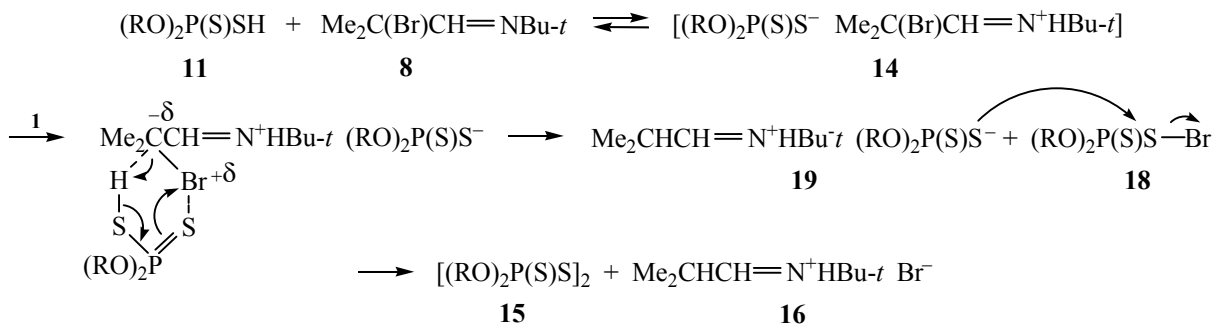
[a] The total content of acid **11a** and disulfide **15a** based on the starting acid.

reduction of organic compounds containing unsaturated or semipolar bond was mentioned. In the literature there is no information on the use of acids **11** to reduce organic halides including C–Hlg bond. It should be noted that in our case the C–Br bond reduction occurred not in the starting imine, but in iminium cation regardless of the anion nature (dithiophosphate or bromide).

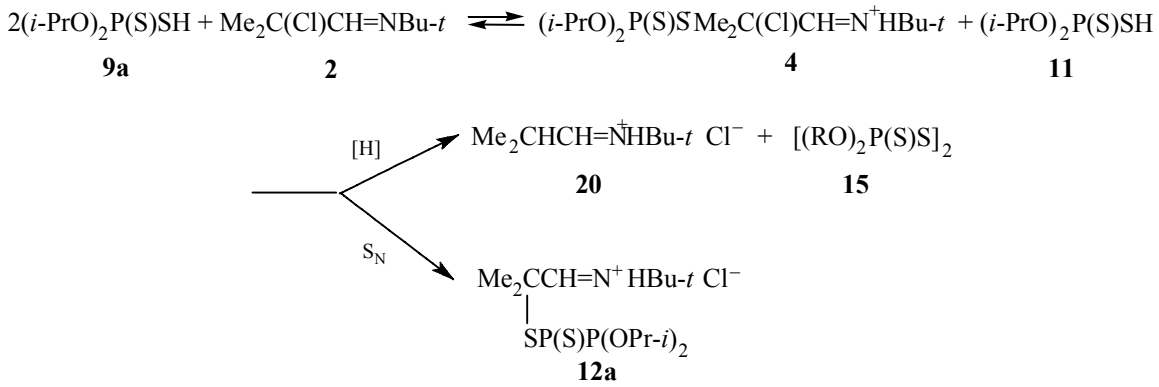
We believe that the conversion of imine **8** into iminium salt **14** increases the halophilic properties of the halogen. The thiophosphoryl sulfur atom and the SH hydrogen are coordinated with the partially positively charged bromine atom and the carbon atom, respectively. As a result of electron transfer through the six-membered ring the bromine atom is substituted by hydrogen, i. e. a reduction occurs of iminium salt cation to form (dialkoxythiophosphoryl)sulfenyl bromide **18**. The interaction of the latter with dithiophosphate anion of new intermediate salt **19** led to formation of bis(dialkoxythiophosphoryl) disulfide **15** (Scheme 4).

After establishing the dependence of the position of the acid ⇄ salt equilibrium on the halogen nature, we studied the reaction of acid **11a** with chlorinated imine **2a** in a ratio of 2 : 1. In this case the second equivalent of acid should be involved into a competing reduction

Scheme 4.



Scheme 5.



process. To a solution in CCl_4 of twofold excess of acid **11a** a solution of chloroimine **2a** in the same solvent was added, and the mixture was maintained at room temperature for 7 days. *N-tert*-Butyl-2-methylpropaniminium chloride **20** and bis(diisopropoxythiophosphoryl) disulfide **15a** were isolated in individual state; about 50% of acid **11a** was consumed in the formation of disulfide **15a**. According to ^{1}H and ^{31}P NMR, the mother liquor after separation of disulfide **15a** contained the substitution product, *N-tert*-butyl-2-(diisopropoxythiophosphorylthio)-2-methylpropaniminium chloride **13a**, and the starting acid **11a** in 4 : 1 ratio (Scheme 5).

In general, according to ^{1}H and ^{31}P NMR spectroscopy, 50% of the acid **11a** was consumed in reduction of chloroimine **2a**, and 40%, in the formation of **13a**; 10% of the acid remained unreacted.

In summary, as in the case of the primary dialkylphosphorus acid adducts to *N*-alkyl-2-chloro- (**1**) and 2-bromoimines (**8**) the further conversion of iminium salts **12** and **14** depended dramatically on the nature of the halogen: chloroiminium salts underwent intramolecular nucleophilic substitution of chlorine atom with dialkoxythiophosphorylthio group, while bromoiminium salts were reduced at the C–Br bond, and the acid was converted to bis(dialkoxythiophosphoryl)disulfide. The reason for this difference lies in the presence of free acid in the reaction medium, which is involved into the reduction of C–Br bond of iminium salts; in the case of chloroimine there was no free acid.

EXPERIMENTAL

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

Reaction of *N-tert*-butyl-2-methyl-2-bromopropanimine 8a with *O*,*O*-diisopropyldithiophosphoric acid 11a. *a*. The reagents ratio 1 : 1. A solution of 1.06 g (5.13 mmol) of imine **8a** in 5 mL of CCl_4 was added dropwise to a solution of 1.1 g (5.13 mmol) of acid **11a** in 5 mL of CCl_4 maintaining the temperature between 0–5°C. Then, the temperature of the reaction mixture was raised to 20°C, the mixture was stirred for 2–3 h, and then allowed to stand at room temperature for 24 h. The resulting crystals were filtered off to give 0.42 g (80%) of *N-tert*-butyl-2-methylpropaniminium bromide **14a**, mp 103–104°C. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm (*J*, Hz): 1.29 d (6H, $\underline{Me}_2CH$, $^3J_{HH}$ 7.0), 1.57 s (9H, CMe_3), 3.75 d. of septets (1H, CH, $^3J_{HH}$ 7.0, $^3J_{HH}$ 8.6), 8.27 d (1H, CH=N, $^3J_{HH}$ 8.6), 14.34 br.s (1H, N^+H). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 19.24 ($\underline{Me}_2CH$), 27.77 ($C\underline{Me}_3$), 32.08 ($Me_2\underline{CH}$), 61.86 ($\underline{C}Me_3$), 180.18 (HC=N). Found, %: C 51.71; H 8.63; N 6.85. $C_8H_{18}BrN$. Calculated, %: C 51.94; H 8.72; N 6.73.

The mother liquor was evaporated in a vacuum (0.08 mmHg), volatile products were collected in a liquid nitrogen trap. According to ^{1}H NMR spectrum, the condensate from traps contained the starting bromoimine. After removal of all the volatile products, disulfide **15a** was isolated. Yield 0.8 g (73%), mp 91°C. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm (*J*, Hz): 1.41 d and 1.43 d (24H, $\underline{Me}_2CH$, $^3J_{HH}$ 6.4), 4.90 d. of septets (4H, CHOP, $^3J_{HH}$ 6.4, $^3J_{PH}$ 12.0). ^{31}P NMR spectrum ($CDCl_3$): δ_P 81.70 ppm.

b. The reagents ratio 1 : 2. To a solution of 2.14 g (10 mmol) of acid **11a** in 15 mL of CCl_4 was added

dropwise a solution of 1.03 g (5 mmol) of the imine **8a** in 5 mL of CCl_4 keeping the temperature between 0–5°C. Then the temperature of the reaction mixture was raised to room temperature and the mixture was stirred for 3 h. On the next day the crystals formed were filtered off. We obtained 0.4 g (78%) *N-tert*-butyl-2-metilpropaniminium bromide **14a**, mp 103–104°C. From the mother liquor 1.8 g (85%) of disulfide **15a** was isolated, mp 91°C (hexane).

Reaction of *N-tert*-butyl-2-methyl-bromopropaniminium bromide 17 with *O,O*-diizopropyldithiophosforic acid 11a. To a suspension of 3.1 g (10.8 mmol) of bromide **17** in 25 mL of CH_2Cl_2 was added dropwise 2.3 g (10.7 mmol) of acid **11a** at 0–2°C. After 10 h, the reaction mixture became homogeneous. After keeping the reaction mixture for 48 h at room temperature the solvent was removed in a vacuum. The solid residue was treated with hexane. Undissolved part was filtered off, washed twice with hexane, and dried in a vacuum to give 2.9 g of a mixture (1 : 1) of iminium salts **16** and **17**. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm (*J*, Hz): ***N-tert*-butyl-2-bromo-2-methylpropaniminium bromide (17)**, 1.73 s (9H, CMe_3), 2.33 s (6H, CMe_2), 8.48 d (1H, $CH=N^+H$, $^3J_{HH}$ 16.4), 14.39 br.s (1H, N^+H); ***N-tert*-butyl-2-methylpropaniminium bromide (16)**, 1.31 d (6H, CMe_2, $^3J_{HH}$ 6.8), 1.59 s (9H, CMe_3), 3.75 m (1H, Me_2CH), 8.22 d.d (1H, $CH=N^+H$, $^3J_{HH}$ 8.4, $^3J_{HH}$ 16.4), 13.59 br.s (1H, N^+H). The combined hexane filtrate was cooled to give 1.7 g (75%) of disulfide **15a** with mp 91°C (hexane).

Reaction of *N-tert*-butyl-2-methyl-2-bromopropanimine 8a with *O,O*-diethyldithiophosphoric acid 11b (1 : 2). A solution of 0.99 g (5.3 mmol) of imine **8a** in 5 mL of CCl_4 was added dropwise to a solution of 1.8 g (9.6 mmol) of acid **11b** in 15 mL of CCl_4 maintaining the temperature between 0–5°C. The reaction mixture was kept at room temperature for 24 h. The resulting crystals were filtered off to give 0.81 g (80.1%) of *N-tert*-butyl-2-methylpropaniminium bromide **16**, mp 103–104°C.

Reaction of *O,O*-diisopropyldithiophosphoric acid 11a with *N-tert*-butyl-2-methyl-2-chloropropanimine 2a. *a.* The reagents ratio 1 : 1. To a solution of 14.1 g (87 mmol) of *N-tert*-butyl-2-methyl-2-chloropropanimine **2a** in 70 mL of CCl_4 with stirring was added dropwise 18.7 g (87 mmol) of *O,O*-diisopropyldithiophosphoric acid **11a** maintaining the temperature between 0–5°C. Then, the temperature was raised to 20°C, and the mixture was kept for 24 h. After removing the solvent, the residue was dissolved in diethyl ether and cooled. The crystals were filtered off to give 20.5 g (81%) of ***N-tert*-butyl-2-(diisopropoxythiophosphorylthio)-2-methylpropaniminium chloride 13a**, mp 147°C. ^{1}H NMR spectrum ($CDCl_3$) δ, ppm (*J*, Hz): 1.18 d (12H, $\underline{Me}_2CHO$, $^3J_{HH}$ 6.1), 1.52 s (9H, CMe_3), 1.85 s (6H, CMe_2), 4.62 d. sept (2H, CHOP, $^3J_{HH}$ 6.1, $^3J_{PH}$ 12.1), 8.76 s (CH=N), 14.25 br.s (1H, N^+H). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm (*J*, Hz): 23.91 d and 23.34 d ($OCH\underline{Me}_2$, $^3J_{PC}$ 5.0), 27.56 (CMe_3), 28.34 d ($SCMe_2$, $^3J_{PC}$ 8.1), 52.09 d (CS, $^2J_{PC}$ 4.0), 63.57 ($\underline{C}Me_3$), 75.32 d ($Me_2\underline{C}HO$, $^2J_{PC}$ 8.1), 177.45 ($CH=N^+$). ^{31}P NMR spectrum ($CDCl_3$): δ_P 85.20 ppm. Found, %: C 44.89; H 8.50; P 8.18; S 16.88. $C_{14}H_{31}ClNO_2PS_2$. Calculated, %: C 44.73; H 8.31; P 8.24; S 17.05.

b. The reagents ratio 2 : 1. A solution of 1.31 g (8.1 mmol) of imine **2a** in 15 mL of CCl_4 was added dropwise to a solution of 3.5 g (16.3 mmol) of acid **11a** in 15 mL of CCl_4 at –10°C. The mixture was allowed to stand for 7 days at room temperature. After removing the solvent, the residue was treated with hexane. The resulting crystals were filtered off, washed twice with diethyl ether, and dried to yield 0.60 g (46%) of **2-methylpropaniminium chloride 20**, mp 139°C. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm (*J*, Hz): 1.26 d (6H, $CHMe_2$, $^3J_{HH}$ 6.8), 1.55 s (9H, CMe_3), 3.77 d. sept (1H, $Me_2\underline{CH}$, $^3J_{HH}$ 6.8, $^3J_{HH}$ 8.8), 7.89 d.d (1H, $\underline{CH}=N^+H$, $^3J_{HH}$ 8.8, $^3J_{HH}$ 16.4), 15.8 br.s (1H, N^+H).

Bis(diisopropoxythiophosphoryl)disulfide **15a** (1.8 g) was isolated from the cooled mother liquor.

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