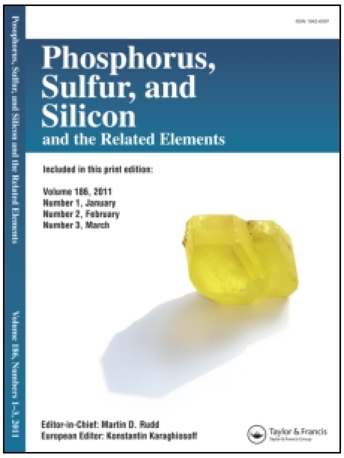

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DITHIOPHOSPHORIC ACIDS AND TETRAPHOSPHORUS DECASULFIDE IN THE SYNTHESIS OF BORON DITHIOPHOSPHATES

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DITHIOPHOSPHORIC ACIDS AND TETRAPHOSPHORUS DECASULFIDE IN THE SYNTHESIS OF BORON DITHIOPHOSPHATES

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The reactions of O,O-dialkyl dithiophosphoric acids with triisobutyl borate, ammonium O,O-diisobutyl dithiophosphate with fluorodiisobutyl borate, and tetraphosphorus decasulfide with triisobutyl borate were studied. On the basis of these studies, new boron derivatives of dithiophosphoric acids were obtained. Low frequency ultrasonic irradiation (22 kHz, power 130 W) leads to reduction in reaction temperature and time in the reactions studied.

Keywords: Dithiophosphoric acids; tetraphosphorus decasulfide; borates; boron dithiophosphates; ultrasound

INTRODUCTION

Boron dihiophosphates with the P(S)SB structural fragment such as O-ethyl-S,S′-bis(o-carborane-9-yl)dithiophosphate, trithiophosphate and diphenyldithiophosphinate have recently been obtained by addition elemental sulfur to the corresponding (o-carborane-9-yl)thiophosphite, dithiophosphite and diphenyldithiophosphinite. [1–5] 2-(O,O-Dialkyl and alkylene dithiophosphato)-1,3,2-dioxaborinanes have been prepared by

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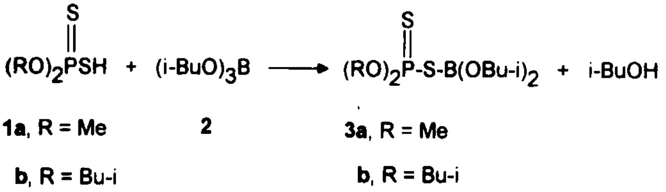
the reactions of ammonium salts of the corresponding dithiophosphoric acids with 2-chloro-1,3,2-dioxaborinanes in refluxing benzene. [6] O,O-Dialkyl and alkylene dithiophosphoric acids react with trimethylaminoborane in benzene at 80°C for 5 h to yield the corresponding dithiophosphates of trimethylaminoborane with the tetracoordinated boron atom. [7] However, it was reported that dithiophosphoric acids do not appear to react with boron derivatives containing the B-O bond such as boric acid, trialkyl borates and 2-alkoxy-1,3,2-dioxaborinanes. [6] Consequently, we have studied the reactions of O,O-dialkyl dithiophosphoric acids with borates under the ultrasonic irradiation conditions and present the results in this article.

RESULTS AND DISCUSSION

The substantial interest in the boron derivatives of dithiophosphoric acids is due to their potential biological activity similarly to other boron derivatives of pentavalent phosphorus, [4, 5] their high reactivity [2, 4, 6] and their use as intermediates to other new organothiophosphorus compounds. [2, 4] In order to obtain boron dithiophosphates in the reactions of dithiophosphoric acids with trialkyl borates we have used low frequency ultrasound (22 kHz, power 130 W). The beneficial effects of synthetic applications of sonochemistry are considered to be acceleration of chemical reactions, the use of less forcing conditions, reduction of induction period, reaction temperature and time. [8, 9] Thus, ultrasound has previously been shown to be an effective means of promoting the reactions of tetraphosphorus decasulfide and 1,3,2,4-dithiadiphosphetane-2,4-disulfides with dialkyl disulfides, thioacetals, acetals, bis(tributylstannyl)sulfide and tributyl(iso-butylthio)stannane. [10] Consequently, the reaction of O,O-dialkyl dithiophosphoric acids **1a,b** with triisobutyl borate **2** has been found to bring about the formation of S-(O,O-diisobutyl)boron O,O-dialkyldithiophosphates **3a,b** in the presence of ultrasound (25–30°C, 0.5 h in the case of **1a** and 60°C, 0.5 h in the case of **1b**) with the elimination of isobutanol which was easily given off from the reaction mixtures by the evaporation at reduced pressure (Equation (1), Tables (I)-(V)).



$$(RO)_2\overset{\overset{\displaystyle S}{\|}}{P}SH + (i\text{-}BuO)_3B \longrightarrow (RO)_2\overset{\overset{\displaystyle S}{\|}}{P}\text{-}S\text{-}B(OBu\text{-}i)_2 + i\text{-}BuOH \qquad (1)$$

1a, R = Me **2** **3a**, R = Me

b, R = Bu-i **b**, R = Bu-i

TABLE I Experimental data and yields of the products obtained

Initial Compounds Quantity, g (mmol)		*Reaction Conditions Temp., °C, Time (h)*	*Product Yield, g (%)*	
1a	3.4 (21.5)/**2** 5.0 (21.7)	25–30/0.5 USI[a]	**3a**[b]	6.0 (88)[c] /3.3 (49)[d]
1b	4.2 (17.3)/**2** 4.0 (17.4)	60/0.5 USI	**3b**	3.4 (49)/2.8 (41)
4	4.5 (17.4)/**5** 3.1(17.6)	80/10.5 15 mL PhH	**3b**[e]	1.5 (22)/0.9 (13)
6	2.9 (6.5)/**2** 12.0 (52.2)	150/3	**3b**[f]	4.8 (46)
6	2.9 (6.5)/**2** 12.0 (52.2)	50–70/20 min USI	**3b**	5.0 (48)

a. Ultrasound irradiation.
b. Yield of product in the reaction 1.
c. Yield of crude product.
d. Yield of isolated product.
e. Yield of product in the reaction 2.
f. Yield of product in the reaction 3.

TABLE II Physical, analytical and ^{31}P NMR spectral data of products obtained

Prod.	*B.p., °C mm Hg)*[a]	n_D^{20}	*Molecular Formula (Mol. mass)*	*Found/(Calc.), %*		*^{31}P NMR, δ, ppm (C_6H_6)*
				B	*P*	
3a	110 (0.05)	1.4355	$C_{10}H_{24}BO_4PS_2$	3.13	9.73	88.9
			(314.0)	(3.44)	(9.86)	
3b[b]	100 (0.04)	1.4549	$C_{16}H_{36}BO_4PS_2$	2.73	7.80	84.8
			(398.1)	(2.72)	(7.78)	

a. Temperature of thermal element of a thin layer distillation apparatus.
b. Data of **3b** obtained in the reaction 1.

TABLE III IR spectral data of the products obtained

Prod.	ν, cm^{-1}
3a	2960, 2930, 2900, 2880 ν (CH_3 as, s; CH_2 as, s; CH); 1380, 1350, 1330 δ (Me_2C gem s); 1045 ν (P-OC); 1015 ν (B-O); 870 ν (B-S); 668 ν (P=S); 525, 495 ν (P-S).
3b	2965, 2945, 2913, 2880 ν (CH_3 as, s; CH_2 as, s; CH); 1358, 1345, 1335 δ (Me_2C gem s); 1042 ν (P-OC); 1010 ν (B-O); 880 ν (B-S); 675 ν (P=S); 575, 555, 533 ν (P-S).

TABLE IV ^1H NMR spectral data of the products obtained

Prod.	CCl_4, δ, *ppm, J, Hz*
3a	0.89 (d, 12H, $\mathbf{CH_3}\mathrm{CHCH_2OB}$, $^3J_{HH}$ 7.0); 1.43–2.11 (m, 211, $\mathrm{(CH_3)_2}\mathbf{CH}\mathrm{CH_2OB}$); 3.48 (d, 4H, $\mathrm{(CH_3)_2CH}\mathbf{CH_2}\mathrm{OB}$, $^3J_{HH}$ 7.0); 3.76 (d, 6H, $\mathbf{CH_3}\mathrm{OP}$, $^3J_{PH}$ 15.0).
3b	0.92 (d, 12H, $(\mathbf{CH_3})_2\mathrm{CHCH_2OB}$, $^3J_{HH}$ 6.0); 1.02 (d, 12H, $(\mathbf{CH_3})_2\mathrm{CHCH_2OP}$, $^3J_{HH}$ 6.0); 1.48–2.18 (m, 2H, $\mathrm{(CH_3)_2}\mathbf{CH}\mathrm{CH_2OB}$; 2H, $\mathrm{(CH_3)_2}\mathbf{CH}\mathrm{CH_2OP}$); 3.48 (d, 4H, $\mathrm{(CH_3)_2CH}\mathbf{CH_2}\mathrm{OB}$, $^3J_{HH}$ 6.0); 3.83 (d. d, 4H, $\mathrm{(CH_3)_2CH}\mathbf{CH_2}\mathrm{OP}$, $^3J_{HH}$ 6.0, $^3J_{PH}$ 8.0).

TABLE V Mass spectral data of the products obtained

Prod.	$i\text{-}C_4H_{10}$, *m/e* (I_{rel}, %)
3a[a]	315 $[M + H]^+$ (30); 283 $[M + H - S]^+$ (60).
3a[b]	314 $[M]^+$ (25); 267 $[M - S - Me]^+$. (10).
3b	243 $[M + 2 H - B(OBu\text{-}i)_2]^+$(10).
3b	398 $[M]^+$. (10).
7	201 $[M + H - 2 Bu\text{-}i]^+$ (17).
7	330 $[M - O]^+$. (10); 257 $[M - O - OBu\text{-}i]^+$. (100); 216 $[M - OBu\text{-}i - Bu\text{-}i]^+$. (10).

a. Chemical ionization, 100 eV.

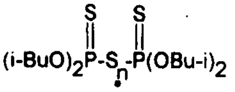
b. Electron impact, 70 eV.

On the basis of the ^{31}P NMR spectral data, we have established that the products **3a,b** were formed as the only organophosphorus compounds in the reaction mixtures when an equimolar ratio of reactants was used as the result of participation of only one isobutoxy group of borate **2** in the reaction 1. The ^{31}P NMR spectra of **3a,b** in benzene solutions reveal singlets at δ 88.9 ppm (for **3a**) and 84.9 ppm (for **3b**). As we can see, the ^{31}P NMR spectral signals of **3a,b** show no significant change with respect to the corresponding free O,O-dialkyl dithiophosphoric acids. [11] These resonances appear in the practically same region as that for 2-(O,O-dialkyldithiophosphato)-1,3,2-dioxaborinanes (δ 82.76–86.43 ppm in CCl_4 solutions). [6]

The ^1H NMR spectra of **3a,b** (Table (IV)) show the characteristic resonances due to the presence of two pairs of alkoxy groups attached both to the phosphorus atom and the boron one. Thus, the methyl protons of two methoxy groups at the phosphorus atom of **3a** (in CCl_4 solution) resonated in the range of δ 3.76 ppm as a doublet with $^3J_{PH}$ 15.0 Hz. The methylene protons of the two isobutoxy group at the boron atom $(CH_3)_2CH\underline{CH_2}OB$ appear as a doublet at δ 3.48 ppm ($^3J_{HH}$ 7.0 Hz). A doublet present in the region of δ 0.89 ppm has been assigned to the methyl protons of two isobutoxy groups at the boron atom $(\underline{CH_3})_2CHCH_2OB$ ($^3J_{HH}$ 7.0 Hz). The corresponding methyl protons of four isobuthoxy groups at the phosphorus and the boron atoms in the ^1H NMR spectrum of **3b** in CCl_4 solution (Table (IV)) appear as two doublets with equal integral intensities at δ_1 0.92 ppm and δ_2 1.02 ppm. The methylene protons of the two isobutoxy groups at the boron atom $(CH_3)_2CH\underline{CH_2}OB$ of **3b** resonate as a doublet at the same region of δ 3.48 ppm ($^3J_{HH}$ 7.0 Hz) as that in **3a**, whereas the similar protons of two isobuthoxy groups at the phosphorus atom $(CH_3)_2CH\underline{CH_2}OP$ of **3b** appear as a doublet of doublets shifted downfield (δ 3.83 ppm, $^3J_{HH}$ 6.0 Hz, $^3J_{PH}$ 8.0 Hz).

Bands of medium intensity present in the range of ν 870–880 cm^{-1} in the IR spectra of **3a,b** (Table (III)) may be attributed to the B-S valence vibrations. These bands appear in practically the same range as that for 2-(O,O-dialkyldithiophosphato)-1,3,2-dioxaborinanes (ν B-S 860–894 cm^{-1}). [6, 12] Bands in the region ν 1045 and 1015 cm^{-1} assigned to the P-OC and the B-O stretching vibrations are overlapping in the case of **3a**. Similar valence vibrations of the P-OC and the B-O bonds of **3b** appear in the range of ν 1042 and 1010 cm^{-1}, respectively. The strong bands at ν 668 and 675 cm^{-1} are due to the P=S stretching vibrations of **3a** and **3b**, respectively. Bands of medium intensity present in the region of ν 525, 495 and 575, 555, 533 cm^{-1} are attributed to the P-S valence vibrations of **3a** and **3b**, respectively.

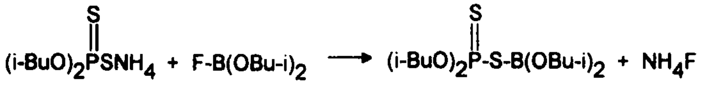
The chemical ionization and electron impact mass spectra of **3a** (Table (IV)) exhibit the mass peaks m/e 315 and 314 due its molecular ions $[M + H]^+$(25%) and $[M]^{+\cdot}$ (10%), respectively. Similarly, the electron impact mass spectrum of **3b** shows the mass peak m/e 398 that may be attributed to its molecular ion $[M]^{+\cdot}$ (10%). Some secondary products were observed by use of electron impact mass spectra of crude reaction mixture of **1b** with borate **2**. Thus, we have also observed mass peaks m/e 450 and 480; those may be assigned to the molecular ions $[M]^{+\cdot}$ of bis(O,O-diisobutyltrithiophosphoryl)sulfide **A** and the corresponding disulfide **B** as a product of addition of the sulfur atom to the molecule of compound **A** (calculated molecular masses M of **A** and **B** are 450 and 480, respectively). This secondary product **A** may be formed via the rupture of the S-P and S-B bonds of **3b**. On the other hand, the possibility of formation of disulfide **B** direct from the initial dithiophosphoric acids **1** must not be ruled out. It should be noted that **A** and **B** were only observed in conditions of recording of the recording of mass spectra.



$$(i\text{-}BuO)_2\overset{S}{\overset{\|}{P}}\text{-}S_n\text{-}\overset{S}{\overset{\|}{P}}(OBu\text{-}i)_2$$

n = 1 (**A**); n = 2 (**B**)

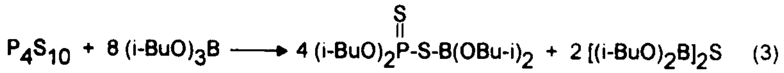
In order to confirm the structure of boron dithiophosphates **3** we decided to obtain them, by different syntheses. Thus, we have used the method developed by the authors [6]and obtained **3b** by refluxing for 10.5 h a benzene suspension of ammonium O,O-diisobutyl dithiophosphate **4** with fluorodiisobutyl borate **5** (Equation (2), Table (I)).



$$(i\text{-}BuO)_2\overset{S}{\overset{\|}{P}}SNH_4 + F\text{-}B(OBu\text{-}i)_2 \longrightarrow (i\text{-}BuO)_2\overset{S}{\overset{\|}{P}}\text{-}S\text{-}B(OBu\text{-}i)_2 + NH_4F \qquad (2)$$

4 **5** **3b**

It should be noted that physical and spectral data of **3b** (in the reaction 2) were identical with those of specimen of **3b** obtained in the course of the reaction 1 (See Experimental). Moreover, we have also synthezised **3b** by the use have called attention to the derivatives of phosphorus(V) of phosphorus sulfide (P_4S_{10}). Thus, we have previously found that tetraphosphorus decasulfide reacts with alkoxides and alkylmercaptides of triorganylsilanes, -germanes, -stannanes and -dialkylarsenites to form S-silyl, germyl, stannyl and arsenic(III) esters of dithiophosphoric and tetrathiophosphoric acids under mild conditions. [13–16] However, trialkyl borates have proven to be less reactive toward P_4S_{10} **6**. Thus, we have found that phosphorus sulfide **6** reacts with triisobutyl borate **2** in the molar ratio 1 : 8 under rather severe conditions (150°C, 3 h) to form the same **3b** and bis(diisobutoxyboron)sulfide **7** (Equation (3), Table (I)).



$$P_4S_{10} + 8\,(i\text{-}BuO)_3B \longrightarrow 4\,(i\text{-}BuO)_2\overset{\overset{\displaystyle S}{\|}}{P}\text{-}S\text{-}B(OBu\text{-}i)_2 + 2\,[(i\text{-}BuO)_2B]_2S \qquad (3)$$

6 **2** **3b** **7**

Diboronsulfide **7** was removed from the reaction mixture by vacuum distillation. Its presence in the volatile distillation fractions was confirmed by use of ^1H NMR and mass spectra (Table (V)). Thus, the electron impact mass spectra of the volatile liquid, collected in a liquid nitrogen cooled trap, from the crude reaction mixture of phosphorus sulfide **6** with borate **2**, show the mass peaks m/e 362 and 216 due to the ions $[M + O]^+$. (48%) (as an oxidation product) and $[M - i\text{-}BuO - i\text{-}Bu]^+$. (10%), respectively of diboronsulfide **7** (calculated molecular mass M of **7** is 346).

The interaction of phosphorus sulfide **6** with borate **2** is accompanied by some pitchy material at high temperatures. Consequently, in order to increase the reactivity of reactants in the reaction 3 we have also used ultrasound. Indeed, the use of ultrasonic technique leads to a reduction in reaction temperature (to 50–70°C) and time (to 20 min) in the reaction 3 compared with that with non-ultrasonic conditions. However, the yield of **3b** remains in the same scale (48%, in the presence of ultrasound) compared with 46% yield of **3b**in the absence of ultrasound (Table (I)). In the case of the reaction 3 the physical and spectral data of **3b** are consistent with that of **3b** obtained from the reactions 1 and 2 (See Experimental).

It should be emphasized that these counter-reactions (Equations (2) and (3)) confirm the structure of boron dithiophosphates **3** formed in the reaction 1. On the other hand, the interaction of P_4S_{10} with borate **2**proceeds with the participation of only one of the three B-O bonds of borate **2** under the conditions used (Equation (3).

Thus, we have developed various methods of synthesizing boron derivatives of dithiophosphoric acids on the bases of dithiophosphoric acids and phosphorus sulfide (P_4S_{10}). The reactions studied are of interest from the point of view of preparative organophosphorus chemistry.

EXPERIMENTAL

The ^{31}P NMR spectra were recorded with a Bruker MSL 400 (162 MHz) instrument in C_6H_6. The ^1H NMR spectra were taken on a Bruker MSL-400 (400 MHz) spectrometer and a Varian T-60 (60 MHz) spectrometer in CCl_4 or C_6D_6. The IR spectra were obtained with a Bruker IFS 113v and an UR-20 infrared spectrophotometers. Mass spectra (EI, 70 eV; CI, 100 eV) were determined on a M 80 B Hitachi chromatomass spectrometer.

Reaction of O,O-Dimethyl Dithiophosphoric Acid 1a with Triisobutyl Borate 2. Typical Procedure

The mixture of **1a** (3.4 g, 21.5 mmol) and **2** (5.0 g, 21.7 mmol) in a glass vessel was irradiated with ultrasound generated by an UZDN-A device (22 kHz, 130 W, direct immersion sonic horn) for 0.5 h at 25–30°C (the reactor was cooled by cold water). The mixture was evaporated at reduced pressure (0.1 and 0.04 mm Hg) at 40°C for 1 h to give crude **3a** (6.0 g, 88%). Pure product **3a** (3.3 g, 49%) was isolated from the residue by means of a thin layer distillation at 110°C (0.05 mm Hg) (See Tables (I) – (V)).

Product **3b** was obtained similarly (See Tables (I) – (V)).

Reaction of Ammonium O,O-Diisobutyl Dithiophosphate 4 with Fluorodiisobutyl borate 5

The solution of **4** (4.5 g, 17.4 mmol) and **5** (3.1 g, 17.6 mmol) in 15 mL of anhydrous benzene was refluxed for 10.5 h. The mixture was filtered. The filtrate was evaporated under vacuum (0.1 and 0.08 mm Hg) at 40°C for 2 h to give crude **3b** (1.5 g, 22%). Product **3b**(3.3 g, 49%) was isolated from the residue by means of a thin layer distillation at 100°C (0.03 mm

Hg), n_D^{20} 1.4551. ^{31}P-NMR (C_6H_6) δ 84.7. Found, % : B 2.52; P 6.59. $C_{16}H_{36}BO_4PS_2$. Calculated, % : B 2.72; P 7.78.

Reaction of Tetraphosphorus Decasulfide 6 with Triisobutyl Borate 2

a. in the absence of ultrasound. The mixture of **6** (2.9 g, 6.5 mmol) and **2** [12.0 g, 52.2 mmol) was stirred at 150°C for 3 h. The mixture was filtered. The filtrate was evaporated at reduced pressure (0.08 mm Hg) at 50°C for 2 h. Product **3b** (4.8 g, 46%) was isolated by common vacuum distillation at 115–117°C (0.04 mm Hg), n_D^{20} 1.4545. ^{31}P NMR (C_6H_6) δ 85.0 ppm. Found, % : B 2.66; P 7.03. $C_{16}H_{36}BO_4PS_2$. Calculated, % : B 2.72; P 7.78.

b. in the presence of ultrasound. The suspension of **6** (2.9 g, 6.5 mmol) and **2** (12.0 g, 52.2 mmol) in 5 mL of anhydrous toluene in a glass vessel was irradiated with ultrasound for 20 min at 50–70°C. The mixture was filtered. The filtrate was evaporated at reduced pressure (0.1 and 0.02 mm Hg) at 50°C for 2 h. Product **3b** (5.0 g, 48%) was isolated from the residue by means of a falling-film distillation at 135–140°C (0.02 mm Hg), n_D^{20} 1.4540. ^{31}P NMR (C_6H_6) δ 85.0 ppm. Found, % : B 2.50; P 7.26. $C_{16}H_{36}BO_4PS_2$. Calculated, % : B 2.72; P 7.78.

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References

[1] E. G. Rys, V. P. Balema, N. N. Godovikov, M. I. Kabachnik, *Izv. Akad. Nauk, Ser. Khim.*, 1653, (1990).
[2] V. P. Balema, E. G. Rys, N. N. Godovikov, M. I. Kabachnik, *Izv. Akad. Nauk. Ser. Khim.*, 2857, (1990).
[3] V. P. Balema, E. G. Rys, N. N. Godovikov, *Izv. Akad. Nauk. Ser. Khim.*, 459, (1992).
[4] V. P. Balema, E. G. Rys, N. N. Godovikov, A. V. Polyakov, Yu. T. Struchkov, *Izv. Akad. Nauk. Ser. Khim.*, 180, (1992).
[5] N. N. Godovikov, V. P. Balema, E. G. Rys, *Usp. Khimii (Russ.)*, **66**. 1125, (1997) and references therein.
[6] B. P. Singh, R. K. Mehrotra, G. Srivastava, *Phosphorus, Sulfur and Silicon*, **60**, 147, (1991).
[7] A. Chaturvedi, P. N. Nagar, G. Srivastava, *Phosphorus., Sulfur and Silicon*, **90**, 229, (1994).
[8] J. P. Lorimer and T. J. Mason, *Chem. Soc. Rev.*, **16**, 275 (1987).
[9] M. A. Margulis, *Russ. J. Phys. Chem.*, **50**, 1 (1976).
[10] I. S. Nizamov, G. G. Garifzyanova and E. S. Batyeva *Phosphorus, Sulfur, Silicon*, **88**, 39 (1994).

[11] M. M. Crutchfield, C. H. Dungan, J. H. Letcher, V. Mark and J. R. Van Wazer, in "P^{31} Nuclear Magnetic Resonance, Topics on Phosphorus Chemistry", Eds., M. Grayson and E. J. Griffith (Interscience Publishers, New York, 1967), Vol. 5.

[12] H. Steinberg, *Organoboron Chemistry*, (Interscience, New York, 1964), Vol. 1.

[13] I. S. Nizamov, V. A. Kuznetzov, E. S. Batyeva, V. A. Al'fonsov and A. N. Pudovik, *Heteroatom Chem.*, **4**, 379 (1993).

[14] I. S. Nizamov, V. A. Kuznetzov, E. S. Batyeva, V. A. Al'fonsov and A. N. Pudovik, *Phosphorus, Sulfur and Silicon*, **79**, 179 (1993).

[15] I. S. Nizamov, V. A. Kuznetzov and E. S. Batyeva, *Phosphorus, Sulfur, and Silicon*, **88**, 67 (1994).

[16] I. S. Nizamov, A. V. Matseevskii, E. S. Batyeva, B. E. Abalonin, I. I. Vandyukova and R. R. Shagidullin, *Heteroatom Chem.*, **8**, 329 (1997).

[17] N. A. Chadaeva, G. Kh. Kamai and K. A. Mamakov, *Zh. Obshch. Khim.*, **36**, 1994 (1966).

[18] M. Wieber and M. Schröpf, *Phosphorus, Sulfur, and Silicon*, **102**, 265 (1995).