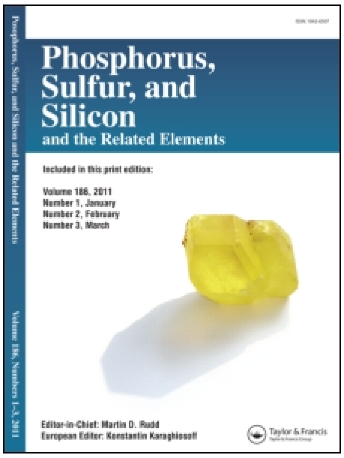

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S-TRIORGANOGERMANIUM ESTERS OF DITHIO- AND TETRATHIOPHOSPHORIC-, AND DITHIO- AND TRITHIOPHOSPHONIC ACIDS

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S-TRIORGANOGERMANIUM ESTERS OF DITHIO- AND TETRATHIOPHOSPHORIC-, AND DITHIO- AND TRITHIOPHOSPHONIC ACIDS

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S-Triphenyl and trialkylgermyl esters of dialkyl dithio- and tetrathiophosphoric acids, and 4-methoxyphenyldithio- and trithiophosphonic acids were easily obtained in the reactions of tetraphosphorus decasulfide and Lawesson's reagent with triphenyl and trialkyl(alkoxy)germanes and (alkylthio)-germanes.

Key words: Tetraphosphorus decasulfide; Lawesson's reagent; alkoxygermanes; alkylthiogermanes.

INTRODUCTION

The chemistry of organic derivatives of groups IVA elements with phosphorus (IV) thioacids is quite well developed. They possess properties of practical use. Thus, S-trimethylsilyl dithiophosphates are thiation reagents for carbonyl compounds and are the intermediates for the synthesis of useful organothiophosphorus compounds.[1] S-Triorganotin dithio- and tetrathiophosphates are used as pesticides and lubricant additives.[2,3]

The S-trialkyl and triaryl silyl, germyl, stannyl and plumbyl esters of linear and cyclic dithiophosphoric acids were obtained by the reactions of dithiophosphoric acids or their salts with silylating, germylating, stannylating and plumbylating agents.[1–6] Organosilicon and organotinthiophosphorus compounds were also obtained in the reactions of 2,4-bis(substituted)-2,4-dithioxo-1,3,2λ^5,4λ^5-dithiadiphosphetanes with heptamethyldisilazane,[7] trimethylsilylazide,[8] trimethylchlorosilane,[9] trimethyl(dimethylamino)silane,[10,11] bis(trimethylsilyl)sulfide,[10] N,N′-bis(trimethylsilyl)-acetamide,[12] bis(trimethylstannyl)sulfide,[13] trimethyl(alkoxy)silanes and trimethyl-(alkylthio)silanes, trialkyl(alkoxy)stannanes and trialkyl(alkylthio)stannanes.[14,15]

We have recently developed facile and efficient methods for the synthesis of S-trialkylsilyl and stannyl esters of dithio- and tetrathiophosphoric acids[16–19] and aryldithio- and trithiophosphonic acids on the basis of the reactions of tetraphosphorus decasulfide $\underline{1}$ and Lawesson's-like reagents with trialkyl(alkoxy) and (alkylthio)silanes, and trialkyl(alkoxy) and (alkylthio)stannanes.[14,15] We have tried to find the boundary of these methods and extended the reactions of $\underline{1}$ and of the 1,3,2,4-dithiadiphosphetane ring of Lawesson's-like reagents to organogermanium derivatives. In this article convenient methods are presented for the synthesis of triphenyl and trialkylgermyl esters of dialkyl dithio- and tetrathiophosphoric acids, and 4-methoxyphenyldithio- and trithiophosphonic acids.

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RESULTS AND DISCUSSION

We assumed that the use of the triaryl or trialkylgermyl protecting groups in protonodonating reagents such as alcohols may lead to S-triaryl or S-trialkylgermyl dithiophosphates directly from 1. In fact we have found that 1 reacts with triphenyl(ethoxy)germane 2 in anhydrous benzene at 20°C for 6 h with the formation of O,O′-diethyl-S-triphenylgermyl dithiophosphate 3 and bis(triphenylgermyl)sulfide 4a (Equation (1), and Table I).

TABLE I

Experimental, physical and analytical data of the products obtained

Initial cpd.		Prod.	Reaction conditions[a] temp. (°C)	time (h)	Yield[b] (%) crude/isolated	B.p.[d], °C (mm Hg) or mp (°C)	^{31}P NMR[e] δ, ppm	Molecular formula (Mol. mass)	Analytical data Found/Calc. (%) C	H	Ge	P	S
1	2	3	20	6	69[c]	98	87.6	$C_{22}H_{25}GeO_2PS_2$ (488.8)	54.88 54.02	5.40 5.17	15.15 14.85	6.67 6.34	13.31 13.08
1	5a	6a	80	0.5	64[c]	200 (0.02)	83.5	$C_{28}H_{33}GePS_4$ (390.7)	55.36 54.09	5.72 5.78	13.00 12.59	5.98 5.37	21.73 22.17
8	5b	9a[f]	20[g]	24[g]	79/51	130 (0.02)	71.2	$C_{12}H_{21}GeOPS_3$ (380.7)	38.52 37.83	5.72 5.57	19.16 19.07	7.32 8.14	25.49 25.19
8	5c	9b[h]	70	1	51/38	145 (0.02)	67.4	$C_{14}H_{25}GeOPS_3$ (408.7)	42.22 41.10	5.63 6.17	17.47 17.76	7.32 7.58	22.77 23.47
8	2	10	20	504	78/65	195 (0.02)	89.3	$C_{27}H_{27}GeO_2PS_2$ (550.8)	58.87 58.83	4.93 4.95	12.93 13.18	5.72 5.62	10.63 11.61

[a] Solvent: benzene.

[b] Yield of crude/isolated product

[c] Yield of isolated product

[d] Temperature of the spiral of the thermal element of thin layer distillation apparatus.

[e] In C_6H_6.

[f] d_4^{20} 1.0838, n_D^{20} 1.6093.

[g] Neat.

TABLE II

IR spectral data (cm^{-1}) of the products obtained

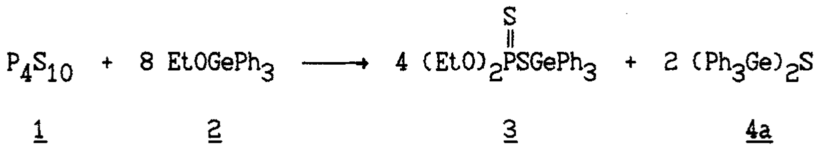
Prod.	C-H[a], Ar	CH_3[a] as, s; CH_2[a] as, s	C=C[a], Ar	P=S[a], PS_2[a] as	P-S[a], PS_2[a] as	$GePh_3$[a] as	Ge-S[a]
3b[b]	3072, 3055	2960, 2932, 2870	1590, 1490	700	546, 516	465	428, 415 405
6a[c]	3073, 3057, 3030, 3015	2967, 2930, 2888	1590, 1490	705	565, 535	468	417
9a[d]	3090, 3070, 3020	2970, 2910, 2880, 2845	1597, 1505	694	538, 515		415
9b[c,d]	3025, 3010	2970, 2945, 2915, 2885, 2848	1603, 1510	697	540, 510		415
10	3075, 3055	2985, 2950, 2910, 2842	1600, 1590	700	535, 519	465	420, 405

[a] Valence vibrations.

[b] In vaseline oil.

[c] Deformation vibrations δ 1390, 1370 cm^{-1} $(CH_3)_2C$ gem.

[d] Deformation vibrations δ 1265 cm^{-1} $CH_3(Ge)$ s.



$$\underset{\underline{1}}{P_4S_{10}} + 8\ \underset{\underline{2}}{EtOGePh_3} \longrightarrow 4\ \underset{\underline{3}}{(EtO)_2\overset{\overset{S}{\|}}{P}SGePh_3} + 2\ \underset{\underline{4a}}{(Ph_3Ge)_2S} \qquad (1)$$

The structure of the product 3 was confirmed by IR (Table II), ^1H NMR (Table III), ^{31}P NMR (Table I), mass-spectral data (Table IV) and microanalyses (Table I).

It is interesting to compare the reactivity of alkoxides of silicon, germanium and tin derivatives towards 1. According to the experimental data of the reactions of trimethyl(ethoxy)silane,[17] triphenyl(ethoxy)germane 2 and trimethyl-(ethoxy)stannane[19] with 1 (reaction conditions: 40–50°C, 6.5 h; 20°C, 6 h and 15°C, 3 h, respectively) the reactivity of these ethoxides increases in the series: silicon, germanium and tin derivatives. This series of reactivity is in accordance with the bond energy of O—E bond, which decreases in the series: Si—O, Ge—O, Sn—O.

We have tried to extend this method to other reagents, in which reactive protons can be substituted by triaryl or trialkylgermyl groups. Thus, we hoped to obtain

TABLE III

^1H NMR spectral data of the products obtained

Prod.	δ, ppm, J, Hz
3	0.92 (t, 6H, $\underline{CH_3}CH_2O$, $^3J_{H-H}$ 7.0), 3.75 (dq, 4H, $CH_3\underline{CH_2}O$, $^3J_{H-H}$ 7.0, $^3J_{P-H}$ 15.5), 6.93-7.60 (m, 15H, $C_6\underline{H_5}$).
6a	0.88 (d, 12H, $\underline{CH_3}CHCH_2$, $^3J_{H-H}$ 6.5), 1.44-1.76 (m, 2H, $CH_3\underline{CH}CH_2$), 2.26 (dd, 4H, $CH_3CH\underline{CH_2}SP$, $^3J_{H-H}$ 6.5, $^3J_{P-H}$ 13.0), 6.80-7.70 (m, 15H, $C_6\underline{H_5}$).
9a	0.93 (s, 9H, $\underline{CH_3}Ge$), 0.98 (t, 3h, $\underline{CH_3}CH_2SP$, $^3J_{H-H}$ 7.2), 2.56 (dq, 2H, $CH_3\underline{CH_2}SP$, $^3J_{H-H}$ 7.2, $^3J_{P-H}$ 15.0), 3.99 (s, 3H, $\underline{CH_3}O$), 6.48 (dd, 2H, 3,5-$H_2C_6\underline{H_2}$, $^3J_{H-H}$ 9.0, $^4J_{P-H}$ 3.0), 7.65 (dd, 2H, 2,6-$\underline{H_2}C_6H_2$, $^3J_{H-H}$ 9.0, $^3J_{P-H}$ 14.0).
9b	0.32 (s, 9H, $\underline{CH_3}Ge$), 1.02 (d, 6H, $\underline{CH_3}CHCH_2$, $^3J_{H-H}$ 7.0), 1.79-2.13 (m, 1H, $CH_3\underline{CH}CH_2$), 2.79 (dd, 2H, $CH_3CH\underline{CH_2}SP$, $^3J_{H-H}$ 7.0, $^3J_{P-H}$ 15.0), 3.88 (s, 3H, $\underline{CH_3}O$), 6.83 (dd, 2H, 3,5-$H_2C_6\underline{H_2}$, $^3J_{H-H}$ 9.0, $^4J_{P-H}$ 3.0), 7.68 (dd, 2H, 2,6-$\underline{H_2}C_6H_2$, $^3J_{H-H}$ 9.0, $^3J_{P-H}$ 13.5).
10	1.27 (t, 3H, $\underline{CH_3}CH_2OP$, $^3J_{H-H}$ 7.2), 3.69-4.33 (m, 2H, $CH_3\underline{CH_2}OP$), 3.83 (s, 3H, $\underline{CH_3}O$), 6.75 (dd, 2H, 3,5-$H_2C_6\underline{H_2}$, $^3J_{H-H}$ 9.0, $^4J_{P-H}$ 4.0), 7.72 (dd, 2H, 2,6-$\underline{H_2}C_6H_2$, $^3J_{H-H}$ 9.0, $^3J_{P-H}$ 10.0).

TABLE IV

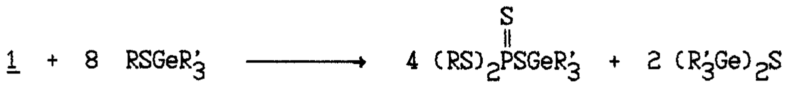
Mass spectral data of the products obtained

Prod.	i-C_4H_{10}, m/e (I_{rel})
3[a]	491 [M]$^{+\cdot}$ (5), 306 [M - 2EtO - PS_2]$^{+\cdot}$ (100).
3[b]	412 [M + H - Ph]$^{+}$ (63), 381 [M + H - Ph - S]$^{+}$ (22), 259 [M + H - 3Ph]$^{+}$ (5).
9a[a]	336 [M - 3Me]$^{+\cdot}$ (15).
9a[b]	368 [M + 2H - S - SEt - $SGeMe_3$]$^{+}$.
9b[a],	234 [M - Bu-i - $GeMe_3$]$^{+\cdot}$ (12), 202 [M - S - Bu-i - $GeMe_3$]$^{+\cdot}$ (73), 170 [M - SBu-i - $SGeMe_3$]$^{+\cdot}$ (17), 138 [M - 3S - Bu-i - $GeMe_3$]$^{+\cdot}$ (75).
9b[b]	365 [M + H - 3Me]$^{+}$ (5), 331 [M + H - Me - 2S]$^{+}$ (33), 290 [M + 2H - Bu-i - 2S]$^{+}$ (35), 258 [M + 2H - Bu-i - 3S]$^{+}$ (100).
10[a]	552 [M]$^{+\cdot}$ (28), 475 [M - EtO - S]$^{+\cdot}$ (47), 443 [M - EtO - 2S]$^{+\cdot}$ (5), 338 [M - $MeOC_6H_4PS$ - OEt]$^{+\cdot}$ (5), 306 [M - $MeOC_6H_4PS$ - OEt - S]$^{+\cdot}$ (100),
10[b]	553 [M + H]$^{+}$ (22), 476 [M + H - EtO - S]$^{+}$ (34), 444 [M + H - EtO - 2S]$^{+}$ (5), 339 [M + H - $MeOC_6H_4PS$ - OEt]$^{+}$ (5), 139 [M + H - 2S - OEt - $GePh_3$]$^{+}$ (34), 108 [M + H - P - 2S - OEt -$GePh_3$]$^{+}$ (41).

[a] Electron impact, 70 eV.

[b] Chemical ionization, 100 eV.

triorganogermanium dialkyl tetrathiophosphates by the reaction of 1 with triaryl or trialkylgermylated thiols. Indeed, we have found that the reaction of 1 with triphenyl(iso-butylthio)germane 5a at 80°C for 0.5 h leads to di-iso-butyl(triphenylgermyl)tetrathiophosphate 6a and 4a (Equation (2), Tables I–IV).



$$\underline{1} + 8\ RSGeR'_3 \longrightarrow 4\ (RS)_2\overset{\overset{S}{\|}}{P}SGeR'_3 + 2\ (R'_3Ge)_2S \qquad (2)$$

5a, R=Bu-i, R'=Ph	6a, R=Bu-i, R'=Ph	4a
b, R=Et, R'=Me	b, R=Et, R'=Me	b, R'=Me
c, R=Bu-i, R'=Me	c, R=Bu-i, R'=Me	

The tetrathiophosphate 6a prepared with this method is a stable compound and was not decomposed during distillation. However, we have been unable to obtain trialkylgermyl homologues 6b,c in pure form as they are thermal unstable compounds. Like dialkyl(trialkylstannyl)tetrathiophosphates[19] the corresponding S-trimethylgermyl dialkyl tetrathiophosphates 6b and 6c (δ_P (C_6H_6) 84.7 and 86.2 ppm, respectively) are formed along with other phosphorus compounds (δ_P (C_6H_6) 78.1 and 78.8 ppm, respectively) detected in the crude reaction mixtures of 1 with trimethyl(ethylthio)germane 5b and trimethyl(iso-butylthio)germane 5c at 20°C for 8–16 h. Perhaps, germyltetrathiophosphates 6b,c are initial reaction products which decompose under the reaction conditions to form secondary products—alkyl bis(trimethylgermyl)tetrathiophosphates 7b,c and trialkyl tetrathiophosphates (Equation (3)).

$$\underline{6b,c} \longrightarrow 0.5\ RS\overset{S}{\overset{\|}{P}}(SGeR'_3)_2 + 0.5\ (RS)_3\overset{S}{\overset{\|}{P}} \qquad (3)$$

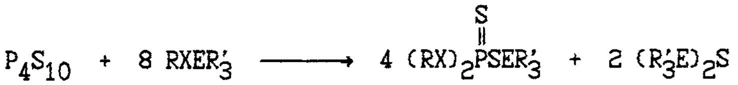
7b, R=Et, R'=Me

c, R=Bu-i, R'=Me

This assumption is confirmed by the mass spectral analysis of a crude reaction mixture of 1 with 5c indicating a mixture of the products of 6c and 7c. The electron impact mass spectrum of the reaction mixture of 1 and 5c shows the mass peaks m/e 405 and 345 which correspond to the fragment *i*-BuSP(S)SGeMe$_3$(SGe) of the product 7c and to the fragment (*i*-BuS)$_2$P(S)SGe of 6c, respectively. Besides, we observed the increase of the intensity of the signal of δ_P (C_6H_6) 78.1 ppm (7b) and the decrease of δ_P (C_6H_6) 84.7 ppm (6b) in the ^{31}P NMR spectra when the reaction mixture of 1 with 5c was heated in a sealed tube at 100°C for 1 h, and at 150°C for 1 h, and at 200°C for 1 h.

Unlike this, S-trimethylsilyl dialkyl tetrathiophosphates are stable compounds.[17] Thus, the thermodynamic stability of silyl, germyl and stannyl tetrathiophosphates decreases in the series: silicon, germanium and tin derivatives. It is remarkable that the reactivity of alkylmercaptides of trialkylsilanes,[17] germanes and stannanes[19] towards 1 (reaction conditions: 50°C, 7 h; 20°C, 8 h; 15°C, 3 h, respectively) increases in the same series.

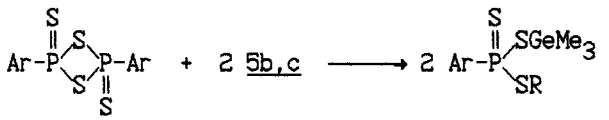
Thus, novel preparative advantageous methods of synthesizing *S*-triorganosilicon, germanium and tin esters of dithio- and tetrathiophosphoric acids were developed on the basis of the reactions of 1 with trialkyl or triaryl(alkoxy)- and (alkylthio)silanes,[16,17] germanes and stannanes.[18,19] A general scheme of these transformations can be conceived (Equation (4)).



$$P_4S_{10} + 8\ RXER'_3 \longrightarrow 4\ (RX)_2\overset{S}{\overset{\|}{P}}SER'_3 + 2\ (R'_3E)_2S \qquad (4)$$

R = Alk; R' = Alk, Ar; X = O, S; E = Si, Ge, Sn

Perhaps, the mechanism of the degradation of 1 with alkoxy- and alkylthiogermanes is similar to those suggested by Roesky and Remmers for the reaction of 1 with trimethylsilyl(dimethyl)amine in which a nucleophilic nitrogen atom attacks a phosphorus atom of 1[10] and the reactions of 1 with alkoxy- and alkylthiosilanes[17]and stannanes.[15] We assumed that during the reaction of 1 with alkoxy- and alkylthiogermanes 2 and 5 four- or six-membered cyclic intermediates may be formed. They may be similar to 2,4-bis(substituted)-2,4-dithioxo-1,3,2λ^5,4λ^5-dithiadiphosphetanes (Davy's or Lawesson's reagents) in their dimer or trimer forms. Consequently, the reactions of Lawesson's reagent with alkoxy- and alkylthiogermanes may be used as a model reaction of some intermediate processes of degradation of 1. As we expected the reaction of Lawesson's reagent 8 with alkylthiogermanes 5b,c has been found to bring about S-trimethylgermyl esters of S-alkyl-4-methoxyphenyl trithiophosphonic acids 9a,c (Equation (5), Tables I–IV).



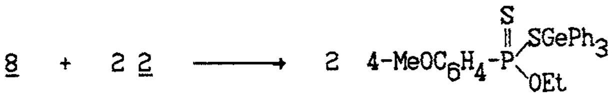
(5)

8, Ar = 4-MeOC$_6$H$_4$

9a, Ar = 4-MeOC$_6$H$_4$, R = Et

b, Ar = 4-MeOC$_6$H$_4$, R = Bu-i

It should be emphasized that formally 9 are the products of the insertion of the monomeric unit, 4-MeOC$_6$H$_4$PS$_2$, of 8 into the S—Ge bond of 5. We extended this convenient method to the reaction of 8 with ethoxygermane 2. This reaction in anhydrous benzene resulted in the formation of O-ethyl S-triphenylgermyl-4-methoxyphenyl phosphonodithioate 10 under mild conditions (20°C, 2 weeks) (Equation (6), Tables I–IV).

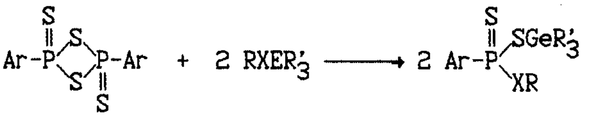


$$8 + 2\ 2 \longrightarrow 2\ 4\text{-MeOC}_6\text{H}_4\text{-P}(\text{=S})(\text{SGePh}_3)(\text{OEt}) \qquad (6)$$

10

We have previously shown that 8 reacts with trialkyl(alkoxy)silanes and (alkoxy)stannanes to form the S-trialkylsilyl and stannyl esters of O-alkyl 4-methoxyphenyl dithiophosphonic acids under mild conditions (15–50°C).[14,15] It is noteworthy that the reactivity of alkoxystannanes towards 8 (reaction conditions: 15°C, 8 h) is higher than that of ethoxygermane 2 (20°C, 2 weeks) and alkoxysilanes (50°C, 2 h).

Thus, on the basis of the results of the reactions of 8 with trialkyl(alkoxy)- and (alkylthio)silanes, germanes and stannanes a general scheme of the fragmentation of the 1,3,2,4-dithiadiphosphetane ring of 8 can be conceived (Equation (7)).



(7)

Ar = 4-MeOC$_6$H$_4$; R = Alk; R' = Alk, Ar; X = O, S; E = Si, Ge, Sn

Thus, the reactions of 1 and 8 with the alkoxy- and alkylthiogermanes 2 and 5 are of interest from the point of view of preparative organothiophosphorus chemistry.

EXPERIMENTAL

^{31}P NMR spectra were recorded with a Bruker MSL-400 (162 MHz) spectrometer, reference external H_3PO_4 (85%). ^1H NMR spectra were run on a Varian T-60 (60 MHz) spectrometer in CCl_4 with $(Me_3Si)_2O$, CH_2Cl_2 and C_6H_6 as internal references. IR spectra were obtained in KBr with a UR-20 spectrophotometer. Mass-spectra (electron impact, 70 eV; chemical ionization, 100 eV) were determined on a M 80 B Hitachi chromato mass spectrometer.

S,S'-Di-iso-butyl-S"-triphenylgermyl tetrathiophosphate 6a. *Typical Procedure.* The mixture of 1 (0.9 g, 2.0 mmol) and 5a (6.8 g, 16.3 mmol) in 10 mL of anhydrous benzene is stirred for 2 h at 80°C. The mixture is evaporated at reduced pressure (0.02 mm Hg) at 40°C for 2 h. 6a (3.0 g, 64%) is isolated from the residue by means of thin layer distillation (see Tables I–IV). 4a (0.5 g, 19%) is isolated similarly; m.p. 137–140°C. ^1H NMR (C_6H_6, δ, ppm): 6.98–7.65 (m, 30 H, C_6H_5). IR (Vaseline oil, ν, cm^{-1}): 480 ν ($GePh_3$ as). Found, %: C 67.46; H 4.34; Ge 22.25; S 5.70. $C_{36}H_{30}Ge_2S$. Calc., %: C 67.56; H 4.74; Ge 22.70; S 5.00.

The product 3 is obtained similarly.

S-Ethyl-S'-trimethylgermyl 4-methoxyphenylphosphonotrithioate 9a. *Typical Procedure.* The mixture of 8 (8.1 g, 20.0 mmol) and 5b (7.2 g, 40.3 mmol) is stirred for 24 h at 20°C. The mixture is filtered. The filtrate is evaporated under vacuum (0.02 mm Hg) at 40°C for 2 h to give crude 9a (12.1 g, 79%). Pure 9a (7.8 g, 51%) is isolated from the residue by means of thin layer distillation (see Tables I–IV).

The products 9b and 10 are obtained similarly (the reactions are performed in anhydrous benzene).

REFERENCES

1. R. A. Cherkasov, G. A. Kutyrev and A. N. Pudovik, *Tetrahedron*, **41**, 2567 (1985).
2. J. L. Lefferts, K. C. Molloy, J. J. Zuckerman, I. Haiduc, C. Guta and D. Ruce, *Inorg. Chem.*, **19**, 1662 (1980) and references therein.
3. B. P. Singh, G. Srivastava and R. C. Mehrotra, *J. Organomet. Chem.*, **171**, 35 (1979) and references therein.
4. R. J. Rao, *Phosphorus, Sulfur, and Silicon*, **53**, 203 (1990).
5. R. J. Rao and H. Wankhade, *Phosphorus, Sulfur, and Silicon*, **70**, 13 (1992).
6. V. V. Ovchinnikov, G. Sh. Malkova, F. Kh. Karataeva and R. A. Cherkasov, *Abstracts of IV Conference on Organometal. Chem.*, (USSR), Kazan, 1988, Part 3, p. 149.
7. W. Zeiss, H. Hunjes, D. Lux, W. Schwarz and H. Hess, *Z. Naturforsch.*, **34b**, 1334 (1979).
8. H. W. Roesky and M. Dietl, *Angew. Chem.*, **85**, 454 (1973).
9. A. Borkowsky, *Dissertation*, Universität Köln (1989).
10. H. W. Roesky and G. Remmers, *Z. Anorg. Allg. Chem.*, **431**, 221 (1977).
11. L. Maier, "Topics in Phosphorus Chemistry" (John Wiley and Sons, New York, 1980), Vol. **10**, pp. 129–169.
12. I. S. Nizamov, L. A. Al'metkina, E. S. Batyeva, V. A. Al'fonsov and A. N. Pudovik, *Izv. Akad. Nauk SSSR. Ser. Khim.*, 2164 (1991).
13. J. Hahn and T. Nataniel, *Z. Anorg. Allg. Chem.*, **543**, 7 (1986).
14. I. S. Nizamov, V. A. Kuznetzov, E. S. Batyeva and V. A. Al'fonsov, *Izv. Rossiiskoi Akad. Nauk. Ser. Khim.*, 2457 (1992).
15. I. S. Nizamov, V. A. Kuznetzov, E. S. Batyeva, V. A. Al'fonsov and A. N. Pudovik, *Heteroatom Chem.*, in print.
16. I. S. Nizamov, V. A. Kuznetzov, E. S. Batyeva, V. A. Al'fonsov and A. N. Pudovik, *Izv. Rossiiskoi Akad. Nauk. Ser. Khim.*, 1933 (1992).
17. I. S. Nizamov, V. A. Kuznetzov, E. S. Batyeva, V. A. Al'fonsov and A. N. Pudovik, *Phosphorus, Sulfur, and Silicon*, **79**, 179 (1993).
18. I. S. Nizamov, V. A. Kuznetzov, E. S. Batyeva, V. A. Al'fonsov and A. N. Pudovik, *Zh. Obshch. Khim.*, **62**, 1665 (1992).
19. I. S. Nizamov, V. A. Kuznetzov, E. S. Batyeva, V. A. Al'fonsov and A. N. Pudovik, *Heteroatom Chem.*, **4**, 379 (1993).