Heteroatom Chemistry
Volume 11, Number 4, 2000

New Methods of Syntheses of Silyl Derivatives of Phosphorus(V) Thioacids on the Basis of Tetraphosphorus Decasulfide, 1,3,2,4-Dithiadiphosphetane-2,4-disulfides, and Bis(trimethylsilyl)acetamide

Il'yas S. Nizamov, Alexey E. Popovich, Elvira S. Batyeva, and Vladimir A. Alfonsov

A. E. Arbuzov Institute of Organic and Physical Chemistry, Russian Academy of Sciences, Arbuzov Str. 8, 420088 Kazan, Russia

Received 2 September 1999; revised 10 February 2000

ABSTRACT: *Mixed O,S-bis- and tris(trimethylsilyl) esters of dithiophosphoric, aryldithiophosphonic and trithiophosphoric acids,* **3**, **7a–c**, *and* **9**, *respectively, were obtained by the reactions of tetraphosphorus decasulfide* **1** *and 1,3,2,4-dithiadiphosphetane-2,4-disulfides* **6a–c** *and* **8** *with bis(trimethylsilyl)acetamide* **2**. © 2000 John Wiley & Sons, Inc. Heteroatom Chem 11:276–280, 2000

INTRODUCTION

S-Trimethylsilyl esters of dithiophosphoric and tetrathiophosphoric acids are efficient intermediates for the synthesis of useful organothiophosphorus compounds [1,2]. There is considerable interest in pentavalent phosphorus thioacids derivatives containing a few $SiMe_3$ groups, due to their possible use as synthetic intermediates for new types of linear and cyclic organothiophosphorus compounds. Tris(trimethylsilyl) tetrathiophosphate and *bis*-(trimethylsilyl) phenyldithiophosphonate containing three or two $S\text{-}SiMe_3$ fragments were earlier obtained in the reactions of bis(trimethylsilyl)sulfide with tetraphosphorus decasulfide and 2,4-diphenyl-1,3,2,4-dithiadiphosphetane-2,4-disulfide, respectively [3]. In this article, new methods are presented for synthesizing di- and trisilyl esters of pentavalent phosphorus thioacids on the basis of the reactions of phosphorus sulfide (P_4S_{10}) and 1,3,2,4-dithiadiphosphetane-2,4-disulfides with disilylated acetamide.

RESULTS AND DISCUSSION

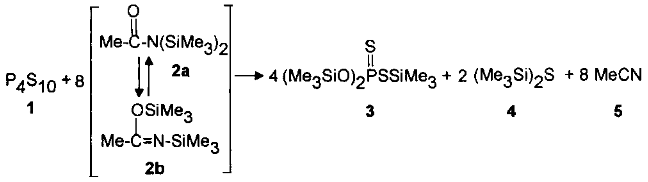
Bis(trimethylsilyl) amides of carboxylic acids have been reported to be efficient silylating agents for amides, ureas, amino acids, hindered phenols, carboxylic acids, enols, chlorides of trivalent and pentavalent phosphorus acids and thioacids [4–9]. Taking into account the rather high silylating ability of silylated amides, we have studied the reactions of disilylated acetamide with phosphorus sulfides. The reaction of tetraphosphorus decasulfide **1** with bis(trimethylsilyl)acetamide **2** that may exist as a mixture of the amide **2a** and imidate **2b** tautomeric forms [4,10–12], at 70–75°C for 2 hours has been found to give *O,O,S*-tris(trimethylsilyl) dithiophos-

---

Correspondence to: Il'yas S. Nizamov.

Contract Grant Sponsor: Russian Foundation for Basic Researches.
Contract Grant Number: 95-03-09739a.

phate **3**, bis(trimethylsilyl)sulfide **4**, and acetonitrile **5** (Reaction 1; Tables 1–5).



Product **3** is a colorless liquid and was isolated by use of a falling-film distillation. Reaction was accompanied by the formation of bis(trimethylsilyl) sulfide **4** and acetonitrile **5**, which were removed from the reaction mixtures by evaporation at reduced pressure. The formation of **4** and **5** were confirmed by ^1H NMR and mass spectral data of volatile distilled fractions and by comparison of their physical constants with data found in the literature [13,14].

TABLE 1 Experimental Data and Yields of the Products Obtained

Initial Compounds Quantity [g (mmol)]		*Reaction Conditions Temp. (°C) /Time (h)*	*Product Yield, g(%)*
1 9.6 (21.6)	/**2** 31.5 (172.9)	70–75/2	**3** 20.8 (69)[b]
6a 9.7 (24.0)	/**2** 9.8 (48.2)	20/7 days	**7a** 12.0 (68)[b] **5** 1.9 (95)[b]
6b 11.6 (26.9)	/**2** 10.9 (53.6)	50/2.5	**7b** 16.4 (81)[b] **5** 2.0 (91)[b]
6c 12.0 (20.0)	/**2** 8.1 (39.9)	20/5	**7c** 8.0 (43)[a]/6.2 (34)[b]
8 3.8 (10.3)	/**2** 4.2 (20.7)	30–40/5	**9** 6.1 (86)[a]

[a] Yield of crude product.
[b] Yield of product isolated by a falling-film distillation.

TABLE 2 Physical, Analytical, and ^{31}P NMR Data of the Products Obtained

					Found/Calc. (%)					
Product	*b.p. °C (mmHg)*[a]	d_4^{20}	n_D^{20}	*Molecular Formula (Mol. mass)*	*C*	*H*	*P*	*S*	*Si*	^{31}P *NMR* $\delta(C_6H_6)$
3	135–140 (0.02)	1.0196	1.4861	$C_9H_{27}O_2PS_2Si_3$	31.35	7.86	8.82	18.79	24.40	58.4
				(346.1)	31.20	7.88	8.95	18.47	24.85	
7a	125–130 (0.02)	1.1129	1.5564	$C_{13}H_{25}O_2PS_2Si_2$	42.67	6.77	8.43	17.59	15.28	75.2
				(364.1)	42.84	6.93	8.51	17.56	15.37	
7b	130–140 (0.02)	1.0726	1.5465	$C_{14}H_{27}O_2PS_2Si_2$	44.23	7.27	7.82	17.14	13.81	75.3
				(378.1)	44.43	7.21	8.19	16.91	14.80	
7c	170 (0.03)		1.5223	$C_{20}H_{39}O_2PS_2Si_2$			6.68		12.11	75.6
				(462.3)			6.70		12.11	
9	140–150 (0.02)[b]		1.5068	$C_{10}H_{27}OPS_3Si_2$	34.99	7.98	8.32	28.56	15.43	58.4
				(346.1)	34.67	7.68	8.95	27.71	16.17	(CCl_4)

[a] Temperature of thermal element of a falling-film distillation apparatus.
[b] Temperature of partial destruction.

TABLE 3 IR Data of the Products Obtained

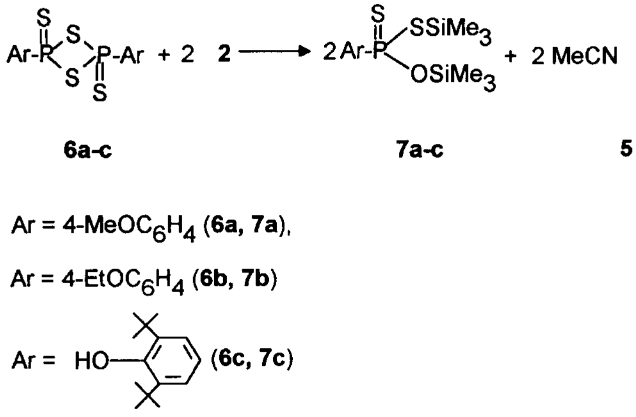
Product	ν, *cm*$^{-1}$
3	2970 ν (CH_3 as); 2910 ν (CH_3 s); 1260 δ [CH_3(Si)s]; 1030 ν (P–O–Si as); 850 ρ (CH_3); 770 ν (P–O–Si s); 647 ν (P=S); 585, 500 ν (P–S, S–Si).
7a	3075 ν (:CH, Ar); 2970, 2910, 2845 ν [(CH_3(O) s), (CH_3(Si)s]; 1598, 1440 ν (C:C, Ar); 1260 δ [CH_3(Si)s]; 1030, 1010 ν (P–O–Si as, SiO–C); 860 ρ (CH_3); 770 ν (P–O–Si s); 627 ν (P=S); 530; 515 ν (P–S, S–Si).
7b	3070 ν (:CH, Ar); 2985, 2967, 2805 ν [CH_3 as, s; CH_2 as, s; (CH_3(Si) s]; 1598 ν (C:C, Ar); 1258 δ [CH_3(Si) s]; 1030, 1005 ν (P–O–Si as, SiO–C); 770 ν (P–O–Si s); 630 ν (P=S); 540, 515 ν (P–S, S–Si).
7c	3630 ν (OH); 3040, 3000 ν (:CH, Ar); 2965, 2910, 2880 [CH_3 as, s; (CH_3(Si) s]; 1580, 1480 ν (C:C, Ar); 1429 δ (CH_3 as); 1365 δ (CH_3 s); 1255 δ [CH_3(Si) s]; 1010 (P–O–Si as); 855 ρ [CH_3(Si)]; 768 ν (P–O–Si s); 700; 650 ν (P=S); 555, 500, 457 ν (P–S, S–Si).
9	1260 δ [CH_3(Si) s]; 1383, 1370 δ (Me_2C gem s); 1030 ν (P–O–Si as); 770 ν (P–O–Si s); 640 ν (P=S, PS_2 as).

TABLE 4 ^{1}H NMR Data of the Products Obtained

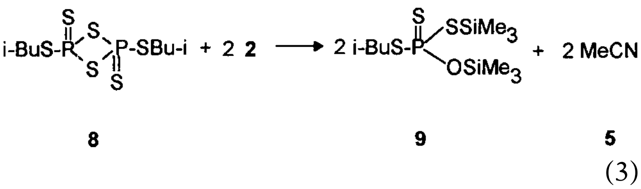
Product	*CCl_4, δ, J [Hz]*
3	0.37 (s, 18H, $\underline{CH}_3SiO$); 0.42 (s, 9H, $\underline{CH}_3SiS$).
7a	0.37 (s, 9H, $\underline{CH}_3SiO$); 0.43 (s, 9H, $\underline{CH}_3SiS$); 3.85 (s, 2H, $\underline{CH}_3OC_6H_4$); 6.87 (d. d, 2H, 3,5-$H_2C_6\underline{H}_2$, $^3J_{HH}$ 9.0, $^4J_{PH}$ 4.0); 7.87 (d. d, 2H, 2,6-$\underline{H}_2C_6H_2$, $^3J_{HH}$ 9.0, $^3J_{PH}$ 15.0).
7b	0.35 (s, 9H, $\underline{CH}_3SiO$); 0.37 (s, 9H, $\underline{CH}_3SiS$); 1.45 (t, 3H, $\underline{CH}_3CH_2O$, $^3J_{HH}$ 7.0); 4.07 (q, 2H, $CH_3\underline{CH}_2O$, $^3J_{HH}$ 7.0); 6.87 (d. d, 2H, 3,5-$H_2C_6\underline{H}_2$, $^3J_{HH}$ 9.0, $^4J_{PH}$ 4.0); 7.86 (d. d, 2H, 2,6-$\underline{H}_2C_6H_2$, $^3J_{HH}$ 9.0, $^3J_{PH}$ 14.5).
7c[a]	0.43 (s, 9H, $\underline{CH}_3SiO$); 0.50 (s, 9H, $\underline{CH}_3SiS$); 1.52 (s, 18H, $(\underline{CH}_3)_3C$); 5.45 (s, 1H, $\underline{OH}$); 8.13 (d, 2H, 2,6-$\underline{H}_2C_6$, $^3J_{PH}$ 16.2).
9	0.33 (s, 9H, $\underline{CH}_3SiO$); 0.43 (s, 9H, $\underline{CH}_3SiS$); 1.07 (d, 6H, $\underline{CH}_3CHCH_2SP$, $^3J_{HH}$ 7.5); 2.77 (d. q, 2H, $CH_3CH\underline{CH}_2SP$, $^3J_{HH}$ 7.5, $^3J_{PH}$ 16.0).

[a] In C_6D_6.

The formation of products containing both $OSiMe_3$ and $SSiMe_3$ groups could be expected in the case of 2,4-diaryl-1,3,2,4-dithiadiphosphetane-2,4-disulfides. In fact, the reaction of dithiadiphosphetanes **6a–c** with disilylacetamide **2** at 20–50°C for 2.5 to 5 hours (20°C for 7 days in the case of **6b**) has brought about the formation of *O,S*-bis(trimethylsilyl) aryldithiophosphonates **7a–c** with the elimination of MeCN **5** (Reaction 2, Tables 1–5). *O,S*-Disilyl dithiophosphonates **7a,b** are pale-yellow liquids, whereas **7c** is a yellow oily liquid. All of the compounds **7a–c** were purified by use of a falling-film distillation.



(2)



(3)

In continuation of our study, we have managed to involve other 1,3,2,4-dithiadiphosphetane-2,4-disulfides, for example, the isobutyl homolog of Davy's reagent **8**, in the reaction with disilylacetamide **2** at 30–40°C for 5 hours (Reaction 3, Tables 1–5). *O,S*-Bis(trimethylsilyl)*S′*-isobutyltrithiophosphate **9** was formed in this reaction, as depicted in Reaction 3.

The structures of **3**, **7a–c**, and **9** were confirmed by IR (Table 3), ^{1}H (Table 4), and ^{31}P NMR (Table 2) and mass spectroscopy (Table 5), as well as by elemental analyses (Table 2). The ^{31}P NMR spectrum of *O,O,S*-trisilyl dithiophosphate **3** in benzene solution reveals a singlet at $\delta = 58.4$, which is intermediate between those of tris(trimethylsilyl) tetrathiophosphate ($\delta = 65.3$ [3]) and *O,O,O*-tris(trimethylsilyl) thionophosphate ($\delta = 32$ [15]). The ^{31}P chemical shift values of *O,S*-disilyl dithiophosphonates **7a–c** are shifted to low field ($\delta = 75.2$–75.6) with respect to that of **3**. It is noteworthy that the ^{31}P resonance of *O,S*-disilyl trithiophosphate **9** ($\delta = 58.4$ in CCl_4 solution) appears in practically the same region as that for O,O,S-trisilyl dithiophosphate **3**.

The ^{1}H NMR spectrum of **3** in CCl_4 solution (Table 4) shows two singlets at $\delta = 0.37$ and $\delta = 0.42$ due to the methyl protons of two $SiMe_3$ groups at the oxygen and the sulfur atoms, respectively, in the integral intensity ratio 2:1. A similar picture was observed in the case of **7a–c** and **9** but the integral intensity ratio of these singlets was 1:1 (see Table 4). This fact seems to confirm the absence of a tautomeric transfer of $SiMe_3$ groups in the molecules of **3**, **7**, and **9** at room temperature. The ^{1}H NMR spectra of **7a–c** and **9** also show characteristic resonances due to the presence of aryl or isobutylthio groups attached to the phosphorus atom. Thus, a doublet of doublets at $\delta = 6.87$ is assigned to two ortho protons of the 3,5-$H_2C_6\underline{H}_2$ group ($^3J_{HH} = 9.0$, $^4J_{PH} = 4.0$), while a doublet of doublets at $\delta = 7.87$ is attributed to the meta protons of the 2,6-$\underline{H}_2C_6H_2$ group ($^3J_{HH} = 9.0$, $^3J_{PH} = 15.0$) of **7a**. The protons of the methoxy group of the 4-$MeOC_6H_4$ substituent of **7a** resonate as a singlet at $\delta = 3.85$, whereas the protons of the ethoxy group of the 4-$EtOC_6H_4$ substituent of **7b** appear as a triplet at $\delta = 1.45$ and a quartet at $\delta = 4.07$ ($^3J_{HH} = 7.0$). In the case of **7c**, two singlets situated

TABLE 5 Mass Spectral Data of the Products Obtained

Product	*i*-C_4H_{10}, *m/e* (I_{rel}, %)
3[a]	274 $[M + H - Me_3Si]^+$ (16); 259 $[M + 2H - Me_3Si - O]^+$ (50).
3[b]	331 $[M - Me]^{+\cdot}$ (10); 316 $[M - 2\,Me]^{+\cdot}$ (100); 301 $[M - 3Me]^{+\cdot}$ (10); 241 $[M - Me_3Si - 2S]^{+\cdot}$ (100)
7a[a]	365 $[M + H]^+$ (19); 292 $[M + H - Me_3Si]^+$ (5); 293 $[M + 2\,H - Me_3Si]^+$ (100)
7a[b]	349 $[M - Me]^{+\cdot}$ (100); 334 $[M - 2\,Me]^{+\cdot}$ (2); 259 $[M - Me_3Si - S]^{+\cdot}$ (100)
7b[a]	307 $[M + 2H - Me_3Si]^+$ (100); 307 $[M + H - Me_3Si]^+$ (13); 274 $[M + H - Me_3Si - S]^{+\cdot}$ (100); 245 $[M + H - Me_3Si - S - Et]^{+\cdot}$ (15)
7b[b]	378 $[M]^{+\cdot}$ (5); 363 $[M - Me]^{+\cdot}$ (13); 273 $[M - Me_3Si - S]^{+\cdot}$ (100)
7c[a]	464 $[M + 2H]^+$ (30); 463 $[M + H]^+$ (3)
7c[b]	448 $[M - Me]^{+\cdot}$ (40); 432 $[M - 2Me]^{+\cdot}$ (100); 417 $[M - 3Me]^{+\cdot}$ (3)

[a] Chemical ionization, 100 eV.
[b] Electron impact, 70 eV.
[c] For Z = 3,5-ditret.-Bu-4-HO-C_6H_2

at $\delta = 1.52$ and 5.42 were assigned to the protons of the *tert*-butyl and hydroxy groups, respectively. The *meta* protons of the 2,6-$\underline{H}_2C_6$ aryl group of **7c** appear as a doublet at $\delta = 8.13$ ($^3J_{PH} = 16.2$). The ^{1}H NMR spectrum of **9** shows a doublet at $\delta = 1.07$ and a doublet of doublets at $\delta = 2.77$ due to the methyl and methylene protons of the iso-butylthio group $(\underline{CH}_3)_2CHCH_2SP$ and $(CH_3)_2CH\underline{CH}_2SP$, respectively ($^3J_{HH} = 7.5$, $^3J_{PH} = 16.0$).

Bands in the region ν 1030–1010 and 770–768 cm^{-1} in the IR spectra of **3**, **7**, and **9** (Table 3) are due to the asymmetrical and symmetrical valence vibrations, respectively, of the P–O–Si bonds. Bands of the valence vibrations of the S–Si bonds are mixed with those of the P–S bonds and appeared in the range of ν 585–530 and 515–500 cm^{-1} of **3** and **7**.

The electron impact mass spectrum of **3** (Table 5) exhibits the mass peaks *m/e* 331, 316, and 301 due to the ions $[M-Me]^{+\cdot}$ (10%), $[M-2Me]^{+\cdot}$ (100%) and $[M-3Me]^{+\cdot}$ (10%), respectively (calculated molecular mass M of **3** is 346.1). The chemical ionization and electron impact mass spectra of **7a**, **7b**, and **7c** show the mass peaks *m/e* 365, 378, and 463 of their molecular ions $[M+H]^+$, $[M]^{+\cdot}$ and $[M+H]^+$, respectively. The mass peaks *m/e* 349, 367, and 448 were also observed in the electron impact mass spectra of **7a**, **7b**, and **7c**, respectively, those being attributable to the ions $[M-Me]^{+\cdot}$. Thus, the Me fragment is split out from the molecules of **3** and **7** under the conditions of recording of the mass spectra. The mass peaks *m/e* 274, 292, and 307 in the chemical ionization mass spectra of **3**, **7a**, and **7b**, respectively, are due to ions $[M+H-Me_3Si]^+$ and $[M+2H-Me_3Si]^+$.

The thermal stability of *O,O,S*-trisilyl dithiophosphate **3** containing the mixture of the $OSiMe_3$ and $SSiMe_3$ groups is not very high. The thermal stability of *O,S*-disilyl dithiophosphonates **7** containing stable P–C bonds is higher than that of **3**. *O,S*-Bis(trimethylsilyl)*S′*-isobutyltrithiophosphate **9** containing the P–S-alkyl group tends to partially decompose at high temperatures and when a falling-film distillation is used in attempted purification at the temperature of the thermal element of the apparatus, 140–150°C (0.02 mmHg).

As we can see, reactions 1, 2, and 3 were accompanied by the elimination of MeCN **5**, indicating a possibility of formation of some 1:1 intermediate adducts of **1** or 1,3,2,4-dithiadiphosphetane-2,4-disulfides **6** and **8** with starting disilylacetamide **2** containing the structural fragments of **2**. The thermal stability of these intermediate addition products seems to be rather low, so these adducts were not isolated. However, the electron impact and chemical ionization mass spectra of the crude reaction mixtures of **6a**, **6b**, and **6c** with **2** reveal the mass peaks *m/e* 389, 391, 419, and 503, 504 that may be assigned to the molecular ions $[M]^{+\cdot}$ (2%), $[M+H]^+$ (6%), $[M']^{+\cdot}$ (6%), $[M'']^{+\cdot}$ (6%) and $[M''+H]^+$ (30%), respectively, of intermediate products of the addition of monomeric units, Z-PS_2, of **6a–c** to **2** (calculated molecular masses M of these adducts are 389.2 (Z = 4-$MeOC_6H_4$), 419 (Z = 4-$EtOC_6H_4$) and 503.3 (Z = 3,5-ditret.-Bu-4-HO-C_6H_2).

Thus, we have established that tetraphosphorus decasulfide and 1,3,2,4-dithiadiphosphetane-2,4-disulfides react with disilylated acetamide to form disilyl esters of phosphorus(V) thioacids containing both $OSiMe_3$ and $SSiMe_3$ groups. We have proposed that the O,S-bis(trimethylsilyl) aryldithiophosphonates **7** obtained may be used as intermediates for the synthesis of a series of heterocyclic organothiophosphorus compounds. These studies are in progress.

EXPERIMENTAL

General Data

The ^{31}P NMR spectra were recorded with a Bruker MSL 400 (162 MHz) instrument in C_6H_6 with 85%

H_3PO_4 as an external reference. The ^1H NMR spectra were taken on a Bruker MSL-400 (400 MHz) spectrometer and a Varian T-60 (60 MHz) spectrometer in C_6D_6 or CCl_4 with $(Me_3Si)_2O$ as an internal reference. The IR spectra were obtained in KBr pellets with an UR-20 infrared spectrophotometer and a Bruker IFS 113v spectrometer. Mass spectra (EI, 70 eV; CI, 100 eV) were determined on an M 80 B Hitachi chromatomass spectrometer.

O,O,S-Tris(trimethylsilyl) Dithiophosphate **3**. Compound **1** (9.6 g, 21.6 mmol) was added portionwise under dry argon with stirring at 20°C to 35.1 g (172.9 mmol) of **2**. The mixture was heated at 70–75 °C for 2 hours with stirring. The mixture was cooled and evaporated at reduced pressure (0.5 and 0.02 mmHg) at 40°C for 2 hours. Product **3** (20.8 g, 69%) was isolated from the residue by means of a falling-film distillation (see Tables 1–5).

Reaction of Lawesson's Reagent **6a** *with Bis(trimethylsilyl)acetamide* **2**. Compound **6a** (9.7 g, 24.0 mmol) was added portionwise under dry argon with stirring at 20°C to 9.8 g (48.2 mmol) of **2**. The mixture was stored at ~20°C for 7 days and evaporated under vacuum (0.5 and 0.01 mmHg) at 40°C for 2 hours with use of a trap cooled by liquid nitrogen. Product **7a** (12.0 g, 68%) was isolated from the residue by means of a falling-film distillation (see Tables 1–5). Distillation of the contents of the liquid nitrogen trap gave **5** (1.9 g, 95%), b.p. 82–83°C, n_D^{20} 1.3449 (cf. lit. [11] b.p. 81–82°C, n_D^{20} 1.3440). The ^1H NMR (CCl_4) δ 1.94 (s, 3H, $\underline{CH_3}$).

Distilled **7a** and **7c** and crude **9** were obtained similarly (see Tables 1–5).

REFERENCES

[1] Cherkasov, R. A.; Kutyrev, G. A.; Pudovik, A. N. Tetrahedron 1985, 41, 2567–2624.
[2] Nizamov, I. S.; Kuznetzov, V. A.; Batyeva, E. S. Phosphorus Sulfur Silicon 1994, 90, 249–257.
[3] Roesky, H. W.; Remmers, G. Z. Anorg Allg Chem 1977, 431, 221–226.
[4] Klebe, J. F.; Finkbeiner, H.; White, D. M. J Am Chem Soc 1966, 88, 3390–3398.
[5] Pudovik, M. A.; Kibardina, L. K.; Pestova, T. A.; Medvedeva, M. D.; Pudovik, A. N. Zh Obshch Khim 1975, 45, 1568.
[6] Pudovik, A. N.; Pudovik, M. A.; Kibardina, L. K.; Medvedeva, M. D. USSR Pat. 52569 Bull Izobr Russ 1976, 31.
[7] Pudovik, M. A.; Kibardina, L. K.; Pudovik, A. N. Zh Obshch Khim 1986, 56, 1908.
[8] Al'fonsov, V. A.; Pudovik, M. A.; Nizamov, I. S.; Trusenyov, A. G.; Batyeva, E. S.; Pudovik, A. N. Izv Akad Nauk SSSR Ser Khim 1987, 2868.
[9] Pudovik, M. A.; Kibardina, L. K.; Pudovik, A. N. Zh Obshch Khim 1991, 61, 1058–1063.
[10] Pump, J.; Rochow, E. G. Chem Ber 1964, 97, 627–635.
[11] Yoder, C. H.; Bonelli, D. Inorg Nucl Chem Lett 1972, 8, 1027.
[12] Yoder, C. H.; Copenhafer, W. C.; DuBeshter, B. J Amer Chem Soc 1974, 96, 4283–4286.
[13] Bažant, V.; Chvalovský, V.; Rathouský, J. Organosilicon Compounds; Publishing House of the Czechoslovak Academy of Sciences: Prague, 1965; Vol. 2(2), p. 195.
[14] Beilsteins Handbuch der Organischen Chemie; Berlin, 1920; Bd. 2. S. 183–184.
[15] Varnavskaya-Samarina, O. A.; Ishmaeva, E. A.; Romanov, G. V.; Nazmutdinov, R. Ya.; Remizov, A. B.; Pudovik, A. N. Izv Akad Nauk SSSR Ser Khim 1978, 363–369.