Ketene organoelement derivatives. Synthesis of bis(1-R-2-oxovinyl)silanes and germanes (R = SiMe₃, GeMe₃, SnMe₃)

S. V. Gruener, * R. N. Ezhov, and V. S. Petrosyan

Department of Chemistry, M. V. Lomonosov Moscow State University, 1 Leninskie Gory, 119992 Moscow, Russian Federation. Fax: +7 (495) 939 5546. E-mail: svgrun@org.chem.msu.ru

Earlier unknown 1,3-bisketene organoelement derivatives $RMeE(C(E'Me_3)=C=O)_2$ (E = Si, Ge; E' = Si, Ge, Sn) have been synthesized by the reaction of bis(alkoxyethynyl)silanes and -germanes with Me₃E'Hal. Bis(1-trimethylsilyl-2-oxovinyl)silane is also formed by the reaction of trimethylsilyl- and (dimethylsilylene)bisketenes with dimethylsilylene bistriflate and trimethylsilyl triflate, respectively. Addition of nucleophiles to the ketene fragment of compounds synthesized has been studied.

Key words: ketenes, alkynyl ethers, organosilicon compounds, organogermanium compounds, organotin compounds.

In continuation of our studies on the properties of bis(alkoxyethynyl)silanes and -germanes,¹⁻³ we studied reactions of the latter with Me₃E'Hal (E' = Si, Ge, Sn). The present work is aimed at obtaining new types of organoelement bisketenes, the first stable representative of which, dimethylsilylenebis(ketene), has been synthesized relatively not long ago.^{1,2}

It is known⁴⁻⁶ that the reaction of silyl(germyl)monoalkoxyacetylenes with Me_3E 'Hal leads to the corresponding biselement-substituted ketenes. We studied reactions of bis(alkoxyethynyl)silanes and -germanes 1 with trimethylsilyl-, trimethylgermyl- and trimethylstannyl iodides 2-4 (Scheme 1). The reaction was carried out by stirring of the reagents in dichloromethane or acetonitrile, which resulted in the corresponding organoelement bisketenes 5 and 6 in good yields.

Unlike the reaction of compounds 2-4 with silicon and germanium monoalkoxyethynyl derivatives,⁴⁻⁶ similar reaction of 1a-e proceeds considerably slower (24–290 h) and requires significant excess of Me₃E'I (>50%) (Table 1).

Initially the reaction presumably proceeds at one of the alkoxyethynyl groups, which was clearly shown by the reaction of **1a** with **2*** in dichloromethane. Monitoring of the reaction course was carried out using the IR spectra of the reaction mixture, namely, from the decrease in intensity of the absorption band for the C=C bond (2190 cm⁻¹) and increase in intensity of the absorption band for the

* Iodotrimethylsilane (2) is an excellent silylating agent for organic carbonyl compounds.⁷

Scheme 1







E⁻ = Si (2, 5a, 6a,c), Ge (3, 6b,d), Sn (4, 5g, 6f)

Com-	Е	R	R´	Com-	Е	R	R´
pound				pound			
1a, 5a, 6a	Si	Me	Me	1e, 6e	Ge	Me	Et
1b, 6b	Si	Me	Et	1e, 6f	Ge	Me	Et
1c, 6c	Si	н	Me	1b, 5g	Si	Me	Et
1d. 6d*	Si	н	Ft				

* Reaction with bromotrimethylgermane.

ketene group in the region $2100-2050 \text{ cm}^{-1}$. Though, the band for the C=C bond almost disappeared after stirring for 88 h, it was unambiguously shown (¹H, ¹³C, and

Published in Russian in Izvestiya Akademii Nauk. Seriya Khimicheskaya, No. 4, pp. 789-793, April, 2009.

1066-5285/09/5804-0805 © 2009 Springer Science+Business Media, Inc.

²⁹Si NMR spectroscopy) that the product distilled *in vacuo*, along with bisketene **6a**, contained \sim 30% of compound with a single ketene group (**5a**).

The IR spectrum of a distilled mixture of compounds **5a** and **6a** exhibits intensive absorption bands at 2190 cm^{-1} (the C=C bond in 5a) and 2095 cm⁻¹ (C=C=O in 5a and 6a), with intensity of the C=C absorption band being much lower than that for C=C=O. The ¹H NMR spectrum, in addition to two singlets at δ 0.13 (Me₃Si-) and 0.25 (Me₂Si–), contains the singlet at δ 3.13 corresponding to the –OMe group in compound 5a. The ¹³C NMR spectrum, in addition to chemical shifts for the carbon atoms in C=C=O (see Ref. 8) in compound 5a, also exhibits signals typical of the carbon atoms of the fragment C=C-OMe. The ²⁹Si spectrum for **5a** contains the resonance signals at δ -21.0 (Me₂Si-) and 20.9 (Me₃Si-), whereas for **6a**, at δ -3.3 (Me₂Si-) and 0.3 (Me₃Si-). A mixture of compounds 5a and 6a was isolated by distillation, rather than a mixture of **1a** and **6a**, which was confirmed by the ²⁹Si NMR spectral data (for $1a \delta - 38.7^{1}$).

In another experiment (a large excess of **2** and longer reaction time), only bisketene **6a** was isolated in 56% yield. When the reaction was run in acetonitrile with molar ratio of reagents 1 : 3, its yield increased (70%) and the reaction time noticeably decreased (see Table 1). Note that in the IR spectrum, there are observed two absorption bands of equal intensity characteristic of the ketene group vibrations, at 2082 and 2100 cm⁻¹. Compounds **6b** and **6c** were synthesized similarly in 27 and 73% yields, respectively.

Further, we studied reaction of acetylene 1d with bromotrimethylgermane. The reaction was carried out in acetonitrile with a large excess of bromotrimethylgermane (50%) (monitoring by the IR spectra of the reaction mixture). In contrast to 1a, the reaction proceeds significantly slower and comes to completion after 528 h (the yield of compound 6d was 48%).

The reaction of compounds **1e** and **3** took place under the same conditions and with the same amounts of starting reagents that in the reaction of **1a** with **2**. The full completion of the reaction required 168 h. In this case, the reaction proceeds considerably faster than the germylation of **1d** with bromotrimethylgermane. Similar reactions were run between **1b**,e and **4**. In case of **1b**, we failed in carrying out the reaction to completion, despite a prolonged (380 h) stirring with a 2-fold excess of **4**. The IR spectrum of the reaction mixture exhibits the absorption bands for acetylene (2180 cm⁻¹) and ketene groups (2050 cm⁻¹). Fractional distillation afforded the product bearing only one ethoxyethynyl group in **5g** (see Scheme 1).

Unlike the reaction of **1b**, the reaction between **1e** and **4** proceeds to completion to form product **6f** (monitoring by the IR spectra of the reaction mixture).

The experimental data obtained can be explained by the fact that in the intermediate product 5g formed, the trimethylstannyl group creates steric hindrance to the attack by the second molecule **4** on the remained ethoxyethynyl group. In the case of **1e**, such hindrance apparently is absent, since the Ge–C= bond distance in **1e** is longer than the Si–C= bond distance in **1b** and the Me₃Sn– group in **1e** does not create steric hindrance.

The reaction of the partial pyrolysis product 1b (see Ref. 1), dimethyl(ethoxyethynyl)silyl ketene (7b), using excess iodotrimethylgermane gave monogermylated bisketene 8 (Scheme 2).

Scheme 2



Conditions for the synthesis of compounds 5a,g, 6a-f, and 8 are given in Table 1.

Molecular structures of compounds **6a**,**b**,**f** were studied by electron diffraction in the gas phase. Conformation analysis was performed using *ab initio* quantum chemical methods and DFT. The theoretical calculations in agreement with the experiment show that compounds under study exist as two conformers of the type *gauche-gauche* and *syn-gauche* relatively to the ketenyl groups.⁹

In the literature, there is described¹⁰ an original way to synthesize bis(trimethylsilyl) ketene by the reaction of trimethylsilyl triflate with trimethylsilyl ketene in the presence of triethylamine. Earlier, we have found¹¹ that reactions of monosilyl ketenes with trialkylsilyl triflates bearing bulky substituents (both in ketenes and in triflates) lead either to the corresponding bis(silyl) ketenes or to a mixture of these ketenes with isomeric silyl ynol ethers. The latter are capable of a slow isomerization into the corresponding bis(silyl) ketenes. These both classes of

Table 1. Synthesis of compounds 5a, 5g, 6a-f, and 8

Com- pound	Starting reagents	Sol- vent	Excess of reagents 2-4 (%)	Reaction time (h)
5a + 6a	1a + 2	CH ₂ Cl ₂	25	88
6a	1a + 2	CH_2Cl_2	50	115
		MeCN	50	24
6b	1b + 3	MeCN	45	220
6c	1c + 2	MeCN	56	290
6d	$1d + Me_3GeBr$	MeCN	50	528
6e	1e + 3	MeCN	50	168
6f	1f + 4	MeCN	90	180
5g	1b + 4	MeCN	100	380
8	7 + 3	MeCN	50	200

compounds are important objects for study in organoelement chemistry.^{12–14} Their relative thermodynamic stability and the use of these compounds in organic and organoelement syntheses are of special interest.^{15,16}

All of this prompted us to study reactions of dimethylsilylenebis(ethylenone) $(9)^{1,2}$ with trimethylsilyl triflate (10) (ratio of the reagents, 1 : 2) in order to explore alternative ways for the synthesis of **6a** and a possible formation of isomeric to it bis(trimethylsiloxyethynyl)dimethylsilane (11) (Scheme 3).



The reaction of compounds 9 and 10 could have been expected to result in the formation of compound 11 with further isomerization into 6a. The reaction was carried out in diethyl ether at -10 °C with subsequent rising the temperature to ambient. Monitoring of the reaction was performed using the IR and ¹³C NMR spectra of the reaction mixture. In 24 h after mixing of the reagents, the IR spectrum exhibited two absorption bands: for the C=C bond at 2190 cm⁻¹ (of medium intensity) and for the ketene group at 2090 cm⁻¹. However, in the ¹³C NMR spectrum of the reaction mixture aliquot, the signals characteristic of 11 were not observed. On standing of the reaction mixture (20 °C), the absorption band at 2190 cm⁻¹ decreased down to entire disappearance after 16 h. Fractional distillation of the reaction mixture afforded compound 6a only.

The reaction of trimethylsilyl ketene (12) with bis-(trifluoromethanesulfonyloxy)dimethylsilane (13) (ratio of reagents, 2 : 1) (see Scheme 3) occured similarly: in the IR spectrum of the reaction mixture (in 24 h after mixing of the reagents), two absorption bands were also recorded, for the C=C bond at 2190 cm⁻¹ and intensive absorption band for the ketene group at 2090 cm⁻¹. Fractional distillation of the reaction mixture also gave compound **6a** only. In both cases, compound **6a** was isolated in low yield (15 and 9%, respectively).

Thus, it can be only suggested that if compound **11** is formed in these reactions, then only as an unstable inter-

mediate, which partially decomposes and partially isomerizes into 6a, which explains the low yield of the ketene isolated.

We have studied reactions of ketenes obtained with nucleophiles. For instance, the reaction of compound **6b** with 2 equiv. of benzylamine in hexane gives N,N'-dibenzyl-2,2'-(dimethylsilylene)bis[2-(trimethylgermyl)acet-amide] (**14**) in good yield (Scheme 4).





The reaction of bisketene **6b** with excess water in dioxane in the presence of catalytic amount of H_2SO_4 gave

Table 2. Characteristics of compounds synthesized

Com- pound	Yield (%)	B.p./°C (<i>p</i> /Torr)	<u>Found</u> Calculat	ed (%)	Molecular formula
		[m.p./°C]	С	Н	
6a	56	45-46	<u>50.56</u>	<u>8.59</u>	C ₁₂ H ₂₄ O ₂ Si ₃
		(0.02)	50.56	8.50	
6b	73	68	<u>38.18</u>	<u>6.42</u>	C ₁₂ H ₂₄ O ₂ Ge ₂ Si
		(0.02)	38.58	6.48	
6c	27	43	<u>46.25</u>	<u>7.93</u>	$C_{11}H_{22}O_2Si_3$
		(0.02)	46.72	8.49	
6d	48	59	<u>37.43</u>	<u>6.29</u>	$C_{11}H_{22}O_2Ge_2Si$
		(0.02)	36.97	6.16	
6e	38	71-72	<u>34.12</u>	<u>5.75</u>	$C_{11}H_{24}O_2Ge_3$
		(0.02)	34.47	5.79	
6d	73	95-98	28.65	<u>4.94</u>	$C_{11}H_{24}O_2GeSn_2$
		(0.02)	28.18	4.69	
5g	22	73	<u>39.45</u>	<u>5.98</u>	C ₁₁ H ₂₀ O ₂ SiSn
		(0.02)	39.91	6.09	11 20 2
8	77	39	46.80	<u>6.08</u>	C ₉ H ₁₆ O ₂ GeSi
		(22)	46.36	6.05	, <u>-</u>
14*	62	[104-105]	<u>53.33</u>	<u>7.45</u>	C ₂₆ H ₄₂ N ₂ O ₂ Ge ₂ Si
			53.15	7.20	
15	40	[133-135]	<u>36.23</u>	<u>6.81</u>	C ₁₁ H ₂₈ O ₄ Ge ₂ Si
			36.81	6.69	

* Found (%): N, 4.73. Calculated (%): N, 4.76.

Com-	IR, v/cm ⁻¹	NMR, δ				
pound		¹ H	¹³ C, { ²⁹ Si}, [¹¹⁹ Sn]			
6a	2082, 2100	0.26 (s, 6 H, Me ₂ Si); 0.14 (s, 18 H, Me ₃ Si)	1.1 (Me ₃ Si); 2.4 (Me ₂ Si); 2.7 (<u>C</u> =C=O); 167.5 (C= <u>C</u> =O); {-3.3 (Me ₂ Si)}; {0.3 (Me ₃ Si)}			
5a + 6a	2190, 2095	0.20 (s, 9 H, Me ₂ Si, 5a); 0.34 (s, 6 H, Me ₂ Si, 5a); 3.13 (s, 3 H, OMe, 6a); 0.13 (s, 18 H, Me ₃ Si, 6a); 0.25 (s, 6 H, Me ₂ Si, 6a)	1.1, 2.5 (ofa Me ₂ Si, 5a); 2.7 ($\underline{C}=C=O$, 5a); 35.0 (Si $\underline{C}=C$, 5a); 65.2 (OMe, 5a); 112.5 (Si $C=\underline{C}$, 5a); 167.8 ($C=\underline{C}=O$, 5a); {-20.9 (Me ₃ Si, 5a)}; {-21.0 (Me ₂ Si, 5a)}; 1.1, 2.5 (ofa Me ₂ Si, 6a); 2.7 ($\underline{C}=C=O$, 6a); 35.0 (Si $\underline{C}=C$, 6a); 65.2 (OMe, 6a); 112.5 (Si $C=\underline{C}$, 6a); 167.5 ($C=\underline{C}=O$, 6a); {20.9 (Me ₃ Si, 6a)}; {-21.0 (Me ₂ Si, 6a)}			
6b	2090	0.28 (s, 18 H, Me ₃ Ge); 0.25 (s, 6 H, Me ₂ Si)	1.6 ($\underline{C}=C=O$); 1.2 (Me ₃ Ge); 2.6 (Me ₂ Si); 167.0 (C= $\underline{C}=O$)			
6c	2090	0.40 (d, 3 H, MeHSi); 0.24 (s, 18 H, Me ₃ Si); 4.74 (k, 1 H, Me <u>H</u> Si)	0.0 (Me ₃ (H)Si); 0.8 (Me ₃ Si); 1.0 (<u>C</u> =C=O); 167.1 (C= <u>C</u> =O); {-20.8 (Me(H)Si)}; {1.2 (Me ₃ Si)}			
6d	2086, 2092	0.27 (s, 18 H, Me ₃ Ge); 0.30 (s, 3 H, Me <u>Si]</u> ; 4.70 (k, 1 H, MeSi(<u>H</u>))	-0.7 (MeSi); 0.5 (<u>C</u> =C=O); 1.0 (Me ₃ Ge); 167.0 (C= <u>C</u> =O); {-22.2 (MeSi)}			
6e	2080	0.28 (s, 18 H, Me ₃ Ge); 0.40 (s, 6 H, Me ₂ Ge)	0.1 (\underline{C} =C=O); 1.4 (Me ₃ Ge); 3.1 Me ₂ Ge); 168.0 (C= \underline{C} =O)			
6f	2050	0.37 (s, 18 H, Me ₃ Sn); 0.55 (s, 6 H, Me ₂ Ge)	-6.5 (Me ₃ Sn); -5.2 (<u>C</u> =C=O); 3.7 Me ₂ Ge; 164.0 (C= <u>C</u> =O); [33.4 (Me ₃ Sn)]			
5g	2060, 2180	0.27 (s, 6 H, Me ₂ Si); 0.37 (s, 9 H, Me ₃ Sn); 1.37 (t, 3 H, OCH ₂ Me); 4.13 (k, 2 H, OCH ₂ Me)	$-6.2 (Me_{3}Sn); 3.0 (Me_{2}Si); 3.7 (\underline{C}=C=O); 14.4 (OCH_{2}\underline{Me}); 36.6 (Si\underline{C}=C); 75.2 (O\underline{C}H_{2}Me); 110.3 (SiC=\underline{C}); 163.6 (C=\underline{C}=O)$			
8	2080, 2090	0.16 (s, 6 H, Me ₂ Si); 0.24 (s, 9 H, Me ₃ Ge); 1.53 (s, 1 H, C <u>H</u> =C=O)	1.3 (Me ₃ Ge); 1.6 (Si(Ge) <u>C</u> =C=O); 2.1 (Me ₂ Si]; 167.0 (Si(Ge)C= <u>C</u> =O); 179.9 (SiCH= <u>C</u> =O)			
14	_	0.31 (s, 18 H, Me ₃ Ge); 0.38 (s, 6 H, Me ₂ Si); 1.57 (s, 2 H, SiC <u>H</u> CO); 4.19 (m, 4 H, NHC <u>H</u> ₂ Ph); 5.03 (t, 2 H, N <u>H</u> CH ₂ Ph); 7.13 (m, 10 H, NHCH <u>2Ph</u>)	0.5 (Me ₂ Si); 0.6 (Me ₃ Ge); 31.1 (Si <u>C</u> HCO); 44.3 (NH <u>C</u> H ₂ Ph); 127.8, 129.0, 129.1, 140.8 (Ph); 174.0 (SiCH <u>C</u> O)			
15	_	0.15 (s, 6 H, Me ₂ Si); 0.24 (s, 18 H, Me ₃ Ge); 1.62 (s, 2 H, SiC <u>H</u> CO); 11.38 (s, 2 H, COO <u>H</u>)	0.0 (Me ₃ Ge); 0.7 (Me ₂ Si); 29.2 (Si <u>C</u> HCO); 175.7 (SiCH <u>C</u> O)			

Table 3. IR and ¹H and ¹³C NMR spectra of compounds 5, 6, 8, 14, and 15

dimethylsilylenebis[(2-trimethylgermyl)acetic] acid (15) (see Scheme 4).

Using **6b** as an example, it was shown that the element-substituted bisketenes react completely with nucleophiles similarly to element-substituted monoketenes, rather somewhat slower.

The constants, yields, and elemental analysis data for compounds **5g**, **6a**—**f**, **8**, **14**, and **15** are given in Table 2, the spectral data, in Table 3.

In conclusion, the reaction of acetylenes 1a-e with trimethylsilicon, -germanium, and -tin halides 2-4 leads to promising silylated, germylated, and stannylated monoand bisketenes.

Experimental

IR spectra were recorded on a Perkin–Elmer 983G and IKS-22 spectrometers (0.1-mm thick cuvette, CaF_2 plates), ¹H, ¹³C, ²⁹Si, and ¹¹⁹Sn NMR spectra were recorded on a Bruker AC-200P (200 MHz), Bruker ARX-250 (250 MHz), and Bruker Avance-400 (400 MHz) spectrometers using CDCl₃, C_6D_6 , and DMSO-d₆ as the solvents. All operations were performed under dry argon. The starting acetylenes **1a**–e and ketene **5** were synthesized according to the procedures described earlier.¹ Iodotrimethylsilane **2**,¹⁷ bromo(iodo)trimethyl-germane,¹⁸ compounds **4**,¹⁹ **12**,²⁰ and **13**²¹ were synthesized following known procedures, compound **10** was purchased from Aldrich.

5-Methoxy-3,3-dimethyl-2-trimethylsilyl-3-silapent-1-en-4yn-1-one (5a) (in a mixture with 6a). Reagent 2 (2.1 g, 10 mmol) was added to compound 1a (0.7 g, 4 mmol) in dichloromethane (6 mL) followed by stirring for 88 h. IR, v/cm⁻¹: 2190 (-C=C-), 2095 (C=C=O); after stirring for another 12 h: 2190 (shoulder of -C=C-), 2095 (C=C=O). Distillation *in vacuo* gave a 5a + 6a product mixture (ratio 1 : 2) (0.8 g, 67%), b.p. 40–42 °C (0.02 Torr).

2,2'-(Dimethylsilylene)bis[(trimethylsilyl)ethylenone] (6a). A. Reagent 2 (2.4 g, 12 mmol) was added to compound 1a (0.7 g, 4 mmol) in dichloromethane (6 mL) followed by stirring for 115 h. Distillation *in vacuo* gave product 6a (0.7 g, 56%), b.p. 45–46 °C (0.02 Torr).

B. A mixture of compound **10** (2.5 g, 12 mmol) and triethylamine (1.8 g, 18 mmol) in anhydrous diethyl ether (10 mL) was added to a solution of compound **9** (0.8 g, 6 mmol) in anhydrous diethyl ether (5 mL) at -10 °C followed by stirring for 24 h at 20 °C. IR of the reaction mixture, v/cm⁻¹: 2090 (Si(C=C=O)₂ from **6a**), 2190 (Si(C=COSi)₂ from **11**). The ethereal layer was separated, the subsequent distillation *in vacuo* gave compound **6a** (0.3 g, 15%), b.p. 47 °C (0.02 Torr). The spectral data of compound obtained completely agree with those obtained as above.

C. A mixture of compound **13** (1.4 g, 4 mmol) and triethylamine (1.2 g, 12 mmol) in anhydrous diethyl ether (10 mL) was added to a solution of compound **12** (0.9 g, 8 mmol) in anhydrous diethyl ether (5 mL) at -10 °C with stirring. The reaction mixture was stirred for 6 days at 20 °C. IR of the reaction mixture, v/cm⁻¹: 2090 (Si(C=C=O)₂ from **6a**), 2190 (Si(C=COSi)₂ from **11**). The ethereal layer was separated, the subsequent fractional distillation gave compound **6a** (0.2 g, 9%), b.p. 47 °C (0.02 Torr). The spectral data of compound obtained completely agree with those obtained as above.

Similarly to the synthesis of compound **6a** (method *A*), the following compounds were obtained: 5-ethoxy-3,3-dimethyl-2-trimethylstannyl-3-silapent-1-en-4-yn-1-one (**5g**), 2,2'-(dimethylsilylene)bis[(trimethylgermyl)ethylenone] (**6b**), 2,2'-(methylsilylene)bis[(trimethylsilyl)ethylenone] (**6c**), 2,2'-(methyl-silylene)bis[(trimethylgermyl)ethylenone] (**6d**), 2,2'-(dimethylgermylene)bis[(trimethylgermyl)ethylenone] (**6e**), and 2,2'-(dimethylgermylene)bis[(trimethylgermyl)ethylenone] (**6f**).

N,*N*'-**Dibenzyl-2**,**2**'-(**dimethylsilylene**)**bis**[**2**-(**trimethylger-myl)acetamide**] (**14**). Benzylamine (0.43 g, 4 mmol) was added to a solution of compound **6b** (0.5 g, 1.3 mmol) in hexane (20 mL) with stirring. A precipitate formed was filtered off to obtain product **14** (0.47 g, 62%) with m.p. 104–105 °C.

2,2'-(Dimethylsilylene)bis[2-(trimethylgermyl)acetic] acid (15). Water (0.05 g, 2.7 mmol) and concentrated sulfuric acid (0.01 g) were added to a solution of compound **6b** (5 g, 1.3 mmol) in 1,4-dioxane (50 mL) with stirring. A precipitate formed was filtered off to obtain diacid **15** (2.3 g, 42%) with m.p. 133-135 °C.

References

- S. V. Gruener, B. Wrackmeyer, R. N. Ezhov, V. S. Petrosyan, *Izv. Akad. Nauk, Ser. Khim.*, 2003, 1641 [*Russ. Chem. Bull.*, *Int. Ed.*, 2003, 52, 1730].
- 2. K. Sung, T. T. Tidwell, J. Am. Chem. Soc., 1996, 118, 2768.
- 3. A. Joffler, G. Himbert, Synthesis, 1992, 495.
- 4. S. V. Ponomarev, M. B. Erman, S. A. Lebedev, S. Ya. Pechurina, I. F. Lutsenko, *Zh. Obshch. Khim.*, 1971, **41**, 127 [*J. Gen. Chem. USSR (Engl. Transl.*), 1971, **41**].
- M. A. Kazankova, V. A. Ilyushin, I. F. Lutsenko, *Zh. Obshch. Khim.*, 1980, **50**, 690 [*J. Gen. Chem. USSR (Engl. Transl.*), 1980, **50**].
- I. V. Efimova, M. A. Kazankova, I. F. Lutsenko, *Zh. Obshch. Khim.*, 1985, 55, 1647 [J. Gen. Chem. USSR (Engl. Transl.), 1885, 55].
- 7. R. D. Miller, D. R. Mckean, Synthesis, 1979, 730.
- Yu. K. Grishin, S. V. Ponomarev, S. A. Lebedev, *Zh. Org. Khim.*, 1974, 404 [J. Org. Chem. USSR (Engl. Transl.), 1974].
- K. B. Borisenko, R. N. Yezhov, S. V. Gruener, H. E. Robertson, D. W. H. Rankin, *Inorg. Chim. Acta*, 2008, 361, 467.
- 10. W. Uhlig, A. Tzschach, Z. Chem., 1988, 28, 409.
- S. V. Ponomarev, A. S. Zolotareva, R. N. Ezhov, Yu. V. Kuznetsov, V. S. Petrosyan, *Izv. Akad. Nauk, Ser. Khim.*, 2001, 1045 [*Russ. Chem. Bull., Int. Ed.*, 2001, **50**, 1093].
- B. L. Groh, G. R. Magrum, T. J. Barton, J. Am. Chem. Soc., 1987, 109, 7568.
- R. L. Danheiser, A. Nishida, S. Savariar, M. P. Trova, *Tetrahedron Lett.*, 1988, 29, 4917.
- 14. S. Akai, S. Kitagaki, T. Naka, K. Jamamoto, Y. Tsusuki, K. Matsumoto, Y. Kita, J. Chem. Soc., Perkin Trans. 1, 1996, 1705.
- H. Kai, K. Iwamoto, N. Chatani, Sh. Murai, J. Am. Chem. Soc., 1996, 118, 7634.
- 16. G. Maas, M. Regitz, R. Rahm, J. Schneider, P. J. Stang, Ch. M. Crittell, J. Chem. Soc., Chem. Commun., 1990, 1456.
- M. G. Voronkov, Yu. I. Khudobin, *Izv. Akad. Nauk SSSR,* Ser. Khim., 1956, 714 [Bull. Acad. Sci. USSR, Div. Chem. Sci. (Engl. Transl.), 1956, 5].
- V. F. Mironov, A. L. Kravchenko, *Izv. Akad. Nauk SSSR*, Ser. Khim., 1965, 1096 [Bull. Acad. Sci. USSR, Div. Chem. Sci. (Engl. Transl.), 1965, 14].
- 19. D. Seyferth, N. Kahlen, J. Org. Chem., 1960, 25, 809.
- L. L. Shchukovskaya, R. I. Pal'chik, A. N. Lazarev, Dokl. Akad. Nauk SSSR, 1965, 164, 357 [Dokl. Chem. USSR (Engl. Transl.), 1965].
- E. J. Corey, H. Cho, Ch. Rucker, D. H. Hua, *Tetrahedron Lett.*, 1981, 22, 3455.

Received November 7, 2007; in revised form October 7, 2008