Dialkylamino- and alkoxytrialkylstannanes as reagents for synthesis of organoboryl (trialkylsilyl) ketenes

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Dialkylamino- and alkoxytrialkylstannanes have been studied as N- and O-nucleophilic reagents in substitution reactions at the B—Br bond. The previously unknown bis(dialkylamino)- and dialkoxyboryl trialkylsilyl ketenes were synthesized. Some peculiarities of the reactivity of these compounds have been studied.

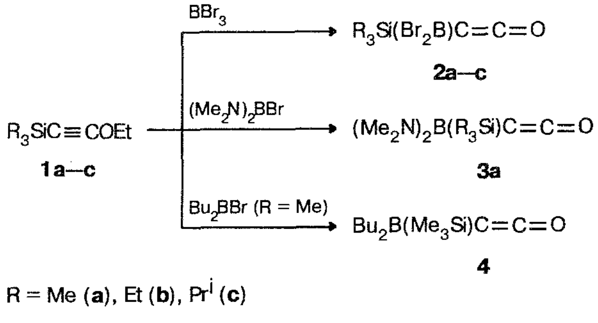
Key words: dialkylaminotrialkylstannane, alkoxytrialkylstannane, synthesis; borylsilyl ketene, methanolysis, aminolysis.

Previously[1] we prepared bis(dialkylamino)boryl trialkylsilyl ketenes by the reaction of dihaloboryl trialkylsilyl ketenes with dialkylamines. In the present work we have studied the interaction of dibromoboryl(trialkylsilyl) ketenes with dialkylamino- and alkoxytrialkylstannanes and the reactions of the resulting ketenes with methanol and dialkylamines.

Ketenes that contain various groups in a molecule are convenient synthons for the synthesis of substituted carbonyl compounds (esters, amides, and ketones), which are of interest for investigating reversible and irreversible 1,3-rearrangements.[2,3] In addition, compounds of this class proved to be useful objects for physicochemical investigations.[4,5]

Symmetrical and asymmetrical bis-element (Si, Ge, and Sn) substituted ketenes are traditionally prepared by the reaction of the corresponding alkoxyacetylenes with organosilicon, -germanium, and -tin halides.[6,7]

We showed that the reactions of trialkylsilyl(ethoxy)acetylenes (**1a—c**) with boron bromides are of preparative value only for the synthesis of dibromoboryl trialkylsilyl ketenes (**2**); reactions of boron monobromides, such as bromobis(dimethylamino)borane and bromodibutylborane, occur slowly and do not go to completion.



R = Me (**a**), Et (**b**), Pr^i (**c**)

Compound **3a** is also formed through the action of iodotrimethylsilane on ethoxyethynylbis(dimethylamino)borane.[8] An attempt to prepare ketene **3a** starting from $Me_3Si(Me_3Sn)C=C=O$ (**5a**) and $(Me_2N)_2BBr$ by transmetallation was unsuccessful, though this method has been used previously[9] for preparing ketene with alkyl substituents at the B atom.

A more general method for the synthesis of bis(dialkylamino)boryl trialkylsilyl ketenes **3** is aminolysis of dibromides **2**.

$$\mathbf{2a{-}c} + 4\,R^1{}_2NH \longrightarrow \underset{\mathbf{3a{-}e}}{(R^1{}_2N)_2B(R_3Si)C=C=O}$$

R = R^1 = Me (**a**); R = Et, R^1 = Me (**b**); R = Pr^i, R^1 = Me (**c**);
R = Me, R^1 = Et (**d**)[1]; R = R^1 = Et (**e**)

The yields of the products depend substantially on the temperature of the reaction and the sizes of the alkyl substituents at the N and Si atoms, since ketenes **3a,b** formed in the reaction can add dimethylamine.[3]

The best results were obtained when (dialkylamino)trialkylstannanes were used as aminating agents.

$$\mathbf{2a{-}c} + 2\,R^2_3SnNR^1_2 \longrightarrow \mathbf{3a{-}c,e}$$

R^2 = Bu (**a,b**); Et (**c**); Me (**e**)

The reactions were carried out at −60 to −10 °C in pentane or ether; the yields of ketenes **3** were 80—98 %.

In order to prepare dialkoxyboryl trialkylsilyl ketenes **6** we studied the interaction of dialkoxyboryl(ethoxy)acetylene with iodotrimethylsilane. However, we could not isolate the expected ketene from the complex mixture of products. Methanolysis of ketene **2a** afforded dimethoxyboryl trimethylsilyl ketene (**6a**) in 20 % yield.

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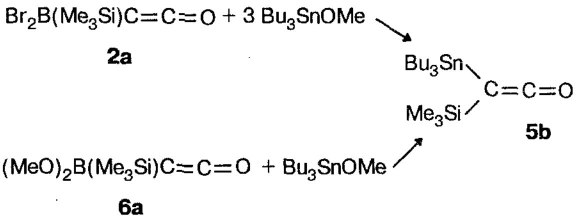
$$\mathbf{2a} + 2\,\mathrm{MeOH} \longrightarrow \underset{\mathbf{6a}}{(\mathrm{MeO})_2\mathrm{B}(\mathrm{Me_3Si})\mathrm{C{=}C{=}O}}$$

When alkoxytributylstannanes were used as O-nucleophiles, ketenes **6** were prepared in 52—86 % yield.

$$\mathbf{2a{-}c} + 2\,\mathrm{Bu_3SnOR^1} \longrightarrow \underset{\mathbf{6a{-}e}}{(\mathrm{R^1O})_2\mathrm{B}(\mathrm{R_3Si})\mathrm{C{=}C{=}O}}$$

$\mathrm{R = R^1 = Me}$ (**a**); $\mathrm{R = Me, R^1 = Et}$ (**b**); $\mathrm{R = Et, R^1 = Me}$ (**c**);
$\mathrm{R = Me, R^1 = SiMe_3}$ (**d**); $\mathrm{R = Pr^i, R^1 = Me}$ (**e**)

The reactions were carried out in pentane or ether at −60 to −40 °C and at a 1 : 2 ratio between the reactants. The yields of ketenes **6** turned out to depend considerably on the experimental conditions and the order in which the reactants were added. In fact, when an ethereal solution of dibromoboryl trimethylsilyl ketene **2a** was added to an ethereal solution of methoxytributylstannane at ~20 °C, a mixture of bromotributylstannane and silyl stannyl ketene (**5b**) was obtained, since an excess of methoxytributylstannane causes cleavage of the B—C bond in the intermediate ketene **6a**. In fact, the reaction of ketene **6a** with methoxytributylstannane gives ketene **5b** in a nearly quantitative yield.



These reactions demonstrate that the quite unusual replacement of the boryl group by the trialkylstannyl fragment readily occurs.

In the series of ketenes **2**, **3**, and **6** synthesized, compounds **3** with dialkylamino groups at the B atom are the most stable. Ketenes **2** containing a dihaloboryl group are the least stable: they rapidly grow dark even at room temperature. Ketenes **6** having a dialkoxyboryl group are intermediate in stability.

The structures of the boron-containing ketenes synthesized were confirmed by the data from ^{1}H and ^{13}C NMR and IR spectroscopy. The IR spectra of compounds **2**, **3**, and **6** exhibit intense absorption bands in the region 2100—2070 cm^{-1} typical of element-substituted ketenes.[10] The signals for the protons of the trialkylsilyl groups in the ^{1}H NMR spectra of these compounds are exhibited in the region 0.32—1.52 ppm and the resonance signals for the protons of the alkoxy and dialkylamino groups are at 2.58—4.1 ppm.

The resonance signals for the C atom of the carbonyl group in the ^{13}C NMR spectra of compounds **2**, **3**, and **6** occur in a relatively low-field region, at 168—172 ppm, which is characteristic of bis-element substituted ketenes.

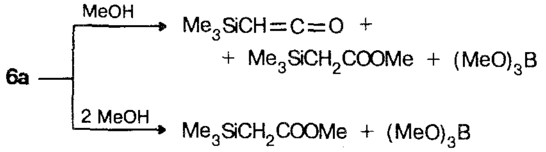
The resonance of the sp^2-hybridized C atoms in compounds **3** and **6**, which have electron-donating *O*-alkyl or *N*-alkyl substituents at the B atom, are displayed in the high-field region. For ketenes **3** these signals are at 4—5 ppm and for compounds **6** they are further displaced upfield by approximately 2 ppm. The presence of the electron-withdrawing dibromoboryl group in compounds **2** results in a downfield shift of the resonance signals for the α-C atoms (29—37 ppm). The C(sp^2) resonance signals in the spectra of ketenes are markedly broadened (30—55 Hz) and are only exhibited at −30 to −55 °C due to the fast scalar relaxation of the C atoms bonded with ^{11}B or ^{10}B quadrupole nuclei. These data attest indirectly that the ketenes under study contain B—C bonds.

In order to synthesize acetic acid derivatives containing boron and silicon, we studied the reactions of ketenes **3a** and **6a** with methanol and dialkylamines. The reaction of **3a** with 1 equiv. of MeOH affords an unseparable mixture of products, and when 2 equiv. of MeOH is added, the ketene is converted into *N,N*-dimethylamide of trimethylsilylacetic acid.

$$\mathbf{3a} + 2\,\mathrm{MeOH} \longrightarrow \mathrm{Me_3SiCH_2CONMe_2} + [(\mathrm{MeO})_2\mathrm{BNMe_2}]$$

Thus, ketenes having dialkylamino groups at the B atom behave as borylating agents with respect to alcohols, like most organoboron compounds.

Treatment of ketene **6a** with methanol gives only products of deborylation.



The IR spectrum of the reaction mixture obtained by adding 1 equiv. of the alcohol to ketene **6a** exhibits the ν(C=C=O) band at 2080 cm^{-1} with practically unchanged intensity and a new ν(CO) band of medium intensity at 1735 cm^{-1}. Spectroscopic analysis of the volatile reaction products (an absorption band of ketene at 2080 cm^{-1} in the IR spectrum and signals at δ 0.33 ($\mathrm{Me_3Si}$), 1.67 (CH=), and 3.65 (OMe) in the ^{1}H NMR spectra) makes it possible to suggest that the mixture contains trimethylsilyl ketene and trimethyl borate. The residue contains methyl trimethylsilylacetate (ν(COOMe) 1735 cm^{-1}, δ 3.7 (OMe), 2.0 ($\mathrm{CH_2}$), and 0.72 ($\mathrm{Me_3Si}$)) and unchanged ketene **6a**. When the second equivalent of methanol is added, the absorption intensity of the C=C=O group decreases and that of the ester group considerably increases. After 20 min, the band at 2080 cm^{-1} entirely disappears.

Previously[3] we reported the reaction of dimethylamine with bis(dialkylamino)boryl trialkylsilyl ketenes and the preparation of boron-silicon substituted derivatives of acetic acid. However, ketene **3c** has low reactivity and does not add dimethylamine with retention of the B—C bond, which is probably due to the steric hindrance caused by the isopropyl group at the Si atom. When excess amine is used, *N,N*-dimethyltriisopropylsilylacetamide (**7**) forms, along with tris(dimethylamino)borane.

It may be suggested that the reaction occurs *via* intermediate triisopropylsilyl ketene formed upon the protolytic cleavage of the B—C bond.

$$\mathbf{3c} \longrightarrow [\mathrm{Pr^i_3SiCH{=}C{=}O}] \longrightarrow$$

$$\longrightarrow \underset{\mathbf{7}}{\mathrm{Pr^i_3SiCH_2CONMe_2}} + \mathrm{(Me_2N)_3B}$$

Compounds **6** react with dimethylamine in a similar way.

$$\mathbf{6a} + \mathrm{Me_2NH} \xrightarrow[-10\ ^\circ\mathrm{C}]{} \left\{\begin{array}{c}\mathrm{Me_3SiCH{=}C{=}O}\\ \mathrm{Me_3SiCH_2CONMe_2}\\ \mathrm{(MeO)_2BNMe_2}\end{array}\right\} \xrightarrow{\mathrm{Me_2NH}}$$

$$\longrightarrow \mathrm{Me_3SiCH_2CONMe_2} + [\mathrm{(MeO)_2BNMe_2}]$$

When 1 mol of dimethylamine is added and the low-boiling products are removed *in vacuo*, the ^{1}H NMR

Table 1. Yields, boiling points, and elemental analysis data for the compounds synthesized

Compound	Method of preparation	Yield (%)	B.p./°C (*p*/Torr)	Found/Calculated (%) C	H	Molecular formula
1c		84	68—73 (1)	69.61 69.61	11.50 11.80	$C_{13}H_{26}OSi$
2c		62	92—94 (0.03)	36.16 35.90	6.29 5.71	$C_{11}H_{21}BBr_2OSi$
3a	*A*	98	54 (1)	50.68 50.99	10.16 9.92	$C_9H_{21}N_2BOSi$
3b	*B*	64	95—96 (1)	56.67 56.72	10.69 10.63	$C_{12}H_{27}N_2BOSi$
	A	80	—	—	—	—
3c	*A*	79	95—100 (0.025)	61.49 60.80	11.44 11.24	$C_{15}H_{33}N_2BOSi$
	B	60	—	—	—	—
3e	*B*	67	105—107 (1)	61.98 61.96	12.05 11.29	$C_{16}H_{35}N_2BOSi$
	A	89	—		—	—
5b	*A*	95	125—128 (1)	50.38 50.63	9.02 9.00	$C_{17}H_{36}OSiSn$
6a	*A*	52	42—46 (7)	44.89 45.19	8.06 8.07	$C_7H_{15}BO_3Si$
6b	*A*	65	50—53 (1)	50.57 50.48	8.98 8.94	$C_9H_{19}BO_3Si$
6c	*A*	86	65—66 (1)	51.75 52.64	9.99 9.28	$C_{10}H_{21}BO_3Si$
6d	*A*	86	55—57 (1)	42.46 43.69	8.68 9.00	$C_7H_{15}BO_3Si_3$
6e	*A*	91	80—85 (1)	57.31 57.77	10.76 10.07	$C_{13}H_{27}BO_3Si$
7		56	90—95 (1)	63.90 64.19 (Si,	11.73 11.92 11.11 11.55)	$C_{13}H_{29}NOSi$

*Note. **A*** denotes the reactions of dibromoboryl trialkylsilyl ketenes with dialkylamino- or alkoxytrialkylstannanes and ***B*** denotes the reactions of dibromoboryl trialkylsilyl ketenes with dialkylamines.

Table 2. Parameters of ^{1}H, ^{13}C NMR, and IR spectra of the resulting compounds

Compound	IR, ν/cm^{-1}	^{1}H NMR, δ		^{13}C NMR, δ			
		R_3Si	R_2N or RO	R_3Si	$\underline{C}$=C=O	C=$\underline{C}$=O	R_2N or RO
1c	2200	1.03 (s)	4.07 (q); 1.43 (t, *J* = 7.5 Hz)	—	—	—	—
2c	2100	1.30 (br.s)		12.34 (CH); 18.39 $(CH)_3$	32.84	167.83	—
3a	2070	0.22 (s)	2.58 (s)	2.1	4.4	170.2	41.80
3b	2075	0.42—1.39 (s)	2.65 (s)	—	—	—	—
3c	2075	1.29 (br.s.)	2.65 (s)	—	—	—	—
3e	2075	0.35—1.32 (m)	4.1 (q); 0.35—1.32 (m)	—	—	—	—
5b	2080	0.22 (Me_3Si); 0.75—1.75 (m, Bu_3Sn)	—	—	—	—	—
6a	2080	0.32 (s)	3.42 (s)	0.58	2.4	171.14	52.15
6b	2080	0.35 (s)	1.12 (t); 3.88 (q)	—	—	—	—
6c	2080	0.59—1.52 (m)	3.45 (s)	4.09 (CH_2); 7.18 (CH_3)	1.5	169.55	51.81
6d	2080	0.39 (s)	0.35 (s)	0.36	6.6	171.72	1.31
6e	2080	1.32 (br.s.)	3.42 (s)	—	—	—	—
7	1620	1.22 (br.s)	1.82 (s, CH_2); 2.65 (d)	—	—	—	—

spectrum of the reaction mixture points to the formation of *N,N*-dimethyltrimethylsilylacetamide and the IR spectrum exhibits the ν(C=C=O) band at 2080 cm^{-1}, along with the amide ν(CO) band. After addition of excess amine the ketene absorption band in the IR spectrum entirely disappears, and silylacetamide can be isolated by fractional distillation.

The properties of the compounds synthesized and the data from elemental analysis are given in Table 1, and the spectral parameters are listed in Table 2.

Experimental

IR spectra were recorded on a UR-20 (Karl Zeiss) spectrophotometer, ^{1}H and ^{13}C NMR spectra were run on Tesla BS-467 and Jeol FX-100 spectrometers, respectively. CCl_4, CD_2Cl_2, and $CDCl_3$ were used as solvents and TMS was used as the internal standard.

Triisopropylsilyl(ethoxy)acetylene (1c). A 1.23 *N* solution of BuLi in pentane (27 mL, 0.033 mol) was added at −10 °C to a solution of ethoxyacetylene (2.3 g, 0.033 mol) in abs. THF (30 mL). The mixture was stirred for 45 min at ~20 °C, chlorotriisopropylsilane (5.6 g, 0.03 mol) in abs. THF (25 mL) was added to it, and stirring was continued for 20 h. The solvent was removed, and abs. pentane (50 mL) was added to the residue. The precipitate was separated by centrifugation. Distillation of the filtrate afforded 5.7 g of acetylene **1c**, n_D^{20} 1.4626.

Dibromoboryl trimethylsilyl ketene (2a) and dibromoboryl triethylsilyl ketene (2b) were prepared by the previously[7] described procedure.

Dibromoboryl triisopropylsilyl ketene (2c). At −50 °C a solution of BBr_3 (4.4 g, 0.018 mol) in abs. pentane (3 mL) was added to a solution of acetylene **1c** (4 g, 0.018 mol) in abs. pentane (5 mL). The low-boiling compounds were evaporated, and the residue was distilled to give 4 g of ketene **2c**.

Bis(dimethylamino)boryl trimethylsilyl ketene (3a). A mixture of acetylene **1a** (3.4 g, 0.024 mol), bromobis(dimethylamino)borane (4.3 g, 0.024 mol), and dibutyl ether (10 mL) was heated for 8 h at 80 °C. Distillation afforded 2.2 g (43 %) of ketene **3a**, b.p. 54 °C (1 Torr).

Dibutylboryl trimethylsilyl ketene (4). A solution of bromodibutylborane (3.6 g, 0.18 mol) in abs. CH_2Cl_2 (3 mL) was added to a solution of acetylene **1a** (2.5 g, 0.018 mol) in abs. CH_2Cl_2 (3 mL), and the mixture was stirred for 36 h at ~20 °C. Vacuum distillation afforded 2.9 g (69 %) of ketene **4** (see Ref. 9), b.p. 70—75 °C (1 Torr).

Trimethylsilyl trimethylstannyl ketene (5a). Trimethylbromostannane (11.3 g, 0.046 mol) in abs. MeCN (14 mL) was added to acetylene **1a** (6.5 g, 0.046 mol). The course of the reaction was monitored by IR spectroscopy (disappearance of the ν(C≡C) absorption band at 2200 cm^{-1}). After 1.5 months 3.5 g (26 %) of ketene **5a** was isolated, b.p. 65—70 °C (10 Torr), n_D^{20} 1.4865 (see Ref. 11).

Trimethylsilyl tributylstannyl ketene (5b). The reaction of ketene **6a** with methoxytributylstannane. Methoxytributylstannane (2.6 g, 0.008 mol) in abs. pentane (2 mL) was added to a solution of ketene **6a** (1.5 g, 0.008 mol). The mixture warmed up to 50 °C. After evaporation of the low-boiling compounds the residue was distilled to give 3.1 g of trimethylsilyl tributylstannyl ketene **5b**, n_D^{20} 1.5048.

The reaction of ketene **2a** with methoxytributylstannane. At −10 °C ketene **2a** (3.8 g, 0.013 mol) in abs. pentane (5 mL) was added to a stirred solution of methoxytributylstannane (12.5 g, 0.039 mol) in abs. pentane (10 mL). After evaporation of the low-boiling compounds the residue was distilled to give 13.5 g of a mixture of bromotributylstannane and trimethylsilyl tributylstannyl ketene **5b**, b.p. 120—125 °C (1 Torr).

Bis(dialkylamino)boryl trialkylsilyl ketenes. *A.* At −60 °C a solution of dimethylaminotributylstannane (16 g, 0.048 mol) in abs. pentane (10 mL) was added to a solution of ketene **2a** (6.8 g, 0.024 mol) in abs. pentane (25 mL). Distillation afforded 5 g (98 %) of ketene **3a**, b.p. 54—60 °C (1 Torr).

Ketenes **3b,c,e** were prepared in a similar way.

B. A solution of ketene **2b** (14.3 g, 0.044 mol) in abs. pentane was added with cooling to −60 °C and intense stirring to a solution of dimethylamine (7.9 g, 0.176 mol) in abs. pentane (40 mL). The precipitate of $Me_2NH \cdot HBr$ was separated by centrifugation. Distillation of the filtrate gave 7.2 g of ketene **3b**.

Ketenes **3c,e** were prepared in a similar way and ketene **3d** was prepared by the previously described[1] procedure.

Dimethoxyboryl trimethylsilyl ketene (6a). Tributylmethoxystannane (10 g, 0.03 mol) in abs. pentane (10 mL) was added at −50 °C to a stirred solution of ketene **2a** (4.4 g, 0.015 mol) in abs. pentane (20 mL). The solvent was evaporated in the vacuum of a water-jet pump and the residue was fractionated to give 1.5 g of ketene **6a**.

Ketenes **6b—e** were prepared in a similar way.

Reactions of ketenes 2a, 3a, and 6a with methanol. A mixture of abs. methanol (2 g, 0.064 mol) and triethylamine (6.5 g, 0.064 mol) was added to a solution of ketene **2a** (9.2 g, 0.32 mol) in 60 mL of abs. pentane. The precipitate of $Et_3N \cdot HBr$ was separated by centrifugation. Distillation of the filtrate afforded 1.2 g (20 %) of ketene **6a**, b.p. 45—46 °C (7 Torr).

Abs. methanol (1.1 g, 0.035 mol) was added dropwise to a solution of ketene **3a** (3.7 g, 0.0175 mol) in 4 mL of abs. ether (the mixture warmed up). The volatile products were removed, and the residue was distilled to give 1.65 g (59 %) of *N,N*-dimethyltrimethylsilylacetamide, b.p. 50—52 °C (1 Torr), n_D^{20} 1.4520 (see Ref. 12), IR, ν/cm^{-1}: 1630 (—CON<).

Methanol (0.24 g, 7.5 mmol) in 1 mL of abs. ether was added to a solution of ketene **6a** (1.4 g, 7.5 mmol) (the mixture warmed up). Additional abs. methanol (0.24 g, 7.5 mmol) was added, and the mixture was stirred for 30 min (the course of the reaction was monitored by IR spectroscopy). Distillation gave 0.7 g (90 %) of trimethylborate, b.p. 65—69 °C[13] and 0.8 g (77 %) of methyl trimethylsilylacetate, b.p. 140—144 °C, n_D^{20} 1.4135 (see Ref. 14).

The reaction of ketenes 3c and 6a with dimethylamine. Dimethylamine (0.2 g, 4.4 mmol) was added to ketene **3c** (1.3 g, 4.4 mmol) in 1 mL of abs. pentane. According to IR spectroscopy, the reaction did not occur. Additional dimethylamine (0.5 mL) was added, and the mixture was allowed to stand in a refrigerator for 3 weeks (the course of the reaction was monitored by IR spectra based on the disappearance of the ketene absorption band at 2075 cm^{-1} and the appearance of an amide band at 1620 cm^{-1}). The volatile compounds were recondensed *in vacuo* into a cooled trap, and the residue was distilled to give 0.6 g of *N,N*-dimethyltriisopropylsilylacetamide (**7**), n_D^{20} 1.4750. Fractionation of the contents of the trap afforded 0.2 g (30 %) of tris(dimethylamino)borane, b.p. 42—44 °C (10 Torr).

At −10 °C, dimethylamine (0.48 g, 11 mmol) in 1 mL of abs. ether was added to a solution of ketene **6a** (1 g, 5.4 mmol) in 1 mL of abs. ether. The volatile products were removed, and the residue was distilled to give 0.6 g (67 %) of trimethylsilyl-*N,N*-dimethylacetamide, b.p. 52—53 °C (1 Torr), n_D^{20} 1.4525.

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