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B.A. Trofimov on the 65th Anniversary of His Birth

Synthesis of 3,3-Dialkoxy-2-trialkylsiloxypropenes

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Abstract—Silylation of 1,1-dialkoxy-2-propanones with trimethyl- and *tert*-butyldimethylsilyl chlorides and trifluoromethanesulfonates in the presence of triethylamine and/or 1,8-diazabicyclo[5.4.0]undec-7-ene in various solvents leads to 2-trialkylsiloxypropenal acetals in up to 60% yield.

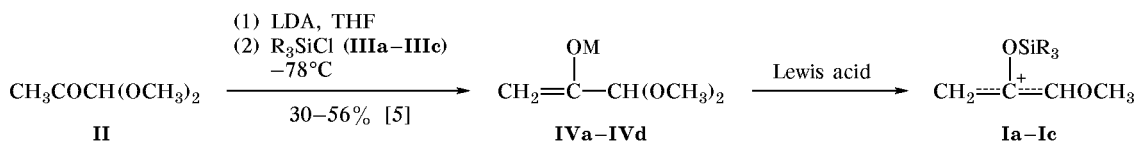
Oxyallyl cations have been used as intermediates in [4+3]-cycloaddition reactions for the synthesis of natural compounds as long as 40 years [1–3]. As a rule, they are generated from α -halo or α,α' -dihalo ketones [2]. Several communications have been published on the application for the same purposes of halogen-free substrates, specifically α -alkoxy-substituted trimethylsiloxyallyl cations **I** [3–7] which are obtained from trialkylsilyl enol ethers derived from α -keto acetals [3, 5–7]. The latter are usually prepared by silylation of α -keto acetals **II** with chlorotrimethylsilane (**IIIa**) (Scheme 1). Here, deprotonation of acetals **II** is effected by lithium diisopropylamide (LDA) at -78°C [3, 5, 7]. Most frequently, silyl enol ethers formed *in situ* were brought into [4+3]-cycloaddition reactions [3, 6, 7]. The low yield of 7-membered cyclic products was sometimes explained by instability of initial sterically hindered silyl ethers [6]. Previously, acetals **IV** were synthesized mainly from dimethyl acetals [3, 5, 6]. Apart from chlorotrimethylsilane, higher chlorotrialkylsilanes **IIIb** and **IIIc** were used in some cases as silylating agents [5, 7].

The physical properties, spectral parameters, and hydrolytic stability of trialkylsilyl ethers derived from methylglyoxal acetals have been studied insufficiently. Therefore, while continuing our studies of α -functionalized vinyl ethers and their heteroanalogs [8], we have developed a procedure which ensures preparation of 3,3-dialkoxy-2-trialkylsiloxypropenes in good yields and examined the stability of the products, depending on the synthesis and isolation conditions.

As substrates we used 1,1-diethoxy- and 1,1-dibutoxypropanones **Va** and **Vb** which were synthesized by the procedure reported by us previously [9]. We examined their silylation with chlorotrialkylsilanes and trialkylsilyl trifluoromethanesulfonates in the presence of organic bases.

Triethylamine is known to be used most frequently as a base in the silylation of carbonyl compounds [10, 11]. However, the silylation with chlorotrimethylsilane in the presence of triethylamine requires fairly severe conditions (heating in boiling DMF for several hours) [12]. Dimethylformamide as solvent is also inconvenient, for its IR absorption is located at

Scheme 1.



I, III, R = Me (**a**), Et (**b**), *i*-Pr (**c**); **IV, M** = Li (**a**), Me₃Si (**b**), Et₃Si (**c**), (*i*-Pr)₃Si (**d**).

Conditions of synthesis and yields of trialkylsilyl ethers $\text{CH}_2=\text{C}(\text{OSiR}_3)\text{CH}(\text{OR}')_2$ (**VIa–VIId**)

Run no.	R_3SiX	R'	Amine (mol %) ^a	Solvent	Temperature, °C	Reaction time, h	Yield, ^b %
1	Me_3SiCl	Et	Et_3N (82)+DBU (6)	CH_2Cl_2	20	192	45 ^c
2	Me_3SiCl	Bu	Et_3N (100)+DBU (28)	CH_2Cl_2	42	7	62
3	Me_3SiOTf	Bu	Et_3N (300)	CCl_4	20	6	38
4	Me_3SiOTf	Et	Et_3N (186)	CCl_4	20	6	30
5	Me_3SiOTf	Bu	DBU (100)	$(\text{CH}_2\text{Cl})_2$	82	1	60
6	Me_3SiOTf	Bu	DBU (100)	CCl_4	75	4.5	53
7	<i>t</i> - BuMe_2SiCl	Et	Et_3N (140)+DBU (25)	CCl_4	75	18	0
8	<i>t</i> - BuMe_2SiCl	Bu	Et_3N (140)+DBU (36)	CCl_4	75	13	0
9	<i>t</i> - $\text{BuMe}_2\text{SiOTf}$	Et	Et_3N (140)+DBU (28)	CCl_4	75	12	52 ^d
10	<i>t</i> - $\text{BuMe}_2\text{SiOTf}$	Bu	Et_3N (140)+DBU (33)	CCl_4	75	13	49 ^d

^a Fraction of the amine with respect to acetal **V**.

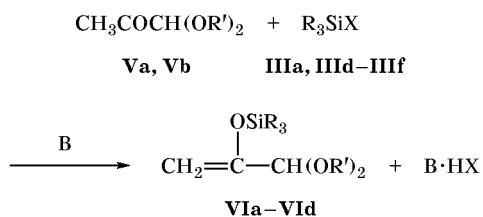
^b According to the ^1H NMR data.

^c Isolated by distillation.

^d After double distillation.

1650 cm^{-1} , i.e., in the region corresponding to absorption of the $\text{C}=\text{C}$ bond in silyl enol ether **VI**. Our preliminary experiments showed that the system $\text{Me}_3\text{SiCl}-\text{Et}_3\text{N}$ is hardly suitable for the silylation of ketones having a bulky substituent in the α -position: the yields were as low as 10–20%.

Scheme 2.



III, $\text{R} = \text{Me}$, $\text{X} = \text{Cl}$ (**a**); $\text{R}_3 = t\text{-BuMe}_2$, $\text{X} = \text{Cl}$ (**d**); $\text{R} = \text{Me}$, $\text{X} = \text{OSO}_2\text{CF}_3$ (**e**); $\text{R}_3 = t\text{-BuMe}_2$, $\text{X} = \text{OSO}_2\text{CF}_3$ (**f**); **V**, $\text{R}' = \text{Et}$ (**a**), Bu (**b**); **B** = Et_3N , DBU; **VI**, $\text{R} = \text{Me}$, $\text{R}' = \text{Et}$ (**a**); $\text{R} = \text{Me}$, $\text{R}' = \text{Bu}$ (**b**); $\text{R}_3 = t\text{-BuMe}_2$, $\text{R}' = \text{Et}$ (**c**); $\text{R}_3 = t\text{-BuMe}_2$, $\text{R}' = \text{Bu}$ (**d**).

Taking into account generally accepted views on the mechanism of silylation of ketones [12–16], we tried to use a stronger base, 1,8-diazabicyclo[5.4.0]-undec-7-ene (DBU). Like 4-dimethylaminopyridine in the silylation of alcohols [13], DBU was added in a catalytic amount (5 to 38 mol %), and the liberated hydrogen chloride was bound with an equimolar or excess amount of Et_3N . In order to ensure preliminary enolization of keto acetals **V** and minimize polymerization of the product, Me_3SiCl was added last to the reaction mixture. As solvent we usually used

carbon tetrachloride which does not dissolve triethylamine hydrochloride, and the latter separated as the upper liquid layer. We thus avoided additional treatment of the reaction mixture with a nonpolar solvent, as it was necessary with the use of DMF or CH_2Cl_2 as solvent [12, 16, 17]. The yields of trimethylsilyl ethers **VIa** and **VIb** reached 50–60% (see table, run nos. 1, 2).

Hergott and Simchen [18] compared the reactivity of silylating agents Me_3SiX toward cyclopentanone and diisopropyl ketone in the presence of Et_3N . According to the authors, the rate constant of the reaction with trimethylsilyl trifluoromethanesulfonate ($\text{X} = \text{OSO}_2\text{CF}_3$) is greater by 8 orders of magnitude than that found for $\text{X} = \text{Cl}$ [18]. The reaction of keto acetals **V** with trimethylsilyl trifluoromethanesulfonate in the presence of Et_3N under comparable conditions [11] afforded trimethyl silyl ethers **VIa** and **VIb** in only 30–38% yield (see table, run nos. 3, 4).

Insofar as a combination of Me_3SiOTf with DBU has been accepted as the most effective silylating system [19] and 1,2-dichloroethane is a solvent accelerating this reaction [11], we performed silylation of keto acetals **V** with Me_3SiOTf in dichloroethane in the presence of DBU. In fact, we observed increase in the reaction rate but not in the yield of the silylation product (see table, run no. 5). In a less polar solvent, carbon tetrachloride, the reaction rate was much lower (run no. 6).

We believe that the lower yield of trimethylsilyl ethers **VIa** and **VIb** in the silylation of ketones **V**, as compared to published data [11], is explained by

hydrolytic instability of the products. In some cases, readily hydrolyzable Me_3SiO group is replaced by more stable sterically crowded groups, e.g., by $t\text{-BuMe}_2\text{SiO}$ [20] whose hydrolytic stability is greater by 4 orders of magnitude than that of Me_3SiO [21]. However, *tert*-butyldimethylsilyl chloride reacts with sterically hindered alcohols much more slowly [22]. In the silylation with $t\text{-BuMe}_2\text{SiCl}$, imidazole [20] or 4-dimethylaminopyridine [13] is usually used as a base. We have found that no expected products are formed in reactions of keto acetals **V** with *tert*-butylchlorodimethylsilane in the presence of an equimolar amount of Et_3N and 25 mol % of DBU (see table, run nos. 7, 8). Analogous results were obtained previously in an attempt to effect silylation of cyclopentanone and some other ketones with chlorotriisopropylsilane in the presence of triethylamine [22].

We succeeded in silylating keto acetals **V** in the presence of DBU using *tert*-butyldimethylsilyl trifluoromethanesulfonate instead of $t\text{-BuMe}_2\text{SiCl}$. As a result, the corresponding silyl ethers **VIc** and **VI d** were obtained in more than 50% yield (see table, run nos. 9, 10). As with trimethylsilyl ethers, washing of the reaction mixture with water (which is a typical procedure in the isolation of trialkylsilyl enol ethers [19, 21, 23]) turned out to be inadmissible. Therefore, as in [17, 19, 23], the reaction mixture was filtered through a layer of aluminum oxide or silica gel (to remove ammonium salts), which led to loss of up to 6% of the silylation product. Trialkylsilyl ethers **VI** are fairly stable in the absence of traces of moisture. They can be distilled under reduced pressure and may be isolated in such a way in 50–60% yield.

EXPERIMENTAL

The ^1H and ^{13}C NMR spectra were recorded on a Bruker DPX-400 spectrometer (400 and 100.6 MHz, respectively) using CDCl_3 as solvent. The chemical shifts were measured relative to tetramethylsilane. The IR spectra were obtained on a Specord 75IR instrument from samples prepared as thin films. Gas chromatographic–mass spectrometric analysis was performed using an HP 5971A mass-selective detector (70 eV) coupled with an HP 5890 gas chromatograph (Ultra-2 column, 5% of phenylmethylsilicone, injector temperature 250°C; oven temperature programming from 70 to 280°C at 20 deg/min; carrier gas helium). All reagents were thoroughly dried and distilled just before use. Methylene chloride, carbon tetrachloride, and 1,2-dichloroethane were distilled over P_2O_5 . Triethylamine was dried over KOH and distilled over calcium hydride. Chlorotrimethylsilane was distilled

in the presence of tributylamine to remove traces of hydrogen chloride. The reaction flask was flame-dried and filled with dry argon (which was preliminarily passed through H_2SO_4 and NaOH). All reactions and distillations were carried out under argon.

Reaction of 1,1-diethoxy-2-propanone (Va) with chlorotrimethylsilane (run no. 1). A mixture of 50 ml of methylene chloride, 3 g (0.029 mol) of triethylamine, 0.36 g (0.002 mol) of DBU, 5.06 g (0.035 mol) of 1,1-diethoxy-2-propanone (**Va**), and 2.72 g (0.025 mol) of chlorotrimethylsilane was stirred for 8 days at room temperature. The progress of the reaction was monitored by IR spectroscopy, following disappearance of the carbonyl absorption band of the initial keto acetal and increase in the intensity of $\text{C}=\text{C}$ absorption. When the reaction was complete, the mixture was filtered from the precipitated salt through a thin layer of silica gel (ICN Silica 60 Å, 63–200 μm , ICN Biomedicals), and the sorbent was washed with hexane–ethyl acetate (20:1). Removal of the solvent, followed by vacuum distillation, gave 3.7 g (49%) of 3,3-diethoxy-2-trimethylsilyloxypropene (**VIa**). The product was purified by preparative GLC using a PAKhV-07 chromatograph (injector temperature 170°C; oven temperature 90°C; 500 \times 1-cm column packed with XE-60 on Chromaton N-AW-HMDS; carrier gas helium), bp 89°C (10 mm), $n_D^{16.5} = 1.4180$. ^1H NMR spectrum, δ , ppm: 0.06 s (9H, SiMe_3), 1.05 t (6H, CH_3CH_2), 3.35 m and 3.45 m (4H, OCH_2), 4.14 s (1H, OCH), 4.40 s (1H, HC=), 4.46 s (1H, HC=). ^{13}C NMR spectrum (CDCl_3), δ_C , ppm: 0.04 (CH_3 , SiMe_3), 15.02 (CH_3), 61.64 (OCH_2), 92.17 ($\text{H}_2\text{C}=\text{C}$), 100.37 (OCHO), 154.17 (COSi). Mass spectrum, m/z (I_{rel} , %): 218 (0) $[M]^+$, 174 (100) $[M - \text{OC}_2\text{H}_4]^+$, 159 (2), 145 (11) $[M - \text{SiMe}_3]^+$, 129 (81) $[M - \text{OSiMe}_3]^+$, 115 (8) $[M - \text{CH}(\text{OEt})_2]^+$, 103 (33) $[\text{CH}(\text{OEt})_2]^+$, 73 (66) $[\text{SiMe}_3]^+$, 59 (22), 47 (28), 43 (17). IR spectrum, ν , cm^{-1} : 2970 s, 2930 s, 2880 s, 1644 s ($\text{C}=\text{C}$), 1440, 1400, 1370, 1250 s ($\text{O}-\text{Si}$), 1100 s ($\text{O}-\text{Si}$), 1060 s, br ($\text{C}-\text{O}-\text{C}$), 850 s, 755 m. Found, %: C 54.85; H 10.03; Si 12.18. $\text{C}_{10}\text{H}_{22}\text{O}_3\text{Si}$. Calculated, %: C 55.0; H 10.15; Si 12.86.

Reaction of 1,1-dibutoxy-2-propanone (Vb) with chlorotrimethylsilane (run no. 2). A mixture of 30 ml of methylene chloride, 5.06 g (0.025 mol) of compound **Vb**, 2.53 g (0.025 mol) of triethylamine, 1.0 g (0.007 mol) of DBU, and 2.72 g (0.025 mol) of chlorotrimethylsilane was stirred for 7 h at 42°C. The mixture was filtered through a layer of aluminum oxide (Woelm neutral TLC, M Woelm Eschwege, Germany; preliminarily dried under reduced pressure), and the sorbent was washed with hexane–ethyl acetate (20:1). The solvent was distilled off, and the residue

was distilled under reduced pressure to obtain 3.87 g (56%) of a fraction with $n_D^{17} = 1.4235$. After triple distillation, we isolated 3,3-dibutoxy-2-trimethylsiloxypropene (**Vib**) with bp 84°C (1 mm), $n_D^{18} = 1.4295$. ^1H NMR spectrum, δ , ppm: 0.2 s (9H, SiMe_3), 0.91 t (6H, CH_3 , Bu), 1.38 m (4H, CH_2), 1.53 m (4H, CH_2), 3.39 m and 3.52 m (4H, OCH_2), 4.23 (1H, OCHO), 4.51 s (1H, HC=), 4.56 s (1H, HC=). Mass spectrum, m/z (I_{rel} , %): 274 (1) $[M]^+$, 202 (39), 159 (17) $[\text{CH}(\text{OBu})_2]^+$, 144 (13), 129 (91), 115 (5) $[M - \text{CH}(\text{OBu})_2]^+$, 103 (33), 73 (71) $[\text{SiMe}_3]^+$, 57 (100) $[\text{Bu}]^+$, 41 (57). IR spectrum, ν , cm^{-1} : 2950 s, 2930 s, 2875 s, 1640 s (C=C), 1740, 1380, 1250 s (O-Si), 1100 s (Si-O), 1070 s, br (C-O-C), 850 s, 755. Found, %: C 61.87; H 10.91; Si 10.22. $\text{C}_{14}\text{H}_{30}\text{O}_3\text{Si}$. Calculated, %: C 61.26; H 10.02; Si 10.23.

Reaction of 1,1-dibutoxy-2-propanone (Vb) with trimethylsilyl trifluoromethanesulfonate (run no. 3). A mixture of 50 ml of carbon tetrachloride, 12.61 g (0.1248 mol) of Et_3N , and 9.23 g (0.0415 mol) of trimethylsilyl trifluoromethanesulfonate was cooled to -20°C , and 8.4 g (0.0415 mol) of compound **Vb** was added. The mixture was allowed to warm up to room temperature and was stirred for 6 h at 20°C . The upper oily (salt) layer was separated, and the bottom layer (more mobile) was distilled under reduced pressure. The product was identical to compound **Vib** (run no. 2) in the ^1H NMR and IR spectra.

The reaction of 1,1-diethoxy-2-propanone (Va) with trimethylsilyl trifluoromethanesulfonate (run no. 4) was carried out as in the previous experiment using 0.0415 mol of compound **Va**, but the more mobile layer before distillation was passed through a small column charged with aluminum oxide to remove residual triethylamine and resinous impurities. The product was identical to compound **Via** (run no. 1) in the ^1H NMR, IR, and mass spectra.

Reaction of 1,1-dibutoxy-2-propanone (Vb) with the system $\text{Me}_3\text{SiOTf-DBU-ClCH}_2\text{CH}_2\text{Cl}$ (run no. 5). A mixture of 25 ml of 1,2-dichloroethane, 0.5 g (0.0025 mol) of acetal **Vb**, 0.38 g (0.0028 mol) of DBU, and 0.55 g (0.0025 mol) of trimethylsilyl trifluoromethanesulfonate was stirred for 15 min at room temperature (23°C) and then for 1 h at 82°C . The product was identical in the ^1H NMR and IR spectra to compound **Vib** obtained in run no. 2.

Reaction of 1,1-dibutoxy-2-propanone (Vb) with trimethylsilyl trifluoromethanesulfonate in the presence of DBU (run no. 6). The reaction was performed in carbon tetrachloride following the above procedure with 0.5 g (0.0025 mol) of acetal **Vb**;

the mixture was heated at the boiling point (75°C); reaction time 4.5 h.

Reaction of 1,1-diethoxy-2-propanone (Va) with *tert*-butyldimethylsilyl trifluoromethanesulfonate (run no. 9). Triethylamine, 1.77 g (0.0175 mol), DBU, 0.53 g (0.0035 mol), compound **Va**, 1.83 g (0.0125 mol), and *tert*-butyldimethylsilyl trifluoromethanesulfonate, 3.49 g (0.0175 mol), were added to 40 ml of carbon tetrachloride. After heating for 12 h at 75°C , the mixture divided into layers. The upper (salt) layer was separated, and the bottom (mobile) organic layer was evaporated. The residue was subjected to double vacuum distillation to isolate 1.7 g ($\sim 52\%$) of 2-*tert*-butyldimethylsiloxy-3,3-diethoxypropene (**Vic**). ^1H NMR spectrum, δ , ppm: 0.15 s (6H, SiMe_2), 0.91 s (9H, CH_3 , *t*-Bu), 1.19 t (6H, CH_3 , Et, $^3J = 7$ Hz), 3.49 d.q and 3.61 d.q (4H, OCH_2 , $^3J = 7$ Hz), 4.26 s (1H, CH), 4.56 s (1H, HC=), 4.63 s (1H, HC=). ^{13}C NMR spectrum, δ_{C} , ppm: -4.77 (CH_3Si), 15.12 (CH_3 , Et), 25.56 (CH_3 , *t*-Bu), 61.49 (OCH_2), 91.89 ($=\text{CH}_2$), 100.24 (OCHO), 154.45 ($=\text{CO}$). Mass spectrum, m/z (I_{rel} , %): 215 (6) $[M - \text{OEt}]^+$, 203 (2), 159 (15), 145 (1) $[M - \text{SiMe}_2\text{Bu-}t]^+$, 129 (100) $[M - \text{OSiMe}_2\text{Bu-}t]^+$, 115 (25) $[\text{SiMe}_2\text{Bu-}t]^+$, 103 (13) $[\text{CH}(\text{OEt})_2]^+$, 75 (20), 57 (21) $[\text{C}_4\text{H}_9]^+$. IR spectrum, ν , cm^{-1} : 2980 s, 2970 s, 2920 s, 1640 s (C=C), 1470 m, 1360 m, 1250 s, br (O-Si), 1100 (O-Si), 1050 s, br (C-O-C), 820, 780. Found, %: C 59.78; H 10.48; Si 10.94. $\text{C}_{13}\text{H}_{28}\text{O}_3\text{Si}$. Calculated, %: C 59.95; H 10.83; Si 10.78.

Reaction of 1,1-dibutoxy-2-propanone (Vb) with *tert*-butyldimethylsilyl trifluoromethanesulfonate (run no. 10). Triethylamine, 1.77 g (0.0175 mol), DBU, 0.62 g (0.0041 mol), compound **Va**, 2.5 g (0.0125 mol), and *tert*-butyldimethylsilyl trifluoromethanesulfonate, 3.45 g (0.0175 mol), were added to 38 ml of carbon tetrachloride, and the mixture was heated for 13 h at 75°C . After cooling, the upper salt layer was separated, and the bottom organic layer was subjected to double vacuum distillation to isolate 1.9 g ($\sim 49\%$) of 2-*tert*-butyldimethylsiloxy-3,3-dibutoxypropene (**Vid**), bp 114°C (1 mm), $n_D^{20} = 1.4385$. ^1H NMR spectrum, δ , ppm: 0.15 s (6H, SiMe_2), 0.9 s (9H, CH_3 , *t*-Bu), 0.88 t (6H, CH_3 , Bu), 1.34 (4H, CH_2), 1.53 (4H, CH_2), 3.41 m and 3.53 m (4H, OCH_2), 4.25 s (1H, CH), 4.55 s (1H, HC=), 4.62 s (1H, HC=). ^{13}C NMR spectrum, δ_{C} , ppm: -4.7 (CH_3Si), 13.96 (CH_3 , Bu), 19.49 (CH_2), 25.64 (CMe_3), 31.89 (CH_2), 65.7 (OCH_2), 91.92 ($=\text{CH}_2$), 100.36 (OCHO), 154.45 ($=\text{CO}$). Mass spectrum, m/z (I_{rel} , %): 259 (1) $[M - \text{Bu}]^+$, 243 (1) $[M - \text{OBu}]^+$, 187 (3) $[M - \text{OBu} - \text{C}_4\text{H}_8]^+$, 171 (3), 159 (1) $[\text{BuOCHOBu}]^+$, 129 (100) $[M - \text{BuO} - 2\text{Bu}]^+$, 101

(5), 73 (8), 57 (27). IR spectrum, ν , cm^{-1} : 2960 s, 2930 s, 2860 s, 1645 s (C=C), 1470 m, 1370 m, 1260 s, br (O-Si), 1100 s (O-Si), 1070 s, br (C-O-C), 830, 790. Found, %: C 64.58; H 11.73; Si 8.84. $\text{C}_{17}\text{H}_{36}\text{O}_3\text{Si}$. Calculated, %: C 64.50; H 11.46; Si 8.87.

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