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Reaction of silyl- and germyl-ketenes with silyldiazomethanes

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Abstract

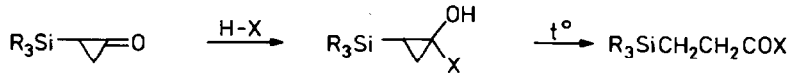
The reaction of trialkylsilyl- and trialkylgermyl-ketenes with trialkylsilyldiazomethanes proceeds stereoselectively to give one of two feasible stereoisomers, the *Z*-2,3-bis(Si,Ge)-substituted cyclopropanone. The addition of nucleophilic reagents such as benzyl alcohol, $LiAlH_4$ or (trimethylsilyl)dialkylphosphites is highly stereoselective and is a convenient method for assigning the stereochemical configuration of 2,3-substituted cyclopropanones.

Introduction

Previously we reported the reaction of silyl- and germyl-ketenes with diazomethane [1,2,3] to afford mono(Si,Ge)-substituted cyclopropanones which were relatively stable compounds under ambient conditions but were at the same time also very reactive [1–5] as compared to the more common cyclopropanones [6].

Like cyclopropanone itself and its derivatives, monosilylcyclopropanones react readily with water, alcohols, dialkylamines, carboxylic acids etc. The resulting addition compounds are easily isomerized by heat e.g. during distillation leading to

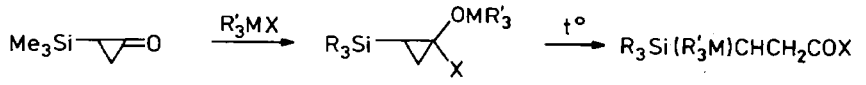
propionic acid derivatives, the products of C^1–C^3 bond cleavage [3].



(1)

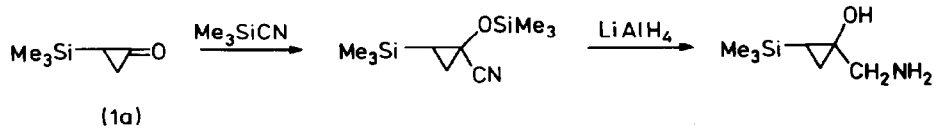
X = OH, OR, NR_2, OCOR

Silyl- and germyl-cyclopropanones also react with a variety of organometallics of the IVB elements, resulting in new organoelement compounds of linear and cyclic structures [3]:

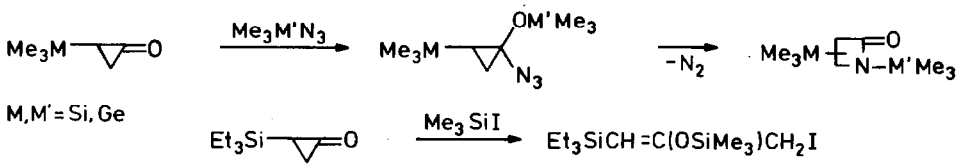


(1)

M = Si, Ge, Sn; X = OR, NR_2

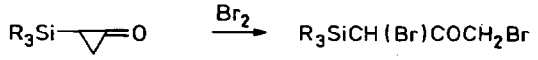


(1a)



M,M' = Si, Ge

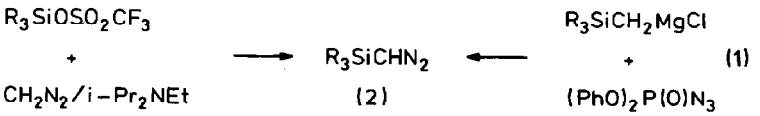
Silylcyclopropanones also undergo C^2–C^3 cleavage when treated with bromine:



In our present paper we report on the syntheses and the structural assignment studies of 2,3-bis(Si,Ge)cyclopropanones.

Results and discussion

We have studied the reaction of trialkylsilyl- and trialkylgermyl-ketenes [3] with trialkylsilyldiazomethanes [2], now readily available reactants due to recent synthetic developments [2]. The following main routes for the syntheses of trimethylsilyldiazomethanes were used [7,8]:



(2)	R_3
a	Me_3
b	$MeEt_2$

A new organometallic diazomethane, methyldiethylsilyldiazomethane, was obtained by a similar reaction.

In earlier experiments we found that one usually had to work at low temperature (−78°C), at relatively high dilutions of reactants and with slow addition of the diazomethane solution to an excess of trimethylsilylketene (**3a**) in order to synthesize mono-element-substituted cyclopropanones(1) [1–3].

Violation of these conditions leads to the formation of the corresponding isomeric silylcyclobutanones owing to subsequent reaction of the cyclopropanones with diazomethane to the partial polymerization of the cyclopropanone.

However, the reaction of trimethylsilyldiazomethane (**2a**) with trimethylsilylketene (**3a**) in an equimolar ratio proceeds smoothly by mixing etherial solutions of the reactants at −10°C and warming to ambient temperatures thus obtaining 2,3-bis(trimethylsilyl)cyclopropanone (**4a**) in 64% yield. Satisfactory elemental analyses were obtained for this compound and its suggested structure was confirmed by IR and ^{1}H, ^{13}C, ^{29}Si NMR spectra together with additional chemical evidence. In the IR spectrum of **4a** an intense absorption band at 1815 cm^{-1} is characteristic and is identified as (>C=O), the valence vibration of the carbonyl group in cyclopropanones [9].

The proton NMR spectrum shows two singlets with integrals 9/1 assigned by us to the protons of two Me_3Si groups at δ 0.12 ppm and two protons of the cyclopropanone ring at δ 1.49 ppm the latter value being similar to that reported for *trans*-2,3-di(t-butyl)cyclopropanone at δ 1.55 ppm [10].

The ^{13}C NMR spectrum supplies only signals corresponding to three types of carbon atom: C^1 (δ 209.9 ppm); C^2, C^3 (δ 12.9 ppm ^{1}J(C,H) 144.6 Hz); Me_3Si (δ −0.58 ppm $^{1}2J$(C,H) 119.5 Hz). The data obtained give clear evidence for the presence of two possible stereoisomers of **4a**; similarly, the following reactions afford only yields of 65–90% for one of two possible stereoisomers of cyclopropanones **4b–4e** (eq. 2).

Physical constants, yields and elemental analyses of compounds **4a–4e** are

Table 1

Properties of cyclopropanones (**4a**)–(**4e**)

Cyclopropanone	Yield (%)	B.p. (°C/mmHg)	n_D^{20}	Formula (mol. mass)	Analysis (Found (calcd.) (%))		
					C	H	Si
2,3-Bis(trimethylsilyl)-cyclopropanone (**4a**)	64	66-67 (2)	1.4535	$C_9H_{20}OSi_2$ (200.42)	53.67 (53.97)	10.12 (10.06)	27.86 (28.02)
2-Trimethylsilyl-3-methyldiethylsilylcyclopropanone (**4b**)	90	95-96 (1)	1.4655	$C_{11}H_{24}OSi_2$ (228.48)	57.56 (57.83)	10.46 (10.59)	24.58 (24.58)
2,3-Bis(methyldiethylsilyl)-cyclopropanone (**4c**)	90	93-94 (0.5)	1.4790	$C_{13}H_{28}OSi_2$ (256.53)	60.45 (60.87)	10.77 (11.02)	21.64 (21.89)
2-Trimethylsilyl-3-triethylgermylcyclopropanone (**4d**)	65	87-88 (0.1)	1.4872	$C_{12}H_{26}GeOSi$ (286.92)	49.86 (50.23)	9.17 (9.13)	–
2-Methyldiethylsilyl-3-triethylgermylcyclopropanone (**4e**)	85	121-122 (0.1)	1.4925	$C_{14}H_{30}GeOSi$	53.29 (53.39)	9.71 (9.60)	– –

Table 2

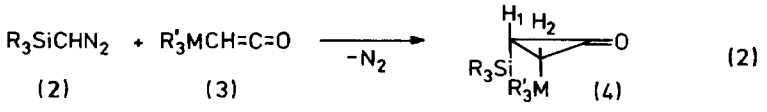
IR and ^{1}H NMR spectral data for cyclopropanones **1a** and **4a–4e**

Compound	IR ν(C=O) (cm^{-1})	^{1}H NMR (in $CDCl_3$; internal ref.; $CHCl_3$, δ (ppm), J(H,H) (Hz) [a]
1a	1815	0.052(s,9H,$SiMe_3$); ABC system: 1.28(m,1H,H^1); 1.63(m,1H,H^2); 1.41(m,1H,H^3); $J(H^1,H^2)$ 16.5; $J(H^1,H^3)$ 11.2; $J(H^2,H^3)$ −10.2
4a	1815	0.12(s,18H,2$SiMe_3$); 1.49(s,2H,H^1,H^2)
4b	1810	0.11(s,9H,$SiMe_3$); 0.037(s,3H,SiMe); 1.47(s,2H,H^1,H^2)
4c	1810	0.046(s,3H,SiMe); 1.47(s,2H,H^1,H^2)
4d	1810	0.10(s,9H,$SiMe_3$); AB system: 1.52(d,1H,H^1); 1.64(d,1H,H^2); $J(H^1,H^2)$ 20.3
4e	1798	0.017(s,3H,SiMe); AB system: 1.50(d,1H,H^1); 1.63(d,1H,H^2); $J(H^1,H^2)$ 20.3

[a] Multiplets of the ethyl groups are in their standard range.

summarized in Table 1, IR and NMR data are collected in Table 2 and compared to those of cyclopropanone **1a**.

In principle, the assignment of the structure of a 2,3-disubstituted cyclopropanone may be achieved on the basis of comparing the magnitude of the coupling constant of protons H^1 and H^2 at the adjacent carbon atoms of the ring:



(2) (3) (4) (2)

(2)	R_3
a	Me_3
b	$MeEt_2$

(3)	R'_3M
a	Me_3Si
b	$MeEt_2Si$
c	Et_3Ge

(4)	R_3	R'_3M
a	Me_3	Me_3Si
b	Me_3	$MeEt_2Si$
c	$MeEt_2$	$MeEt_2Si$
d	Me_3	Et_3Ge
e	$MeEt_2$	Et_3Ge

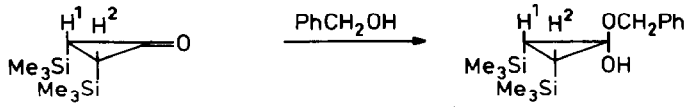
The 200 MHz ^{1}H NMR spectrum of cyclopropanone **4b** with different substituents (Me_3Si, $MeEt_2Si$), run in different solvents $CDCl_3$ and C_6D_6, exhibits only a singlet for the cyclopropane protons whereas protons H^1 and H^2 of the cyclopropanones **4d** and **4e** are not equivalent: their spectra are characterized by an AB-system with J_{AB} 20.3 Hz.

This value of the coupling constant suggests a *cis*-structure for compounds **4**. However, further evidence supporting this assumption was needed since we had no spectra of the *trans*-isomers to compare with. This piece of evidence was then obtained during our investigation of addition reactions of several nucleophiles, e.g. benzyl alcohol, $LiAlH_4$, (trimethylsilyl)dialkylphosphites, to the carbonyl group: as a result, the C^1-atom of the cyclopropanone became pseudo symmetric in the addition product.

Reaction of cyclopropanone (4a) with benzyl alcohol

The reaction was found to proceed exothermally forming adduct **5** in 90% yield (after purification) eq. 3.

The signals of the two Me_3Si groups, the cyclopropane protons, the OH and OCH_2 groups are observed in the ^1H NMR spectrum as singlets in a 18/2/1/2 ratio:



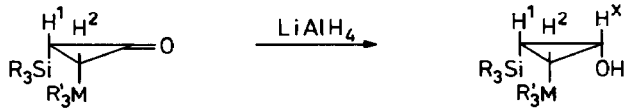
(4a) (5) (3)

Atom C^1 of the adduct **5** is not asymmetric giving only evidence for the *cis*-configuration of the Me_3Si-groups; a *trans*-configuration of the Me_3Si groups would induce chirality on C^1 of the ring resulting in a non-equivalence of the diastereotopic methylene protons [10] and the Me_3Si groups.

Taking into account the spectroscopic evidence after addition of the nucleophile to cyclopropanone (**1a**) [3] we assume that in the isomer under consideration the Me_3Si and OH groups are in a *cis*-configuration. Its formation may be explained by steric hindrance exerted by two bulky organoelement residues: thus addition is preferentially from the opposite side of the cyclopropanone ring plane.

Reactions of cyclopropanones 4a, 4b and 4e with $LiAlH_4$

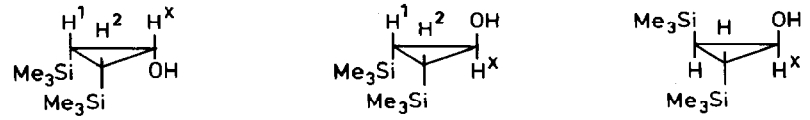
Cyclopropanones **4a**, **4b** and **4e** react smoothly with $LiAlH_4$ to give the corresponding cyclopropanols **6** (eq. 4):



(6) (4)

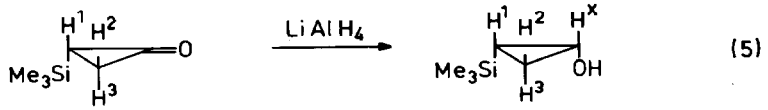
(6)	R_3	R'_3M
a	Me_3	Me_3Si
b	Me_3	$MeEt_2Si$
c	$MeEt_2$	Et_3Ge

The ^1H NMR spectrum of **6a** shows singlets for Me_3Si (at δ 0.06 ppm) and OH (at δ 1.48 ppm) groups, also signals of the A_2X system of the cyclopropane protons with J_{AX} 6.6 Hz. Therefore we assume that **6a** is one of the three possible stereoisomers, viz. that of *Z,Z*-configuration, all three H atoms of the ring are in the *cis*-position:



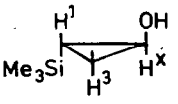
Z,Z-(6a) *E,Z*-(6a) *Z,E*-(6a)

The alternative *Z,E*-**6a** structure, with a *trans*-configuration of the Me_3Si groups, would appear in the ^1H NMR spectrum with two singlets for the non-equivalent Me_3Si groups and with an ABX pattern for the ring protons. We discarded structure *E,Z*-**6a** considering the fact that the value of J_{AX} must be equal to about 3–4 Hz as detected in a ^1H NMR study of the isomeric 2-trimethylsilylcyclopropan-1-ols (**7**), which were obtained as a mixture of the *Z,E*-isomers (70/30) after reduction of the cyclopropanone **1a** with $LiAlH_4$ [5]:



(5)

70%, *Z*-(7), $J(H^1,H^X)$ 7 Hz



30%, *E*-(7), $J(H^1,H^X)$ 4.0 Hz

For similar reasons cyclopropanol **6b** is ascribed the *Z,Z*-structure: in the ^1H NMR spectrum of cyclopropanol **6c** there are signals of an ABX system of the cyclopropane ring protons with J_{AB} 13.4 Hz, $J_{AX} = J_{BX} = 6.5$ Hz (see Table 3). The ^{13}C and ^{29}Si NMR spectra also confirm the predominant formation of only one stereoisomer (reaction 4).

In the ^{13}C NMR spectrum of **6a** three signals are present: C^1 (δ 55.86 ppm), C^2, C^3 (δ 11.33 ppm) Me_3Si (δ 0.8 ppm) and ^{29}Si NMR spectrum of **6a** with δ 0.97

Table 3

IR and ^1H NMR data for cyclopropanols **6** and **7**

Compound	IR ν(C=O) (cm^{-1})	^1H NMR (in $CDCl_3$; internal ref.: $CHCl_3$, δ (ppm), J(H,H) (Hz) [a]
6a	3390	0.06(s,18H,2Me_3Si); 1.48(s,1H,OH); A_2X system: −0.1(d,2H,H^1,H^2); 3.99(t,1H,H_X); $J(H^{1,2},H^X)$ 6.6
6b	3480	0.12(s,9H,Me_3Si); 0.07s,3H,MeSi); 1.63(s,1H,OH); A_2X system: −0.1(d,2H,H^1,H^2); 4.07(t,1H,H^X); $J(H^{1,2},H^X)$ 6.5
6c	3480	0.04(s,3H,MeSi); 1.46(s,1H,OH); ABX system: −0.1(q,1H,H^1); 0.19(q,1H,H^2); $J(H^1,H^2)$ 13.4; 4.05(t,1H,H^X); $J(H^{1,2},H^X)$ 6.5
Z-**7**	3350	0.04(s,9H,Me_3Si); 1.58(s,1H,OH); ABCX system: −0.38(m,1H,H^1; 0.48(m,1H,H^3); −0.66(m,1H,H^2); 3.72(m,1H,H^X); ($J(H^1,H^2)$ 11.4; $J(H^1,H^3)$ 4.6; $J(H^1,H^3)$ 8.7; $J(H^2,H^3)$ −4.6; $J(H^1,H^X)$ 7.0; $J(H^2,H^X)$ 6.0; $J(H^3,H^X)$ 2.9
E-**7**		−0.08(s,9H,Me_3Si); 1.64(s,1H,OH); ABCX system: 0.16(m,1H,H^1); −0.70(m,1H,H_2); 0.34(m,1H,H_3); 3.36(m,1H,H^X); $J(H^1,H^2)$ 11.9; $J(H^1,H^3)$ 8.3; $J(H^2,H^3)$ −4.7; $J(H^1,H^X)$ 4.0; $J(H^2,H^X)$ 2.6; $J(H^3,H^X)$ 5.7

[a] See ref. for Table 2.

Table 4

Properties of cyclopropanols **6a–6c**

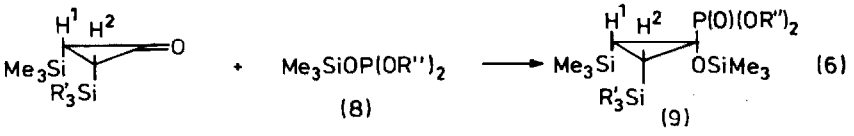
Cyclopropanol	Yield %	B.p. (°C/ mmHg)	n_D^{20} (M.p. (°C))	Formula (mol. mass)	Analysis (Found (calcd.) (%))		
					C	H	Si
2,3-Bis(trimethylsilyl)-cyclopropan-1-ol (**6a**)	74	55–56/1	(55–56)	$C_9H_{22}OSi_2$ (202.43)	53.28 (53.40)	10.82 (10.95)	27.36 (27.74)
2-Trimethylsilyl-3-methyl-diethylsilylcyclopropan-1-ol (**6b**)	92	75–76/1	1.4735	$C_{11}H_{26}OSi_2$ (230.48)	57.80 (57.32)	11.67 (11.37)	24.26 (24.37)
2-Methyldiethylsilyl-3-triethylgermylcylopropan-1-ol (**6c**)	80	110–112/2	1.4930	$C_{14}H_{32}GeOSi$ (316.99)	52.88 (53.04)	10.06 (10.18)	– –

ppm. Physical constants, yields and elemental analyses of **6a–6c** are listed in Table 4.

Reaction of cyclopropanones 4a, 4b and 1a with (trimethylsilyl)dialkylphosphites (8)

Reactions of the cyclopropanones **4a** and **4b** with silylphosphites (**8**) normally take several hours at ambient temperature affording the corresponding addition products **9a**, **9b** in 90% yield. In accordance with ^1H, ^{13}C, ^{31}P NMR data the compounds **9** are only obtained as one isomer (see Tables 5, 6, and eq. 6).

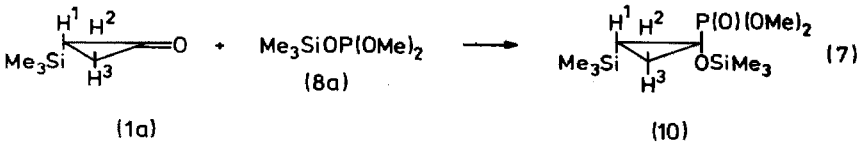
The appearance of a doublet for the cyclopropane protons H^1 and H^2 in the ^1H NMR spectrum confirms their *cis*-configuration; a *trans*-configuration of the same protons would induce an ABX spin system (X being the phosphorus atom).



(8)	R''
a	Me
b	Et

(9)	R'_3	R''
a	Me_3	Me
b	$MeEt_2$	Et

By comparing spectral data of $^3J(P,H)$ 12.8 Hz in these spectra with that obtained previously by us for the adduct of cyclopropanone **1a** with silylphosphite **8a** (eq. 7), the positions of the phosphorus atom and the cyclopropane protons were identified:



$^3J(P,H^1)$ 13.7 Hz, $^3J(P,H^2)$ 9.9 Hz, $^3J(P,H^3)$ 5.3 Hz

Table 5

^{1}H NMR data for compounds **9a**, **9b** and **10**

Compound	^{1}H NMR (in $CDCl_3$; internal ref.: $CHCl_3$, δ (ppm), J(H,H), ^3J(P,H) (Hz) [a]
9a	−0.01(s,18H,2Me_3SiC); 0.0(s,9H,Me_3SiO); 0.51(d^3,J(P,H) 12.8, H^1,H^2); 3.77(d, ^3J(P,H) 10.5, 6H,2$POCH_3$)
9b	0.11(s,9H,Me_3SiC); 0.05(s,3H,MeSi); 0.17 (s,9H,Me_3SiO); 0.54(d, ^3J(P,H) 12.8, 2H,H^1,H^2
10	0.03(s,9H,Me_3SiC); 0.07(s,9H,Me_3SiO); 3.79(d, ^3J(P,H) 10.5, 3H; $POCH_3$, 3.77(d, ^3J(P,H) 10.5,3H,$POCH_3$); ABCX system (X-P atom): 0.26(m,1H,H^1); 1.16(m,1H,H^2); 0.66(m,1H,H^3); J(H^1,H^3) 12.2; J(H^1,H^3) 9.3; J(H^2,H^3) −4.8; ^3J(P,H$_1$) 13.7; ^3J(P,H^2) 9.9; ^3J(P,H^3) 5.3

[a] See footnote for Table 2.

Table 6

^{13}C NMR data for compounds **10**, **11a** and **11b**

Compound	^{13}C NMR (solvent: $CDCl_3$, δ (ppm), J(C,P), Hz)		
	C^1	C^2	C^3
10	54.1,d(227.1)	10.8,s	15.7,d(3.9)
11a	58.3,d(223.1)	15.3,s	13.2,d(0.8)
11b	52.6,d(225.6)	11.1,s	15.2,d(3.4)

Table 7

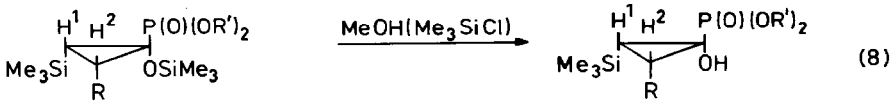
Properties of cyclopropylphosphonates **9–11**

Compound	Yield %	B.p. (°C/mmHg)	n_D^{20} (M.p., °C)	Formula (mol. mass)	Analysis (Found (Calcd.) (%))	
					C	H
Dimethyl [1-trimethylsiloxy--2,3-bis(trimethylsilyl)/] cyclopropyl phosphonate (**9a**)	88	102–103/0.1	1.4585 (55–56)	$C_{14}H_{35}O_4PSi_3$ (382.64)	43.98 (43.95)	8.74 (9.22)
Diethyl (1-trimethylsiloxy-2--trimethylsilyl-3-methyldiethyl-silyl)cyclopropylphosphonate (**9b**)	91	135–136/0.5	1.4660	$C_{18}H_{43}O_4PSi_3$ (438.75)	49.11 (49.28)	9.75 (9.88)
Dimethyl (1-trimethylsiloxy-2--trimethylsilyl)cyclopropyl-phosphonate (**10**)	88	99–100/1	1.4485	$C_{11}H_{27}O_4PSi_2$ (310.51)	42.13 (42.55)	9.02 (8.76)
Diethyl (1-hydroxy-2-trimethyl-silyl-3-methyldiethylsilyl)cyclo-propylphosphonate (**11a**)	[a]	150–151/1	(72–73)	$C_{15}H_{35}O_4PSi_4$ (366.57)	49.36 (49.14)	9.86 (9.62)
Dimethyl (1-hydroxy-2-trimeth-ylsilyl(cyclopropylphosphonate (**11b**)	[a]	115–116/0.1	(83–84)	$C_8H_{19}O_4PSi$ (238.26)	40.28 (40.32)	8.04 (8.04)

[a] Quantitative yield; analyzed without additional purification.

These results are in good agreement with data reported for the *cis/trans* coupling constants ^3J(P,H) of other cyclopropyl phosphonates [12].

In **9b** and **10** Me_3Si groups are quantitatively cleaved off by methanol in the presence of catalytic amounts of Me_3SiCl:



(8)

(9b), (10) (11)

(11)	R	R'
a	$MeEt_2Si$	Et
b	H^3	Me

The ^1H NMR spectra of the hydroxy derivatives **11** clearly show that their configuration is the same as in the initial siloxy compounds. Signals for the phosphorus atoms in **9–11** are found in the same range as those of the cyclopropyl phosphonates (δ 26 ppm) [13].

^{13}C chemical shift data of the cyclopropane carbon atoms are almost identical to those reported for substituted siloxycyclopropanes [14].

Chemical data presented in this paragraph are also in good agreement with an assumed *Z*-configuration of the cyclopropanones **4**. Yields, physical constants and elemental analyses of compounds **9–11** are listed in Table 7.

Experimental

IR spectra: UR-20 (Nujol mull). NMR spectra: Bruker CPX-200, Bruker AM-360 ^{13}C and ^{29}Si NMR spectra: Bruker WP-80; ^{31}P NMR: Varian FT-80A (H_3PO_4 as external standard).

Cyclopropanones (4). To a solution of ketene **3** in 25 ml ether, 0.022 mol of silyldiazomethane in 5 ml ether is added. After warming to room temperature, nitrogen evolves from the reaction mixture. The ether is pumped off, distillation of the liquid residue affords **4** (see: Table 1).

1-Hydroxy-1-benzyloxy-2,3-bis(trimethylsilyl)cyclopropane (5). 1.2 g (0.006 mol) of **4a** and 0.65 g (0.006 mol) of benzyl alcohol are mixed at room temperature: after distillation results in 1.7 g (90%) of **5**, b.p. 123–125 °C/0.1 mmHg; m.p. 53–55 °C (uncorr.). IR: (ν, cm^{-1}) 3420 (OH). ^1H NMR: 0.16 (s, 18H; $2 \times Me_3Si$); 0.67 (s, 2H; H^1, H^2); 2.7 (s, 1H, OH): 4.73 (s, 2H; OCH_2). (Found: C, 61.91; H, 9.06; Si, 18.12. $C_{16}H_{28}O_2Si$ calcd.: C, 62.28; H, 9.15; Si, 18.20%.

Cyclopropanols 6a–6c. An etherial suspension of 0.74 g (0.0193 mol) $LiAlH_4$ in 75 ml ether is refluxed for $\frac{1}{2}$ h; after cooling to 5 °C, 0.0193 mol of **4**, dissolved in 5 ml ether, are added dropwise with vigorous stirring. After a period of 1 h the reaction mixture is treated with 10 ml wet ether, then with 10 ml of water. The etherial layer is dried with $MgSO_4$. The ether evaporated and distillation of the residue affords **6** (see Table 4).

Siloxycyclopropylphosphonates 9a, 9b and 10. A mixture of equimolar amounts of **4** and **8** is kept at room temperature for 5 h: **9a**, **9b** are then isolated by distillation. Adduct **10** is obtained by mixing equimolar amounts of **1a** and **8a** at −30 °C, followed by immediate distillation (see Table 7).

Hydroxycyclopropylphosphonates ***11a, 11b.*** To a mixture of 0.0027 mol of **9b** or **10** in 0.5 ml methanol a drop of freshly distilled Me_3SiCl is added; at about 7 mmHg pressure volatile substances are removed and further pumping at 1 mmHg for 1 h results in an analytically pure sample of **11.**

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