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Electrochemical synthesis of cyclic alkylsilanes

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Abstract

The electrochemical reduction of aliphatic α,ω-dibromides in the presence of polychlorosilanes of the formula R_nSiCl_{4-n} ($n = 0, 2$) was shown to afford heterocyclic silicon compounds in good yield (up to 91%). In contrast to non-electrochemical methods of synthesis of silacycloalkanes, based on the ring closure of terminal unsaturated compounds, the electrochemical route does not produce α-methylated byproducts and the heterocycle formation occurs quite selectively. The yield of cyclic organosilicon compounds goes through a maximum for 1,1-dimethyl-1-silacyclopentane (91%) and roughly decreases for 1,1-dimethyl-1-silacyclobutane (18%) and 1,1-dimethyl-1-silacycloheptane (57%). The formation of 5-silaspiro[4,4] nonane by the electrochemical process occurs with high selectivity despite the multitude of possible reaction pathways and the high probability of polymer formation due to the high functionality of the silicon. The relatively high selectivity of the electrochemical ring closure is suggested to be due to the orientating effect of an electrode in the course of an irreversible reduction of a C–Hal bond in the monosilylated intermediate. A possible mechanism for the process is discussed.

Keywords: Silicon; Electrochemistry; Electrochemical synthesis

1. Introduction

Usually, the heterocyclic organosilicon compounds are obtained by processes that involve intramolecular hydrosilylation, e.g. by ring closure reactions upon addition of dialkylsilanes to 1,5-hexadiene [1], by cyclization of compounds bearing terminal $CH_2=CH-$ and $\equiv Si-H$ groups in the presence of hexachloroplatinic acid or by heating the reaction mixture without a catalyst under pressure [2–6]. Reaction of dichlorosilanes with terminal di-Grignard or alkyldilithium reagents has also been reported [7,8]. The procedure is based on hydrosilylation of unsaturated compounds usually gives a mixture of cyclic products with n and $n-1$ C atoms in the cycle, as a consequence of the presence of two potential centres in the double bond [1,3,5].

Bicyclic silaspiro compounds have also been prepared by the reaction of unsaturated compounds with $SiCl_4$ (in a stepwise manner) or with $(RO)_2SiCl_2$ in a one-pot process [9,10]. The synthesis of silaspiranes by use of a pentacoordinated silicon complex obtained by depolymerization of silica has also been described [11].

In the past decade the electrochemical reduction of halogen derivatives in the presence of chlorosilanes has been shown to be quite an efficient way of preparing carbosilanes bearing a new Si–C bond [12–18]. In the course of electroreduction of some mixed organosilicon dihalogenides such as $ClCH_2(CH_3)_2SiCl$ and $Cl(CH_3)_2SiCH_2CH_2Si(CH_3)_2Cl$ the formation was observed, along with silicon-containing acyclic products, of mono- [19,20], di- [15,18,20] and polysilylated [15,17] silicon heterocycles.

We have reported previously the electrochemical preparation of 1,1-dimethyl-1-silacyclopentane [21] and because of the interest in new methods of making of silicon containing heterocycles we have now studied their formation under electrolytic conditions.

2. Results and discussion

It is known that the two-electron electrochemical reduction of compounds bearing $\equiv C-X$ or $\equiv Si-X$ (X = halogen) bonds gives the corresponding $\equiv C^-$ and $\equiv Si^-$ anions in appropriate aprotic solvents. These

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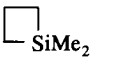
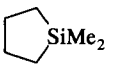
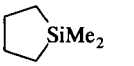
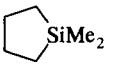
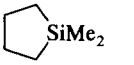
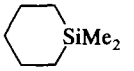
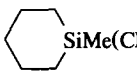
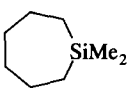
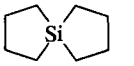
anions, being strong nucleophiles, can react with the starting halides as well as with other electrophiles in the solution, and so the electrochemical reduction of a mixture of alkyl and silyl halogenides in solution must inevitably result in the formation of various cross-products. Thus the problem consists of the selective electrogeneration of a nucleophile in the presence of appropriate halide which acts as a Nu-trapping agent. Unfortunately, the electrophilicity of the halogenated substrate is very often correlated with the ease of its cathodic reduction.

Linear alkylbromides are reduced in DMF/Bu_4NBF_4 in the range of potentials of −1.4 to 2.5 V vs. SCE [22–24], whereas dichlorodialkylsilanes are normally reducible at more negative potentials, though the values of E_p ($E_{1/2}$) reported for this process are very variable e.g. −0.6 V [25,26], −2.08 V [18], −2.28 V [17,27] and −2.5 V [16] all vs. SCE.

Since the $E_{1/2}$ values for reduction of 1,3-dibromopropane, 1,4-dibromobutane and 1,5-dibromopentane are −1.90, −1.99 and −2.10 V vs. SCE, respectively [19,24], one can generate carbanions rather than silylanions in a relatively selective manner by reducing a mixture of ≡C–Br and ≡Si–Cl in the potential-controlled mode at low current density at the bottom of the dibromide reduction wave.

Silicon heterocycles were obtained in two operational modes, i.e. either in an electrolytic cell with cathodic

Table 1
Electrosynthesis of cyclic alkylsilanes [a]

Run	Chain-forming α,ω-dihalide	Chlorosilane	Product	Electricity (F mol^{-1}) [b]	Yield (%) [c]
1	$Br(CH_2)_2Br$	$(CH_3)_2SiCl_2$	–	2.4	– [d]
2	$Br(CH_2)_3Br$	$(CH_3)_2SiCl_2$		4.0	18
3	$Cl(CH_2)_3Cl$	$(CH_3)_2SiCl_2$	–	4.0	–
4	$Br(CH_2)_3Br$	$(CH_3)CH_2CHSiCl_2$		4.2	7
5	$Br(CH_2)_2CH(CH_3)Br$	$(CH_3)_2SiCl_2$	–	4.0	–
6	$Br(CH_2)_4Br$	$(CH_3)_2SiCl_2$		4.0	91
7	$Br(CH_2)_4Br$	$(CH_3)_2SiCl_2$		4.0	43 [e]
8	$Br(CH_2)_4Br$	$(CH_3)_2SiCl_2$		4.1	72 [f]
9	$Cl(CH_2)_4Cl$	$(CH_3)_2SiCl_2$		4.0	12
10	$Br(CH_2)_5Br$	$(CH_3)_2SiCl_2$		4.2	68
11	$Br(CH_2)_5Si(CH_3)_2Cl$	–		2.0	87
12	$Br(CH_2)_5Br$	$(CH_3)CH_2CHSiCl_2$		4.1	83
13	$Br(CH_2)_6Br$	$(CH_3)_2SiCl_2$		4.1	57
14	$Br(CH_2)_4Br$	$SiCl_4$		4.2 [g]	24

[a] THF/DMF (1:1), 0.02 M Et_4NBr, other products are not shown.
[b] $E_{app} = E_{1/2} - 100$ mV (a potential at the base of the reduction wave for the dibromide).
[c] Based on alkylhalide consumed.
[d] $CH_2=CH_2$ was formed.
[e] $E_{app} = -2.6$ V vs. SCE.
[f] C of $Br(CH_2)_4Br$ = C of Me_2SiCl_2 = 0.025 mol l^{-1}.
[g] Q = 4.2 F mol^{-1} even though the ratio of the concentration of the dibromide to that of the chlorosilane is 2:1.

and anodic compartments divided by a sintered glass diaphragm or in an undivided cell fitted with an Al-sacrificial anode. The oxidation potentials of cyclic products are sufficiently positive (e.g. $E_p = 2.66$ V vs. SCE for 1,1-dimethyl-1-silacyclohexane) for the compounds not to be affected by oxidation.

A presence of Al^{3+} cations in the solution does not alter the process substantially, the yields of cyclic products with or without Al^{3+} assistance are comparable and any conclusive trends were not observed.

The results of the electrosynthesis of cyclic silaalkanes are presented in Table 1.

When the reduction of one C–Br bond requires 2 F mol^{-1} of electricity, the formation of the cycle under electrolysis conditions can be represented as consisting of two consecutive reduction steps or in the following scheme:

$$Br(CH_2)_nCH_2Br \xrightarrow[-Br^-]{+2e} Br(CH_2)_n\bar{C}H_2 \xrightarrow[-Cl^-]{RR'SiCl_2} Br(CH_2)_nCH_2SiRR'Cl \quad (1)$$

$$Br(CH_2)_nCH_2SiRR'Cl \xrightarrow[-Br^-]{+2e} \bar{C}H_2(CH_2)_nSiRR'Cl \xrightarrow[-Cl^-]{} \begin{matrix} R' \\ R \end{matrix}\!\!>\!Si\!<\!(CH_2)_{n+1} \quad (2)$$

$n = 2 - 5$

After a nonexhaustive electrolysis (ca. 2 F mol^{-1} of electricity passed) monosilylated derivatives are observed in the mixture, proving that they are half-products in the total four-electron process of heterocycle formation. The electrochemical reduction of 1,1-dimethyl-1-chloro-6-bromo-1-silahexane under similar conditions leads to 1,1-dimetyl-1-silacyclohexane in 87% yield. In addition, plots for the 4 F mol^{-1} process of the product distribution (obtained by a chromatographic analysis) show the characteristic form for the accumulation and consumption of the monosilylated product, which has a maximum at ca. 1.7–1.8 F mol^{-1} (Fig. 1). As can be seen from Table 1, the overall yield of the one-step process is lower than that of the stepwise procedure.

A relatively high selectivity in the formation of the cyclic compounds (taking into account the multitude of possible reaction pathways due to the polyfunctionality of the substrates) is explained by the fact that the electrolysis is carried out at low current density and in the presence of a great excess of an electrophilic substrate in the close-to-electrode layer; in other words the electrolysis is curried out under conditions such that the diffusion of components limits the rate of the process less than does the slow rate of electrogeneration of a nucleophile at the cathode. In addition, the final cyclic product formation is probably a thermodynamically-controlled process.

In considering the selectivity of formation of heterocycles, one must take into account the heterogeneous nature of an electrochemical process together with the

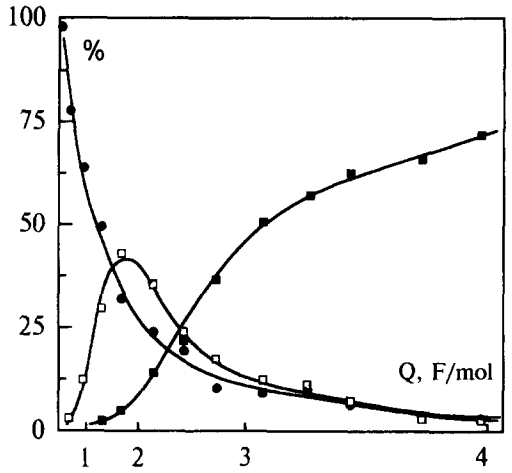
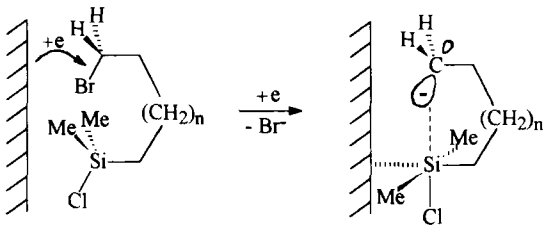


Fig. 1. Variations of the product distribution during the electroreduction of $Br(CH_2)_5Br + Me_2SiCl_2$ in THF/DMF (1:1) + 0.02 M Et_4NBr. ● $Br(CH_2)_5Br$, □ $Br(CH_2)_5SiMe_2Cl$; ■ 1,1-dimethyl-1-silacyclohexane. Q is the quantity of electricity passed during the potential-controlled electrolysis; the scale is linear with time.

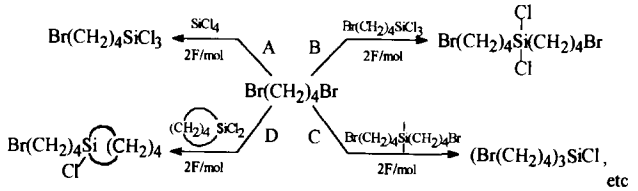
dissociative nature of the electron transfer for both compounds, i.e. for C–Br [22,23] as well as Si–Cl [28,29]. These factors determine the orientation of the molecule of silylated intermediate at the cathode surface (where both electrophilic centres are directed towards the electrode) that favours the ensuing intramolecular S_N2-type process over the intermolecular reaction. Thus, besides being a source of electrons, the electrode also serves as a kind of "matrix" for the nucleophilic reaction. Moreover, the nucleophilic action of the cathode causes the Si to adopt a 5- or 6-coordinated configuration, which is also favourable for nucleophilic attack, as has been shown for hydrolysis of chlorosilanes and the S_N2-Si reactions [30,31].



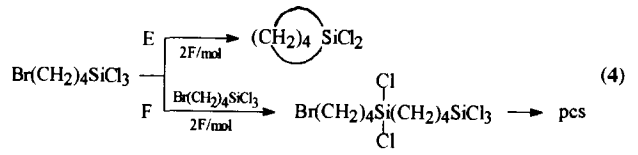
It is interesting to note that a two-fold increase in the concentration of the reagents causes the yield of silylated heterocycles to fall by 20% (see Table 1, run 8), which indicates an increasing participation of polymerisation (which is a second order process). However, regardless of the assistance of the cathode, the alkylation of the chlorosilane does not take place solely in the surface, and evidently the reaction layer is markedly thicker than the distance from the electrode to the outer Helmholtz plane [32]. Nevertheless, the operation of some dominant factor is consistent with the relatively

high yield of the bicyclic product 5-silaspiro[4,4]nonane (24%), since its formation under electrolysis conditions requires many steps and at first sight would not be expected to be very selective.

For the sake of simplicity, an interaction by the scheme $\equiv C^- + \equiv CBr$ will be neglected in the following discussion, in addition, the relative rates of alkylation of $\equiv CBr$ and $\equiv SiCl$ (for $(CH_3)_3SiCl$) substrates by the electrogenerated $\equiv C^-$-anion differ by more than one order of magnitude ($k_{(C^- + SiCl)}/k_{(C^- + CBr)} = 11.4$), as has been demonstrated by an electrochemical competition reaction method [33]. After passing of the first 2 F mol^{-1} of electricity (of the 4 F mol^{-1} required by the stoichiometry), the concentration of the intermediate ω-bromoalkyl-α-trichlorosilane is approximately equal to that of the starting dibromide (Eq. (3A)). At this point concurrence of reactions B, C, E and F takes place:



(3)



(4)

When the monoalkylated ω-bromo product is to be consumed in more than one reaction (in at least B, E and F), the rapid fall in its concentration in the solution means that reactions B and C become less important. It should be noted that only reactions B, E and D can lead to the silaspirane desired. Once the first alkylation has taken place, the intramolecular second C–Si bond formation resulting in a 5-membered cycle must occur somewhat more readily than the intermolecular reaction.

In fact of course, the process is more complex than the simplified version presented above. It is clear, nevertheless, that in order to obtain the highest selectivity towards the silaspirane formation, ω-bromoalkyl-α-trichlorosilane should be made (in 2 F mol^{-1} process with an excess of $SiCl_4$) separately and then used for the two-step electrochemical annelation (see Table 1).

The increased concentration of silylated half-product in the close-to-electrode layer is due to a great affinity of organic silicon compounds for the metallic surface of an electrode [34]. As a consequence, its preferential reduction, in comparison with that of non-silylated bromides, also contributes to the increased yield of the silaspirane.

A radical mechanism for the Si–C bond formation must be rejected, because the $E^0_{BrBu^{\bullet}/BrBu^-}$ value is lower than the $E_{1/2}$ of $Br(CH_2)_4Br$ ($Br(CH_2)_4Br$ shows a single two-electron irreversible wave in contrast to tert- and sec-BuI reduction, where a separated wave of butyl radical reduction appears [22]), and the process occurs with a large overpotential which rules out participation by the radicals [35]. The same is true for the reduction of $\equiv SiCl$. Thus both $\equiv C^{\bullet}$ and $\equiv Si^{\bullet}$ radicals, formed by the reductive cleavage of corresponding bonds, must be immediately reduced to form $\equiv C^-$ and $\equiv Si^-$ anions, respectively.

The occurrence of Nu-attack of $\equiv C^-$ on $\equiv SiCl$ rather than the radical way of the formation of the Si–C bonds is confirmed by the above-mentioned relative rates of alkylation of CBr and SiCl bonds [33] and by the acute lowering in the yield of the cyclic products upon increase in the electrolysis potential (see Table 1, run 7). Indeed, under the reduction of SiCl at potentials

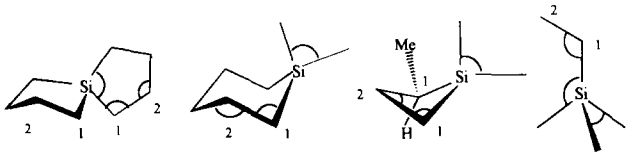
Table 2
Bond angles (°) and distances (Å) and E_{ster} values for silicon heterocycles $(CH_3)_2Si(CH_2)_n$ [a]

	n						Spirane
	3	4	4 [b]	5	6	7	
C(1')–Si–C(1)	56.344	80–81 [c]	91.642	95.512	103.324	110.848	97.373
Si–C(1)–C(2)	61.828	85.237	61.133 [d]	103.481	111.212	115.911	102.523
			57.283 [d]				
C(1)–C(2)–C(3)		101.199	125.347	108.338	113.027	114.913	108.744
C–Si–C [e]	120.936	108 [c]	109.256	111.556	110.616	108.906	97.370
Si–C(1)	1.918	1.898 [c]	1.939 [d]	1.898	1.891	1.887	1.880
			1.813 [d]				
C(1)–C(2)	1.811	1.542	1.538 [d]	1.541	1.537	1.534	1.543
			1.792 [d]				
E_{ster} (kcal mol^{-1}) [f]	173.12	12.842	74.615	8.727	3.621	9.061	21.411

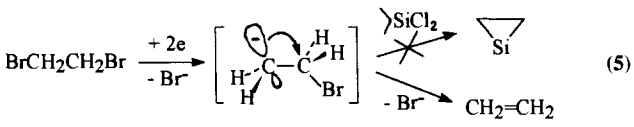
[a] No account is taken of any effect due to the solvent. For Me_3SiEt the corresponding values are: ∠Si–C(1)–C(2) 114.692°, *l*Si–C(1) 1.894 Å, *l*C(1)–C(2) 1.533 Å, ∠H_3C–Si–CH_3 109.471°, E_{ster} = 1.63 kcal mol^{-1}. [b] 1,1,2-Trimethyl-1-silacyclobutane. [c] Taken from Ref. [38]. [d] For Me-substituted and non-substituted C(1) carbon respectively. [e] For *exo*-cyclic C–Si–C angle in monocyclic compound. [f] Ref. [39].

at the beginning of its limit current and higher, the surface concentration of SiCl ($C^s_{\equiv SiCl}$) is zero and a gradient of $C_{\equiv SiCl}$ within the diffusion layer takes place, on the rising part of the reduction curve this gradient increases with an increase in the applied potential. Owing to the nonuniformity of the electrode surface and the distribution of electric field lines in large-scale electrolysis [36], the rising part of a potentiostatic curve extends further toward the negative potentials than in voltammetric experiments. Therefore, the higher the value of E_{app}, the lower the likelihood of obtaining the cyclic carbosilanes. For the same reason, there is a fall in the yield of the cyclic product when the α,ω-dichloro derivative is used as the chain-forming agent in place of the dibromide (Table 1, run 9).

In addition to the fact that the geometry of starting and intermediate monosilylated compounds governs the formation of the cycles, the geometry of the cycle itself must also apparently play an important rôle. In fact, consideration of the strain energies for the parent carbocyclic compounds [37], using angles and bond lengths calculated for cyclic silaalkanes in question (Table 2), shows that the steric strain in the latter is, in general, in line with the yield of the silicon heterocycle. In Table 2, the atoms are labelled as follows:



The fact that we failed to obtain 1,1-dimethyl-1-silacyclopropane by the electrolysis of 1,2-dibromoethane in the presence of $(CH_3)_2SiCl_2$ can be attributed to the extremely strained nature of the three-membered cycle of the product (which is, however, less strained than cyclopropane itself) on the one hand. However, there is the possibility of another reaction pathway in the case of 1,2-dibromoethane, that involving an intramolecular S_N2 process, as in reaction (5)



(5)

Ethylene was indeed collected in the liquid nitrogen trap after the electrolysis, and whereas in the reaction mixture some silylated products were found, but no trace of silacyclopropane was detected. Some volatile Si-containing products were trapped during the electroreduction of $ClSi(CH_3)_2CH_2CH_2Cl$, but we did not detect silacyclopropane among them.

The low yield of 1,1-dimethyl-silacyclobutane could be due to the preferential reduction of 1,3-dibromides at the cathode as a result of a "concerted mechanism" in which the carbocyclic products are formed by synchronous reduction of two C–Br bonds at the electrode [40].

An attempt to obtain the substituted cycle 1,1,2-trimethyl-1-silacyclobutane, starting from 1,3-dibromobutane, was also unsuccessful, probably because the ring in 1,1,2-trimethyl-1-silacyclobutane is significantly more strained as a result of repulsion between CH_3 and $SiCH_3$ groups (see Table 2). In addition, the fact that the primary CBr site of 1,3-dibromobutane is more easily reduced than the secondary $CH(CH_3)Br$ site, owing to the larger partial positive charge on its carbon, means that the first silylation occurs on this centre. Thus, ring closure by internal Nu-attack on SiCl is now disfavoured because the second Nu site is sterically hindered.

In conclusion, the features in the electrosynthesis of silicon heterocycles can be rationalised as follows. The yield of cyclic products is determined, on one hand, by the steric strain of the product itself, and on the other by the accessibility of the electrophilic centre to nucleophilic attack or, in other words, by the distance required for annelation between these two centres. Both of these factors depend on the chain length of the starting α,ω-dibromide. The multitude of possible reaction pathways arising from the presence of other electrophilic centres, as well as the electrolysis potential and concentration of the reagents (both in the bulk and in the close-to-electrode layer) are also of importance in the overall effectiveness of the process. The strain in the cycle seems to be a determining factor only in the formation of 4-membered silacyclobutanes. For larger cycles, the increasing chain length of the monomer precursor raises the probability of intermolecular interaction to give oligomers, and thus the other factors become more important, as in the case of ring closure by intramolecular hydrosilylation [3].

3. Experimental details

3.1. General comments

Voltammetric measurements were carried out using a PU-I polarograph. Large scale potential-controlled electrolyses were performed using a P-5827M potentiostat in a three-electrode system. A Ag/0.1 M $AgNO_3$ in CH_3CN reference electrode was used, separated from the solution by an electrolytic bridge filled with 0.02 M Et_4NBF_4/CH_3CN solution. A Pt cylinder cathode was used, but titanium or stainless steel may also be used.

A solution of tetraethylammoniumbromide (TEAB) and chlorosilane present in a small excess (in THF/

DMF, 1:1) was introduced into the electrolysis cell by a syringe. A preliminary electrolysis was effected until the hydrogen evolution had ceased, then the dihalogenide was added to give a concentration of 0.01 mol l^{-1}. All operations were performed under dry N_2. After passage of the required amount of electricity, the electrolysis was stopped and the solution was added to cool water. The organic layer was extracted three times with pentane, washed with 10% aqueous $KHCO_3$ solution and dried over anhydrous Na_2SO_4. Fractional distillation gave the final products.

The identities of the products were established by NMR ^1H spectroscopy (Tesla 60 MHz spectrometer), elemental analysis, and various physical properties.

The GLC analyses were carried out with a LHM-80 MD chromatograph equipped with 3 m × 3 mm column packed with 7% dihexylphthalate on Chromosorb N-AW.

3.2. Reagents, solutions and products

The DMF was dried for 3 d over drops of freshly melted K_2CO_3 then twice distilled with benzene used in a 1/20 ratio with respect to the volume of the DMF. The first fractions were rejected. The third distillation under reduced pressure was carried out immediately prior to use. THF was distilled over $LiAlH_4$ and then from benzophenone/Na.

The TEAB was dried and stocked over P_2O_5 in vacuo.

All dihalides compounds and chlorosilanes were distilled before use, the latter over Mg turnings.

3.2.1. 1,1-Dimethyl-1-silacyclobutane

B.p. 80–81°C; n_D^{20} 1.4310; d_4^{20} 0.765 (lit. b.p. 79–80°C [41]). ^1H NMR (δ ppm, TMS, $CDCl_3$): 2.10 (m, 2H), 1.00 (t, 4H), 0.17 (s, 6H). Anal. found: C, 60.17%; H, 11.93%. Calc. for $C_5H_{12}Si$: C, 59.91%; H, 12.07%.

3.2.2. 1-Methyl,1-vinyl-1-silacyclobutane

B.p. 109–110°C; n_D^{20} 1.4614; d_4^{20} 0.832 (lit. b.p. 112°C [41]). ^1H NMR (δ ppm, TMS, $CDCl_3$): 5.87 (m, 3H), 2.10 (m, 2H), 1.02 (m, 4H), 0.29 (s, 3H). Anal. found: C, 63.96%; H, 10.79%. Calc. for $C_6H_{12}Si$: C, 64.20%, H, 10.78%.

3.2.3. 1,1-Dimethyl-1-silacyclopentane

B.p. 104–105°C; n_D^{20} 1.4356; d_4^{20} 0.776 (lit. b.p. 107°C; n_D^{25} 1.4335 [7]; b.p. 102°C; n_D^{30} 1.4288 [2];). ^1H NMR (δ ppm, TMS, $CDCl_3$): 1.54 (m, 4H), 0.52 (m, 4H), 0.1 (s, 6H). Anal. found: C, 62.92%; H, 12.46%. Calc. for $C_6H_{14}Si$: C, 63.07%; H, 12.35%.

3.2.4. 1,1-Dimethyl-1-silacyclohexane

B.p. 132–133°C, n_D^{20} 1.4393; d_4^{20} 0.806 (lit. b.p. 133°C; n_D^{25} 1.4380; d^{25} 0.798 [7]). ^1H NMR (δ ppm, TMS, $CDCl_3$): 1.45–1.85 (m, 6H), 0.57 (m, 4H), 0.03 (s, 6H). Anal. found: C, 65.11%; H, 12.34%. Calc. for $C_7H_{16}Si$: C, 65.54%; H, 12.57%.

3.2.5. 1,1-Dimethyl-1-silacycloheptane

B.p. 45°C/12 mm Hg; n_D^{20} 1.4527; d_4^{20} 0.814 (lit. b.p. 163–164°C; n_D^{20} 1.4535; d^{20} 0.8268 [1]). ^1H NMR (δ ppm, TMS, $CDCl_3$): 1.59–1.63 (m, 8H), 0.74 (m, 4H), 0.02 (s, 6H). Anal. found: C, 67.34%; H, 12.57%. Calc. for $C_8H_{18}Si$: C, 67.52%; H, 12.75%.

3.2.6. 5-Silaspiro[4,4]nonane

B.p. 64–65°C/12 mm Hg; n_D^{20} 1.4870; d_4^{20} 0.906 (lit. b.p. 103°C/90 [10], 178.5°C [7]; n_D^{25} 1.4860; d^{25} 0.899 [7]). ^1H NMR (δ ppm, TMS): 1.51 (m, 8H), 0.4–1.1 (m, 8H). Anal. found: C, 68.17%; H, 11.37%. Calc. for $C_8H_{16}Si$: C, 68.49%; H, 11.49%.

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