

Proton and Carbon-13 Nuclear Magnetic Resonance Characteristics of Substituted Silacyclopentanes

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The preparation and separation of the isomers of 1,2,5-trimethyl-1-silacyclopentane and 2,5-dimethyl-1-phenyl-1-silacyclopentane are described. Proton and ^{13}C chemical shifts were used to establish the structure of the *E,E*, *E,Z/Z,E*, and *Z,Z* isomers.

Geometric isomers of cyclic organosilicon compounds have been used extensively by various authors to study the stereochemical pathway with respect to silicon in reactions in which this element is involved.¹⁻⁷ In general, the results are in good agreement with the results obtained with acyclic chiral silicon compounds except in the case of silacyclobutanes, when angle strain causes reactions to proceed with retention of configuration at silicon, where inversion is normally preferred.³ The assignment of configuration of the two geometric isomers of 1,2-dimethyl-1-silacyclopentane, *E* and *Z*, was based on a comparison of chemical shifts of silicon methyls, C-2 methyls, and silicon protons in the proton NMR⁶ and silicon methyls and C-2 methyls in the ^{13}C NMR spectrum.⁸ Methyl groups in a *cis* configuration shield each other more effectively than when they are *trans*, while a C-2 methyl group shields a silicon proton more effectively when in a *cis* than when in a *trans* configuration. Similar effects are operative in *cis*- and *trans*-1,2-dimethylcyclopentane.⁹ This argument, although very tempting, seemed to us not unobjectionable considering the flexibility of the silacyclopentane ring and the ease with which bulky groups can escape steric crowding by conformational changes. Furthermore the reported shift differences are small. It was therefore of interest to study the related system of 1,2,5-trimethyl-1-silacyclopentane, in which, in the case of the *Z,Z* isomer, the silicon methyl group cannot escape steric crowding without causing severe interaction between the two carbon methyls and in which, therefore, any argument concerning relative positions of substituents on the silacyclopentane ring, based on spectroscopic properties, should be more clear cut. In any case, the shift differences between the *Z,Z* and *E,E* isomer should be significantly increased, while the *E,Z/Z,E* isomer is expected to lie between these extremes.

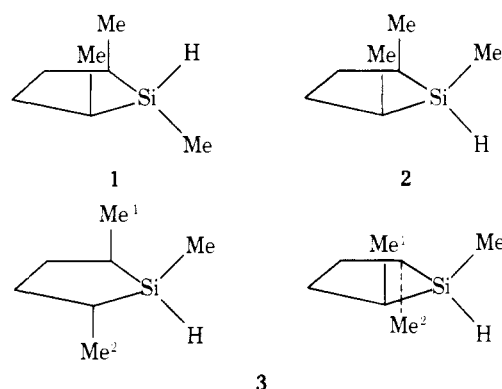
Results and Discussion

1,2,5-Trimethyl-1-silacyclopentane was obtained by reaction of the di-Grignard reagent derived from 2,5-dibromohexane with methylchlorosilane. Yields varied considerably, but never exceeded 27%. When the reaction was performed on a 0.1–0.2 molar scale, yields were optimal. However, when the reaction was scaled up (0.4–0.6 mol), not only did the yields fall to 14 and 5%, respectively, but also the isomeric composition of the product mixture, roughly 1:2:1 when on a 0.1–0.2 molar scale, changed to ~1:1:0 (only traces of one isomer detectable by gas chromatography) when performed on the larger scale. At least nine byproducts of higher boiling point were formed together with oligomeric and polymeric material usually encountered in this reaction. These were not further investigated. We assume that in these cases some of the 1,2,5-trimethyl-1-silacyclopentane, being exposed to a large excess of unreacted di-Grignard, reacted to form higher molecular weight products, the isomer with the least steric hindrance around the reactive silicon–hydrogen bond reacting preferentially. When the dibromide and methylchlorosilane were added simultaneously to a mixture of magnesium in

ether, yields did not improve greatly.

Alternatively, 1,2,5-trimethyl-1-silacyclopentane was prepared in a three-step reaction sequence from 5-chloro-1-hexene via 5-(chloromethylsilyl)-1-hexene and 5-(methylsilyl)-1-hexene, which was ring closed using catalytic amounts of hexachloroplatinic acid, in an overall yield of 5%. As 5-chloro-1-hexene is not readily accessible, this sequence was not further pursued. The identity of the products thus obtained with the products from the reaction of the di-Grignard reagent of 2,5-dibromohexane with methylchlorosilane was established by their retention times on GC columns and their spectroscopic properties (NMR).

1,2,5-Trimethyl-1-silacyclopentane obtained by either of the above mentioned methods consists of a mixture of four isomers, *E,E* (1), *Z,Z* (2), and *E,Z/Z,E* (3). The latter (3) form



an enantiomeric pair. The mixture of isomers A, B, and C, in the order of increasing boiling points and retention times by GC analysis, was separated into its components by (a) preparative GC and (b) careful spinning band distillation. Method a gave pure A, B, and C (pure by NMR), while method b gave only mixtures, enriched in one or two isomers (A/B/C = 50:49:1; 39:58:3; 14:79:7; 11:48:41; 9:23:70; 3:18:79).

Structure Assignments

Proton NMR shifts, obtained at 270 MHz, and ^{13}C NMR shifts, obtained at 67.8 MHz, for A, B, and C are given in Tables I and II, respectively. The assignment of proton NMR spectra is based on selective decoupling experiments. Irradiation on the silicon methyl doublet at δ 0.03 ($J = 3.76$ Hz) in C causes the broad signal of the silicon hydrogen (δ 3.96) to sharpen to a broad triplet ($J \sim 3$ Hz) by elimination of the silicon methyl couplings. Irradiation of δ 3.96 collapses the silicon methyl doublet to a sharp singlet. The complex signal group at $\delta \sim 1.2$ is due to H-2 and H-5. Saturation here results in a sharp quartet for the silicon hydrogen ($J = 3.76$ Hz). (The effect on the carbon methyl was not observable because of its close resonance position to that of H-2.) Analogous decoupling experiments were performed with the other isomers (A and B). The assignment of ^{13}C spectra follows from ^{13}C – ^1H coupled spectra, leaving the assignment of C_2 against C_5 , C_3

Table I. Proton Chemical Shifts of 1,2,5-Trimethyl-1-silacyclopentanes^a

	A ^b	B ^c	C ^d
SiH	3.62 (m)	3.76 (m)	3.96 (m)
SiCH ₃	0.15 (d, <i>J</i> = 3.32 Hz)	0.08 (d, <i>J</i> = 3.32 Hz)	0.03 (d, <i>J</i> = 3.76 Hz)
CCH ₃	1.01 (d, <i>J</i> = 6.86 Hz)	1.01 (d, <i>J</i> = 7.08 Hz)	0.99 (d, <i>J</i> = 7.52 Hz)
		1.11 (d, <i>J</i> = 7.08 Hz)	(d, <i>J</i> = 7.52 Hz)
H _{2,3,3',4,4',5}	1.3–2.1	1.3–2.1	1.2–1.8

^a In parts per million relative to Me₄Si. ^b Registry no.: 68212-43-1. ^c Registry no.: 68295-62-5. ^d Registry no.: 68295-63-6.

Table II. ¹³C Chemical Shifts of 1,2,5-Trimethyl-1-silacyclopentanes^a

	A	B	C
SiCH ₃	-5.5	-8.4	-10.8
CCH ₃	17.4	15.2; 16.9	15.6
C ₂ , C ₅	20.1	17.8; 21.1	16.8
C ₃ , C ₄	34.7	35.6; 36.2	34.4

^a In parts per million relative to Me₄Si.

against C₄, and Me¹ against Me² in B as the only ambiguity.

A and C show only one resonance absorption for C₂/C₅, C₃/C₄, and Me¹/Me² in the ¹³C spectrum and only one carbon methyl doublet in the proton spectrum, while the nonequivalence of C₂ and C₅, C₃ and C₄, and Me¹ and Me² in B is shown by separate signals in proton and ¹³C spectra. It is therefore clear that B, which was also formed in twice the amount of each of the other isomers, is the enantiomeric *Z,E/E,Z* mixture.

The assignment of configuration, i.e., *Z,Z* or *E,E*, to A and C is less obvious. Following the usual argument concerning mutual shielding it can be seen from Table I that the proton chemical shifts follow a consistent pattern. Isomer C has the most shielded silicon methyl and least shielded silicon hydrogen, while the opposite is the case for isomer A. The difference in the carbon methyl proton shifts ($\Delta\delta$ 0.02) is not significant, although a larger difference might have been expected from a comparison with B, where the carbon methyls differ considerably ($\Delta\delta$ 0.10). Most reasonably the "unique" carbon methyl proton shift (δ 1.11) of B is that for (*E*)-methyl Me² in 3, which has neither a 1,2-*cis*-nor a 1,3-*cis*-methyl group. The ¹³C shift values of the carbon methyl groups are at highest field in C and lowest field in A, with those of B lying between. Likewise the ¹³C shift values of the silicon methyls are at highest field in C and lowest field in A, with again an intermediate value in B. All this information points strongly to the assignment of A to the *E,E* and C to the *Z,Z* structure. Furthermore for A a NOE of ~8% is observed, while none could be detected in C, when the carbon methyls were irradiated and the integrals of the silicon-hydrogen resonances were measured. This demonstrates the closer proximity of carbon methyls to silicon hydrogen in A.

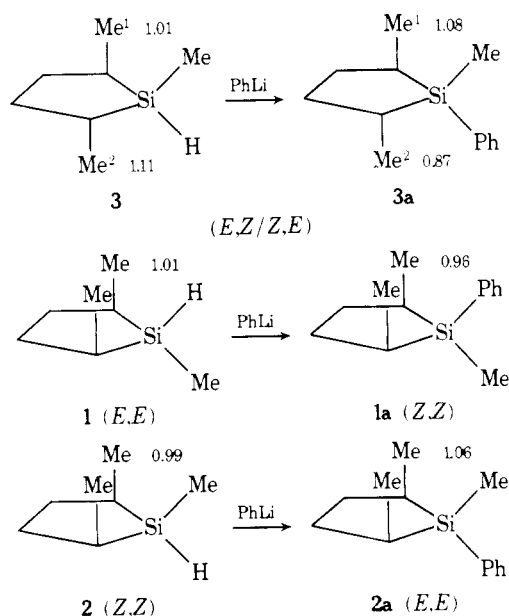
These assignments are further supported by the increased chemical reactivity of C with respect to A. As mentioned earlier, when in the course of synthesis of 1,2,5-trimethyl-1-silacyclopentane the isomeric mixture is exposed to an excess of unreacted Grignard reagent for extended periods, the isomeric ratio of ~1:2:1 (A/B/C) changes to ~1:1:0. This certainly indicates an increased reactivity of the silicon hydrogen due to lower steric hindrance to front-side attack in C with respect to the other isomers.

Table III. Proton Chemical Shifts of 1,2,5-Trimethyl-1-phenyl-1-silacyclopentanes^a

	A-phenylated ^b	B-phenylated ^c	C-phenylated ^d
SiCH ₃	0.35 (s)	0.27 (s)	0.23 (s)
CMe	0.96 (d, <i>J</i> = 7.08 Hz)	1.08 (d, <i>J</i> = 7.30 Hz)	1.06 (d, <i>J</i> = 7.52 Hz)
		0.87 (d, <i>J</i> = 6.41 Hz)	
H _{2,3,3',4,4',5} aromatic	1.1–2.1 7.2–7.6	1.1–2.1 7.0–7.3	1.1–2.1 7.3–7.6

^a In parts per million relative to Me₄Si. ^b Registry no.: 68212-44-2. ^c Registry no.: 68296-96-8. ^d Registry no.: 68295-64-7.

Further evidence for the correctness of structure assignment is provided by the conversion of A (1), B (3), and C (2) into their phenyl derivatives (1a, 3a, and 2a, respectively), a re-



action which is reported to proceed stereospecifically with 100% retention.⁴ (Note that the *E,E* isomer on replacement with retention of hydrogen by phenyl yields the *Z,Z* isomer and conversely.)

As can be seen from Table III, in which the proton NMR data of the phenyl derivatives of A, B, and C are given, the introduction of a phenyl group into B (3) causes a significant upfield shift in one carbon methyl group ($\Delta\delta$ 0.24 or 0.14 ppm). It is reasonable to assume that it is the "unique" methyl group (Me² in 3) which experiences the shielding effect when converted into 3a. The shift is thus $\Delta\delta$ 0.24 upfield for Me², while $\Delta\delta$ 0.07 downfield for Me¹, when 3 is converted into 3a. A similar upfield shift is expected in the case of the *E,E* (1) isomer going to the *Z,Z* phenyl derivative (1a) against a small downfield shift for the *Z,Z* isomer (2 to 2a). The reaction product obtained when isomer A was reacted with phenyllithium shows its carbon methyl resonance in the proton NMR spectrum at 0.96 ppm, 0.05 ppm upfield from A, while the reaction product of isomer C absorbs at 1.06 ppm, 0.07 ppm downfield from C. While the agreement with the predicted value is good for the *Z,Z* isomer (2a), the upfield shift due to the phenyl group in the *E,E* isomer (1a) is considerably smaller than expected, although in the anticipated direction. Conformational aspects clearly need to be taken into consideration. It is precisely the *Z,Z* phenyl isomer (1a) in which the full shielding effect may not be operative. The ¹³C chem-

Table IV. ^{13}C Chemical Shifts of 1,2,5-Trimethyl-1-phenyl-1-silacyclopentanes^a

	A-phenylated	B-phenylated	C-phenylated
SiCH ₃	-3.6	-6.7	-9.0
CCH ₃	15.9	15.4; 15.8	15.9
C ₂ , C ₅	19.7	20.1; 22.3	19.7
C ₃ , C ₄	34.7	35.7; 36.3	34.7

^a In parts per million relative to Me₄Si.**Table V.** Proton Chemical Shifts of 1-Phenyl-2,5-dimethylsilacyclopentanes^a

	X ^b	Y ^c	Z ^d
SiH	4.28 (t, <i>J</i> = 3.68 Hz)	4.13 (t, <i>J</i> = 2.65 Hz)	4.44 (t, <i>J</i> = 3.37 Hz)
CMe	1.20 (d, <i>J</i> = 6.63 Hz) 0.90 (d, <i>J</i> = 7.08 Hz)	1.20 (d, <i>J</i> = 6.63 Hz)	1.00 (d, <i>J</i> = 7.74 Hz)
aliphatic	1.2-1.8	1.2-1.8	1.2-1.8
aromatic	7.3-7.6	7.3-7.6	7.3-7.6

^a In parts per million relative to Me₄Si. ^b Registry no.: 68212-45-3. ^c Registry no.: 68295-65-8. ^d Registry no.: 68295-66-9.**Table VI.** ^{13}C Chemical Shifts of 1-Phenyl-2,5-dimethyl-1-silacyclopentanes^a

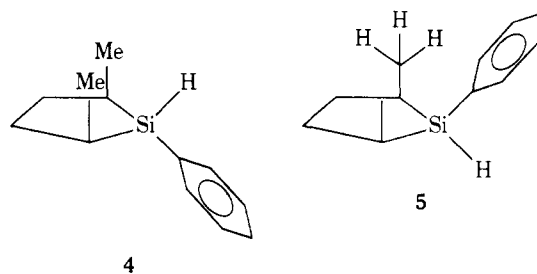
	X	Y	Z
CCH ₃	15.7; 16.9	17.5	16.2
C ₂ , C ₅	19.4; 20.6	20.1	18.3
C ₃ , C ₄	35.8; 36.8	35.1	34.9

^a In parts per million relative to Me₄Si.

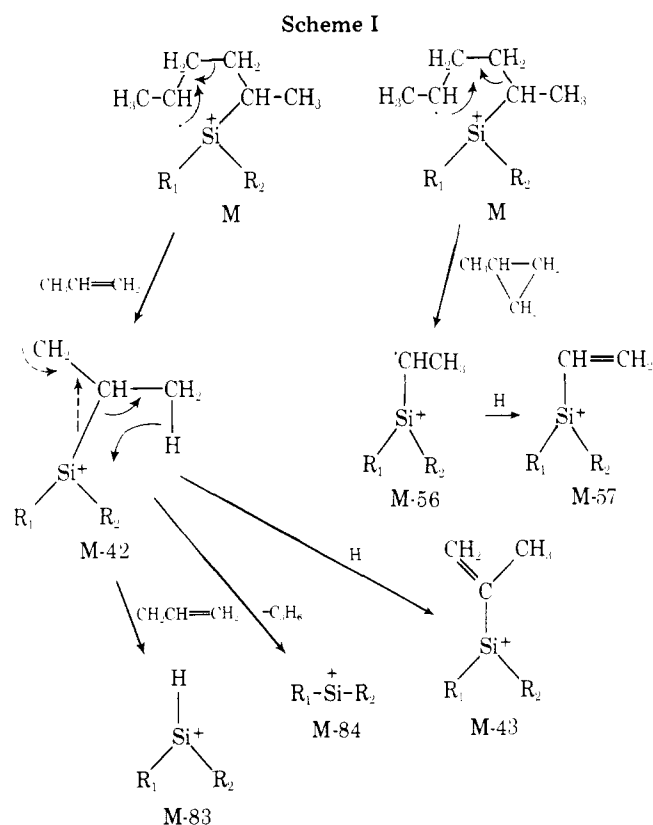
ical shifts of the phenyl derivatives of A, B, and C are given in Table IV.

To confirm the effect of the phenyl group on chemical shifts in the silacyclopentane ring system, the isomers of 2,5-dimethyl-1-phenyl-1-silacyclopentane were prepared from the di-Grignard compound of 2,5-dibromohexane and phenyldichlorosilane in ~24% yield. The isomers X, Y and Z (in the order of increasing boiling points and retention times on GC) were formed in relative amounts of 2:2:1. The deviation from the statistical distribution, as encountered in the case of 1,2,5-trimethyl-1-silacyclopentane, may be due to different formation rates, arising from differences in steric factors. Alternatively, the isomers formed in statistical distribution may have reacted preferentially with excess Grignard reagent. By careful spinning band column distillation it was only possible to obtain fractions enriched in one or two of the isomers. (Thus X/Y/Z = 75:25:0; 48:48:24; 25:50:25; or 8:35:54). Proton NMR spectral data are given in Table V and ^{13}C data in Table VI. Assignment of structures is again based on a comparison of shift values. (Unfortunately, the coupling constants of the silicon proton to the C₂ and C₅ protons, found to be *J* = 2.65 and 3.37 Hz for Y and Z respectively, are uninformative because of the flexibility of the silacyclopentane ring.) X is easily picked out as the *E,Z,Z,E* isomer because of the nonidentity of the carbon methyl groups and of the C₂/C₅ and C₃/C₄ pairs. Y, with the most shielded silicon-proton resonance, can be identified as the *E,E* isomer, Z, with the least shielded silicon-proton resonance, as the *Z,Z* isomer. The other proton and ^{13}C data are in conformity with these assignments. The shielding effect of the phenyl substituent on the two methyl groups is more pronounced than that of a methyl substituent. The difference in proton chemical shifts

of the carbon methyls between the *E,E* and *Z,Z* isomers is 0.20 ppm for 2,5-dimethyl-1-phenyl-1-silacyclopentane, while only 0.02 ppm for 1,2,5-trimethyl-1-silacyclopentane. The shielding/deshielding effect of the benzene ring depends on the different spacial positions of the protons with respect to the benzene ring. It can be seen that the *Z,Z* isomer (5) can assume a sterically favorable conformation in which the carbon methyl protons are held in positions close to the center of the aromatic ring. In this the induced ring currents cause shielding, while in the *E,E* isomer (4) the deshielding effect predominates.



Mass spectrometry does not differentiate between the stereoisomers of substituted 2,5-dimethyl-1-silacyclopentanes. Pure samples of the isomers from preparative GC all show identical mass spectra. A relatively small molecular ion peak (24%) is observed with fragments corresponding to *M* - 42 (100%), *M* - 43 (26%), *M* - 56 (26%), *M* - 57 (24%), and *M* - 83 (24%). Very similar fragmentation patterns are observed for 1,1,2,5-tetramethyl-1-silacyclopentane, 2,5-dimethyl-1-phenyl-1-silacyclopentane, and 1,2,5-trimethyl-1-phenyl-1-silacyclopentane. The results are best rationalized by assuming the formation of an open chain radical cation, with loss of stereochemistry, which may lose a molecule of propene (*M* - 42), then an allyl radical (*M* - 83) or another molecule of propene (*M* - 84), or alternatively one molecule of methyl cyclopropane (*M* - 56), as shown in Scheme I.



Experimental Section

All reactions were performed in flame-dried vessels flushed with dry nitrogen prior to the experiment. Ether was predried with calcium chloride, refluxed over sodium, and distilled. Reaction products were distilled using a short (~10 cm) fractionating column and their purity was checked by analytical GC (Varian Aerograph 200, SGE GSB/SE 30/S SCOT, glass capillary column with a minimum effective plate number of 20,000). The proton NMR spectra were determined on a Jeol JNM-MH-100 instrument (100 MHz) and at the National NMR center, Canberra (270 MHz). ¹³C spectra were obtained at 67.89 MHz at the National NMR center, Canberra. All spectra were run in CDCl₃ solutions with Me₄Si as internal standard, except where stated otherwise. Analyses were carried out by the Australian Microanalytical Service, Melbourne, and the Microanalytical Unit, University of Queensland. Mass spectra were recorded at 70 eV on an AEI. MS9 spectrometer.

1,2,5-Trimethyl-1-silacyclopentane (1, 2 and 3). Method a: from 2,5-Dibromohexane. The Grignard reagent prepared from dibromohexane¹⁰ (48 g; 0.2 mol) and magnesium (10.4 g, 0.44 mol) in dry ether (300 mL) was reacted with methylchlorosilane (23 g, 0.2 mol), and the mixture was refluxed for 2 h. Salts were removed by filtration and washed with hexane. The residue gave the title compound (6.8 g, 0.05 mol, 27%), bp 61–76 °C (100 mm). Alternatively, 2,5-dibromohexane and methylchlorosilane were added simultaneously to magnesium in dry ether (6.1 g, 24%).

Method b: from 5-Chloro-1-hexene. To excess lithium aluminum hydride (1.5 g) in dry ether (50 mL) was added 5-(chloromethylsilyl)-1-hexene, prepared as described¹¹ (5 g, 31 mM), and the mixture was refluxed overnight. Excess lithium aluminum hydride was destroyed by water. The organic phase yielded 5-(methylsilyl)-1-hexene (2.5 g, 20 mM, 63%): by 73 °C (100 mm); NMR (neat) δ 4.9–5.7 (m, 1 H, vinyl), 4.4–4.8 (m, 2 H, vinyl), 3.2–3.6 (m, 2 H, SiH), 4–2.0 (m, 8 H, aliphatic), –0.24 (t, 3 H, J = 4 Hz, SiCH₃).

Anal. Calcd for C₇H₁₆Si: C, 65.5; H, 12.6. Found: C, 65.7; H, 12.5.

Ring Closure. To 5-(methylsilyl)-1-hexene (2.5 g, 20 mM) in AR grade hexane (50 mL) was added catalytic amounts of hexachloroplatinic acid and the mixture was refluxed overnight. Distillation yielded 1,2,5-trimethyl-1-silacyclopentane (0.8 g, 6.5 mM, 33%), bp 65–76 °C (100 mm).

Anal. Calcd for C₇H₁₆Si: C, 65.5; H, 12.6. Found: C, 66.1; H, 13.2.

Separation into E,E, Z,Z and E,Z/Z,E Isomers (1, 2, 3). Method a. The mixture was separated by preparative GC using a 20 ft × 0.5 in. SE-30 column at 70 °C with a flow rate of 15 mL/min He. E,E (A), E,Z/Z,E (B), and Z,Z (C) had retention times of 4.3, 4.8, and 6.0 min, respectively.

Method b. A Nester/Faust Auto annular Teflon spinning band distillation column was used with a theoretical plate efficiency of >150 at atmospheric pressure. The reflux ratio was adjusted to 100:1.

The mass spectra of the three isomers were identical: *m/e* (rel intensity) 128 (24), 113 (3), 99 (7), 86 (100), 85 (26), 72 (26), 71 (24), 59 (33), 58 (83), 45 (24), 43 (24); exact mass of molecular ion peak; 128.1016. Calcd for C₇H₁₆Si: 128.1021.

1,2,5-Trimethyl-1-phenyl-1-silacyclopentane (1a, 2a, and 3a). Method a. To a solution of excess phenyllithium, prepared by bromobenzene (2 g) and lithium (0.2 g) in dry ether (50 mL), was added 1,2,5-trimethyl-1-silacyclopentane (E,E:E,Z/Z,E:Z,Z = 14:79:7) (0.56 g, 4.4 mM) and the mixture was refluxed overnight. Excess phenyllithium was hydrolyzed with ice. The organic phase yielded, by distillation, the title compound [0.6 g, 2.9 mM, 67%, bp 62 °C (1 mm)]. Similarly were prepared the phenyl derivatives of the E,E (1) and Z,Z (2) isomers. The isomeric composition of the phenyl derivatives was the same as that of the silicon hydride, within experimental error. Thus were obtained Z,Z:E,Z/Z,E:E,E = 14:79:7; 50:49:1; 9:23:70, respectively.

Method b: via 5-(Methylphenylsilyl)-1-hexene. To a Grignard solution prepared from bromobenzene (1.94 g, 12.4 mM) and magnesium (0.33 g, 13.8 mM) in dry ether (50 mL) 5-(chloromethylsilyl)-1-hexene (see above) (2.0 g, 12.4 mM) was added and the mixture was refluxed overnight. Salts were hydrolyzed and the organic phase gave, by distillation, 5-(methylphenylsilyl)-1-hexene (1.5 g, 7.4 mM, 60%):

bp 78–80 °C (10 mm); NMR (neat) δ 6.6–7.4 (m, 5 H, aromatic), 5.0–5.8 (m, 1 H, vinyl), 4.3–3.9 (m, 2 H, vinyl), 4.03 (q, 1 H, J = 4 Hz, SiH), 3–2.2 (m, 8 H, aliphatic), –0.02 (3 H, J = 4 Hz, SiCH₃).

Anal. Calcd for C₁₃H₂₀Si: C, 76.4; H, 9.9. Found: C, 76.9; H, 10.2.

Ring Closure. 5-(Methylphenylsilyl)-1-hexene (1.5 g, 7.4 mM) was refluxed in AR grade hexane in the presence of catalytic amounts of hexachloroplatinic acid for 4 h. Distillation gave an isomeric mixture of 1,2,5-trimethyl-1-phenyl-1-silacyclopentanes (~1:2:1) (1.0 g, 4.9 mM, 67%). The components of this mixture were shown to be identical with the products obtained by method a by a comparison of their proton NMR properties as well as retention times on GC: mass spectrum *m/e* (rel intensity) 204 (79), 189 (18), 162 (74), 161 (26), 148 (42), 147 (50), 121 (100), 120 (58), 105 (42), 69 (29), 55 (34); exact mass of molecular ion peak, 204.1330; calcd for C₁₃H₂₀Si, 204.1334.

Anal. Calcd for C₁₃H₂₀Si: C, 76.4; H, 9.9. Found: C, 76.5; H, 10.0.

1,1,2,5-Tetramethyl-1-silacyclopentane. The compound prepared by the action of methylolithium (0.05 M) on 1,2,5-trimethyl-1-silacyclopentane (2.5 g, 0.02 M) in dry ether in 22% yield (bp 61–66 °C (60 mm) [lit.¹⁰ 145–150 °C (760 mm)]) was shown to be 1,1,2,5-tetramethyl-1-silacyclopentane by a comparison of NMR and MS data with the data quoted.¹⁰

2,5-Dimethyl-1-phenyl-1-silacyclopentane. To the di-Grignard reagent prepared from 2,5-dibromohexane (52 g; 0.21 mol) and magnesium (11.6 g, 0.48 mol) in dry ether (300 mL) was added phenyldichlorosilane (36 g; 0.20 mol) in dry ether (100 mL) and the mixture was refluxed overnight. Salts were removed by filtration and washed with hexane. The residue was distilled to give the title compound (9.8 g, 0.05 mol, 24%), bp 68–78 °C (1 mm). The mass spectrum of the isomeric mixture showed peaks at *m/e* (rel intensity) 190 (100), 148 (69), 147 (24), 134 (35), 133 (36), 121 (29), 120 (47), 112 (45), 107 (53), 106 (42), 105 (53), 97 (24), 84 (33).

Anal. Calcd for C₁₂H₁₈Si: C, 75.7; H, 9.5. Found: C, 75.6; H, 9.6.

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Registry No.—2,5-Dibromohexane, 24774-58-1; methylchlorosilane, 75-54-7; 5-chloro-1-hexene, 927-54-8; 5-(chloromethylsilyl)-1-hexene, 68212-46-4; 5-(methylphenylsilyl)-1-hexene, 68238-00-6; 1,1,2,5-tetramethyl-1-silacyclopentane, 55956-01-9; phenyldichlorosilane, 1631-84-1; 2,5-bis(bromomagnesium)hexane, 68212-47-5; bromophenylmagnesium, 100-58-3; 1,2,5-trimethyl-1-silacyclopentane, 68295-67-0.

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