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Synthesis and Reduction of Octasilyl[4]radialene. Structure and Dynamic Study of [4]Radialene Dianion with Eight-Center Ten-Electron π-System

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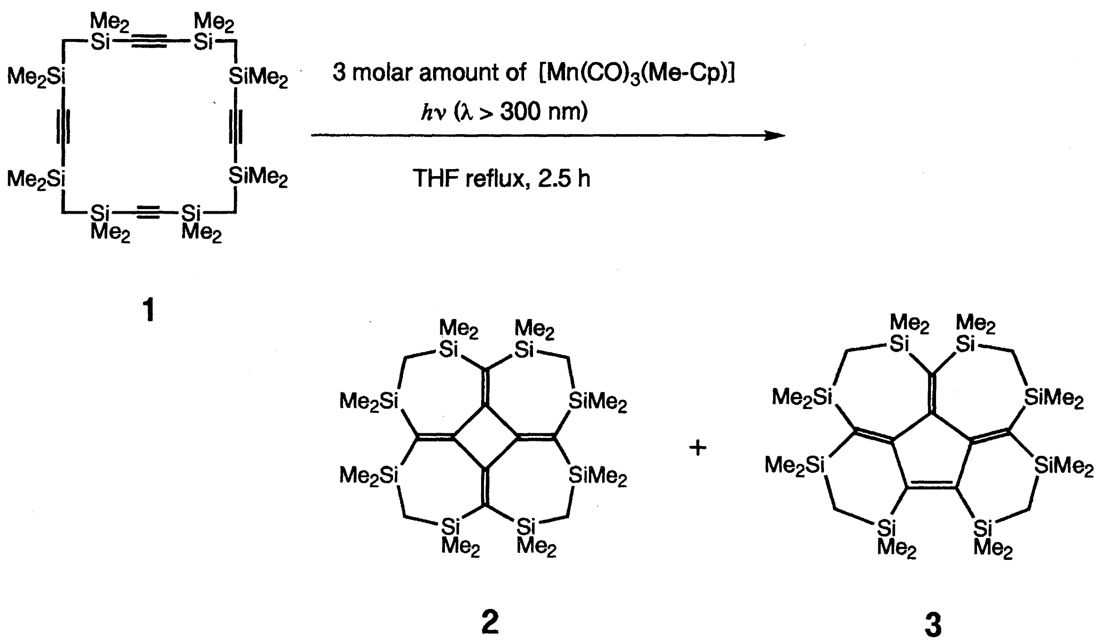
The intramolecular reaction of 3,3,5,5,8,8,10,10,13,13,15,15,18,18,20,20-hexadecamethyl-3,5,8,10,13,15,18,20-octasilacycloicosa-1,6,11,16-tetrayne (**1**) with three molar amounts of [Mn(CO)$_3$(Me-Cp)] in THF under photochemical and refluxing conditions produced octasilyl[4]radialene derivative (**2**). The reaction of **2** with alkali metals (Li, Na, and K) in THF gave dialkali metal salts of the corresponding dianion (**4**) with an eight-center ten-electron π-system. The molecular structure of dilithium salt of octasilyl[4]radialene dianion (**4a**) has been established by X-ray crystallography. The two lithium atoms are located above and below and are bonded to the atoms of the radialene framework to give a bis-CIP structure. The structural parameters of **4a** are discussed in comparison to those of **2**. The structure of **4a** in solution has also been discussed on the basis of NMR spectroscopic data. The two Li$^+$ ions of **4a** are not fixed to the π-skeleton in toluene-d_8, but are fluxional, giving a symmetric structure (bis-CIP) on the NMR time scale. In a solvating medium such as THF-d_8, one of the Li$^+$ ions dissociates to yield an ion pair (CIP and SSIP). Some evidence for the Li$^+$ ion walk on the π-skeleton is demonstrated.

Over the last few years, structural studies of various anions, which can be either a solvent-separated ion pair (SSIP) or solvent-shared contact ion pair (CIP), have been reported in both crystal and solution states.1) The polarity of the solvent has been recognized as important factor for the SSIP and CIP formations. However, in most of these systems, the dynamic behavior of the counter cation in solution has not been made clear.2) We have shown that the introduction of a silyl group to the π-electron system causes remarkable steric and electronic perturbations.3) One of the most interesting features is the ability to form a dianion by reduction with alkali metals. We previously reported successful syntheses of the dilithium salts of various silyl-substituted ethylenes,4) silyl-substituted styrenes,5) silyl-substituted benzenes,6) silyl-substituted dimethylenecyclobutene dianions,7) and the lithium salt of persilyl-substituted cyclopentadienide.8) Most recently, we have reported the tetralithium salt of a trimethylenecyclopentene tetraanion derivative as a silyl-substituted eight-center twelve-electron π-system.9) Herein, we wish to report a detailed study of dialkali metals of [4]radialene dianion (**4**) as a new silyl-substituted eight-center ten-electron π-system, together with the crystal structures of dilithium salt of octasilyl[4]radialene dianion (**4a**) and a precursor of the [4]radialene derivative (**2**). We also report the unique dynamic behavior of a novel Li$^+$ ion walk on the skeleton of **4a** in solution.10)

Results and Discussion

Synthesis of 2. Radialene has received considerable attention due to its special arrangement of π-electrons. Various radialene derivatives have been reported, but the synthetic methods are still very limited.11) The silyl-substituted radialene derivative is hitherto unknown. Previously, we reported the intramolecular cyclotrimerization of the silacyclotriynes with transition metal complexes to give various persilylated π-electron systems, such as a dimethylenecyclobutene derivative.12) The persilylated [4]radialene derivative (**2**) has been synthesized in a similar manner via the intramolecular reaction of 3,3,5,5,8,8,10,10,13,13,15,15,18,18,20,20-hexadecamethyl-3,5,8,10,13,15,18,20-octasilacycloicosa-1,6,11,16-tetrayne (**1**) linked by silmethylene chains with [Mn(CO)$_3$(Me-Cp)].

A mixture of the macrocyclic tetrayne (**1**) and three molar amounts of [Mn(CO)$_3$(Me-Cp)] in THF was irradiated with a 500 W high-pressure mercury lamp under the refluxing temperature of THF to produce yellow crystals of the persilylated [4]radialene derivative (**2**) in 11% yield (Scheme 1). The reaction may involve disilylvilnylidene complex as a reactive intermediate.13) The reaction mechanism to produce **2** remains unclear; nevertheless, a quadruple 1,2-silyl shift must be involved for the formation of **2**. Compound **2** is a first example of the formation of [4]radialene via the tetramerization of acetylene units. However, the use of one molar amount of



Scheme 1.

the manganase complex resulted in the formation of trimethylenecyclopentene derivative (**3**)9) (17% yield), arising from a triple 1,2-silyl shift, together with a small amount of **2**.

Structure of 2. [4]Radialene has a particular arrangement of 8π-electrons with a symmetric framework. We have performed the X-ray diffraction of **2**. Figure 1 shows the molecular structure of **2** determined by the X-ray diffraction method. Selected bond lengths, bond angles, and dihedral angle are listed in Table 1. The central four-membered ring of **2** is not planar, but has a considerably distorted conformation with a puckered angle of 32.5°. The puckered angle of **2** is quite similar to those of reported: 26.5° for perchloro- and 34.7° for perphenyl-substituted [4]radialenes.11f,14) The internal bond angles of the four-membered ring of **2** range from 87.6(4) to 87.9(4)° (av 87.7°), and the sum of the bond angles is 357.8° (357.7 to 358.0° for C1, C2, C3, and C4). The appreciable bond alternation between the single and double bonds of the π-skeleton of **2** shows the structural feature as a cross conjugated diene. The average lengths of C–C single and double bonds are 1.520(9) and 1.350(9) Å, respectively. The four seven-membered rings containing the $Me_2SiCH_2SiMe_2$ fragments have twist and boat conformations.

NMR data of **2** show a symmetric structure in solution on the NMR time scale. Thus, in the ^{1}H NMR spectrum of **2** at room temperature, only one signal is found at 0.14 and 0.05 ppm for the methyl and methylene protons, respectively. In the ^{13}C NMR spectrum, the signal is observed at 2.1 and 10.8 ppm for the methyl and methylene carbon atoms, respectively. The endocyclic and exocyclic quaternary ^{13}C NMR signals appear at 134.7 and 167.5 ppm, respectively. Only one ^{29}Si signal is observed at −7.8 ppm.

Two Electron Reduction of 2 with Alkali Metals. [4]-Radialene dianion by metalating 1,2-dimethyl-3,4-bismethylenecyclobutene with *n*-BuLi/KO^{t}Bu or *n*-BuLi/TMEDA

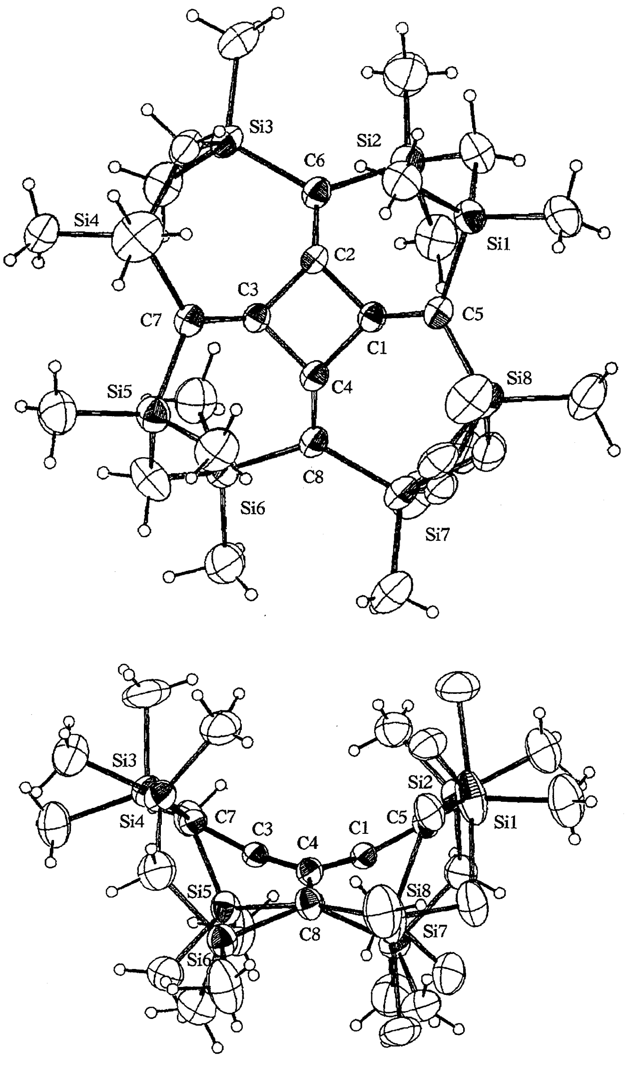


Fig. 1. ORTEP drawing of **2**: upper, top view; below, side view.

Table 1. Selected Bond Distances (Å), Angles (deg), and Dihedral Angle (deg) of **2**[a)]

Bond distances			
C1–C2	1.519(9)	C1–C4	1.520(9)
C1–C5	1.361(9)	C2–C3	1.517(9)
C2–C6	1.350(9)	C3–C4	1.523(9)
C3–C7	1.354(9)	C4–C8	1.333(9)
C5–Si1	1.884(7)	C5–Si8	1.868(7)
C6–Si2	1.882(7)	C6–Si3	1.887(7)
C7–Si4	1.878(7)	C7–Si5	1.882(7)
C8–Si6	1.892(7)	C8–Si7	1.869(9)
Bond angles			
C2–C1–C4	87.6(4)	C2–C1–C5	132.3(6)
C4–C1–C5	137.8(6)	C1–C2–C3	87.9(4)
C1–C2–C6	133.6(6)	C3–C2–C6	136.3(6)
C2–C3–C4	87.6(4)	C2–C3–C7	137.4(5)
C4–C3–C7	133.0(5)	C1–C4–C3	87.6(4)
C1–C4–C8	135.4(6)	C3–C4–C8	134.7(6)
Dihedral angle			
C2–C1–C4/C2–C3–C4	32.5		

a) Atomic numbers are given in Fig. 1. Standard deviations are in parentheses.

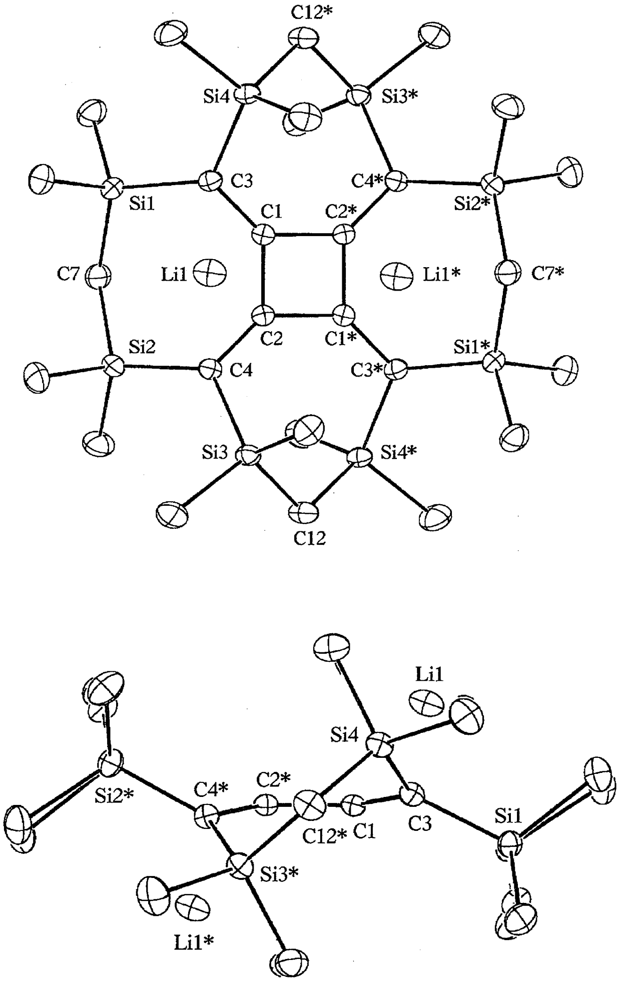
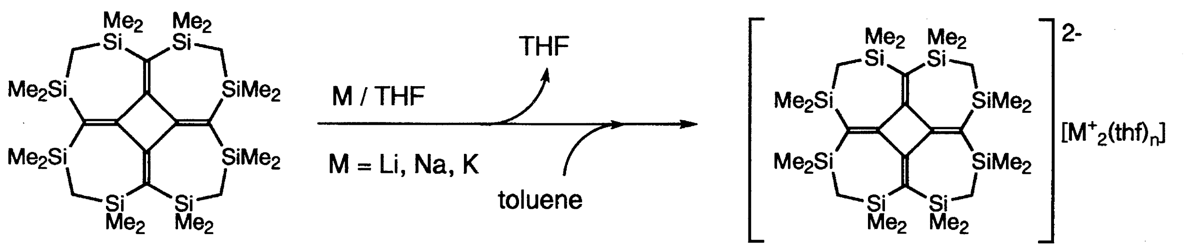


Fig. 2. ORTEP drawing of **4a** (THF molecules are omitted for the clarity): upper, top view; below, side view.

has been reported,[15)] but its characterization is only by the quenching experiments. The reaction of **2** with excess lithium metal in dry oxygen-free THF at room temperature led to the formation of a red solution of the dianion of **2**. The two electron reduction occurred quantitatively and completed within 1 h. After the solvent was removed in vacuo, dry degassed toluene was introduced by vacuum transfer. Crystallization from a toluene solution afforded air- and moisture-sensitive dark red crystals of dilithium salt of the dianion (**4a**) containing two molecules of THF (Scheme 2).

The [4]radialene derivative (**2**) also underwent a two electron reduction, not only by lithium, but also by sodium and potassium in THF to give red solutions of the dianions of **4b** (Na) and **4c** (K) within 1 h. The structures of **4a** (Li), **4b** (Na), and **4c** (K) were deduced by NMR spectroscopy as well as by X-ray diffraction for **4a** (Li).

Crystal Structure of 4a. Figure 2 shows the molecular structures of **4a**, which has been confirmed by X-ray crystallography. The structure shows that the molecule has an inversion center at the center of the four-membered ring of C1–C2–C1*–C2*. The dilithium **4a** has a monomeric structure and forms contact ion pairs (bis-CIP) in the crystals. One THF molecule is coordinated to each lithium atom. Li1 and Li1* are located above and below the ring, and are bonded to the four carbon atoms of the radialene skeleton as well as to the oxygen atoms of THF. The distance between Li1 and the four carbon atoms (C1, C2, C3, and C4) ranges from 2.245(2) to 2.315(2) Å (av 2.279 Å). The two



2

4a: M = Li
4b: M = Na
4c: M = K

Scheme 2.

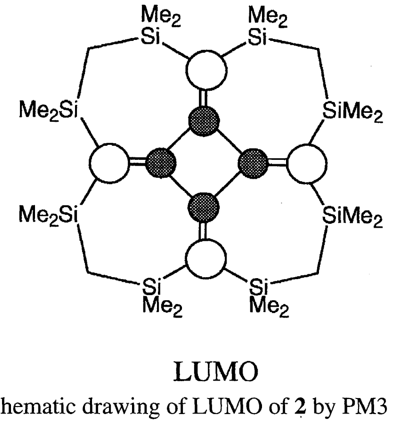
7-membered rings that are "para" to each other and contain the $Me_2SiCH_2SiMe_2$ fragments have a twist conformation, and the other two have a boat conformation. Selected bond lengths, bond angles, and dihedral angle are listed in Table 2.

Comparison of the structural features of **2** and **4a** is quite interesting. Eight carbon atoms of the π-skeleton of **4a** are almost coplanar, following a two-electron reduction due to the delocalization of the negative charge. The four-membered ring is planar and almost square, as determined by the internal bond angles of 89.8(1) to 90.2(1)° (av 90.0°) and the sum of the bond angles of 358.9° (358.8 to 358.9° for C1, C2, C1*, and C2*). The average length of the exocyclic bonds (C1–C3 and C2–C4) is elongated by about 0.06 Å relative to those of **2** (1.350(9) Å (av) for **2** and 1.409(1) Å (av) for **4a**). In contrast, the average length of endocyclic bonds (C1–C2 and C1–C2*) is shortened by about 0.04 Å (1.520(9) Å (av) for **2** and 1.479(1) Å (av) for **4a**). Thus, the endocyclic bonds in **4a** are bonding, whereas the exocyclic bonds are antibonding. Therefore, the geometry of **4a** reflects the nature of the LUMO of **2**, as shown in Fig. 3.[16] The bond lengths of the Si–C bonds (Si1–C3, Si2–C4, Si3–C4, and Si4–C3) for **4a** are slightly shortened compared to those of **2** due to the delocalization of the negative charge onto the silicon centers by p π–σ* conjugation (1.880(7) Å (av) for **2** and 1.860(1) Å (av) for **4a**).

Table 2. Selected Bond Distances (Å), Angles (deg), and Dihedral Angle (deg) of **4a**[a)]

	Bond distances		
C1–C2	1.473(1)	C1–C2*	1.485(1)
C1–C3	1.406(1)	C2–C4	1.412(1)
C3–Si1	1.854(1)	C4–Si2	1.851(1)
C4–Si3	1.867(1)	C3–Si4	1.868(1)
Li1–C1	2.246(2)	Li1–C2	2.245(2)
Li1–C3	2.315(2)	Li1–C4	2.309(2)
	Bond angles		
C1–C2–C1*	90.2(1)	C2–C1–C2*	89.8(1)
C2–C1–C3	134.1(1)	C1–C2–C4	133.9(1)
C3–C1–C2*	135.0(1)	C4–C2–C1*	134.7(1)
	Dihedral angle		
	C1–C2–C1*/C1–C2*–C1*	0.0	

a) Atomic numbers are given in Fig. 2. Standard deviations are in parentheses.



LUMO

Fig. 3. Schematic drawing of LUMO of **2** by PM3 calculation.

Structure of 4 in Toluene-d_8. The structure of **4a** in toluene-d_8 has been deduced on the basis of ^1H, ^{13}C, ^{29}Si, and ^6Li NMR spectra. These NMR data on the structure of **4a** indicate a symmetric bis-CIP formation in solution. In the ^1H NMR spectrum of **4a**, only one signal is observed at 0.32 and 0.13 ppm for the methyl and methylene protons, respectively, together with the signals due to THF. As well, only one ^{13}C NMR signal is observed at 6.0 and 9.5 ppm for the methyl and methylene carbon atoms, respectively. Of particular interest are the chemical shifts of the ^{13}C NMR signals of the exocyclic (C3, C4, C3*, and C4*) and endocyclic (C1, C2, C1*, and C2*) carbon atoms. The former ^{13}C NMR signal is observed at 73.2 ppm. This has shifted significantly upfield relative to that of **2** ($\Delta\delta = 94.3$). On the other hand, the latter has shifted downfield and appears at 182.8 ppm ($\Delta\delta = 48.1$). The ^{13}C NMR spectral data suggest that the negative charge is largely delocalized on the four exocyclic carbon atoms in the π-skeleton of **4a**. The ^{29}Si signal is observed at −11.8 ppm, which has shifted upfield relative to that of **2** ($\Delta\delta = 4.0$). This suggests that the negative charge is stabilized by the eight silyl groups in **4a**. The ^6Li NMR spectrum of **4a** yields only one signal at −0.66 ppm. On the basis of these spectral data, it is quite reasonable to assume that the two Li^+ ions of **4a** are not fixed to the π-skeleton, but are fluxional; the two Li^+ ions are walking on the π-skeleton of the radialene on the NMR time scale (Fig. 4). However, the variable-temperature NMR experiment can not be conducted in toluene-d_8. Due to lowering the temperature, the crystals of bis-CIP have precipitated to hamper the dynamic NMR study. The symmetric structure in toluene-d_8 was also deduced on the bases of the ^1H, ^{13}C, and ^{29}Si NMR spectra of the disodium **4b** and the dipotassium **4c**.

Structure of 4 in THF-d_8. The interesting Li^+ walk on the π-skeleton is evidenced in THF-d_8. In a solvating medium such as THF-d_8, one of the Li^+ ions (either Li1 or Li1*) above the [4]radialene ring dissociates to yield an ion pair (CIP and SSIP) (Scheme 3). The ^6Li NMR spectrum of **4a** shows two signals with the same intensity at −0.38

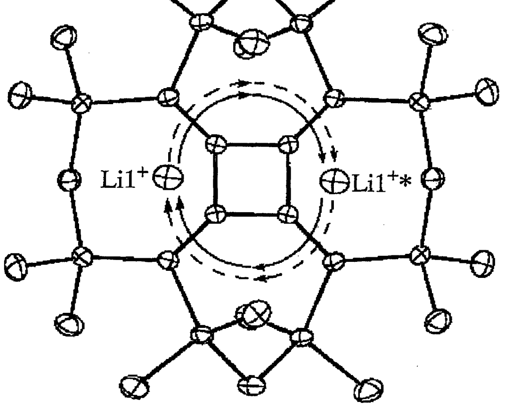
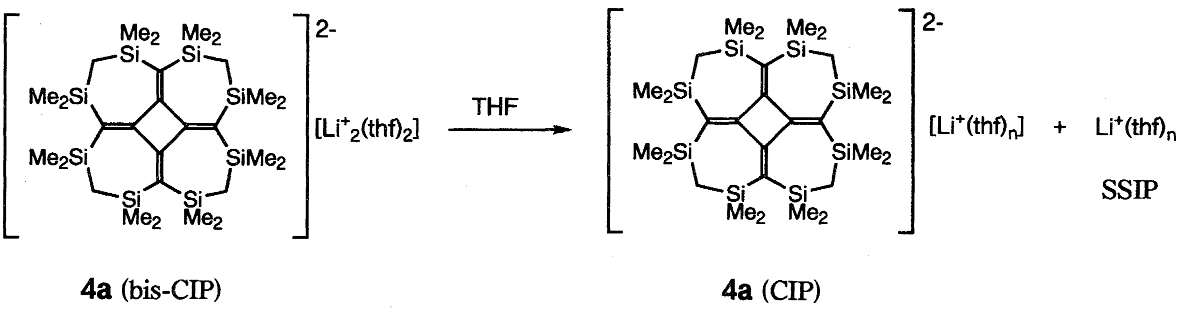


Fig. 4. Schematic representation of the lithium walk on the π-skeleton of **4a** (bis-CIP) in toluene-d_8 (298 K).



Scheme 3.

and −0.66 ppm; the ratio of the peaks is independent of the concentration (0.03 to 0.13 M, 1 M = 1 mol dm^{-3}). The former signal is assigned to the THF solvated species, Li$^+$(thf)$_n$, whereas the latter is assigned to the Li$^+$ ion that is bonded to the π-skeleton.[4b)] The signal at −0.38 ppm assigned to the solvated Li$^+$ ion grows thanks to the ^{6}LiBr added due to the rapid exchange. However, the intensity of the signal at −0.66 ppm (CIP) bound to the π-framework remains unchanged. No exchange of the two Li$^+$ ions was observed in the temperature range of 173 to 298 K. Thus, one Li$^+$ ion of **4a** forms a SSIP and the other one forms a CIP in THF-d_8. Consequently, the π-skeleton of **4a** has a different environment above and below the plane; methylene group ^{1}H doublet signals, with the geminal coupling of 12.6 Hz, and two ^{13}C signals of SiMe$_2$ can be seen at room temperature. The ^{13}C signal of methylene carbon atoms is found at 11.4 ppm. The endocyclic and exocyclic quaternary carbon atoms are found at 180.7 and 66.8 ppm, respectively. Only one ^{29}Si signal is observed at −13.5 ppm. These spectroscopic data clearly reveal that the Li$^+$ ion of CIP coordinates with one side of the radialene framework and that it can walk freely on the π-skeleton (Fig. 5).

Upon lowering the temperature, the ^{13}C signals of the endocyclic and exocyclic carbon atoms broadened and split into two singlets (185.9, 177.8 ppm and 83.5, 50.2 ppm, respectively). The temperature-dependent change of the ^{13}C endocyclic carbons in THF-d_8 must have resulted from the dynamics of Li$^+$ walk on the [4]radialene framework. Experimental and simulated spectra for the ^{13}C NMR signals of the endocyclic carbon atoms are shown in Fig. 6. From the Arrhenius and Eyring plots, the values of $E_a = 54.9$ kJ mol^{-1}, $\Delta H^{\ddagger} = 52.7$ kJ mol^{-1}, and $\Delta S^{\ddagger} = 19.0$ J mol^{-1} K^{-1} for the Li$^+$ walk can be estimated. The present lithium walk is accompanied by the twist-boat interconversion of the 7-membered rings that contain the Me$_2$SiCH$_2$SiMe$_2$ fragments, which leads to a high barrier to internal migrations of the Li$^+$ ion. At 173 K, the Li$^+$ ion walk of the CIP is suppressed so that Li$^+$ is fixed at one site of the framework (Fig. 7), giving eight carbon signals of SiMe$_2$ and three methylene carbons (9.7, 10.4, and 11.4 ppm), two endocyclic carbons (185.9 and 177.8 ppm), and two exocyclic carbons (83.5 and 50.2 ppm). In the ^{29}Si NMR spectrum, four signals were observed at −15.7, −14.3, −12.6, and −11.6 ppm. The Li$^+$ walk again starts on raising the temperature.

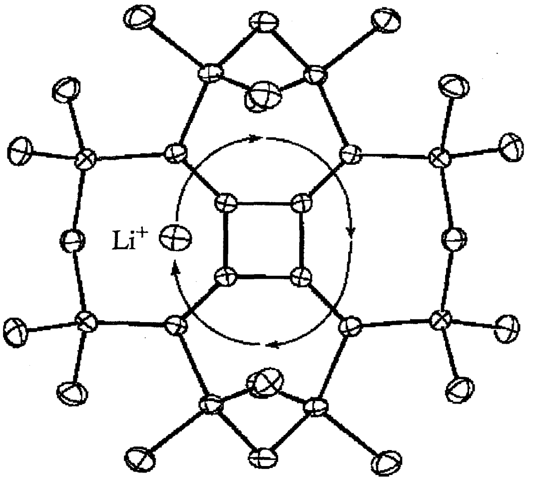


Fig. 5. Schematic representation of the lithium walk on the π-skeleton of **4a** (CIP) in THF-d_8 (298 K).

Experimental

General Procedure. ^{1}H NMR spectra were recorded on a Bruker AC-300 FT NMR spectrometer. ^{13}C, ^{29}Si, and ^{6}Li NMR spectra were collected on a Bruker AC-300 at 75.5, 59.6, and 44.2 MHz, respectively. ^{6}Li NMR spectra are referenced to 1 M LiCl in methanol/toluene-d_8 or 1 M LiCl in THF-d_8. Mass spectra were obtained on a Shimadzu QP-1000. Electronic spectra were recorded on a Simadzu UV-2100 spectrometer. The sampling of **4a** for X-ray crystallography was carried out by using a Giken Engineering Service GBX-1200 gas-replacement type glove box. Tetrahydrofuran and hexane were dried and distilled from sodium benzophenone ketyl. These solvents were further dried and degassed over a potassium mirror in vacuo prior to use. Toluene-d_8 and THF-d_8 were dried over molecular sieves, and then transferred into a tube covered with potassium mirror prior to use. Lithium-6 (95 atom%) metal was commercially available (Aldrich Chemical Company). 3,3,5,5,8,8,10,10,13,13,15,15,18,18,20,20-Hexadecamethyl-3,5,8,10,13,15,18,20-octasilacycloicosa-1,6,11,16-tetrayne (**1**) was prepared by the reported method.[17)]

Octasilyl[4]radialene (2). A mixture of **1** (301 mg, 0.49 mmol) and [Mn(CO)$_3$(Me-Cp)] (320 mg, 1.47 mmol) in THF (30 ml) was irradiated with a 500 W high-pressure mercury lamp for 2.5 h through the cut off filter ($\lambda > 300$ nm) under the refluxing temperature of THF. After removal of the manganese complex, the reaction mixture was chromatographed on silica gel to produce yellow crystals of **2** in 11% yield, along with a small amount of **3**. Mp 219—220 °C; ^{1}H NMR (CDCl$_3$) $\delta = 0.05$ (s, 8 H, CH$_2$), 0.14 (s, 48 H, CH$_3$); ^{13}C NMR (CDCl$_3$) $\delta = 2.1$ (CH$_3$), 10.8 (CH$_2$), 134.7 (C), 167.5 (C); ^{29}Si NMR (CDCl$_3$) $\delta = -7.8$; UV (hexane) λ_{max}/nm (ε) 229 (19500), 338 (14100), 444 (600). Found: C, 54.35;

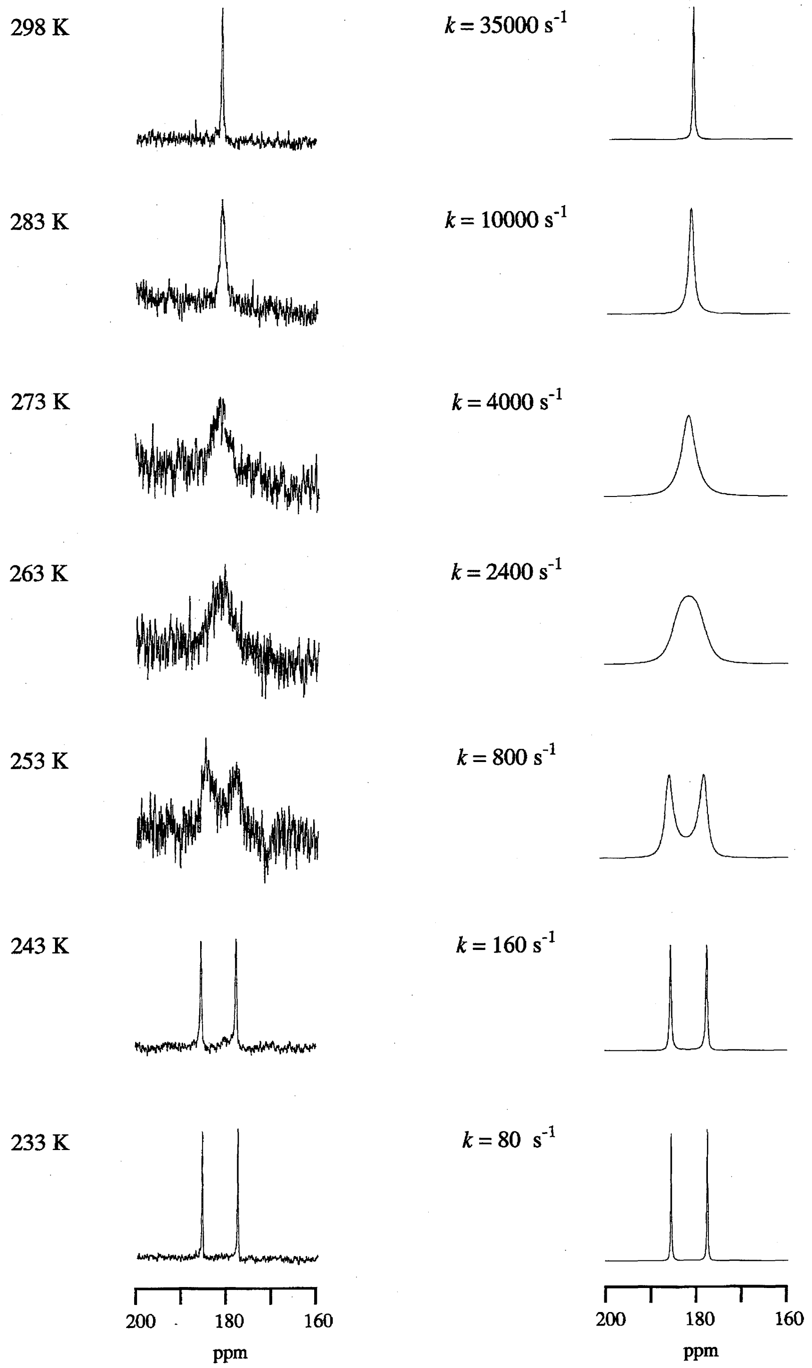


Fig. 6. Observed (left) and simulated (right) ^{13}C (125 MHz) line shapes of the endocyclic carbons of **4a** in THF-d_8.

H 8.99%. Calcd for $C_{28}H_{56}Si_8$: C, 54.47; H 9.14%.

Dilithium Salt of Octasilyl[4]radialene Dianion (4a). The crystals of **2** (20 mg, 0.03 mmol) and lithium metal (30 mg, 4.3 mmol) were put in a sealed tube with a magnetic stirrer. After degassing, dry oxygen-free THF (1 ml) was introduced by vacuum transfer and stirred at room temperature to give a red solution of the dianion of **2** within 1 h. After the solvent was removed in vacuo, degassed toluene was introduced by vacuum transfer. Then after removing the lithium metal, the solution was cooled to afford dark red crystals of **4a** quantitatively. ^{1}H NMR (C_7D_8) $\delta = 0.13$ (s, 8 H, CH_2), 0.32 (s, 48 H, CH_3), 1.26 (br. s, 8 H, THF), 3.46 (br. s, 8 H, THF); ^{13}C NMR (C_7D_8) $\delta = 6.0$ (CH_3), 9.5 (CH_2), 25.3 (THF), 69.7 (THF), 73.2 (C), 182.8 (C); ^{29}Si NMR (C_7D_8) $\delta = -11.8$; ^{6}Li NMR (C_7D_8) $\delta = -0.66$.

NMR Spectral Data of 4a in THF-d_8 at 298 K. ^{1}H NMR (THF-d_8) $\delta = -0.23$ (d, $J = 12.6$ Hz, 4 H, CH_2), -0.17 (d, $J = 12.6$ Hz, 4 H, CH_2), 0.04 (s, 48 H, CH_3); ^{13}C NMR (THF-d_8) $\delta = 6.4$ (CH_3), 6.9 (CH_3), 11.4 (CH_2), 66.8 (C), 180.7 (C); ^{29}Si NMR (THF-d_8) $\delta = -13.5$; ^{6}Li NMR (THF-d_8) $\delta = -0.66$, -0.38.

Disodium Salt of Octasilyl[4]radialene Dianion (4b). This was obtained by a procedure similar to the synthesis of **4a** as dark red crystals. ^{1}H NMR (THF-d_8) $\delta = -0.22$ (s, 8 H, CH_2), 0.04 (s, 48 H, CH_3); ^{13}C NMR (THF-d_8) $\delta = 7.2$ (CH_3), 11.6 (CH_2), 66.5 (C), 182.0 (C); ^{29}Si NMR (THF-d_8) $\delta = -14.0$.

Dipotassium Salt of Octasilyl[4]radialene Dianion (4c). This was obtained by a procedure similar to the synthesis of **4a** as dark

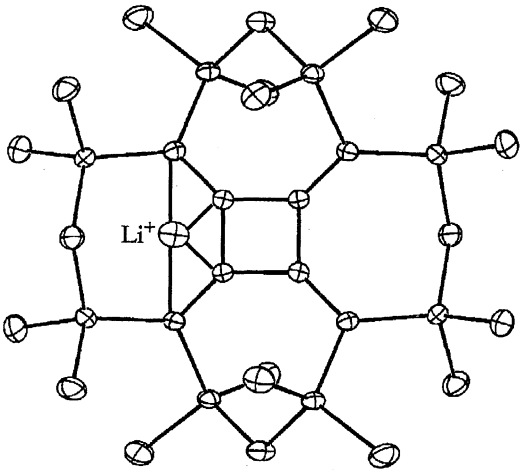


Fig. 7. Structure of **4a** in THF-d_8 at 173 K.

red crystals. ^{1}H NMR (THF-d_8) $\delta = -0.17$ (s, 8 H, CH_2), 0.07 (s, 48 H, CH_3); ^{13}C NMR (THF-d_8) $\delta = 6.9$ (CH_3), 11.1 (CH_2), 70.6 (C), 182.8 (C); ^{29}Si NMR (THF-d_8) $\delta = -14.0$.

X-Ray Crystallography. Single crystals of **2** and **4a** for X-ray diffractions were grown from an ethanol solution and a toluene solution, respectively. The X-ray crystallographic experiments of **2** were performed on a Rigaku-Denki AFC 5R diffractometer equipped with graphite-monochromatized Mo$K\alpha$ radiation ($\lambda = 0.71069$ Å). Crystal data for **2**: MF = $C_{28}H_{56}Si_8$, MW = 617.43, monoclinic, $a = 18.935(7)$, $b = 9.106(2)$, $c = 22.829(2)$ Å, $\beta = 91.53(2)°$, $V = 3935(2)$ Å^3, space group = $P2_1/c$, $Z = 4$, $D_{\text{calcd}} = 1.042$ g cm^{-3}. The final R factor was 0.0787 ($R_w = 0.0767$) for 4070 reflections with $F_o > 3\sigma$ (F_o). The X-ray crystallographic experiments of **4a** were performed on a DIP2020 image plate diffractometer equipped with graphite-monochromatized Mo$K\alpha$ radiation ($\lambda = 0.71073$ Å). Crystal data for **4a**: MF = $C_{36}H_{72}Li_2O_2Si_8$, MW = 767.47, monoclinic, $a = 9.506(1)$, $b = 22.105(1)$, $c = 11.869(1)$ Å, $\beta = 112.197(1)°$, $V = 2309.20(1)$ Å^3, space group = $P2_1/c$, $Z = 2$, $D_{\text{calcd}} = 1.164$ g cm^{-3}. The final R factor was 0.031 ($R_w = 0.038$) for 4208 reflections with $I_o > 2\sigma(I_o)$. Tables of positional and thermal parameters and complete interatomic distances and angles have been deposited as Document No. 71036 at the Office of the Editor of Bull. Chem. Soc. Jpn.

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