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CHEMISTRY OF SILOLES. THE REACTIONS OF SILOLES WITH ORGANOLITHIUM REAGENTS

MITSUO ISHIKAWA, TATSURU TABOHASHI, HIROSHI SUGISAWA, KUNIO NISHIMURA and MAKOTO KUMADA

Department of Synthetic Chemistry, Faculty of Engineering, Kyoto University, Kyoto 606 (Japan)

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Summary

The chemical behaviour of siloles toward various organolithium reagents in THF has been investigated. The reaction of 1-methyl-1-(trimethylsilyl)-, 1-phenyl-1-(trimethylsilyl)- and 1,1-bis(trimethylsilyl)dibenzosilole (I, II and III) with a large excess of an alkyllithium such as methyllithium or butyllithium afforded 1,1-dialkyldibenzosiloles in quantitative yields. Treatment of I with an excess of phenyllithium gave a mixture of 1-methyl-1-phenyl- and 1,1-diphenyldibenzosilole quantitatively, while with an excess of tert-butyllithium, I afforded 1,1-dimethyl- and 1-tert-butyl-1-methyldibenzosilole in low yield. Similar treatment of I and II with 1 equiv. of methyl- or butyl-lithium yielded a mixture of the corresponding mono- and dialkyl-substituted dibenzosiloles. 1-Methyl-3,4-diphenyl-1,2,5-tris(trimethylsilyl)silole reacted with methyllithium in THF to give 1,1-dimethyl-3,4-diphenyl-2,2,5-tris(trimethylsilyl) silole. Similarly, both 2,4-diphenyl-1,1,3,5-tetrakis(trimethylsilyl)silole and 4,5-diphenyl-1,1,2,3-tetrakis(trimethylsilyl)silole with methyllithium afforded two isomers of 1-methyl-2,4-diphenyl-1,2,3,5-tetrakis(trimethylsilyl)-1-silacyclopent-3-ene in a ratio of 3 : 2 in high yields.

Introduction

Despite rapid developments within the last few years in the chemistry of siloles [1–13], very little interest has been shown in the chemical behavior of siloles toward organolithium reagents [9,11]. Recently, we have found that the reaction of 1-methyl-1-(trimethylsilyl)dibenzosilole with an excess of an alkyllithium such as methyl- or butyl-lithium in THF affords a transmetallation product, 1,1-dialkyldibenzosilole in almost quantitative yield * [11]. In contrast to this dibenzosilole, the reaction of 1-methyl-3,4-diphenyl-1,2,5-tris(trimethylsilyl)silole with methyllithium in THF, followed by hydrolysis, gives 1,1-dimethyl-3,4-diphenyl-2,2,5-tris(trimethylsilyl)silole in high yield [11].

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* Reactions which probably belong to the same category as ours have been reported by Gilman et al., see refs. 14–17.

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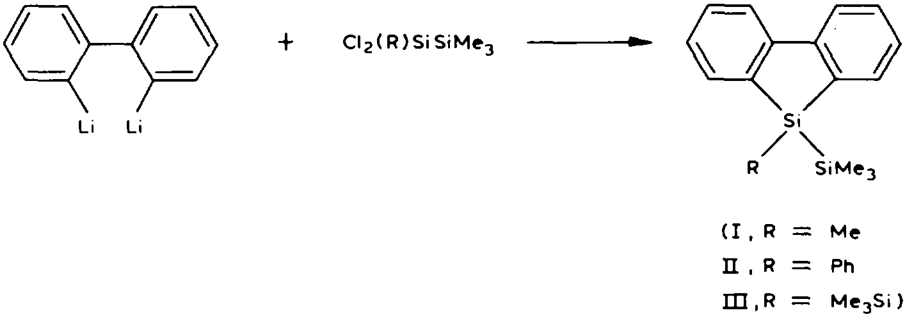
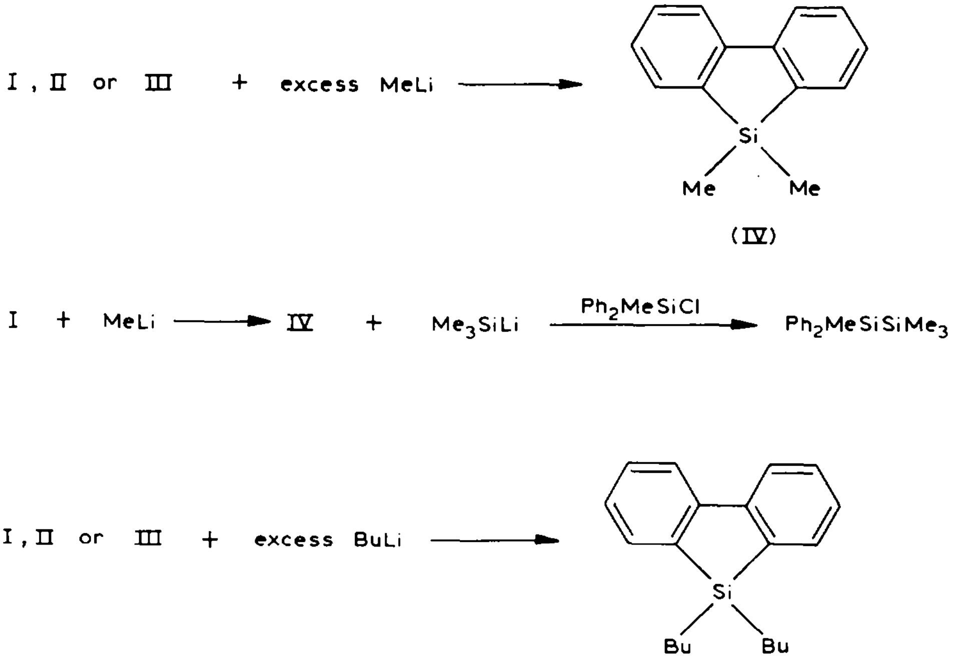
In an effort to learn much more about the chemistry of siloles, we have examined the reaction of several types of silole with organolithium reagents.

Results and discussion

1-Methyl-1-(trimethylsilyl)- and 1-phenyl-1-(trimethylsilyl)-dibenzosilole (I and II) were prepared from the reaction of 2,2'-dilithiobiphenyl with the corresponding 1,1-dichlorodisilanes in high yields. 1,1-Bis(trimethylsilyl)dibenzosilole (III) was synthesized from the reaction of 2,2-dichlorohexamethyltrisilane and 2,2'-dilithiobiphenyl in excellent yield.



When dibenzosilole I was treated with a 2.5-fold excess of methyllithium in THF at −78°C and warmed up to room temperature, a dark green solution was obtained. Hydrolysis of this solution afforded 1,1-dimethyldibenzosilole (IV) [17,18] quantita-



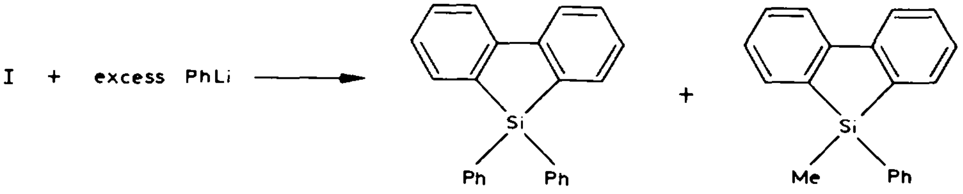
(V)

SCHEME 1

tively, indicating that substitution of the trimethylsilyl group by methyllithium took place as shown in Scheme 1. In this reaction, trimethylsilyllithium was expected to be formed. In fact, we could trap the trimethylsilyllithium as 1,1,1,2-tetramethyldiphenyldisilane using methyldiphenylchlorosilane as a quencher. Thus, the reaction of I with a 1.5-fold excess of methyllithium in THF at − 100°C, followed by treatment of the mixture with methyldiphenylchlorosilane, produced IV and 1,1,1,2-tetramethyldiphenyldisilane in 71 and 62% yield, respectively, in addition to a 12% yield of dimethyldiphenylsilane.

When dibenzosilole II was treated with a 2.5-fold excess of methyllithium in THF, compound IV was obtained as a single product. Similar reaction of silole III with an excess of methyllithium again afforded IV in quantitative yield. Furthermore, with a 2.5-fold excess of butyllithium, dibenzosiloles I, II and III yielded 1,1-dibutyldibenzosilole (V) quantitatively. These results indicate that both substituents on the silicon atom in the dibenzosilole ring can be replaced by methyl- or butyl-lithium.

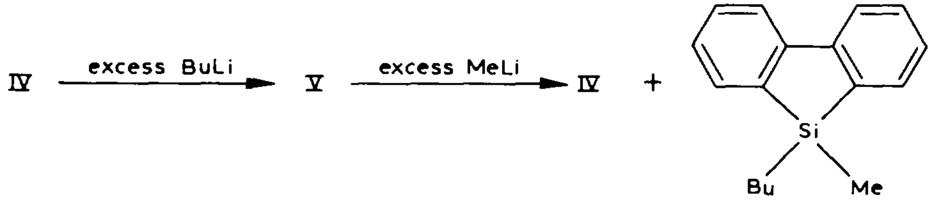
The present transmetallation is not restricted to the alkyllithium reagents. Thus, the reaction of I with a 2.6-fold excess of phenyllithium under the same conditions gave two products in almost quantitative yield. In this case, however, 1,1-diphenyldibenzosilole (VI) [19] was obtained only in 25% yield. 1-Methyl-1-phenyldibenzosilole (VII) was found to be the major product (75% yield). Such a difference in product distribution between the reaction with methyl- or butyllithium and that with phenyllithium may be attributed to the reactivity of the lithium reagent.



(VI) (VII)

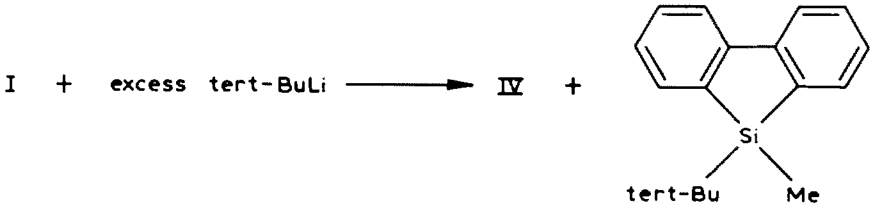
Interestingly, the reaction of 1,1-dimethyldibenzosilole IV with a 2.5-fold excess of butyllithium at room temperature afforded V quantitatively, while V with a 2.5-fold excess of methyllithium at reflux temperature gave IV and 1-butyl-1-methyldibenzosilole (VIII) in 10 and 40% yield, in addition to 37% of the starting dibenzosilole V. These results may also be attributed to the different reactivities of methyl- and butyl-lithium.

A lithium reagent having a bulky alkyl group, such as tert-butyllithium, also



(VIII)

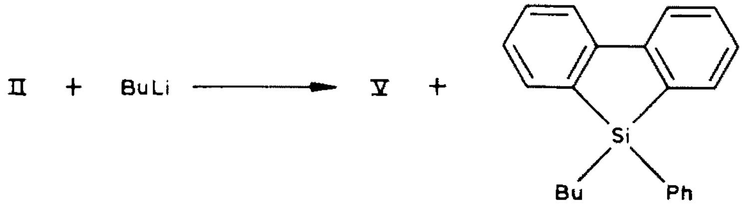
reacted with I to give transmetallation product; however, tert-butyl-substituted dibenzosilole was obtained only in low yield. When I was treated with a 2.5-fold excess of tert-butyllithium, 1-tert-butyl-1-methyldibenzosilole (IX) was obtained in 18% yield, in addition to 12% of IV. In this reaction, no di-tert-butyldibenzosilole could be detected by either GLC or spectroscopic analysis. The formation of IV can be explained in terms of the reaction of IX with methyllithium.



(IX)

The reaction conditions and yields of the products are listed in Table 1, and ^{1}H NMR chemical shifts, elemental analysis and mass and IR spectral data for isolated siloles are shown in Tables 2 and 3.

Next, we investigated the reaction of I and II with 1 equiv. of alkyllithium. In these reactions, a mixture of mono- and di-substituted dibenzosiloles was always obtained. When we treated I with 1 equiv. of butyllithium, 1,1-dibutyl- and 1-butyl-1-methyldibenzosilole, V and VIII, were obtained in 15 and 63% yield, respectively, in addition to 14% of the starting dibenzosilole I. Similar reaction of II with 1 equiv. of methyllithium gave IV and VII in 25 and 59% yield, respectively. In this reaction, 16% of the starting II was recovered unchanged. Compound II with 1 equiv. of butyllithium again afforded V and 1-butyl-1-phenyldibenzosilole (X) in 26 and 40% yield, together with 26% of the starting dibenzosilole II.



(X)

All the solutions obtained from the reaction of the dibenzosiloles with organolithium reagents in THF exhibit a deep green color. In order to learn whether or not radical species such as a radical anion are present in the solution, we determined the ESR spectrum of the solution. However, the reaction mixture prepared from I and butyllithium showed no ESR signal in the range of −70° to 0°C, indicating the absence of a stable radical in the colored solution.

To explain the results obtained, we propose a five-coordinate silicon species (A) as a key intermediate for the transmetallation of the dibenzosiloles with organolithium reagents. For example, the reaction of I with an organolithium reagent leading to the mono-substituted dibenzosilole is shown in Scheme 2.

In marked contrast to the dibenzosilole, the reaction of 1-methyl-3,4-diphenyl-

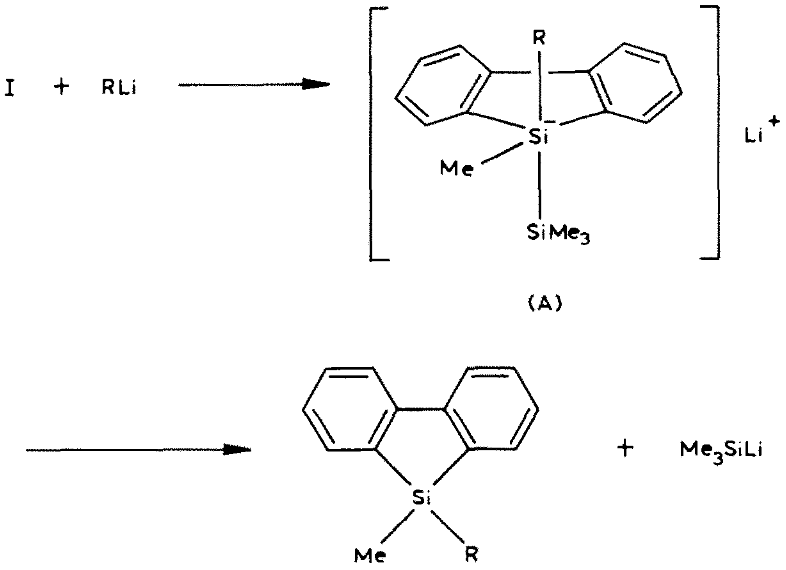
TABLE 1

REACTION OF SILOLES WITH ORGANOLITHIUM REAGENTS

Silole (g (mmol))	Organolithium reagent (ml (mmol))	Time (h)	THF (ml)	Product (yield, %)
I	MeLi			
0.0446 (0.17)	0.5 (0.41)	1	3.0	IV (100)
II	MeLi			
0.2045 (0.62)	2.0 (1.80)	1	5.0	IV (100)
III	MeLi			
0.0790 (0.24)	0.5 (0.60)	1	3.0	IV (100)
I	BuLi			
0.1818 (0.68)	1.0 (1.60)	1	5.0	V (100)
II	BuLi			
0.0444 (0.13)	0.2 (0.32)	1	3.0	V (100)
III	BuLi			
0.1279 (0.39)	0.6 (0.96)	1	5.0	V (100)
I	PhLi			
0.1186 (0.44)	1.0 (1.15)	2	5.0	VI (25), VII (75)
I	t-BuLi			
0.5140 (1.92)	2.3 (4.80)	2	5.0	I (14), IV (12), IX (18)
I	MeLi			
0.1470 (0.55)	0.5 (0.60)	1	2.0	IV (100)
I	BuLi			
0.2594 (0.96)	0.6 (0.96)	1	2.0	I (14), V (15), VIII (63)
II	MeLi			
0.5738 (1.74)	1.5 (1.79)	1	3.0	II (16), IV (25), VII(59)
II	BuLi			
0.4753 (1.44)	0.9 (1.44)	1	3.0	II (26), V (26), X (40)
IV	BuLi			
0.9662 (4.59)	7.0 (11.0)	2	10.0	V (100)
V	MeLi			
0.1559 (0.53)	1.1 (1.30)	10[a]	5.0	IV (10), V (37), VIII (40)
XI	MeLi			
0.4340 (0.93)	3.0 (3.60)	4	5.0	XV (7), XIV (70)
XII	MeLi			
0.4900 (0.94)	2.5 (3.00)	2	10.0	XVIII (46), XIX (30)
XIII	MeLi			
0.2327 (0.44)	1.3 (1.56)	1	5.0	XVIII (52), XIX (35)

[a] Reflux temperature.

1,2,5-tris(trimethylsilyl)silole (XI) [10], and 3,5-diphenyl-1,1,2,4-tetrakis(trimethylsilyl)silole (XII) [20] and its isomer, 4,5-diphenyl-1,1,2,3-tetrakis(trimethylsilyl)silole (XIII) [20] with methyllithium gave rearranged addition products. Thus, the addition of an excess of methyllithium in ether solution to silole XI in THF at −78°C, warming up slowly to room temperature, followed by hydrolysis of the resulting dark red solution gave 1,1-dimethyl-3,4-diphenyl-2,2,5-tris(trimethylsilyl)-1-silacyclopent-3-ene (XIV) in 70% yield, besides 1,1-dimethyl-3,4-diphenyl-2,5-bis(trimethylsilyl)silole (XV) (7% yield). The production of XIV can be best explained in terms of the transient formation of a five-coordinate intermediate (B), followed by rearrangement of a trimethylsilyl group attached to the pentavalent silicon atom to an



SCHEME 2

TABLE 2

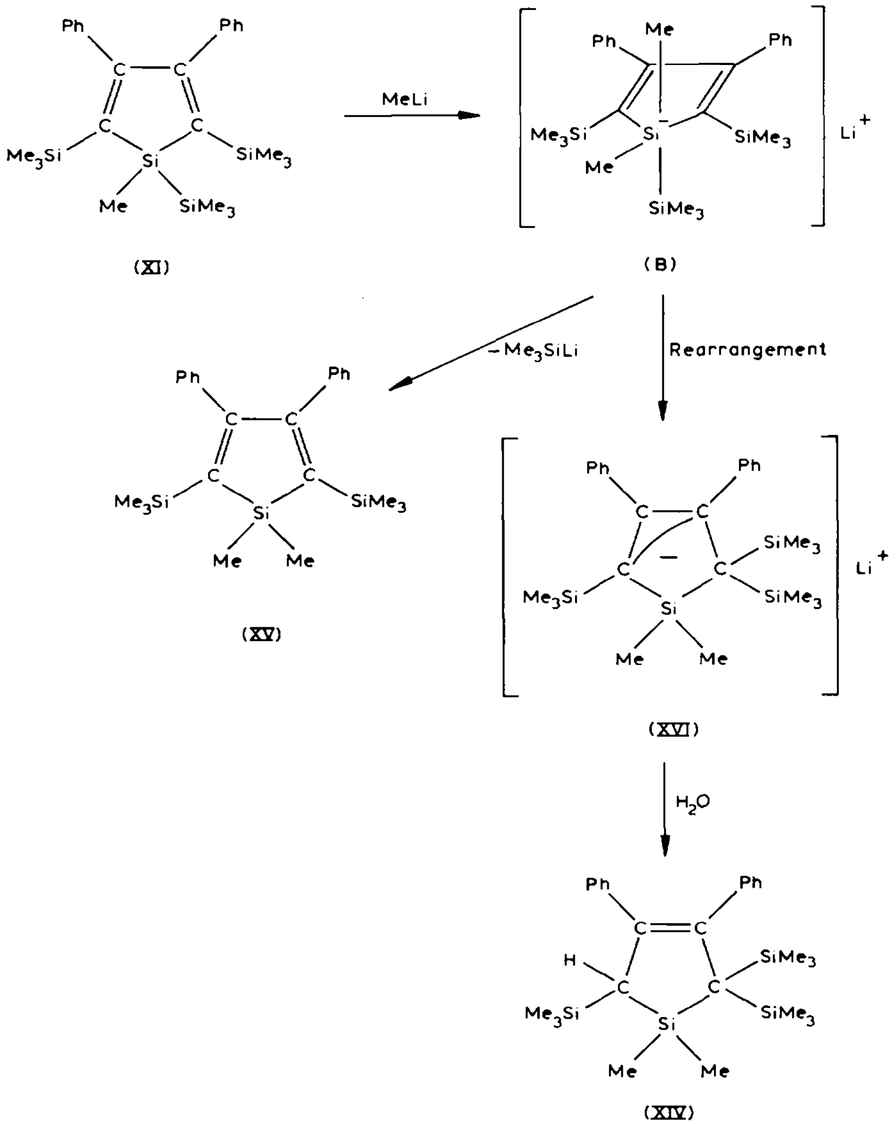
^{1}H NMR CHEMICAL SHIFTS FOR SILOLES

Compound	Chemical shifts, δ (ppm) in CCl_4
IV	0.41(Me$_2$Si, s, 6H), 7.15–7.90(ring protons, m, 8H)
V	0.70–1.05(CH$_3$C and CH$_2$Si, m, 10H), 1.15–1.55(CH$_2$CH$_2$, m, 8H), 7.10–7.85(ring protons, m, 8H)
VI	7.05–7.80(ring protons, m, 18H)
VII	0.71(MeSi, s, 3H), 7.00–7.80(ring protons, m, 13H)
VIII	0.40(MeSi, s, 3H), 0.75–1.10(CH$_3$C and CH$_2$Si, m, 5H), 1.20–2.05(CH$_2$CH$_2$, m, 4H), 7.0–7.8 (ring protons, m, 8H)
IX	0.41(MeSi, s, 3H), 1.00(Me$_3$C, s, 9H), 7.12–7.88(ring protons, m, 8H)
X	0.75–0.95(CH$_3$C, m, 3H), 1.15–1.50(CH$_2$CH$_2$CH$_2$, m, 6H), 7.00–7.75(ring protons, m, 13H)
XIV [a]	−0.24(Me$_3$Si, s, 9H), 0.10(Me$_3$Si, s, 9H), 0.21(Me$_3$Si, s, 9H), 0.40(MeSi, s, 3H), 0.51(MeSi, s, 3H), 1.85(HC, s, 1H), 6.5–7.0(ring protons, m, 10H)
XVIII [b]	−0.38(Me$_3$Si, s, 9H), −0.29(Me$_3$Si, s, 9H), −0.22(Me$_3$Si, s, 9H), 0.39 (Me$_3$Si, s, 9H), 0.51(MeSi, s, 3H), 1.85(HC, s, 1H), 6.8–7.3(ring protons, m)
XIX [b]	−0.55(Me$_3$Si, s, 9H), −0.29(Me$_3$Si, s, 9H), −0.27(Me$_3$Si, s, 9H), 0.41(Me$_3$Si, s, 9H), 0.55(MeSi, s, 3H), 1.94(HC, s, 1H), 6.8–7.3(ring protons, m)

[a] ^{13}C NMR chemical shifts for XIV, 0.9(Me$_3$Si), 2.8(MeSi), 3.3(MeSi), 3.6(Me$_3$Si), 5.0(Me$_3$Si), 26.4(CH(SiMe$_3$)), 27.8(C(SiMe$_3$)$_2$), 125.1, 125.6, 126.2, 126.9, 129.7, 131.6, 142.2, 142.4, 142.7, 143.6(olefinic and phenyl ring carbons); ^{29}Si NMR chemical shifts for XIV, 20.6(SiMe$_3$–CH(Si)), 2.4 (SiMe$_3$–C(SiMe$_3$)(Si)), −0.1(SiMe$_2$), −2.0(SiMe$_3$–C(SiMe$_3$)(Si)).

[b] ^{13}C NMR chemical shifts for XVIII and XIX, −1.3(Me$_3$Si), 0.0(Me$_3$Si), 0.4 (MeSi), 0.9(Me$_3$Si), 1.6(MeSi), 3.4(Me$_3$Si), 4.0(Me$_3$Si), 29.9(CH(SiMe$_3$)), 33.8(CPh(SiMe$_3$)), 46.0(CPh(SiMe$_3$)), 123.7, 126.8, 127.1, 127.6, 128.2, 129.0, 130.1, 130.6, 143.6, 144.9, 145.8, 148.8, 156.5(olefinic and phenyl ring carbons); ^{29}Si NMR chemical shifts for XVIII, 22.3(SiMe$_3$–CPh(Si)), 1.7(SiMe$_3$–CH(Si)), −2.7(SiMe$_3$–C=C), −8.0(SiMe–(SiMe$_3$)), −17.5(SiMe$_3$–Si). For XIX, 13.8(SiMe$_3$–CPh(Si)), 0.2(SiMe$_3$–CH(Si)), −1.7(SiMe$_3$–C=C), −7.2(SiMe–(SiMe$_3$)), −17.0(SiMe$_3$–Si).

adjacent unsaturated carbon atom, while elimination of trimethylsilyllithium from the intermediate B leads to the formation of XV. Lithium compound XVI could be detected by ^{1}H NMR spectroscopic analysis of the resulting solution [11] (Scheme 3).



SCHEME 3

In this reaction, no products arising from the addition of methyllithium across the silacyclopentadiene ring could be detected. The addition of butyllithium across the silacyclopentadiene ring has recently been reported by Jutzi and Karl [9].

TABLE 3

ELEMENTAL ANALYSES, MELTING POINTS, MASS AND IR SPECTRAL DATA FOR ISOLATED SILOLES

Compound		Found (calcd.) (%)		M.p. (°C)	M^+	IR (cm^{-1})
		C	H			
IV	$C_{14}H_{14}Si$	-	-	56-57	210	1430,1260,1245
V	$C_{20}H_{26}Si$	81.35(81.56)	8.92(8.90)	-	294	1430,1260
VI	$C_{24}H_{18}Si$	-	-	-	334	1430,1425
VII	$C_{19}H_{16}Si$	83.64(83.77)	5.81(5.92)	66	272	1430,1260,1250
VIII	$C_{17}H_{20}Si$	80.49(80.89)	7.77(7.99)	-	252	1455,1430,1255 1245
IX	$C_{17}H_{20}Si$	80.73(80.89)	8.08(7.99)	68-70	252	-
X	$C_{22}H_{22}Si$	83.74(84.02)	7.17(7.05)	-	314	1460,1430,1260
XIV	$C_{27}H_{44}Si_4$	67.27(67.42)	9.44(9.22)	118	480	1493,1441,1263, 1250
XVIII [a] XIX	$C_{29}H_{50}Si_5$	64.80(64.61)	9.59(9.35)		538	1485,1440,1267, 1250

[a] A mixture of XVIII and XIX.

The structure of XIV was confirmed by spectroscopic as well as elemental analysis. All the spectral data for XV were identical with those of an authentic sample prepared by an independent route [10].

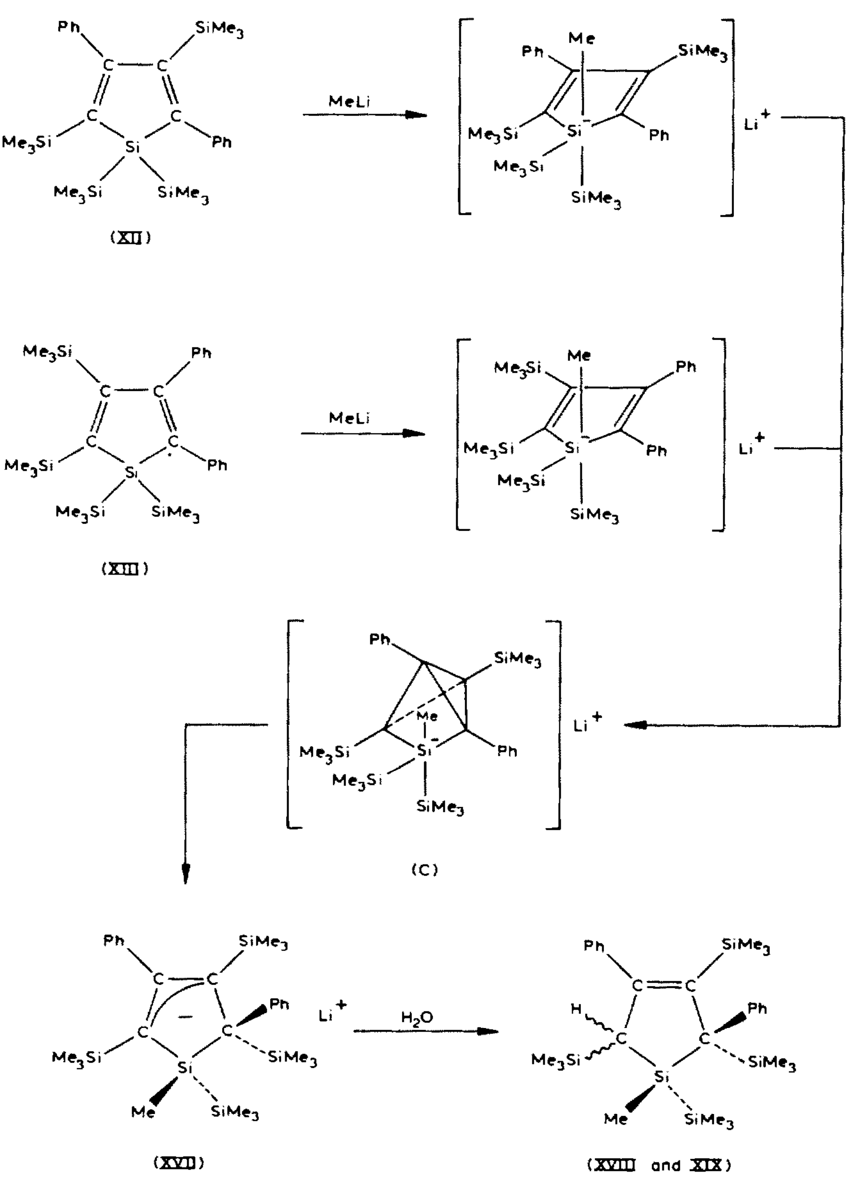
Similar treatment of 3,5-diphenyl-1,1,2,4-tetrakis(trimethylsilyl)silole XII with methyllithium in THF again afforded a dark red solution. The ^1H NMR spectrum of this solution showed five singlets at δ -0.54, -0.47, -0.36, 0.32 and 0.35 ppm with relative intensities of 3 : 3 : 3 : 3 : 1, due to four different kinds of trimethylsilyl protons and methylsilyl protons, indicating the presence of a single lithium compound (XVII). Hydrolysis of this solution, however, gave a mixture consisting of two isomers of 1-methyl-2,4-diphenyl-1,2,3,5-tetrakis(trimethylsilyl)-1-silacyclopent-3-ene (XVIII and XIX) in a ratio of 3 : 2 in 76% combined yield. Unfortunately, all attempts to separate XVIII from XIX were unsuccessful. Compounds XVIII and XIX were identified tentatively by ^1H NMR, ^{13}C NMR, ^{29}Si NMR, mass and IR spectroscopic methods.

Surprisingly, silole XIII reacted with methyllithium to give the same lithium compound as that from XII with methyllithium. Indeed, the ^1H NMR spectrum of the resulting dark red solution prepared from XIII with methyllithium in THF was identical with that of lithium compound XVII. As can be expected, hydrolysis of the solution gave a 3 : 2 mixture consisting of two isomers of XVIII and XIX in 87% yield. The reaction mechanism leading to the products, XVIII and XIX, is not yet clear, but it seems likely that a tricyclic five-coordinate species such as C would be involved as an intermediate as shown in Scheme 4.

Experimental

General procedure

Proton NMR spectra were determined at ambient temperature with a JEOL Model JNM-MH-100 spectrometer using carbon tetrachloride solutions containing



SCHEME 4

cyclohexane (δ 1.44 ppm) as internal standard. Mass spectra were obtained on a JEOL Model JMS-D 300 equipped with a JMA-2000 data processing system. The ionizing voltage was 24 eV for all compounds. Tetrahydrofuran was dried over sodium-potassium alloy and distilled just before use.

Preparation of 1-methyl-1-(trimethylsilyl)dibenzosilole (I)

In a 100-ml two-necked flask fitted with a condenser and a dropping funnel were placed 6.2 g (19.8 mmol) of 2,2′-dibromobiphenyl in 30 ml of dry ether. To this were added 26 ml (41.6 mmol) of butyllithium-hexane solution at −78°C. The mixture was stirred for 3 h at room temperature, and 3.8 g (20.2 mmol) of 1,1-dichlorotetramethyldisilane in 20 ml of ether were then added to the mixture without cooling. The mixture was heated to reflux for 3 h and the mixture was hydrolyzed with water. After the solvent was evaporated, the residue was distilled under reduced pressure (1 Torr) to give 5.0 g (94% yield) of I, m.p. 57°C; IR (cm^{-1}) 1430, 1255, 1245; M^+ 268; ^{1}H NMR (δ, ppm) 0.05 (Me_3Si, s, 9H), 0.45 (MeSi, s, 3H), 7.15–7.95 (ring protons, m, 8H) (Found: C, 71.48; H, 7.59. $C_{16}H_{20}Si_2$ calcd.: C, 71.57; H, 7.51%).

Preparation of 1-phenyl-1-(trimethylsilyl)dibenzosilole (II)

To a solution of 2,2′-dilithiobiphenyl prepared from 3.5 g (11.2 mmol) of 2,2′-dibromobiphenyl and 15 ml (24.0 mmol) of butyllithium-hexane solution in 20 ml of ether were added 2.8 g (11.2 mmol) of 1,1-dichloro-1-phenyltrimethyldisilane at room temperature. After work-up in the usual manner, distillation under reduced pressure (1 Torr) gave 3.4 g (92% yield) of II, m.p. 54°C; IR (cm^{-1}) 1435, 1260, 1245; M^+ 330; ^{1}H NMR (δ, ppm) 0.15 (Me_3Si, s, 9H), 7.10–7.95 (ring protons, m, 13H) (Found: C, 76.35; H, 6.96. $C_{21}H_{22}Si_2$ calcd.: C, 76.30; H, 6.71%).

Preparation of 1,1-bis(trimethylsilyl)dibenzosilole (III)

To a solution of 2,2′-dilithiobiphenyl prepared from 4.0 g (12.8 mmol) of 2,2′-dibromobiphenyl and 16 ml (25.6 mmol) of butyllithium-hexane solution were added 3.2 g (13.0 mmol) of 2,2-dichlorohexamethyltrisilane. After work-up in the usual manner, distillation under reduced pressure (1 Torr) gave 4.0 g (96% yield) of III, m.p. 60–61°C; IR (cm^{-1}) 1430, 1250, 1240; M^+ 326; ^{1}H NMR (δ, ppm) 0.12 (Me_3Si, s, 18H), 7.00–7.85 (ring protons, m, 8H) (Found: C, 65.82; H, 8.22. $C_{18}H_{26}Si_3$ calcd.: C, 66.18; H, 8.02%).

Reaction of dibenzosiloles with organolithium reagents

The following is typical of the procedures used. In a 25-ml two-necked flask were placed 44.6 mg (0.166 mmol) of I in 3 ml of THF. The flask was cooled to −78°C. To this was added 0.45 ml (0.41 mmol) of methyllithium-ether solution at the same temperature. The mixture was warmed up to room temperature and the resulting dark green solution was stirred for 1 h at room temperature. The mixture was hydrolyzed with water. The yield of product IV was determined by GLC using cetane as an internal standard. Pure IV was isolated by preparative GLC. The reaction conditions and yields of the products obtained are listed in Table 1.

Trapping of trimethylsilyllithium

In a 25-ml two-necked flask were placed 183.5 mg (0.68 mmol) of dibenzosilole I in 10 ml of THF. The flask was cooled to −100°C. To this was added 0.4 ml (1.02 mmol) of methyllithium-ether solution. The mixture was stirred at −100 to −78°C for 4 h. Methyldiphenylchlorosilane (159.4 mg, 0.69 mmol) was added to the lithium solution, and the mixture was then warmed up to room temperature. The mixture was hydrolyzed with water. GLC analysis of it showed the presence of IV (71% yield) and 1,1,1,2-tetramethyldiphenyldisilane (62% yield). 1,1,1,2-Tetramethyldiphenyldisilane was isolated by preparative GLC. All the spectral data of this compound were identical with those of an authentic sample [21].

Reaction of siloles with methyllithium

The following is typical of the procedures used. To 0.4900 g (0.94 mmol) of silole XII in 10 ml of THF were added 2.5 ml (3.0 mmol) of methyllithium-ether solution at room temperature. The mixture was stirred at room temperature for 2 h, and hydrolyzed with water. The organic layer was separated and washed with water, and then dried over potassium carbonate. After evaporation of the solvent, the residue was analyzed by GLC as being XVIII and XIX (76% combined yield). The residue was chromatographed, and the ratio of XVIII and XIX was determined by ^{1}H NMR to be 3 : 2.

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