

Synthesis of 1,1-Bis(*n*-butyl, *t*-butyl)-2,3,4,5-Tetraphenyl-1-Silacyclopentadiene and NMR Study of Their 2,5-Carbodianions

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Abstract

1,1-Bis(*n*-butyl)-2,3,4,5-tetraphenyl-1-silacyclopentadiene (**3**) and 1,1-bis(*t*-butyl)-2,3,4,5-tetraphenyl-1-silacyclopentadiene (**4**) are synthesized from the reaction of the versatile silole dianion (**2**) with *n*-butyl bromide and *t*-butyl bromide. Reduction of (**3**) and (**4**) with an excess of lithium to give 1,1-bis(*n*-butyl)-2,5-dilithio-2,3,4,5-tetraphenyl-1-silacyclopenta-3-ene (**6**) and 1,1-bis(*t*-butyl)-2,5-dilithio-2,3,4,5-tetraphenyl-1-silacyclopenta-3-ene (**7**). ¹³C-NMR study of two 2,5-carbodianions (**6** and **7**) shows *tert*-carbanion at 73.18 and 78.12 ppm respectively. Two bulky *tert*-butyl groups in (**7**) increase the inversion barrier at the *tert*-carbanion, line broadenings of *tert*-butyl groups in ¹H and ¹³C-NMR spectrum are observed.

Key words: Silole, *tert*-Carbanion, 2,5-Carbodianion, Inversion

1. Introduction

In last decades a lot of group 14 metalloles have been prepared^[1-6]. Especially their anions/dianions have been reported to be aromatic systems in experimental^[7-25] and theoretical studies^[26-31] except trimethylsilyl group substituted silole and germole anions^[10,18,20]. C_α, C_β, C_{αi}, C_{βi} atoms of 2,3,4,5-tetraphenyl substituted group 14 metallole anions/dianions are participate in π-delocalization of the negative charge on Si atom to give chemical shift changes ongoing from group 14 metalloles to their anions/dianion^[9,10,12]. Lately siloles have been attracted by the unique electronic structures^[32] and novel aggregation-induced emission (AIE) characteristics^[33]. Recently the reduced silole species of 2,5-dilithio-1-silacyclopenta-3-ene have been utilized as a new route to synthesize a silole-containing hyperbranched poly(silylenevinylene)^[34,35]. However there are few reports for the reduction of siloles to 2,5-carbodianion species since W.H. Atwell and coworkers had reported for silole anions and dianions^[36]. Herein we report NMR study of 1,1-bis(*n*-butyl/*t*-butyl)-2,5-dilithio-2,3,4,5-tetraphenyl-1-silacyclopenta-3-ene.

2. Experimental Section

2.1. General Procedures

All reactions were performed under an inert nitrogen atmosphere using standard Schlenk techniques. Air sensitive reagents were transferred in a nitrogen-filled glove box. THF and ether were distilled from sodium benzophenone ketyl under nitrogen. Hexane and pentane were stirred over concentrated H₂SO₄ and distilled from CaH₂. NMR spectra were recorded on JEOL GSX270 and GSX400 spectrometers. GC-MS and solid sample MS data were obtained on a Hewlett-Packard 5988A GC-MS system equipped with a methyl silicon capillary column. Elemental analyses were done by Desert Analytics (Tucson, AZ, USA).

2.2. 1,1-bis(*n*-butyl)-2,3,4,5-tetraphenyl-1-silacyclopentadiene (**3**).

1,1-dichloro-2,3,4,5-tetraphenyl-1-silacyclopentadiene (0.57 g, 1.25 mmol) was sonicated with an excess of lithium for 2 hr. Then the remaining lithium was removed by filtration to give the red purple solution of the silole dianion. The solution was added to THF solution of 1-bromo-butane with stirring at room temperature for 2 hrs to give yellow solution. After removing solvent under vacuum the remained yellow solid was extracted with hexane. The concentrated solution was kept in refrigerator for couple of days to

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provide yellow crystals. Yield: 0.56 g (90%); mp 85°C (lit.[43], mp 81°C).

2.3. 1,1-Bis(*t*-butyl)-2,3,4,5-tetraphenyl-1-silacyclopentadiene (4).

1,1-dichloro-TPSCp (0.55 g, 1.21 mmol) was sonicated with an excess of lithium in THF for 2 hr. Then the remaining lithium was removed by filtration to give the red purple solution of (2). The solution was added to THF solution of *t*-butylbromide with stirring at room temperature for 5 hrs to give yellow solution. After removing solvent under vacuum the remained yellow solid was extracted with ether. The concentrated solution was kept in refrigerator for couple of days to provide pale yellow crystals of 1,1-bi(1-*t*-butyl-2,3,4,5-tetraphenyl-1-silacyclopentadiene) (5). The filtrated solution was concentrated under vacuum, then it was kept in refrigerator for couple of days to give yellow crystals of 1,1-bis(*t*-butyl)-2,3,4,5-tetraphenyl-1-silacyclopentadiene (4). Yield: 0.33 g (54%); mp 169-171°C, ¹H-NMR (CDCl₃, ref; ext. TMS = 0.00 ppm), 1.16 (s, Me, 18H), 6.68-7.15 (m, 20H), ²⁹Si-NMR (CDCl₃, ref; ext. TMS=0.00), 16.49; Anal Calcd for C₃₆H₃₈Si: C, 86.69; H, 7.68, Found: C, 86.71; H, 7.75. 1,1-Bi(1-*t*-butyl-2,3,4,5-tetraphenyl-1-silacyclopentadiene) (5) Yield: 0.19 g (18%)^[8].

2.4. NMR Study of the Reduction of (3) and (4) with Lithium in THF-*d*₆.

The respective 1,1-bis(*n*-butyl)-TPSCp (3) (0.025 g,

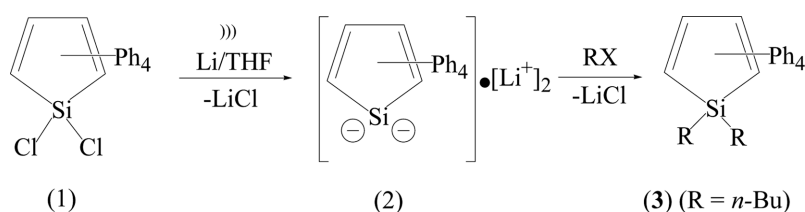
0.05 mmol) and 1,1-bis(*t*-butyl)-TPSCp (4) (0.025 g, 0.05 mmol) was transferred into 5 mm NMR tube, they were sonicated with an excess of lithium in THF-*d*₆ for 1.5 hr to give red-purple solution. Then ¹H, ¹³C, and ²⁹Si-NMR spectroscopic study was performed. 1,1-Bis(*n*-butyl-2,5-dilithio-2,3,4,5-tetraphenyl-1-silacyclopentadiene) (6); ¹H-NMR (THF-*d*₆, ref; ext. TMS = 0.00 ppm), 0.83 (t, CH₃, 6H), 0.90 (m, CH₂, 4H), 1.36 (sept, CH₂, 4H), 1.52 (m, CH₂, 4H), 6.68-7.15 (m, 20H). 1,1-Bis(*t*-butyl-2,5-dilithio-2,3,4,5-tetraphenyl-1-silacyclopentadiene) (7); ¹H-NMR (THF-*d*₆, ref; ext. TMS = 0.00 ppm), 1.21 (brd s, Me, 18H), 6.68-7.15 (m, 20H).

3. Results and Discussion

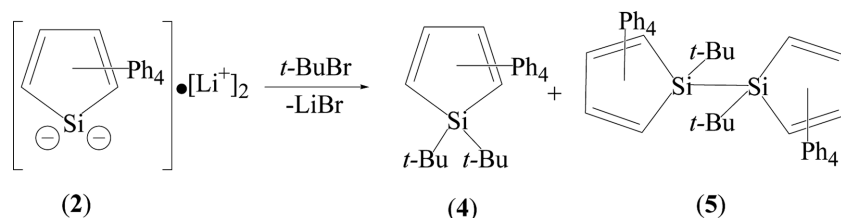
3.1. Preparation of 1,1-bis(*n*-butyl/*t*-butyl)-2,3,4,5-tetraphenyl-silacyclopentadiene

1,1-Bis(*n*-butyl)-2,3,4,5-tetraphenyl-silacyclopentadiene [1,1-bis(*n*-butyl)-TPSCp] (3) is prepared from the reaction of 2,3,4,5-tetraphenyl-1-silacyclopentadienide dianion (2) with *n*-bromobutane in good yield. The silole dianion (2) is generated by the sonication of 1,1-dichloro-TPSCp (1) in THF with lithium^[11].

However, in the case of *tert*-butyl bromide, 1,1-bis(*t*-butyl)-TPSCp (4) is provided with 1,1-bi(1-*t*-butyl-TPSCp) (5) in the ratio of 3 to 1. On the contrary it was reported that the silole dianion of [TPSCp]²⁻•[Na⁺]₂ was reacted with *tert*-butyl chloride to give (5) as major^[8].

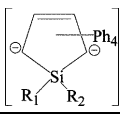
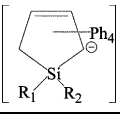


(1)



(2)

Table 2. ^{13}C -NMR chemical shifts

	$\text{R}_1 = \text{Me}$ $\text{R}_2 = \text{Me}$	$\text{R}_1 = \text{Me}$ $\text{R}_2 = \text{H}$	$\text{R}_1 = n\text{-Bu}$ $\text{R}_2 = n\text{-Bu}$	$\text{R}_1 = t\text{-Bu}$ $\text{R}_2 = t\text{-Bu}$		$\text{R}_1 = \text{H}$ $\text{R}_2 = \text{H}$	$\text{R}_1 = \text{Me}$ $\text{R}_2 = \text{H}$	$\text{R}_1 = \text{N}(\text{SiMe}_3)_2$ $\text{R}_2 = \text{H}$
M^+	$\text{M} = \text{Li}$	$\text{M} = \text{Li}$	$\text{M} = \text{Li}$	$\text{M} = \text{Li}$	M^+	$\text{M} = \text{K}$	$\text{M} = \text{K}$	$\text{M} = \text{Na}$
ppm	77.4	76.42	73.18	78.12	ppm	77.78	86.20	89.77
ref	[38]	[39]	This work		ref	[40]	[40]	[41]

In THF- d_8 , reference = 25.30 ppm.

Table 2. ^{29}Si -NMR chemical shifts

(4) ^a	(6) ^b ($\text{R} = n\text{-Bu}$) ^b	(7) ^b ($\text{R} = t\text{-Bu}$) ^b
16.49	-0.27	13.69

^aIn CDCl_3 , reference; external TMS as standard; ^bIn THF- d_8 , reference; external TMS as standard.

R_1, R_2 -2,3,4,5-tetraphenyl-1-silacyclopenta-3-ene ($\text{R}_1 = \text{R}_2 = \text{H}$; $\text{R}_1 = \text{Me}$, $\text{R}_2 = \text{H}$ ^[40]; $\text{R}_1 = \text{N}(\text{SiMe}_3)_2$, $\text{R}_2 = \text{H}$ ^[40]).

Upon lithiation of (4) to (7) the chemical shift of ^{29}Si -NMR in (4) does not change much, but it depends on the substituent of Si atom.

3.4. Inversion of *tert*-carbanions in (6) and (7)

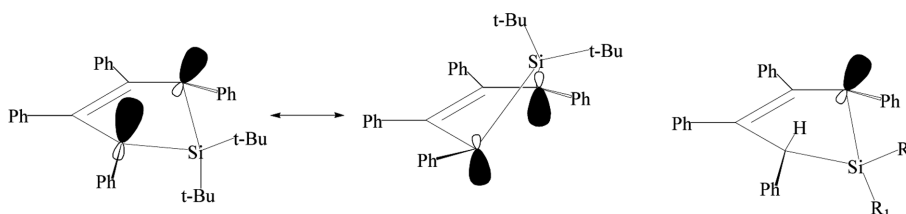
Before lithiation two *tert*-butyl groups of (4) in ^1H -NMR are observed as a sharp singlet at 1.16 ppm, but unexpectedly those of (7) are observed as broad singlet at 1.21 ppm. Moreover the *tert*-butyl groups of (7) in ^{13}C -NMR are observed as a broad doublet at 31.5 ppm and 33.3 ppm with no *tert*-carbon peak to be assigned, those of (4) are observed sharp singlet at 20.79 ppm for *tert*-C and 29.01 ppm for methyl groups. This may be to the slow inversion rate at two *tert*-carbanions in (7) due to the bulkiness of two *tert*-butyl groups substituted on Si atom. However in the case of (6) with two *n*-butyl groups it seems to be fast inversion at two *tert*-carbanions since there is no observable line broadening ^1H and

^{13}C -NMR of (6). In the cases of (6) and (7) there are two *tert*-carbanions with no tetra valence bonds at 5-position, those lower the inversion barriers each other. Even though two bulky *tert*-butyl groups in (7) increase the barrier, they still do slow inversion to broaden the lines of *tert*-butyl groups in ^1H and ^{13}C -NMR spectrum.

On the contrary no inversion at 2-carbanion for 1,1- R_1, R_2 -2-lithio-2,3,4,5-tetraphenyl-1-silacyclopenta-3-enide ($\text{R}_1 = \text{Me}$, $\text{R}_2 = \text{H}$; $\text{R}_1 = \text{N}(\text{SiMe}_3)_2$, $\text{R}_2 = \text{H}$) was reported^[42]. They have one *tert*-carbanion with tetra valence bonds at 5-position, which increases the barrier to prevent the inversion.

4. Conclusions

1,1-Bis(*n*-butyl)-2,3,4,5-tetraphenyl-1-silacyclopentadiene (3) and 1,1-bis(*t*-butyl)-2,3,4,5-tetraphenyl-1-silacyclopentadiene (4) are synthesized from the reaction of the versatile silole dianion (2) with *n*-butyl bromide and *t*-butyl bromide. Reduction of (3) and (4) with an excess of lithium in THF- d_8 by sonication gives the respective 1,1-bis(*n*-butyl)-2,5-dilithio-2,3,4,5-tetraphenyl-1-silacyclopenta-3-enide (6) and 1,1-bis(*t*-butyl)-2,5-dilithio-2,3,4,5-tetraphenyl-1-silacyclopenta-3-enide (7). Two bulky *tert*-butyl groups in (7) increase the inversion barrier at the *tert*-carbanions, line broadenings of *tert*-butyl groups in ^1H and ^{13}C -NMR spectrum are observed.

Scheme 1. Inversion of *tert*-carbanions

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