Journal of Organometallic Chemistry 696 (2011) 982–985

Contents lists available at ScienceDirect

Journal of Organometallic Chemistry

journal homepage: www.elsevier.com/locate/jorganchem

Note

Synthesis and structure of 9,10-bis(2,4,6-triisopropylphenyl)-9,10-dihydro-9,10-disilaanthracene

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ARTICLE INFO

Article history:
Received 17 August 2010
Accepted 5 October 2010
Available online 13 October 2010

Keywords:
Disilaanthracene
Bulky substituent
Wurtz-type coupling
Crystal structure
Theoretical calculations

ABSTRACT

We prepared a 9,10-dihydro-9,10-disilaanthracene (DSA) derivative **4** bearing a bulky aryl substituent, a 2,4,6-triisopropylphenyl (Tip) group, on the silicon atoms via a Wurtz-type coupling reaction between two molecules of (2-chlorophenyl)(2,4,6-triisopropylphenyl)silane (**3**) in a head-to-tail fashion. The structural determination of *cis*-**4** and *trans*-**4** was examined using ^{1}H NMR spectroscopy and theoretical calculations. In addition, the molecular structure of *cis*-**4** was unambiguously determined by X-ray crystallography. The tricyclic DSA skeleton of *cis*-**4** adopts a folded structure with a boat-like central ring in which the rotation about the Si–Tip bond is restricted. In contrast, theoretical calculations suggest that the tricyclic DSA skeleton of *trans*-**4** has an almost planar structure.

1. Introduction

9,10-Dihydro-9,10-disilaanthracenes (DSAs) have attracted considerable attention as precursors of organosilicon reactive intermediates such as silyl anions [1,2] and silyl radicals [3]. We [4,5] and Chatgilialoglu and coworkers [6] independently reported that DSA derivatives can be used as radical-reducing agents, and the DSA framework is essential to generate the silyl radical efficiently [7]. An important goal in this area is the synthesis of 9,10-disilaanthracene, which is a novel silaaromatic compound. There have been some reports on the transient generation of the related 9-silaanthracene [8–10] and 1,4-disilabenzene [11–14]. We also reported the thermal generation and chemical trapping of the transient 9-silaanthracenes [15]. In 2002, Tokitoh and coworkers successfully isolated 9-silaanthracene, which was kinetically stabilized by a bulky substituent [16]. However, there have been no reports on the isolation or chemical trapping of the disilaaromatic 9,10-disilaanthracene. A simple route to 9,10-disilaanthracene involves the thermal extrusion of molecular hydrogen from the corresponding dihydride (DSA). In addition, the existence of bulky substituents on the silicon atoms is expected to improve the stability of the reactive silaaromatic species.

In this paper, we report the synthesis and structure of 9,10-bis(2,4,6-triisopropylphenyl)-9,10-dihydro-9,10-disilaanthracene (**4**), the first DSA derivative bearing bulky aryl substituents on the silicon atoms.

2. Results and discussion

Generally, the DSA framework is constructed through a halogen–metal exchange reaction of bis(2-halophenyl)silane followed by ring closure with an appropriate chlorosilane or hydrosilane (see Scheme 1, route A) [17,18]. However, our initial attempt to obtain bis(2-chlorophenyl)(2,4,6-triisopropylphenyl)silane (**2**) via a coupling reaction of (2,4,6-triisopropylphenyl)silane ($TipSiH_3$) with two equivalents of (2-chlorophenyl)lithium (prepared by lithiation of 1-bromo-2-chlorobenzene (**1**) at −95 °C) was unsuccessful, probably because of steric effects. Instead, we obtained a 1:1 coupling product **3** in a 79% yield. Therefore, we focused our attention to the Wurtz-type coupling between two molecules of 2-chlorophenylsilane **3**; Gilman and coworkers had previously synthesized some DSA derivatives via this reaction [19]. When the intermolecular cyclization reaction of **3** was performed using molten sodium in refluxing toluene, 9,10-bis(2,4,6-triisopropylphenyl)-9,10-dihydro-9,10-disilaanthracene (**4**) was obtained as a 43:57 mixture of the *cis* and *trans* isomers in a 21% yield. *Cis*-**4** is thermodynamically more stable than the *trans* isomer by 1.13 kcal/mol according to calculations at the B3LYP/6-31G(d,p) level of theory, as described below. The slight predominant formation of *trans*-**4** can be attributed to the kinetically controlled ring closure reaction, as observed in the synthesis of the corresponding 9,10-di-*tert*-butyl derivatives reported by Kyushin and coworkers [3].

Cis-**4** and *trans*-**4** were partially separated by silica gel column chromatography and were isolated in 7% and 9% yields, respectively. Characterizations of these new DSA derivatives included ^{1}H, ^{13}C, and ^{29}Si NMR; IR; MS; and elemental analyses. In particular,

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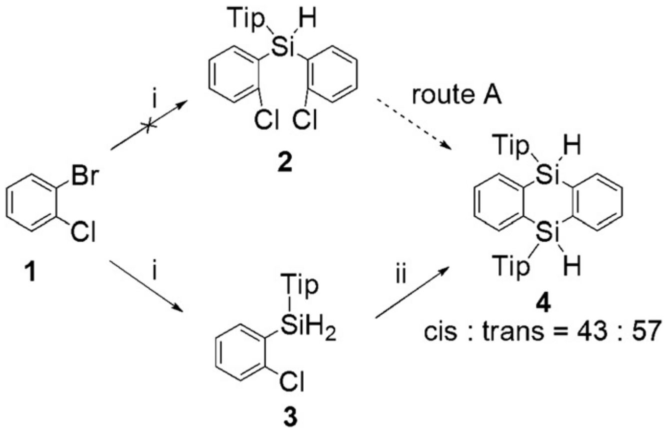
doi:10.1016/j.jorganchem.2010.10.010



Scheme 1. Preparation of DSA **4**. Reagents and conditions: (i) BuLi, THF, −95 °C, then $TipSiH_3$, 79%; (ii) Na, toluene, reflux, 21%. Tip = 2,4,6-triisopropylphenyl.

X-ray crystallographic analysis unambiguously confirmed the structure of *cis*-**4**.

Fig. 1 illustrates the crystal structure of *cis*-**4**. The central ring of DSA adopts a boat-like conformation in which each silicon atom has a pseudoaxial hydrogen and a pseudoequatorial Tip group. The phenyl groups of the Tip groups are oriented perpendicular to the C1—C6—C7—C12 plane. The tricyclic DSA framework has a folded structure with a dihedral angle (also known as a butterfly angle) of 157°, which is within the range of reported values for similar compounds (9,9,10,10-tetramethyl-9,10-dihydro-9,10-disilaanthracene [20] and 9,10-dihydro-9,10-disilaanthracene, 156° [21]; *cis*-9,10-di-*tert*-butyl-9,10-dihydro-9,10-disilaanthracene, 161° [3]).

In the ^{1}H NMR spectrum of *cis*-**4**, we observed three types of isopropyl groups over the observed temperature range (22—110 °C). The assignment of proton resonances was achieved by NOESY and decoupling experiments. The two ortho isopropyl substituents of the Tip group hinder the rotation about the Si—Tip bond and are discriminated on the NMR time scale. As shown in Fig. 1, one of the ortho isopropyl groups lies inside the folded DSA framework and therefore within its shielding region. As a result, the methyl proton signal of the internal isopropyl group resonated at a higher field (δ 0.66) to some degree. Furthermore, we observed an unexpected H—H long-range coupling (*ca.* 1 Hz) between Si—H and the methine proton of the external isopropyl group through five bonds, also indicating the restricted Si—Tip bond rotation. The through-space distances of these hydrogens in the solid phase were 2.202 Å and 2.223 Å for H1—H13 and H2—H50, respectively, which are shorter than the sum of van der Waals radii (2.4 Å). These findings indicated that the solution structure of *cis*-**4** is rigid and almost identical to the crystal structure.

In contrast, the ^{1}H NMR spectrum of *trans*-**4** indicated two types of isopropyl groups over the observed temperature range –90—22 °C), though the signals assigned to the ortho isopropyl groups were rather broad even at 22 °C, probably because of slow rotation about the Si—Tip bond. The above observation indicates the chemical equivalency of the two Tip groups as well as the two ortho isopropyl groups. It is not clear whether the reason for the chemical equivalency of the two Tip groups is rapid ring-flapping of the boat-like central ring or a rigid chair-like conformation of the central ring as observed in the *trans*-9,10-di-*tert*-butyl derivative [3].

X-ray crystallographic analysis of *trans*-**4** is expected to resolve the above mentioned uncertainty; however, our repeated attempts to refine the structure to a satisfactory resolution failed. Therefore, we carried out theoretical calculations on *cis*-**4** and *trans*-**4** using Gaussian 03 [22]. The geometries were fully optimized and characterized by frequency analysis at the B3LYP/6-31G(d,p) level. Fig. 2 shows the results. Including the zero-point vibrational energy (ZPVE) correction scaled by 0.9804 [23], *cis*-**4** is 1.13 kcal/mol more stable than *trans*-**4**. The calculated stretching vibrational frequencies of the Si—H bond in *cis*-**4** and *trans*-**4** are 2148 and 2132 cm^{-1} (scaled by 0.9613 [24]), respectively, which are in good agreement with the experimental data (2150 cm^{-1} for *cis*-**4** and 2124 cm^{-1} for *trans*-**4**). The optimized structure of *cis*-**4** is in good agreement with the X-ray structure, also validating the calculations. Unlike *cis*-**4**, the DSA ring of *trans*-**4** is almost planar (mean deviation 0.032 Å). The two silicon atoms are slightly displaced above and below the DSA plane, and the central ring adopts a flat chair conformation with the two Tip groups in pseudoequatorial positions. These results are consistent with the observations concerning the chemical equivalency of the Tip groups.

Since the obtained DSA derivative is expected to serve as a potential precursor for a novel silaaromatic 9,10-disilaanthracene, we performed flow thermolysis of *cis*-**4** in the presence of various trapping reagents. However, we were unable to isolate the products that would indicate the formation of the expected silaaromatics. Further investigations of the generation of 9,10-disilaanthracene are ongoing.

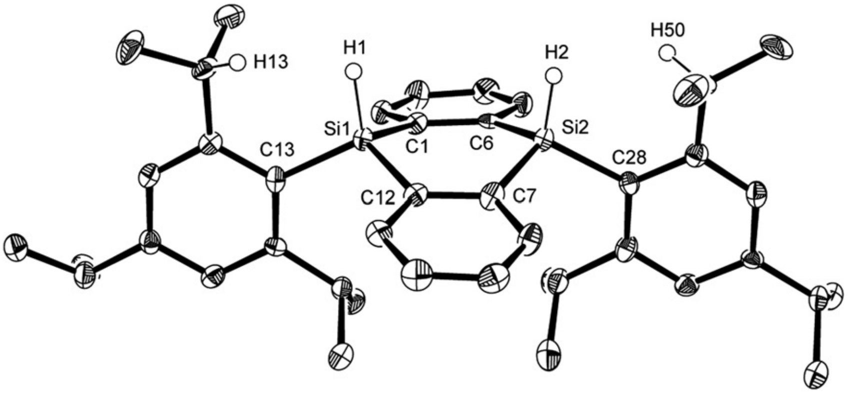
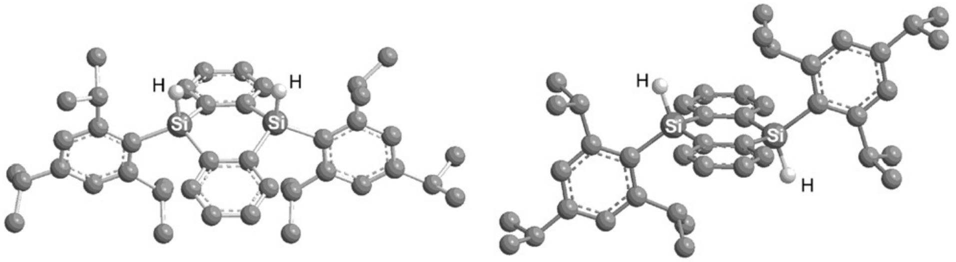


Fig. 1. X-ray crystal structure of *cis*-**4**. Hydrogen atoms except for H1, H2, H13, and H50 are omitted for clarity. Selected bond distances [Å] and angles [°]: Si1—H1 1.433, Si2—H2 1.355, Si1—C1 1.879(2), Si1—C12 1.882(2), Si1—C13 1.898(2), Si2—C6 1.872(2), Si2—C7 1.884(2), Si2—C28 1.885(2), C1—Si1—H1 107.1, C12—Si1—H1 105.3, C13—Si1—H1 111.1, C6—Si2—H2 105.9, C7—Si2—H2 105.8, C28—Si2—H2 112.0, C1—Si1—C12 109.95(11), C1—Si1—C13 110.29(11), C12—Si1—C13 112.87(9), C6—Si2—C7 110.45(10), C6—Si2—C28 112.66(11), C7—Si2—C28 109.76(10).



cis-**4**
(ΔE = 0 kcal/mol)

trans-**4**
(ΔE = 1.13 kcal/mol)

Fig. 2. Optimized structures and ZPVE-corrected relative energies of *cis*-**4** and *trans*-**4** computed at the B3LYP/6-31G(d,p) level. Hydrogen atoms, except those attached to the silicon atoms, are omitted for clarity.

3. Conclusion

We have synthesized the highly crowded 9,10-bis(2,4,6-triisopropylphenyl)-9,10-dihydro-9,10-disilaanthracene (**4**) via the intermolecular Wurtz-type coupling of 2-chlorophenylsilane derivative **3**. The structures of *cis*-**4** and *trans*-**4** were confirmed using X-ray crystallography, ^{1}H NMR spectroscopy, and theoretical calculations and found to have different features. The central ring of *cis*-**4** has a boat-like conformation, whereas that of *trans*-**4** is almost planar. The introduction of a Tip group provides steric crowding around the silicon atom; in particular, the rotation about the Si–Tip bond of the *cis* isomer is significantly restricted even in solution.

4. Experimental

4.1. General

Melting points were determined using a Yamato MP-21 melting point apparatus with open capillaries and are uncorrected. ^{1}H, ^{13}C, and ^{29}Si NMR spectra were measured in $CDCl_3$ solution using a Varian Mercury plus 400 spectrometer at 400, 100, and 79 MHz, respectively. All chemical shifts are reported as δ-values (ppm) relative to residual chloroform (δ_H 7.26), the central peak of $CDCl_3$ (δ_C 77.0), or tetramethylsilane (δ_{Si} 0.0). *J*-Values are given in Hz. GC–MS (EI) was measured by the direct combination of GC (Hewlett–Packard GC 5890 Series II with a 25 m × 0.25 mm methyl silicone capillary column) and a JEOL JMS-AX-500 spectrometer with a DA7000 data system. Infrared spectra were recorded using a JASCO IR Report-100 spectrometer. Elemental analyses were performed using a PerkinElmer 2400 Series II Analyzer.

4.2. Preparation of (2-chlorophenyl)(2,4,6-triisopropyl)phenylsilane (3)

Butyllithium in hexane (1.6 M, 63 mL, 100 mmol) was added to a solution of 2-chlorobromobenzene (**1**, 19.2 g, 100 mmol) in THF (100 mL) and diethyl ether (100 mL) at -95 °C under an argon atmosphere. After the above mixture was stirred for an additional 1 h, a solution of (2,4,6-triisopropylphenyl)silane (23.4 g, 100 mmol) in THF (100 mL) and diethyl ether (100 mL) was added dropwise into it and the resulting solution was stirred for 1 h. Then the reaction mixture was allowed to warm up to room temperature and quenched with a mixture of concentrated HCl (20 mL) and ethanol (50 mL). The mixture was diluted with diethyl ether and the organic layer was washed with brine, dried over $MgSO_4$, and evaporated. The residue was purified by column chromatography on silica gel (hexane) to give silane **3** (27.2 g, 79%) as a colorless solid, mp 78–79 °C. ^{1}H NMR ($CDCl_3$) δ 1.19 (d, $J = 7$ Hz, 12H), 1.29 (d, $J = 7$ Hz, 6H), 2.92 (sep, $J = 7$ Hz, 1H), 3.22 (sep, $J = 7$ Hz, 2H), 5.07 (s, 2H), 7.09 (s, 2H), 7.16 (m, 1H), 7.22 (m, 1H), 7.36 (m, 1H), 7.38 (m, 1H). ^{13}C NMR δ 23.8, 24.4, 34.3, 34.4, 121.1, 123.2, 126.1, 128.7, 131.3, 133.1, 137.6, 141.4, 151.5, 156.4. ^{29}Si NMR δ -54.6. IR (KBr) ν_{Si-H} 2164 cm^{-1}. HRMS *m*/*z* 344.1719 (M^{+}, calcd for $C_{21}H_{29}ClSi$ 344.1727). Anal. Calcd for $C_{21}H_{29}ClSi$: C, 73.11; H, 8.47. Found C, 73.11; H, 8.67.

4.3. Synthesis of 9,10-bis(2,4,6-triisopropylphenyl)-9,10-dihydro-9,10-disilaanthracene (4)

Silane **3** (17.9 g, 51.9 mmol) was added to a refluxing mixture of sodium (3.44 g, 150 mmol) in toluene (60 mL), and the mixture was refluxed overnight. Then, the mixture was cooled in an ice bath, quenched with ethanol, and diluted with diethyl ether. The organic layer was washed with water, dried over $MgSO_4$, and evaporated. Direct analysis of the reaction mixture by ^{1}H NMR spectroscopy showed that the ratio of the *cis* and *trans* isomers was 43:57. The residue was separated by column chromatography on silica gel (hexane). The former fraction contained the *cis* isomer (1.17 g, 1.90 mmol, 7%) and further elution gave a mixture of the *cis* and *trans* isomers (780 mg, 1.26 mmol, 5%). Finally, the *trans* isomer (1.44 g, 2.33 mmol, 9%) was obtained from the latter fraction. The total yield of compound **4** was 21%.

cis-**4**, mp 235–237 °C. ^{1}H NMR ($CDCl_3$) δ 0.66 (d, $J = 7$ Hz, 12H), 1.32 (d, $J = 7$ Hz, 12H), 1.37 (d, $J = 7$ Hz, 12H), 2.68 (sep, $J = 7$ Hz, 2H), 2.95 (sep, $J = 7$ Hz, 2H), 3.86 (d sep, $J = 1$ and 7 Hz, 2H), 5.84 (d, $J = 1$ Hz, 2H), 6.99 (s, 2H), 7.21 (s, 2H), 7.27 (m, 4H), 7.42 (m, 4H). ^{13}C NMR ($CDCl_3$) δ 23.8, 24.2, 24.9, 33.3, 34.2. 34.9, 120.7, 122.0, 125.2, 128.3, 135.1, 141.4, 151.4, 156.9, 157.5. ^{29}Si NMR ($CDCl_3$) δ -46.7. IR (KBr) ν_{Si-H} 2150 cm^{-1}. HRMS *m*/*z* 616.3935 (M^{+}, calcd for $C_{42}H_{56}Si_2$ 616.3921). Anal. Calcd for $C_{42}H_{56}Si_2$: C, 81.75; H, 9.15. Found C, 81.40; H, 9.26.

trans-**4**, mp 247–249 °C ^{1}H NMR ($CDCl_3$) δ 1.00 (d, $J = 7$ Hz, 24H), 1.26 (d, $J = 7$ Hz, 12H), 2.88 (sep, $J = 7$ Hz, 2H), 3.55 (sep, $J = 7$ Hz, 4H), 5.86 (s, 2H), 7.01 (s, 4H), 7.24 (m, 4H), 7.49 (m, 4H). ^{13}C NMR ($CDCl_3$) δ 23.8, 24.3, 34.3, 34.4, 121.3, 127.5, 128.0, 135.4, 142.2, 151.1, 156.5. ^{29}Si NMR ($CDCl_3$) δ -47.1. IR (KBr) ν_{Si-H} 2124 cm^{-1}. HRMS *m*/*z* 616.3911 (M^{+}, calcd for $C_{42}H_{56}Si_2$ 616.3921). Anal. Calcd for $C_{42}H_{56}Si_2$: C, 81.75; H, 9.15. Found C, 81.35; H, 9.18.

4.4. X-ray structure determination of cis-4

Crystal data were collected using a Rigaku RAXIS RAPID imaging plate area detector with graphite monochromated Cu-Kα radiation. An empirical absorption correction was applied, which resulted in transmission factors ranging from 0.742 to 0.901. The data were corrected for Lorentz and polarization effects. A correction for secondary extinction [25] was applied (coefficient = 93.535004). The structure was solved by direct methods [26] and expanded using Fourier techniques [27]. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model.

Crystal data for *cis*-**4**: $C_{42}H_{56}Si_2$, $M = 617.08$, crystal dimensions $0.10 \times 0.08 \times 0.06\ \mathrm{mm}^3$, monoclinic, $a = 16.8703(3)$, $b = 12.8553(2)$, $c = 18.3357(3)$ Å, $\beta = 109.3719(10)^\circ$, V 3751.40(12) Å^3, $T = 123.1$ K, space group *P21/c* (*#14*), $Z = 4$, $\rho_{\mathrm{calcd}} = 1.092\ \mathrm{gcm}^{-3}$, μ (Cu-Kα) $= 1.040\ \mathrm{mm}^{-1}$, 33663 reflections measured, 6777 independent reflections ($R_{int} = 0.058$). The final R_1 values were 0.0485 ($I > 2\sigma(I)$). The final $wR(F^2)$ values were 0.1386 (all data). The goodness of fit on F^2 was 0.958.

4.5. Theoretical calculations

The structures of *cis*-**4** and *trans*-**4** were fully optimized with the 6-31G(d,p) basis set using the three-parameter functional of Becke (B3) [28], augmented by the correlation functional of Lee, Yang, and Parr (LYP) [29]. The B3LYP vibrational analyses were performed for each optimized structure to characterize the stationary points as minima and to calculate the ZPVE. All calculations were performed with Gaussian 03 [22].

Appendix A. Supplementary material

CCDC 787292 contains the supplementary crystallographic data for the structural analysis of *cis*-**4**. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via http://www.ccdc.cam.ac.uk/data_request/cif.

Appendix A. Supplementary material

Supplementary material associated with this article can be found in the online version, at doi:10.1016/j.jorganchem.2010.10.010.

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