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Short Communication

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Unexpected photochemical reactions of 2-aryl-2-ethynyltrisilane derivative

Abstract: The synthesis of cyclic disilene **10** was attempted by photolysis of 2-alkynyltrisilane **9**. As a result, silylsilylene **14** was generated, along with the elimination of the alkynylsilane moiety, in the photolysis of 2-alkynyltrisilane **9**, similar to the photolysis of alkynyldisilane. In addition, the product **14** trapped by triethylsilane underwent further photolysis to produce hydrosilylene **16**.

Keywords: photolysis; silylene; trisilane.

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Disilenes are the silicon analogue of carbon-carbon double-bond compounds, and the first isolation of tetramesityldisilene was reported by West et al. (1981). Since then, various types of disilenes have been synthesized, and their characters have been revealed in detail (for reviews, see Okazaki and West, 1996; Kira and Iwamoto, 2006; Lee and Sekiguchi, 2010). There remains an ever-increasing interest in the challenging study of the interaction of organic π-conjugated systems and the silicon-silicon double-bond units (Bejan and Scheschkewitz, 2007; Fukazawa et al., 2007; Kinjo et al., 2007; Sasamori et al., 2008; Iwamoto et al., 2009). In that field, we have succeeded in the synthesis and isolation of the first example of a silicon analogue of (*E*)-endiyne, (*E*)-1,2-dialkynyldisilenes **1**, and revealed that conjugative interaction exists between the Si=Si and C≡C units (Sato et al., 2010).

Those compounds were synthesized by the reduction of dihalosilanes **2** (Scheme 1). In the reactions, however, only (*E*)-isomer was generated, and the formation of (*Z*)-isomers could not be detected. (*Z*)-1,2-Dialkynyldisilenes are expected to show through-space interaction between the two alkynyl groups, resulting in electronic properties different from those of (*E*)-isomers. To synthesize the (*Z*)-1,2-dialkynyldisilenes selectively, we attempted the reduction of compound **3**, the two ethynylsilane moieties of which were linked by tether (for cyclic disilenes, see Sakurai et al., 2002; Tanaka et al., 2006; Abe et al., 2010), but the desired disilene **4** could not be synthesized (Scheme 2) (Mizuhata et al., 2015). The reaction afforded the cyclic 1,2-dihydrodisilane **5** in low yield, where the chlorine atoms on the silicon atoms were fully replaced by hydrogen atoms derived from solvent and/or benzylic protons of **3**. This result suggests that hydrogen abstraction occurred by radical species generated in the stepwise reduction of chlorosilane moieties.

In this particular case, the reduction of the corresponding dichlorosilane, the most general method for the generation of a silylene (or a disilene), was found to be not appropriate. We then decided to adopt another synthetic method for a disilene or a silylene without using a reducing agent, i.e., the photolysis of the corresponding trisilane derivative. By using this method, many disilenes, including the first disilene, tetramesityldisilene, have been successfully synthesized (for reviews, see Okazaki and West, 1996; Kira and Iwamoto, 2006; Lee and Sekiguchi, 2010). The photolysis of linear-trisilanes giving disilenes proceeds via the initial formation of a silylene, followed by its dimerization (Ishikawa and Kumada, 1981). In addition, Apeloig et al. (1994) generated alkynylsilylenes **6**–**8** in a low-temperature glass matrix using photolysis of linear-trisilane bearing an alkynyl group, and its formation was suggested by the trapping reaction with triethylsilane (Et_3SiH) (Scheme 3). Based on these results, we designed and synthesized compound **9**, the 2-alkynyltrisilane moieties of which were linked, and attempted the photolysis of **9** to synthesize cyclic disilene **10** (Scheme 4). In this paper, we discuss the unexpected elimination of alkynylsilane rather than disilane in the photoreaction of **9**.

The synthesis of the precursor, compound **9**, was performed. 1,2-Bis(ethynyldimethylsilyl)benzene **11** was prepared by a reaction of 1,2-bis(bromodimethylsilyl)benzene

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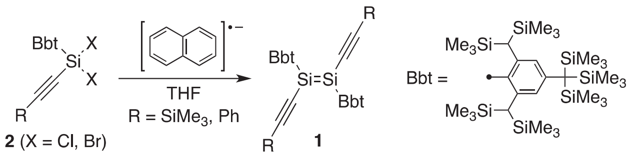
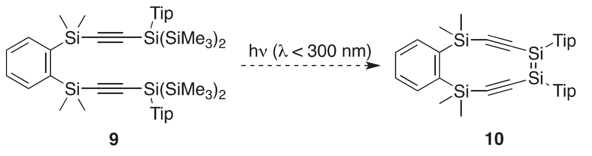
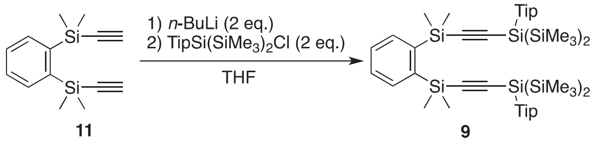


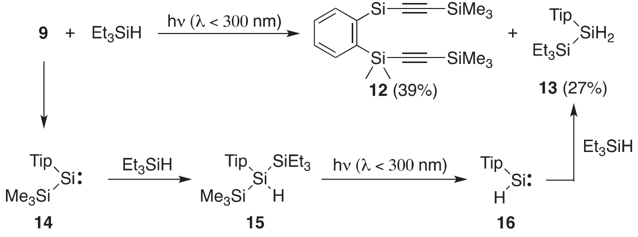
Scheme 1: Syntheses of (*E*)-1,2-dialkynyldisilenes **1**.



Scheme 4: Strategy for the synthesis of **10**.



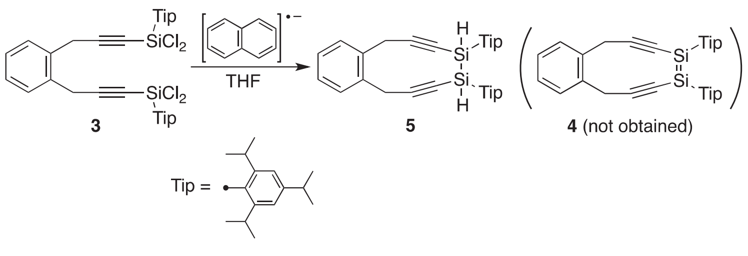
Scheme 5: Synthesis of **9**.



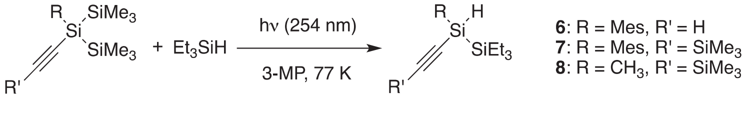
Scheme 6: Photolysis of **9** in the presence of triethylsilane.

(Cavelier-Frontin et al., 1991) with ethynylmagnesiumbromide in THF (11% yield) as a pale yellow viscous liquid. The synthesis of **9** was achieved by the reaction of the dilithio compound generated by the treatment of **11** with *n*-butyllithium followed by treatment with $TipSi(SiMe_3)_2Cl$ (Tip=2,4,6-triisopropylphenyl) (Archibald et al., 1992) in 23% yield as a viscous liquid (Scheme 5). Compounds **9** and **11** were characterized by NMR spectroscopy together with elemental analysis.

To check the generation of silylene from **9** via the elimination of disilane described in Scheme 3, we performed the photolysis of compound **9** in the presence of Et_3SiH, which is commonly used as a trapping reagent for silylenes. A *n*-hexane solution of precursor **9** and Et_3SiH was placed in a quartz sealed tube and irradiated with a low-pressure mercury lamp at -78°C for 24 h (Scheme 6). As a result, compound **12** bearing trimethylsilylethynyl moieties and triethylsilyldihydrosilane **13** were obtained at 39% and 27%, respectively. Both compounds were characterized by NMR spectroscopy together with elemental analyses. The formation of compound **13** suggests the generation of silylsilylene **14** via the elimination of alkynylsilane (Scheme 6). That is, the generated silylene **14** was trapped by Et_3SiH to afford trisilane **15**, and it was photolyzed again to give hydrosilylene **16** in the next step. Silylene **16** was trapped by Et_3SiH to afford dihydrosilane **13**. Whereas hydrosilylenes are important intermediates in view of bearing a primitive substituent, hydrogen, there are few examples of the generation (Hong et al., 1994; Sasamori et al., 2007; Agou et al., 2012). As far as we know, only one example was reported for the generation of hydrosilylene by the photolysis of oligosilane (Hong et al., 1994). In that report, photolysis of $MeSi(SiMe_3)_3$ in the



Scheme 2: Attempted synthesis of cyclic 1,2-dialkynyldislene **4**.



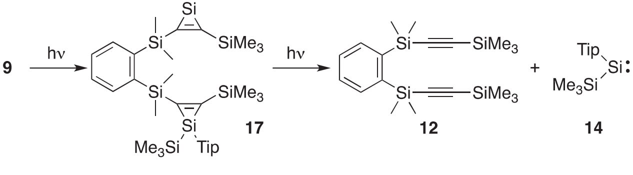
Scheme 3: Generation of alkynylsilylenes **6**–**8**.

presence of Et_3SiH afforded $Me(H)Si(SiMe_3)(SiEt_3)$ mainly (36%), together with 1,1-dihydrodisilane, $H_2(Me)SiSiEt_3$, in 1.5% yield, which suggests the generation of hydromethylsilylene from the initial product, $Me(H)Si(SiMe_3)(SiEt_3)$. In the example using compound **9**, monohydrosilane **15** was not observed, and the yield of dihydrosilane **13** was much higher than that in the previous report. This result suggests that the Tip-substitution is effective for the generation of hydrosilylene. Actually, photoreaction of $TipSi(SiMe_3)_2H$ (Archibald et al., 1992) in the presence of Et_3SiH proceeded cleanly and afforded dihydrosilane **13** in 39% yield. When compound **9** was irradiated under the same conditions in the absence of Et_3SiH, only a complicated mixture was obtained. Its ^{29}Si NMR spectrum indicated the existence of (*Z*)-$Tip(Me_3Si)Si{=}Si(SiMe_3)Tip$ (at 97.8 ppm) (Archibald et al., 1992), but no other signal was observed in the lower field region other than this signal.

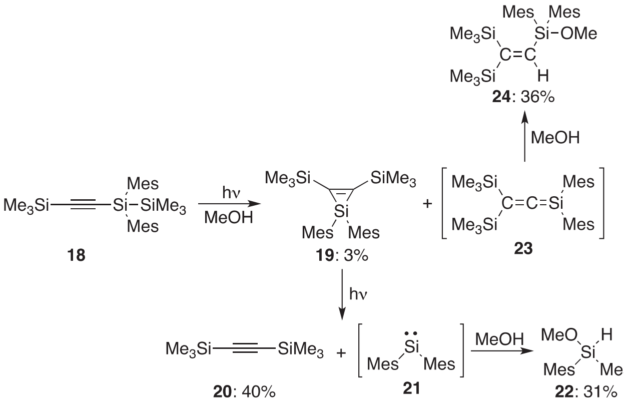
In the photolysis of compound **9**, alkynylsilane, not disilane, was eliminated from the central silicon atom of trisilane moieties. This result marked a sharp contrast with the results reported by Apeloig et al. (1994) described above. Neither the trapped compound of alkynylsilylene nor the compound bearing triethylsilylacetylene terminus were observed in these experiments.

A possible mechanism for the generation of **12** is proposed in Scheme 7. Compound **17** containing silacyclopropene moieties is generated, and then the photolysis of compound **17** may give silylene **14** and compound **12**. A similar reaction mechanism is reported in the photoreaction of alkynyldisilane **18** in the presence of methanol by Ishikawa, Kumada, and their co-workers (Scheme 8) (Ishikawa et al., 1980; Ishikawa and Kumada, 1981). Silacyclopropene **19** could be isolated, and the following photolysis gave bis(trimethylsilyl)acetylene **20** and dimesitylsilylene **21**, the generation of which was suggested by the isolation of **22**. In this reaction, generation of 1-silaallene **23** was suggested as a side reaction by the formation of **24**. It is likely due to this type of side-reaction that the yields of **12** and **13** were low, similar to the photolysis of **9**.

In summary, silylsilylene **14** was observed to generate in the photolysis of 2-alkynyltrisilane **9** via the elimination of alkynylsilane rather than disilane. Judging from the results obtained in this work and those shown in Scheme 3, the elimination of alkynylsilane from 2-alkynyltrisilane is competitive with that of disilane, and the selectivity could change depending on the reaction conditions or small differences in the substituents. For the synthesis of dialkynyldisilene using photolysis, protection of the alkynyl moieties is considered to be necessary. In addition, hydrosilylene **16** was observed to generate via compound **15**. The yield of the trapped compound, dihydrosilane **13**, was still low, but much higher than the previous result, indicating this methodology possibly presents an alternative way to generate a hydrosilylene.



Scheme 7: Possible mechanism for the generation of **12** and **14**.



Scheme 8: Photolysis of alkynyldisilane **18** in the presence of methanol.

Experimental section

General

All experiments were performed under an argon atmosphere unless otherwise noted. All solvents were purified by standard methods and/or The Ultimate Solvent System (Pure Process Technology, Nashua, New Hampshire, USA) (Pangborn et al., 1996) prior to use. ^1H-NMR (300 MHz), ^{13}C-NMR (75 MHz), and ^{29}Si-NMR (59 MHz) spectra were measured in C_6D_6 (Cambridge Isotope Laboratories, Inc., Tewksbury, Massachusetts, USA) with a JNM AL-300 spectrometer (JEOL resonance Inc., Tokyo, Japan). A signal due to C_6D_5H (7.15 ppm) was used as an internal standard in ^1H-NMR, and a signal due to C_6D_6 (128 ppm) was used in ^{13}C-NMR. Multiplicity of signals in ^{13}C-NMR spectra was determined by DEPT technique. ^{29}Si-NMR (59 MHz) spectra were measured in C_6D_6 with a JEOL JNM AL-300 spectrometer using the signal for $SiMe_4$ (0 ppm) in C_6D_6 as an external standard. Gel permeation liquid chromatography (GPLC) was performed on an LC-908, LC-918, or LC-908-C60 (Japan Analytical Industry Co., Ltd., Tokyo, Japan) equipped with JAIGEL 1H and 2H columns (eluent: chloroform). All melting points were determined on a micro-melting-point apparatus (Yanaco, Kyoto, Japan). Elemental analyses were carried out at the Microanalytical Laboratory of the Institute of Chemical Research, Kyoto University. Preparative thin-layer chromatography (PTLC) was performed with Kieselgel 60 PF254 (Merck KGaA, Darmstadt, Germany, Art. No. 7747).

Synthesis of 1,2-bis(ethynyldimethylsilyl)benzene (11)

A solution of 1,2-bis(hydrodimethylsilyl)benzene (12.6 g, 64.8 mmol, Tokyo Chemical Industry Co., Ltd., Tokyo, Japan) in CCl_4 (50 mL) was added to a solution of bromine (7.6 mL, 150 mmol, NACALAI TESQUE, Inc., Kyoto, Japan) in CCl_4 (50 mL) and cooled by ice-bath while maintaining an argon atmosphere. Argon gas was bubbled through the mixture for a further 30 min before removing the solvent in vacuum. The residue was used for the next reaction without further purification. To that compound was added ethynylmagnesiumbromide solution (0.5 M THF solution, 370 mL, 185 mmol, Sigma-Aldrich Co. LLC, St. Louis, Missouri, USA) at room temperature, and the mixture was stirred at room temperature for 3 days. The reaction mixture was treated with aqueous sodium hydrogen carbonate (Wako Pure Chemical Industries, Ltd., Osaka, Japan), and the organic layer was extracted with *n*-hexane. After the removal of volatile substances, the residue was subjected to silica gel chromatography (eluent: *n*-hexane) to afford **11** (1.75 g, 7.22 mmol, 11%). **11**: pale yellow viscous liquid; ^{1}H-NMR (300 MHz, C_6D_6, rt, δ in ppm) 0.57 (s, 12H), 2.15 (s, 2H), 7.17–7.20 (m, 2H), 7.95–8.00 (m, 2H); ^{13}C-NMR (75 MHz, C_6D_6, rt, δ in ppm) 2.1 (CH_3), 90.4 (C), 96.6 (C), 128.9 (CH), 136.4 (CH), 142.3 (C); ^{29}Si-NMR (59 MHz, C_6D_6, rt, δ in ppm) -59.2 ($SiMe_2$); HRMS (EI) m/z calcd for $C_{14}H_{18}Si_2$ ([M]$^+$): 242.0947, found: 242.0946; Anal. calcd for $C_{14}H_{18}Si_2$: C, 69.35; H, 7.48%, found: C, 69.34; H, 7.45%.

Synthesis of 1,2-bis(((1,1,1,3,3,3-hexamethyl-2-(2,4,6-triisopropylphenyl)trisilan-2-yl)ethynyl)dimethylsilyl)benzene (9)

To a THF solution (10 mL) of **11** (328 mg, 1.35 mmol) was added *n*-butyllithium (1.60 M in *n*-hexane, 0.850 mL, 1.36 mmol, KANTO chemical co., Inc., Tokyo, Japan) at -78°C, and the mixture was stirred at -78°C for 30 min. The resulting solution was added to a THF solution (10 mL) of TipSi($SiMe_3$)$_2$Cl (1.12 g, 2.71 mmol) at room temperature, and the mixture was stirred at room temperature for 12 h. The solvents were removed under reduced pressure. *n*-Hexane was added to the residue, and the resulting suspension was filtered through Celite® to remove inorganic salts. The reaction mixture was purified by GPLC (eluent: $CHCl_3$) to afford **9** (308 mg, 0.309 mmol, 23%). **9**: viscous liquid; ^{1}H-NMR (300 MHz, C_6D_6, rt, δ in ppm) 0.37 (s, 36H), 0.69 (s, 12H), 1.18 (d, *J*=7.0 Hz, 12H), 1.34 (d, *J*=6.5 Hz, 24H), 2.76 (sept, *J*=7.0 Hz, 2H), 3.72 (sept, *J*=6.5 Hz, 4H), 7.14 (s, 4H), 7.26–7.32 (m, 2H), 8.26–8.32 (m, 2H); ^{13}C-NMR (75 MHz, C_6D_6, rt, δ in ppm) 0.5 (CH_3), 2.5 (CH_3), 24.0 (CH_3), 26.1 (CH_3), 34.5 (CH), 35.2 (CH), 116.6 (C), 118.9 (C), 121.9 (CH), 126.3 (C), 128.8 (CH), 137.1 (CH), 142.6 (C), 149.9 (C), 156.5 (C); ^{29}Si-NMR (59 MHz, C_6D_6, rt, δ in ppm) -12.1, -20.6, -68.4; Anal. calcd for $C_{56}H_{98}Si_8$: C, 67.53; H, 9.92%, found: C, 67.48; H, 9.74%.

Photolysis of 9

A solution of **9** (333 mg, 0.334 mmol) and triethylsilane (3.0 mL, 19 mmol, Sigma-Aldrich Co. LLC, St. Louis, Missouri, USA) in *n*-hexane (4 mL) contained in a quartz sealed tube was exposed to a low-pressure mercury lamp (SUV110PL-10 with UVB-110, SEN LIGHTS CORPORATION, Osaka, Japan) while immersed in a -78°C bath for 24 h. All of the volatile materials were removed under reduced pressure. The reaction mixture was separated by GPLC (eluent: toluene) to afford oligomeric products and the mixture of **12** and **13**. The following PTLC (eluent: *n*-hexane) gave **12** (51 mg, 0.13 mmol, 39%) and **13** (62 mg, 0.18 mmol, 27%). **12**: viscous liquid; ^{1}H-NMR (300 MHz, C_6D_6, rt, δ in ppm) 0.16 (s, 18H), 0.66 (s, 12H), 7.17–7.20 (m, 2H), 7.99–8.02 (m, 2H); ^{13}C-NMR (75 MHz, C_6D_6, rt, δ in ppm) 0.0 (CH_3), 2.5 (CH_3), 114.7 (C), 116.8 (C), 128.8 (CH), 136.3 (CH), 142.7 (C); ^{29}Si-NMR (59 MHz, C_6D_6, rt, δ in ppm) -18.9, -21.4; HRMS (FAB) m/z calcd for $C_{20}H_{34}Si_4$ ([M]$^+$): 386.1738, found: 386.1737; Anal. calcd for $C_{20}H_{34}Si_4$: C, 62.10; H, 8.86%, found: C, 61.97; H, 8.77%. **13**: viscous liquid; ^{1}H-NMR (300 MHz, C_6D_6, rt, δ in ppm) 0.72 (q, *J*=8.4 Hz, 6H), 0.98 (t, *J*=8.4 Hz, 9H), 1.20 (d, *J*=6.9 Hz, 6H), 1.33 (d, *J*=6.7 Hz, 12H), 2.77 (sept, *J*=6.9 Hz, 1H), 3.33 (sept, *J*=6.7 Hz, 2H), 4.52 (s, 2H), 7.12 (s, 2H); ^{13}C-NMR (75 MHz, C_6D_6, rt, δ in ppm) 4.6 (CH_2), 8.2 (CH_3), 24.1 (CH_3), 24.5 (CH_3), 34.7 (CH), 35.3 (CH), 121.0 (CH), 124.7 (C), 150.4 (C), 155.7 (C); ^{29}Si-NMR (119 MHz, C_6D_6, rt, δ in ppm) -5.7, -82.8; HRMS (DART) m/z calcd for $C_{21}H_{34}Si_4$ ([M+H]$^+$): 349.2741, found: 349.2725; Anal. calcd for $C_{20}H_{34}Si_4$: C, 72.33; H, 11.56%, found: C, 72.08; H, 11.56%.

Photolysis of TipSi($SiMe_3$)$_2$H

A solution of TipSi($SiMe_3$)$_2$H (239 mg, 0.632 mmol) and triethylsilane (2.0 mL, 12 mmol) in *n*-hexane (6.0 mL) contained in a quartz sealed tube was exposed to a low-pressure mercury lamp while immersed in a -78°C bath for 24 h. All of the volatile materials were removed under reduced pressure. The following PTLC (eluent: *n*-hexane) gave **13** (86 mg, 0.25 mmol, 39%).

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References

Abe, T.; Iwamoto, T.; Kira, M. A stable 1,2-disilacyclohexene and its 14-electron palladium(0) complex. *J. Am. Chem. Soc.* **2010**, *132*, 5008–5009.

Agou, T.; Sugiyama, Y.; Sakai, H.; Furukawa, Y.; Takagi, N.; Guo, J.-D.; Nagase, S.; Hashizume, D.; Tokitoh, N. Synthesis of kinetically stabilized 1,2-dihydrodisilenes. *J. Am. Chem. Soc.* **2012**, *134*, 4120–4123.

Apeloig, Y.; Karni, M.; West, R.; Welsch, K. Electronic spectra of ethynyl- and vinylsilylenes: experiment and theory. *J. Am. Chem. Soc.* **1994**, *1116*, *9719–9729*.

Archibald, R. S.; van den Winkel, Y.; Millevolte, A. J.; Desper, J. M.; West, R. New 2,4,6-triisopropylphenyl-substituted disilenes. *Organometallics* **1992**, *11*, 3276–3281.

Bejan, I.; Scheschkewitz, D. Two Si–Si double bonds connected by a phenylene bridge. *Angew. Chem., Int. Ed.* **2007**, *46*, 5783–5786.

Cavelier-Frontin, F.; Jacquier, R.; Paladino, J.; Verducci, J. N-bis-silylation of α-amino acids: “benzostabases” as amino protecting group. *Tetrahedron* **1991**, *47*, 9807–9822.

Fukazawa, A.; Li, Y.; Yamaguchi, S.; Tsuji, H.; Tamao, K. Coplanar oligo(*p*-phenylenedisilenylene)s based on the octaethyl-substituteds-hydrindacenyl groups. *J. Am. Chem. Soc.* **2007**, *129*, 14164–14165.

Hong, S. K.; Boo, B, H.; Rhee, H. J.; Kang, S. K.; Kim, J. R.; Kwak, Y.-W. Photochemistry of tris (trimethylsilyl)methylsilane. *Bull. Korean Chem. Soc.* **1994**, *15*, 68–74.

Ishikawa, M.; Kumada, M. Photochemistry of organopolysilanes. *Adv. Organomet. Chem.* **1981**, *19*, 51–95.

Ishikawa, M.; Nishimura, K.; Sugisawa, H.; Kumada, M. Photolysis of organopolysilanes. The synthesis and reactions of stable silacyclopropenes. *J. Organomet. Chem.* **1980**, *194*, 147–158.

Iwamoto, T.; Kobayashi, M.; Uchiyama, K.; Sasaki, S.; Nagendran, S.; Isobe, H.; Kira, M. Anthryl-substituted trialkyldisilene showing distinct intramolecular charge-transfer transition. *J. Am. Chem. Soc.* **2009**, *131*, 3156–3157.

Kinjo, R.; Ichinohe, A.; Sekiguchi, A.; Takagi, N.; Sumitomo, M.; Nagase, S. Reactivity of a disilyne $RSi{\equiv}SiR$ ($R{=}Si_{i}Pr[CH(SiMe_3)_2]_2$) toward π-bonds: stereospecific addition and a new route to an isolable 1,2-disilabenzene. *J. Am. Chem. Soc.* **2007**, *129*, 7766–7767.

Kira, M.; Iwamoto, T. Progress in the chemistry of stable disilenes. *Adv. Organomet. Chem.* **2006**, *54*, 73–148.

Lee, V. Ya.; Sekiguchi, A. Heavy analogs of alkenes, 1,3-dienes, allenes and alkynes: Multiply bonded derivatives of Si, Ge, Sn, Pb. In *Organometallic Compounds of Low Coordinate Si, Ge, Sn and Pb: From Phantom Species to Stable Compounds*. Wily: Chichester, UK, 2010; Chapter 5.

Mizuhata, Y.; Egawa, Y.; Sasamori, T.; Tokitoh, N. Synthesis of 1,2-dialkynyldisilanes incorporated in 10-membered-ring system. *Heterocycles* **2015**, *90*, 1111–1123.

Okazaki, R.; West, R. Chemistry of stable disilenes. *Adv. Organomet. Chem.* **1996**, *39*, 231–273.

Pangborn, A. B.; Giardello, M. A.; Grubbs, R. H.; Rosen, R. K.; Timmers, F. J. Safe and convenient procedure for solvent purification. *Organometallics* **1996**, *15*, 1518–1520.

Sakurai, H.; Iimura, T.; Matsumoto, S.; Sanji, T. Intramolecular dimerization of silylenes leading to novel cyclic disilenes. *Chem. Lett.* **2002**, *31*, 22–23.

Sasamori, T.; Ozaki, S.; Tokitoh, N. Unexpected reaction of an overcrowded 9,10-dihydroanthrylchlorosilane leading to the formation of a dibenzo-7-silanorbornadiene derivative. *Chem. Lett.* **2007**, *36*, 588–589.

Sasamori, T.; Yuasa, A.; Hosoi, Y.; Furukawa, Y.; Tokitoh, N. 1,2-Bis(ferrocenyl)disilene: A multistep redox system with an Si=Si double bond. *Organometallics* **2008**, *27*, 3325–3327.

Sato, T.; Mizuhata, Y.; Tokitoh, N. 1,2-dialkynyldisilenes: silicon analogues of (E)-enediyne. *Chem. Commun.* **2010**, *46*, 4402–4404.

Tanaka, R.; Iwamoto, T.; Kira, M. Fused tricyclic disilenes with highly strained Si–Si double bonds: addition of a Si–Si single bond to a Si–Si double bond. *Angew. Chem. Int. Ed.* **2006**, *45*, 6371–6373.

West, R.; Fink, M. J.; Michl, J. Tetramesityldisilene, a stable compound containing a silicon-silicon double bond. *Science* **1981**, *214*, 1343–1344.