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NOVEL REACTIONS OF STERIC ENCUMBERED 1,4-DILITHIO-1,3-BUTADIENE WITH GROUP 14 ELECTROPHILES: FORMATION AND STRUCTURE OF STABLE DIHYDROXYGERMOLE

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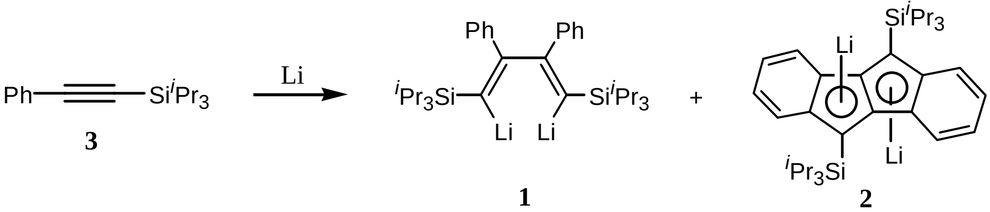
Abstract – Reactions of 1,4-dilithio-1,3-butadiene **1** having bulky silyl ligands at the 1,4-positions with group 14 electrophiles were examined. Reactions of 1,4-dilithio-1,3-butadiene **1** with tetraethoxygermane gave diethoxygermole **5**, which was hydrolyzed to give stable dihydroxygermole **16**. The X-ray diffraction analysis of dihydroxygermole **16** revealed intermolecular hydrogen bonds. The reaction of **1** with stannous chloride was also examined.

INTRODUCTION

1,4-Dilithio-1,3-butadiene, which can be prepared from phenylacetylene with lithium[1] and 1,4-diiodo-1,3-butadiene with alkyllithium reagents,[2] is often employed as the starting compound for the synthesis of metal-containing carbocyclic π-systems, for example, group 14 metalloles.[3] In the course of our studies on the synthesis of reactive species with a 1,3-butadiene skeleton,[4] we became interested in the preparation of 1,4-dilithio-1,3-butadienes having bulky ligands at the 1,4-positions. Very recently, we reported on the novel 1,4-dilithio-1,3-butadiene **1** that has bulky silyl substituents in the 1,4-positions (Scheme 1).[5] On the other hand, the germinal 1,1-diols of the carbon versions are so unstable that the equilibrium is completely shifted from the 1,1-diols to the corresponding ketones and water. In contrast, 1,1-diols of silicon analogs are stable enough to be isolated and many examples have already appeared.[6] With regard to the germanium analogs, although some recent examples of stable 1,1-dihydroxygermane have been already reported,[7] the chemistry of 1,1-dihydroxygermane remains largely unexplored. In efforts to functionalize 1,4-dilithio-1,3-butadienes, we examined 1,4-dilithio-1,3-butadiene **1** with some group 14 electrophiles. Here we report on the formation and structure of a stable dihydroxygermole, a novel type of 1,1-dihydroxygermanes.

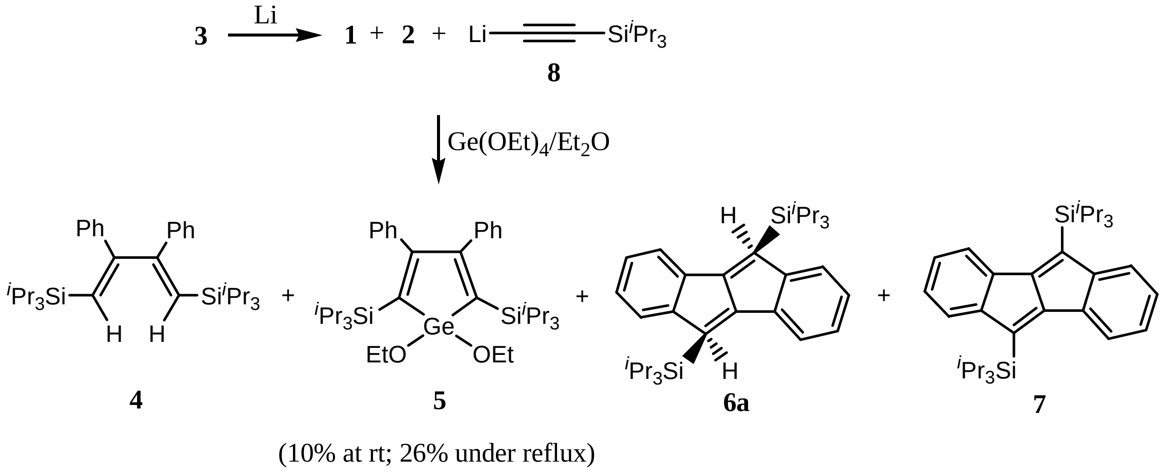
RESULTS AND DISCUSSION

(a) Reaction of 1,4-Dilithio-1,3-butadiene 1 with Chlorosilanes. Hexachlorodisilane was considered be a useful reagent for the preparation of 1,4-dimetalla-cyclohexa-1,3-dienes, which could be potential precursors of six-membered reactive species having two heavier group 14 atoms. 1,4-Dilithio-1,3-butadiene **1**, prepared in situ by the reduction of phenylsilylacetylene **3** (Scheme 1),[8] was treated with hexachlorodisilane in toluene at low temperature. However, the resulting mixture was complex; the ^{1}H NMR spectrum showed a broad signal at 1.0-1.6 ppm, and only 1,3-butadiene **4** (*vide infra*) was identified. Although the reaction of **1** with tetrachlorosilane was also examined, the reaction was so complex that no identifiable products were obtained. We concluded that chlorosilanes were very reactive toward **1** and/or the expected products of dichlorosilane derivatives were unstable under the reaction conditions used.

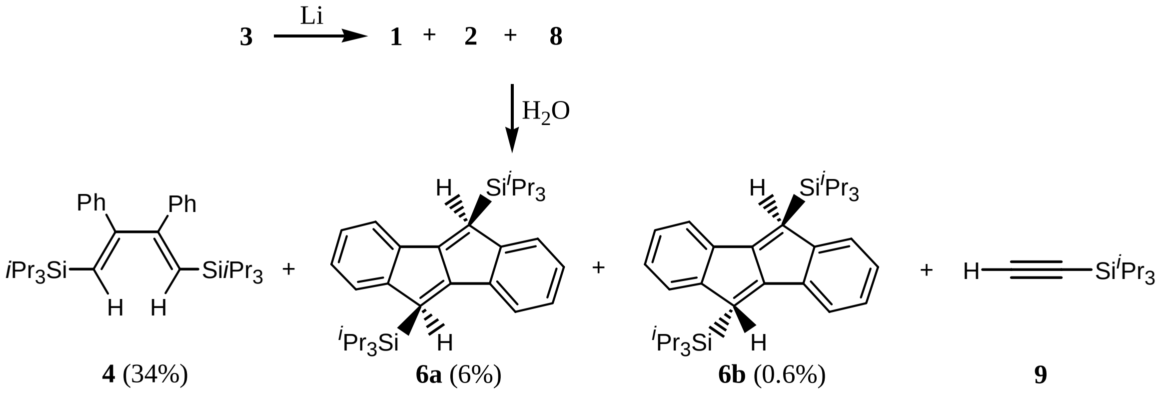


Scheme 1

(b) Reaction of 1,4-Dilithio-1,3-butadiene 1 with Tetraethoxygermane. Since the reactions of **1** with chlorosilanes were complex, we next examined the reaction of **1** with commercially available tetraethoxygermane. Tetraethoxygermane is thought to be less reactive than tetrachlorogermane, because ethoxide is a poor leaving group compared with chloride. After treatment of **1** with excess tetraethoxygermane in ether at rt, diethoxygermole **5** was obtained in 10% yield, together with a mixture containing 1,3-butadiene **4**, *cis*-dihydrodibenzopentalene **6a** and dibenzopentalene **7** (Scheme 2). The yield of **5** was improved to 26%, by heating the reaction mixture under reflux after the treatment of **1** with tetraethoxygermane in ether at rt (Scheme 2). In these reactions, no compounds derived from a byproduct, ethynyllithium **8** (*vide infra*), were obtained. The structure of **5** was confirmed by NMR spectroscopy and elemental analysis. Compounds, **4** and **6**, were alternatively prepared by treatment of the reaction mixture formed after the reduction of phenylsilylacetylene **3** with H_2O (Scheme 3).[9] The stereochemistry of **4** was determined to be the *Z,Z*-form, by comparing its ^{1}H NMR spectrum with that of analogous to that of the corresponding 1,4-diiodo derivative,[5] while that of **6** was finally confirmed by X-ray diffraction analysis. Although the ^{1}H NMR spectrum of the crude product showed the formation of triisopropylsilylacetylene (**9**), derived from the corresponding ethynyllithium **8**, it could not be isolated because of its relatively low boiling point[11] and the yield of **9** could not be determined. The formation of **5**, upon using an excess of tetraethoxygermane suggests that ring closure of the intermediate formed from **1** and an equivalent of tetraethoxygermane should be faster than further addition of tetraethoxygermane to the intermediate.

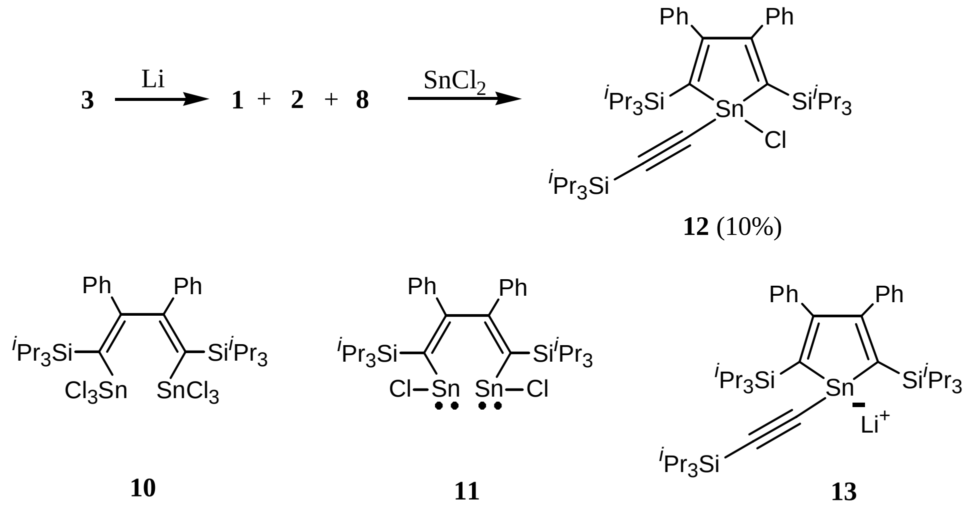


Scheme 2

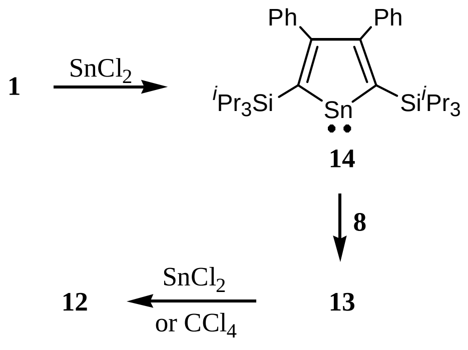


Scheme 3

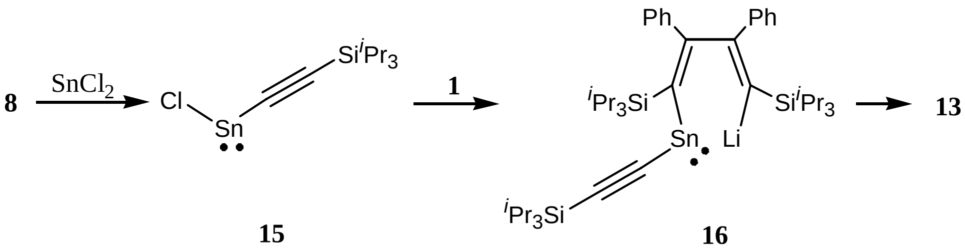
(c) Reaction of 1,4-Dilithio-1,3-butadiene 1 with Stannous Chloride. To prepare the 1,4-trichlorostannyl derivative **10**, 1,4-dilithio-1,3-butadiene **1** was treated with stannous chloride at low temperature and the reaction mixture then treated with carbon tetrachloride at –50 ºC. The reason for using the carbon tetrachloride was because the expected chlorostannylene intermediate **11** would react with carbon tetrachloride to give **10**.[12] However, 1-chloro-1-silylethynylstannole **12** was obtained in 10% yield (Scheme 4). In order to elucidate the mechanism for the formation of **12**, the reaction was monitored using ^{119}Sn NMR spectroscopy. After treatment of **1**, generated from **1** in a mixture with **2** and **8**, with stannous chloride, the ^{119}Sn NMR spectrum of the resulting mixture in C_6D_6 revealed two signals at –32 ppm and –110 ppm. The former signal was assigned to **12**, suggesting that compound **12** was already formed from the reaction of **1** and stannous chloride. The latter signal was located in a region similar to those of the 1-aryl-substituted tetraphenylstannole anions,[13] and was therefore assigned to the stannole anion **13**. A possible mechanism for the formation of **12** is proposed in Scheme 5. Reaction of **1** with stannous chloride afforded the stannylidene intermediate **14**, which further reacted with ethynyllithium **8** to afford the stannole anion **13**. Since the final product **13** was already formed after treatment of **1**, generated from **1** in a mixture with **2** and **8**, with stannous chloride, as evidenced by ^{119}Sn NMR analysis, it was proposed that the stannole anion **13** reacted with the remaining stannous chloride to afford **12**. However, since the stannole anion **13** is also observed in the reaction mixture, the reaction pathway in which the stannole anion **12** reacts with carbon tetrachloride to give **12** would be also involved. Alternatively, ethynyllithium **8** would react with stannous chloride to afford chlorostannylene intermediate **15**, which further reacted with **1** to give the stannole anion **13** *via* another stannylene intermediate **16** (Scheme 6).



Scheme 4

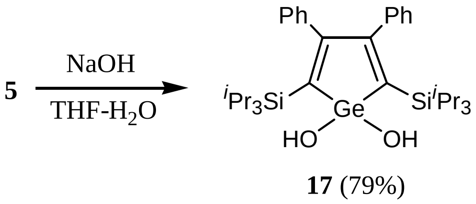


Scheme 5



Scheme 6

(d) Formation of Dihydroxygermole 17 from Diethoxygermole 5. Heating diethoxygermole **5** and sodium hydroxide in THF-H_2O under reflux gave dihydroxygermole **17**, stable under aerobic conditions, in 79% yield (Scheme 6), the structure of which was established by NMR and IR spectroscopy, and finally confirmed by X-ray diffraction analysis. In the ^1H NMR spectrum, a signal assignable to hydroxyl protons was observed at 1.67 ppm. In IR spectra measured both in solution (CCl_4) and in the solid state (KBr), two broadened absorptions were observed at about 3400 and 3650 cm^{-1}. The former absorption was assigned to the stretching vibration of hydroxy bonds involved in intermolecular hydrogen bonds,[14] while the latter absorption was assigned to the stretching vibration of hydroxy bonds without intermolecular hydrogen bonds.[7c,15] While 1,1-dihyrdoxy-2,3,4,5-tetraphenylgermole was so unstable that it underwent trimerization via dehydration to afford the corresponding cyclotrigermoxane,[16] the bulky triisopropylsilyl groups prevent the dihydroxygermole **17** from oligomerization.



Scheme 7

(e) X-Ray Diffraction Analysis of Dihydroxygermole 17. The molecular structure of **17** is shown in Figure 1. The geometry of the germole ring, with alternation of the C–C bonds, is quite similar to those reported previously.[16,17] The Ge–O bond length (1.774(2) Å) in **17** is quite similar to those of $t\mathrm{Bu}_2\mathrm{Ge(OH)}_2$ (av. 1.778 Å),[14] but slightly shorter than those of $(2,6\text{-MesC}_6H_3)_2\mathrm{Ge(OH)}_2$ (av. 1.790 Å).[7a] In the unit cell, two molecules face each other with an O–O distance of about 2.96 Å (Figure 2), suggesting hydrogen-bonding interactions. This is slightly longer than those found in $t\mathrm{Bu}_2\mathrm{Ge(OH)}_2$ (2.775-2.849 Å).[14]

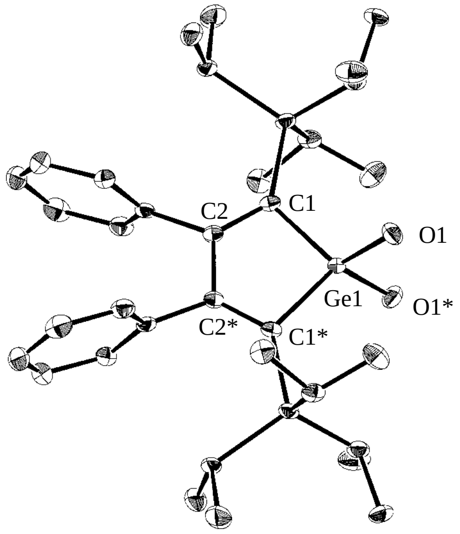


Figure 1. *ORTEP* drawing of dihydroxygermole **17** with thermal ellipsoids plots (40% probability for non-hydrogen atoms). All hydrogen atoms were omitted for clarity. Selected bond lengths (Å) and angles (deg), Ge1-O1: 1.774(2); Ge1-C1: 1.976(3); C1-C2: 1.347(4); C2-C2*: 1.541(6); O1-Ge1-O1*: 101.06(15); C1-Ge1-C1*: 94.39(18).

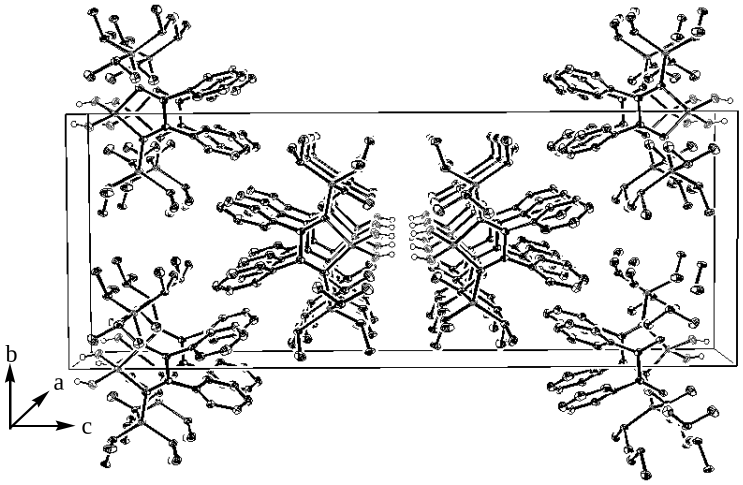


Figure 2. Unit cell of dihydroxygermole **17**. All hydrogen atoms except for those of the OH groups were omitted for clarity.

SUMMARY

Reactions of 1,4-dilithio-1,3-butadiene **1** having bulky silyl ligands at the 1,4-positions with tetrachlorosilane and hexachlorodisilane gave complex mixtures. However, 1,4-dilithio-1,3-butadiene **1** was treated with tetraethoxygermane to give diethoxygermole **5**, which was hydrolyzed to give stable dihydroxygermole **17**. The molecular structure of **17** was confirmed by X-ray diffraction analysis, which revealed intermolecular hydrogen bonds. Reaction of 1,4-dilithio-1,3-butadiene **1** with stannous chloride followed by treatment of the reaction mixture with carbon tetrachloride gave

chloroethynylstannole **12**, which was derived from ethynyllithium **8**. The ethynyllithium **8** was formed by reduction of **3** with lithium as a byproduct, although the generation efficiency could not be estimated.

EXPERIMENTAL

General Procedures: All experiments were performed under an argon atmosphere. Diethyl ether (Et_2O), THF, benzene and benzene-d_6 were distilled over sodium/benzophenone. Lithium dispersion was used for reduction of phenylsilylacetylene **3**. ^{1}H NMR (400 MHz), ^{13}C NMR (101 MHz) and ^{119}Sn NMR (149 MHz) spectra were recorded on a Bruker DRX-400 or a Bruker DPX-400 spectrometer in $CDCl_3$ or benzene-d_6. The multiplicities of signals in ^{13}C NMR given in parentheses were deduced from DEPT spectra. Wet column chromatography (WCC) was carried out with Kanto silica gel 60N. Preparative gel permeation chromatography (GPC) was carried out on an LC-918 (Japan Analytical Ind. Co., Ltd.) with JAIGEL-1H and -2H columns with chloroform ($CHCl_3$) as the eluant. Infrared spectra were measured at rt on a JASCO FT/IR-460 plus. All the melting points were determined on a Mitamura Riken Kogyo MEL-TEMP apparatus and were uncorrected. Elemental analyses were carried out at the Microanalytical Laboratory of Molecular Analysis and Life Science Center, Saitama University.

Reaction of 1,4-Dilithio-1,3-butadiene 1 with Hexachlorodisilane. A mixture of lithium (22 mg, 3.12 mmol) and phenyl(triisopropylsilyl)acetylene (**3**) (206 mg, 0.80 mmol) in Et_2O (1.5 mL) was stirred at rt for 4 h. To the resulting mixture was added Et_2O (10 mL), and the remaining lithium was filtered off through a glass filter. After removal of ether in vacuo, to the residue was added toluene (5.0 mL) and hexachlorodisilane (0.10 mL, 0.53 mmol) at –85 ºC. After the mixture was warmed to rt over 17 h, removal of volatile substances and materials insoluble in benzene followed by concentration of the filtrate afforded a crude product (188 mg). The ^{1}H NMR spectrum of the crude product revealed a broadened signal at 1.0-1.6 ppm and only 1,3-butadiene **4** was identified.

Reaction of 1,4-Dilithio-1,3-butadiene 1 with Tetrachlorosilane. A mixture of lithium (43 mg, 6.24 mmol) and phenyl(triisopropylsilyl)acetylene (**3**) (548 mg, 2.12 mmol) in Et_2O (2.1 mL) was stirred at rt for 4 h. The remaining lithium was filtered off through a glass filter. After removal of Et_2O in vacuo, to the residue was added toluene (5.0 mL) and the resulting solution was added to a toluene (3.0 mL) solution of tetrachlorosilane (3.0 mL, 26.1 mmol) at –105 ºC. After the mixture was warmed to rt over 15 h, removal of volatile substances and materials insoluble in CH_2Cl_2 followed by concentration of the filtrate afforded a crude product (674 mg). The ^{1}H NMR spectrum of the crude product was so complicated that no products could be identified.

Reaction of 1,4-Dilithio-1,3-butadiene 1 with Tetraethoxygermane. *(a) at room temperature.* A mixture of lithium (47 mg, 6.73 mmol) and phenyl(triisopropylsilyl)acetylene (**3**) (595 mg, 2.30 mmol) in

Et_2O (2.3 mL) was stirred at rt for 5 h. The resulting mixture was added to an Et_2O (5.0 mL) solution of tetraethoxygermane (1.56 mL, 7.03 mmol) at –85 ºC. After the reaction mixture was warmed to rt over 15 h, removal of volatile substances and materials insoluble in CH_2Cl_2 afforded a crude product (254 mg). The crude product was subjected to WCC (eluant; hexane : ethyl acetate = 10 : 1) to give 1,1-diethoxy-3,4-diphenyl-2,5-bis(triisopropylsilyl)germole (**5**) (75 mg, 10%) together with a mixture containing 1,3-butadiene **4**, *cis*-dihydrodibenzopentalene **6a** and dibenzopentalene **7**.[5] **5**: ^1H NMR($CDCl_3$): δ 0.88-0.96(m, 6H), 1.01(d, *J*=7 Hz, 36H), 1.26(t, *J*=7 Hz, 6H), 3.89(q, *J*=7 Hz, 4H), 6.81-6.84(m, 4H), 6.94-6.97(m, 6H); ^{13}C NMR($CDCl_3$): δ 13.56(d), 18.45(q), 19.30(q), 59.09(t), 126.37(d), 126.61(d), 129.04(d), 131.21(s), 142.12(s), 167.35(s). Anal. Calcd for $C_{38}H_{62}GeO_2Si_2$: C, 67.15; H, 9.19. Found: C, 66.28; H, 9.40.

(b) Heating under reflux. A mixture of lithium (60 mg, 8.66 mmol) and phenyl(triisopropylsilyl)acetylene (**3**) (741 mg, 2.87 mmol) in Et_2O (2.8 mL) was stirred at rt for 4 h. The resulting mixture was diluted by Et_2O (6.0 mL) and then added to Et_2O (4.0 mL) solution of tetraethoxygermane (1.92 mL, 8.66 mmol) at –85 ºC. After the reaction mixture was heated under reflux for 14 h, removal of volatile substances and materials insoluble in CH_2Cl_2 afforded a crude product (1.768 g). The crude product was subjected to WCC (eluant; hexane : EtOAc = 20 : 1) to give 1,1-diethoxy-3,4-diphenyl-2,5-bis(triisopropylsilyl)germole (**5**) (253 mg, 26%) together with a mixture containing 1,3-butadiene **4**, *cis*-dihydrodibenzopentalene **6a** and dibenzopentalene **7**.[5]

Alternative Preparation of 1,3-Butadiene 4 and Dihydrodibenzopentalenes 6. A mixture of lithium (177 mg, 25.4 mmol) and phenyl(triisopropylsilyl)acetylene (**3**) (2.212 g, 8.56 mmol) in Et_2O (8.6 mL) was stirred at rt for 6 h. After treatment of the reaction mixture with H_2O (10 mL), the organic layer was extracted with Et_2O, dried over anhydrous magnesium sulfate and concentrated to give a crude product (1.635 g). The ^1H NMR spectrum of the crude product revealed the formation of triisopropylsilylacetylene.[11] The crude product was purified by WCC (eluant; hexane), GPC and recrystallization to give (Z,Z)-2,3-diphenyl-1,4-bis(triisopropylsilyl)-1,3-butadiene (**4**) (761 mg, 34%), *cis*-5,10-dihydro-5,10-bis(triisopropylsilyl)dibenzo[*a,e*]pentalene (**6a**) (123 mg, 6%) and *trans*-5,10-dihydro-5,10-bis(triisopropylsilyl)dibenzo[*a,e*]pentalene (**6b**) (13 mg, 0.6%). **4**: mp 102-103 ºC(recrystallized from hexane+EtOH). ^1H NMR($CDCl_3$): δ 0.59-0.70(m, 6H), 0.80(d, *J*=7 Hz, 36H), 5.33(s, 2H), 7.21-7.30(m, 10H); ^{13}C NMR($CDCl_3$): δ 11.90(d), 18.90(q), 126.95(d), 127.29(d), 129.47(d), 129.77(d), 142.99(s), 161.47(s). Anal. Calcd for $C_{34}H_{54}Si_2$: C, 78.69; H, 10.49. Found: C, 78.68; H, 10.74. **6a**: mp 140-141 ºC(recrystallized from hexane+EtOH). ^1H NMR($CDCl_3$): δ 1.07(d, *J*=7 Hz, 36H), 1.44(sept, *J*=7 Hz, 6H), 3.82(s, 2H), 7.08(dd, *J*=8, 8 Hz, 2H), 7.24(dd, *J*=8, 8 Hz, 2H), 7.51(d, *J*=8 Hz, 2H), 7.53(d, *J*=8 Hz, 2H); ^{13}C NMR($CDCl_3$): δ 12.29(d), 19.22(q), 19.24(q), 36.01(d), 120.00(d), 122.79(d), 124.38(d), 124.99(d), 141.62(s), 151.10(s), 153.19(s). Anal. Calcd for $C_{34}H_{52}Si_2$: C, 79.00; H, 10.14. Found: C, 79.21; H, 10.34. **6b**: mp 278 ºC(recrystallized from hexane+EtOH). ^{1}H NMR($CDCl_3$): δ 0.88(d, *J*=7 Hz, 18H), 0.91(d, *J*=7 Hz, 18H), 1.31(sept, *J*=7 Hz, 6H), 4.14(s, 2H), 7.11(ddd, *J*=1, 8, 8 Hz, 2H), 7.24(ddd, *J*=1, 8, 8 Hz, 2H), 7.51(d, *J*=8 Hz, 2H), 7.57(d, *J*=8 Hz, 2H); ^{13}C NMR($CDCl_3$): δ 11.81(d), 18.50(q), 18.51(q), 35.32(d), 120.25(d), 122.79(d), 124.20(d), 124.74(d), 140.81(s), 149.72(s), 150.66(s). Anal. Calcd for $C_{34}H_{52}Si_2$: C, 79.00; H, 10.14. Found: C, 77.96; H, 10.11.

Reaction of 1,4-Dilithio-1,3-butadiene 1 with Stannous Chloride. A mixture of lithium (72 mg, 10.3 mmol) and phenyl(triisopropylsilyl)acetylene (**3**) (893 mg, 3.46 mmol) in Et_2O (3.6 mL) was stirred at rt for 4 h. The resulting mixture was diluted by Et_2O (7.2 mL) and added to a THF (6.0 mL) solution of stannous chloride (1.094 g, 5.77 mmol) at –85 ºC and the reaction mixture was then warmed to –50 ºC over 2 h. After treatment of the resulting mixture with CCl_4 (4.0 mL) at –50 ºC, the reaction mixture was warmed to rt over 10 h. Removal of volatile substances and materials insoluble in hexane afforded a crude product (708 mg). The crude product was subjected to GPC to afford 1-chloro-3,4-diphenyl-2,5-bis(triisopropylsilyl)-1-triisopropylsilylethynylstannole (**13**) (137 mg, 10%). **13**: ^{1}H NMR($CDCl_3$): δ 0.82-0.93(m, 6H), 0.99(d, *J*=4 Hz, 18H), 1.01(d, *J*=4 Hz, 18H), 1.12(br s, 21H), 6.79-6.83(m, 4H), 6.90-6.96(m, 6H); ^{13}C NMR($CDCl_3$): δ 11.22(d), 13.96(d, *J*(Sn-C)=55 Hz), 18.50(q), 19.48(q), 19.66(q), 112.73(s, *J*(Sn-C)=448, 473 Hz), 118.33(s, *J*(Sn-C)=73 Hz), 126.39(d), 126.50(d), 126.71(d), 129.23(d), 129.46(d), 134.48(s, *J*(Sn-C)=270, 282 Hz.), 142.59(s, *J*(Sn-C)=176, 184 Hz), 166.60(s, *J*(Sn-C)=115 Hz); ^{119}Sn NMR($CDCl_3$): δ -31.6. Anal. Calcd for $C_{45}H_{73}ClSi_3Sn$: C, 63.40; H, 8.63. Found: C, 63.40; H, 8.81.

Formation of Dihydroxygermole 16 from Diethoxygermole 5. A mixture of diethoxygermole **5** (63 mg, 0.084 mmol) and sodium hydroxide (336 mg, 8.41 mmol) in THF (5.0 mL) and H_2O (5.0 mL) was heated under reflux for 9 h. After being cooled to rt, the organic layer was extracted with CH_2Cl_2 and dried over anhydrous magnesium sulfate. After removal of the solvent, a crude product (63 mg) was purified by WCC (eluant; hexane : EtOAc = 10 : 1) to give 1,1-dihydroxy-3,4-diphenyl-2,5-bis(triisopropylsilyl)germole (**16**) (42 mg, 79%). **16**: mp 133.5-134.5 ºC (recrystallized from hexane). ^{1}H NMR($CDCl_3$): δ 0.85-0.95(m, 6H), 1.00(d, *J*=7 Hz, 36H), 1.67(s, 2H), 6.81-6.83(m, 4H), 6.92-6.98(m, 6H); ^{13}C NMR($CDCl_3$): δ 13.23(d), 19.36(q), 126.50(d), 126.59(d), 129.07(d), 130.95(s), 141.55(s), 167.14(s). IR (CCl_4): 3645(OH), 3350-3550(br, OH) cm^{-1}; IR (KBr): 3641(OH), 3200-3600(br, OH) cm^{-1}. Anal. Calcd for $C_{34}H_{54}GeO_2Si_2$: C, 65.49; H, 8.73. Found: C, 65.01; H, 8.80.

X-Ray Crystallography of 6a and 17. Data were collected at on Bruker SMART APEX diffractometer fitted with Mo-Kα radiation (λ = 0.71073 Å). The structures were solved by direct-methods (SHELXS-97)[18] and refinement (non-hydrogen atoms with anisotropic displacement parameters, H atoms in their calculated positions and with a weighting scheme of the form $w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$ where $P = (F_o^2 + 2F_c^2)/3)$ was by full-matrix least-squares procedures on F^2 (SHELXL-97).[19] Crystal data are listed in Table 2 and molecular structures were drawn with ORTEP-II.[20] **6a**: $C_{34}H_{52}Si_2$, FW=516.94; 103 K; crystal system, monoclinic; space group, $P2_1/n$; a=7.5220(5), b=22.3043(15), c=18.2951(12) Å; β=92.865(2) °; V=3065.6(4) Å^3; Z=4; D_{calc}=1.120 g cm^{-3}; R_1=0.057 ($I > 2\sigma(I)$, 6174 reflections), $_wR_2$=0.140 (for all reflections) for 7291 reflections and 337 parameters; GOF=1.107. CCDC-700912 contains the supplementary crystallographic data for this compound. **17** $C_{34}H_{54}GeO_2Si_2$, FW=623.54; 103 K; crystal system, orthorhombic; space group, $I222$; a=7.9861(14), b=12.904(2), c=33.827(7) Å; V=3485.9(11) Å^3; Z=4; D_{calc}=1.188 g cm^{-3}; R_1=0.034 ($I > 2\sigma(I)$, 2802 reflections), $_wR_2$=0.083 (for all reflections) for 3167 reflections and 232 parameters; GOF=1.063. CCDC-700913 contains the supplementary crystallographic data for this compound. These data can be obtained free of charge at www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (internat.) +44-1223/336-033; Email: deposit@ccdc.cam.ac.uk).

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