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Studies on Organosilicon Reactive Intermediates. VI. Photolysis of Trisilanes Containing Heterocycles

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ABSTRACT

*Both 2-phenyl-2-furylhexamethyltrisilane (**1**) and 2-phenyl-2-thienylhexamethyltrisilane (**2**) were synthesized by conventional organometallic reactions. The photolysis of **1** in the presence of 2,3-dimethyl-1,3-butadiene led to normal silylene-olefin addition and silylene C–H insertion reactions. However, when **2** was photolyzed in a methanol-cyclohexene system, an apparent radical reaction occurred. We suspect that the sulfur atom of the thienyl group strongly stabilizes a silyl radical. This result was supported by both identifying its typical radical reaction products and examining the ESR spectrum of its adduct with a radical trap.*

INTRODUCTION

Ever since Ishikawa and Kumada reported the generation of dimethylsilylene via photolysis of dodecamethylcyclohexasilane in 1970 [1], more and more silylenes have been generated by photolysis of polysilanes. This method has come to be recognized as a useful silylene source in silylene structure and reaction studies.

Although this method is widely used, relatively little is known about the mechanism. In 1974, Sakurai et al. reported that, when dimethylsilylene was extruded from a trisilacycloheptane system, the *cis-trans* stereochemistry was retained [2]. This result is consistent with a concerted extrusion process, but it also may be the consequence of the fact that the silyl radicals are configurationally stable. West and co-workers reported that, when a saturated linear polysilane was photolyzed in CCl_4, only CCl_3-CCl_3 was obtained at low retention time in GLC. They concluded that silyl radicals must be involved in the process [3].

Another point of interest for us was the electronic configurational state of photolytically generated silylene. The so-called "Skell rule" states [4] that singlet carbenes add to olefins with retention of stereochemistry, whereas triplet carbenes do not. But unlike carbenes, for which both singlet and triplet ground states are known, most known silylenes have singlet ground states [5]. Thus far, only Ando et al. have reported a nonstereospecific addition [6]. They found that the bulky dimesityl and bis(2,4,6-triisopropylphenyl)silylene gave nonstereospecific adducts. However, their work has been refuted by Conlin and co-workers [7]. In search of triplet ground state silylenes, Boudjouk et al. [8] and Pae et al. [9] have generated di-*t*-butylsilylene and diadamantylsilylene, respectively, hoping that the bulky substituent groups on the silylene would stabilize the triplet ground state silylene. They found that both silylenes have a high degree of stereospecificity for the olefin addition reactions and concluded that the bulky substituted silylenes still have the singlet ground state.

Thus, given the interest in the mechanism of the generation and electronic configuration of photolytically generated silylenes, we began to ex-

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Dedicated to Prof. Yao-Zeng Huang on the occasion of his eightieth birthday.

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amine silylenes having a heterocyclic substituent, hoping that the unshared electrons and the richer electron density in the heterocycle might have some effect on the empty orbital of silicon and thus change the silylene's electronic configuration and reaction mechanisms.

RESULTS AND DISCUSSION

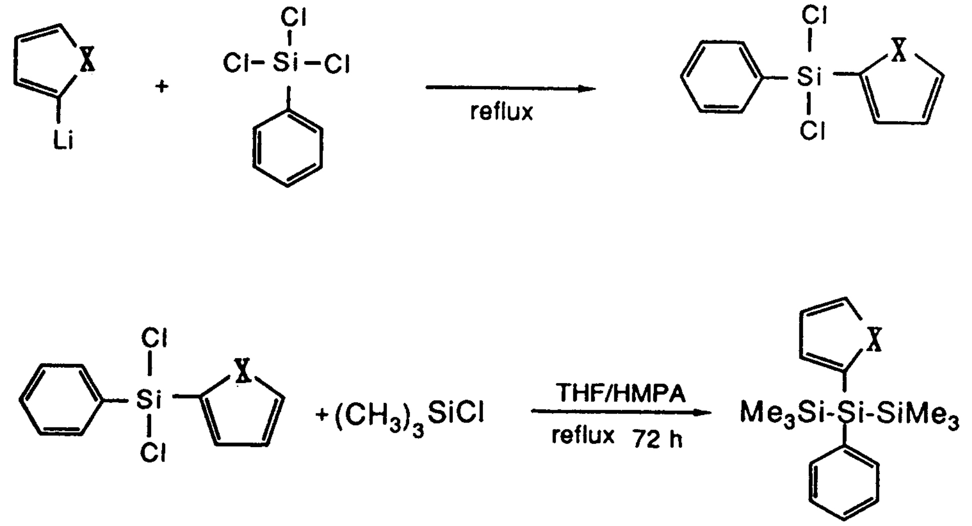
The silylene precursors were prepared by the method shown in Scheme 1.

Upon irradiation of **1** in the presence of 2,3-dimethyl-1,3-butadiene, three products were obtained: hexamethyldisilane **3** (40%) and two expected silylene addition and insertion products **4** (26%) and **5** (24%) [10]. In addition, photolysis of **1** in the presence of cyclohexene and methanol in pentane yielded hexamethyldisilane **3** and furylcyclohexylphenylmethoxylsilane **6** (26.3%) which might have been formed by addition of the photochemically generated silylene to the C=C double bond of cyclohexene and then subsequent ring-opening of the silirane by methanol (Scheme 2). However, when we irradiated **2** under the same conditions, different results were obtained (Scheme 3).

We immediately surmised that either phenylthienylsilylene is a triplet silylene or that the reaction products arose from reactions of a silylenoid intermediate. Thus, we undertook an experiment in which we attempted to capture reactive intermediates by carrying out the photolysis in the presence of a large excess of CCl_4 or $CBrCl_3$ in pentane solution. The main products were trimethylsilylphenylthienylchlorosilane (**13**) or trimethylsilylphenylthienylbromosilane (**14**) and hexachloroethane. The isolation of these products showed that the Si–Si bond of compound **13** or **14** did not undergo further homolytic cleavage when subjected to photolysis. Since a good yield of trimethylsilylphenylthienylsilane, which is a typical radical extraction product, was obtained from the photolysis of **2** in cyclohexene-methanol, formation of a triplet silylene or the occurrence of a silylenoid mechanism is unlikely.

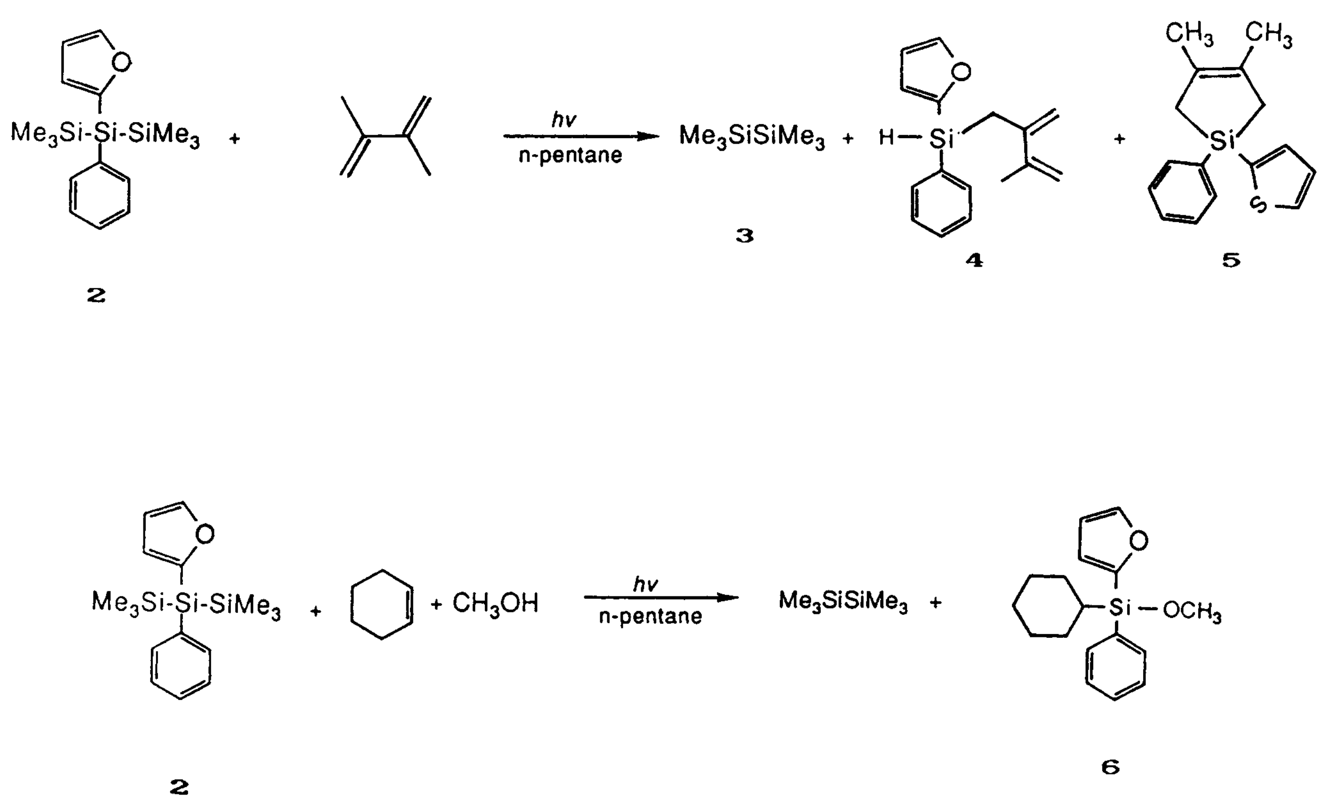
We then tried to gain an understanding of a possible radical mechanism of this reaction by use of the following experiments. Photolysis of **2** was carried out in methanol or in cyclohexene (Scheme 5). The results suggested that an initially formed trimethylsilylphenylthienylsilyl radical could abstract hydrogen from either methanol or cyclohexene.

We believe that compound **10** arises from the reaction of the trimethylsilylphenylthienylsilyl radical with methanol [11]. Further cleavage of the Si–Si bond in compound **9** and subsequent abstraction of hydrogen by the phenylthienylsilyl radical give phenylthienylsilane (**7**). If the trimethylsilylphenylthienylsilyl radical adds to cyclohexene and the subsequent radical abstracts hydrogen from cyclohexene, the compound **11** is obtained. Further cleavage of the Si–Si bond of compound **9** and the subsequent radical addition to cyclohexene and the loss of a hydrogen can then give product **15**.



X =O (1) ; S (2)

SCHEME 1



SCHEME 2

In the presence of a radical trap, hydroquinone, the radical reaction is stopped at the primary stage. In the presence or absence of methanol and cyclohexene, the only product, trimethylsilylphenylthienylsilane (**9**), was obtained in a high yield.

Now, a question emerges. What is the reasonable argument for the difference between the photolytic reactions of **1** and **2**? The only difference in these substrates is the replacement of the oxygen atom by a sulfur atom. A possible explanation is that the large $\sigma^{\cdot}_{JJ}$ value [12] of sulfur shows that it has a great stabilizing effect on a free radical.

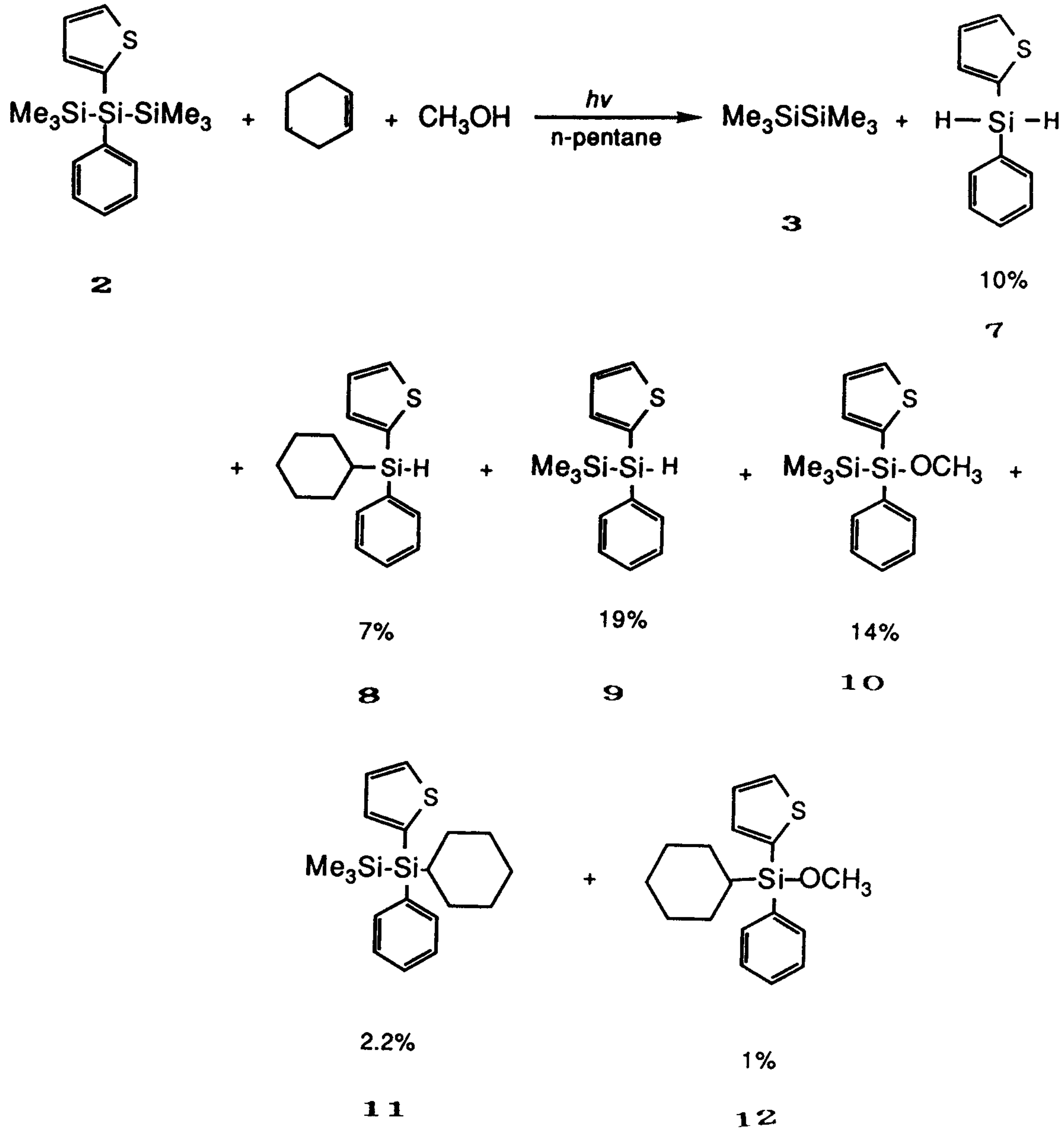
In order to confirm the initial formation of the silyl radical, we used ESR spectroscopy to detect the presence of a trapped product of the photolysis of the trisilane **2**. After irradiation of trisilane **2** for 1 hour, a trapping agent, 2-methyl-2-nitrosopropane, was added and the ESR spectrum of the trapped radical shown in Figure 1 was obtained.

The hyperfine splitting value a and the value of g are comparable with those of a typical nitroxide in an organic solvent, which confirms that it is the trapped product of a radical with 2-methyl-2-nitrosopropane. Since the bimolecular disproportionation of the trimethylsilyl radical is relatively fast [13], the ESR signal must come from the trimethylsilylphenylthienylsilyl radical only.

EXPERIMENTAL SECTION

Melting points were uncorrected. Elemental analyses were performed on a Perkin-Elmer 240C elemental analyzer. Infrared spectra were recorded on Shimadzu IR 408 and Perkin-Elmer 983G spectrophotometers. Nuclear magnetic resonance spectra were recorded on a JNM-PMX 60 SI (60 MHz, JEOL) spectrometer. Mass spectra were obtained on a Finnigan-MAT 4501 GC/MS spectrometer. Electron spin resonance spectra were taken on a Bruker ER-200D-SRC spectrometer. Analytical gas chromatograph was performed on a Shanghai 103 gas chromatograph (flame ionization detector) with nitrogen as carrier gas, using a 0.25-mm diameter and 45-M length SE-30 capillary column, and data were calculated on an HP 3390A integrator. Preparative gas chromatography was performed on a Shanghai 102 gas chromatograph with a thermal conductivity detector, hydrogen being used as carrier gas. The collections were conducted on a 6.3 mm × 3 m 20% SE-30 aluminum column.

Anhydrous ether and tetrahydrofuran (THF) were heated with sodium and distilled under nitrogen before use. Hexamethylphosphoramide (HMPA) was purified by reduced pressure distillation and then dried with calcium hydride. *n*-Pentane and methanol were distilled and then dried with molecular sieves. Cyclohexene was distilled



SCHEME 3

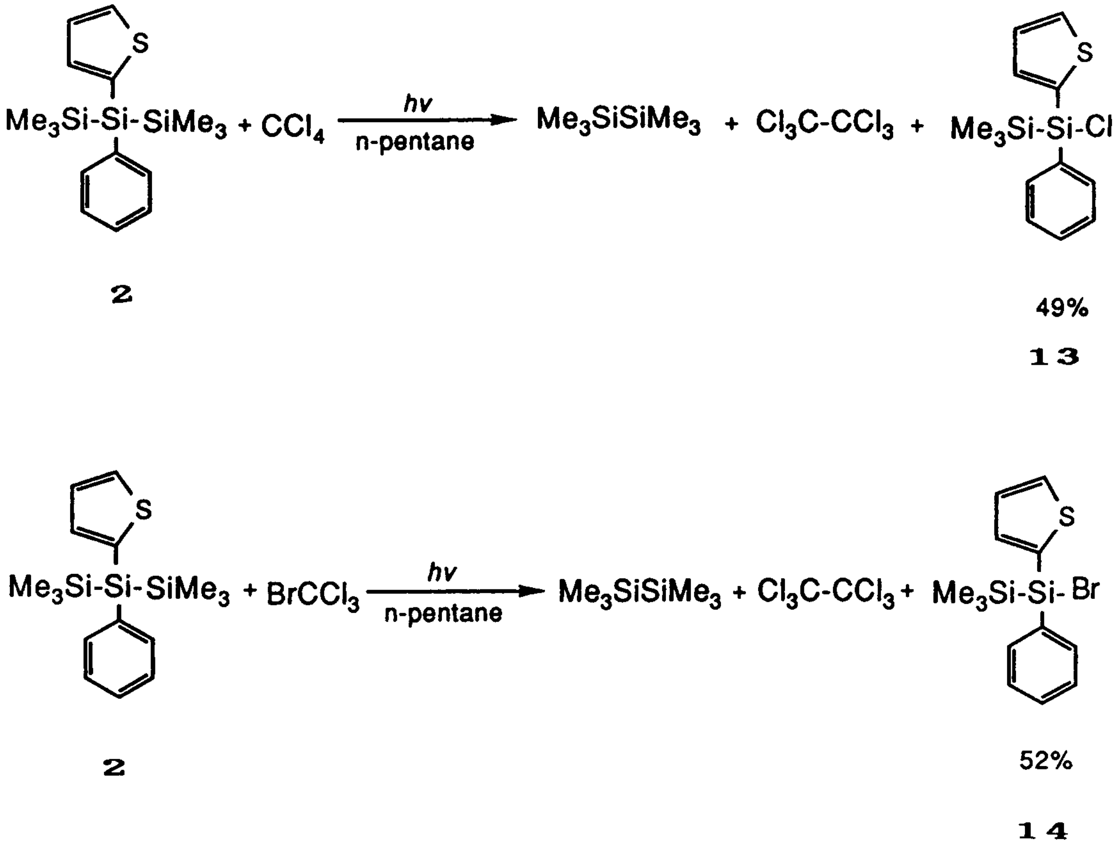
before use. 2-Methyl-2-nitrosopropane was purchased from the SIGMA company.

Phenyl-(α-Thienyl)dichlorosilane, $C_6H_5(C_4H_3S)SiCl_2$

A solution of 105.8 g of phenyltrichlorosilane (80 mL, 0.5 mol) in 60 mL of anhydrous ether was added into a 500 mL three-necked round-bottomed flask. A 200 mL hexane solution containing 0.26 mol of α-thienyllithium was added dropwise at 45°C with stirring. The reaction mixture was heated and stirred for another 11 hours. After the solvent had been distilled, the product was distilled under reduced pressure (ca. 5 mm) at 130–180°C, and 40.4 g (yield 60%, based on thienyllithium) of phenyl-(α-thienyl)dichlorosilane was obtained. δ_H (CCl_4, CH_2Cl_2 as inner standard): 7.10–7.82 (m, C_6H_5 and C_4H_3S).

*2-Phenyl-2-(α-Thienyl)hexamethyltrisilane $Me_3SiSi(C_6H_5)(C_4H_3S)$ $SiMe_3$ (**2**)*

In a 500 mL three-necked round-bottomed flask 8.4 g (0.35 g-atom) of magnesium, 60 mL of HMPA, and 110 mL of THF were mixed. A mixture of 40.4 g of phenyl-(α-thienyl)dichlorosilane (0.16 mol) and 38.5 g of trimethylchlorosilane (45 mL, 0.35 mol) was added dropwise with stirring under nitrogen. When the reaction mixture was heated to 80°C, the re-



SCHEME 4

action started. The reaction mixture was heated and stirred for another 72 hours.

This reaction mixture was treated with 50 mL of 10% NH_4Cl solution, and a few drops of 0.2N HCl were added to dissolve unreacted magnesium. Then the reaction mixture was washed with water and extracted with ether, and the ether solution was dried over Na_2SO_4. After filtration and evaporation of the solvent, the products were distilled under reduced pressure. Trisilane **2** distilled at 160–180°C (3 mm); yield 12 g (23.0%).

A pure sample was collected from a 3 m 20% SE-30 chromatography column. λ_{max} (EtOH): 260 nm. ν_{max}: 3050 (m), 2900 (m), 1585 (vw) and 1480 (w), 1425 (m), 1400 (m), 1245 (s), 1210 (m), 1092 (s) cm^{-1}. δ_H (CCl_4, CH_2Cl_2 as inner standard): 0.29 [18H, s, 6 × (CH_3Si)], 7.17–7.66 (8H, m, C_6H_5 and C_4H_2S). *m/z* (%): 334 (M^+, 30, 25), 73 (Me_3Si^+, 100).

Anal.: Calcd for $C_{16}H_{26}Si_3S$: C, 57.42; H, 7.83. Found: C, 57.62; H, 7.88.

Photolysis of **2** *in Cyclohexene and Methanol*

A mixture of 208 mg (0.62 mmol) of **2**, 2 mL of cyclohexene, 2 mL of methanol, and 15 mL of pentane was placed in a quartz tube equipped with a greaseless gas-tight Teflon vacuum stopcock. This solution was repeatedly degassed by the freeze-thaw method. The tube was closed, and the reaction mixture was irradiated for 12 hours in a water-cooled bath using a medium-pressure 500 W mercury lamp (Shanghai Yaming). After irradiation, the tube was placed in a liquid nitrogen bath and opened and the solvent was removed under ordinary pressure. The residue was analyzed by VPC, and the pure products were collected by preparative gas chromatography.

*Hexamethyldisilane $Me_3SiSiMe_3$ (**3**)*

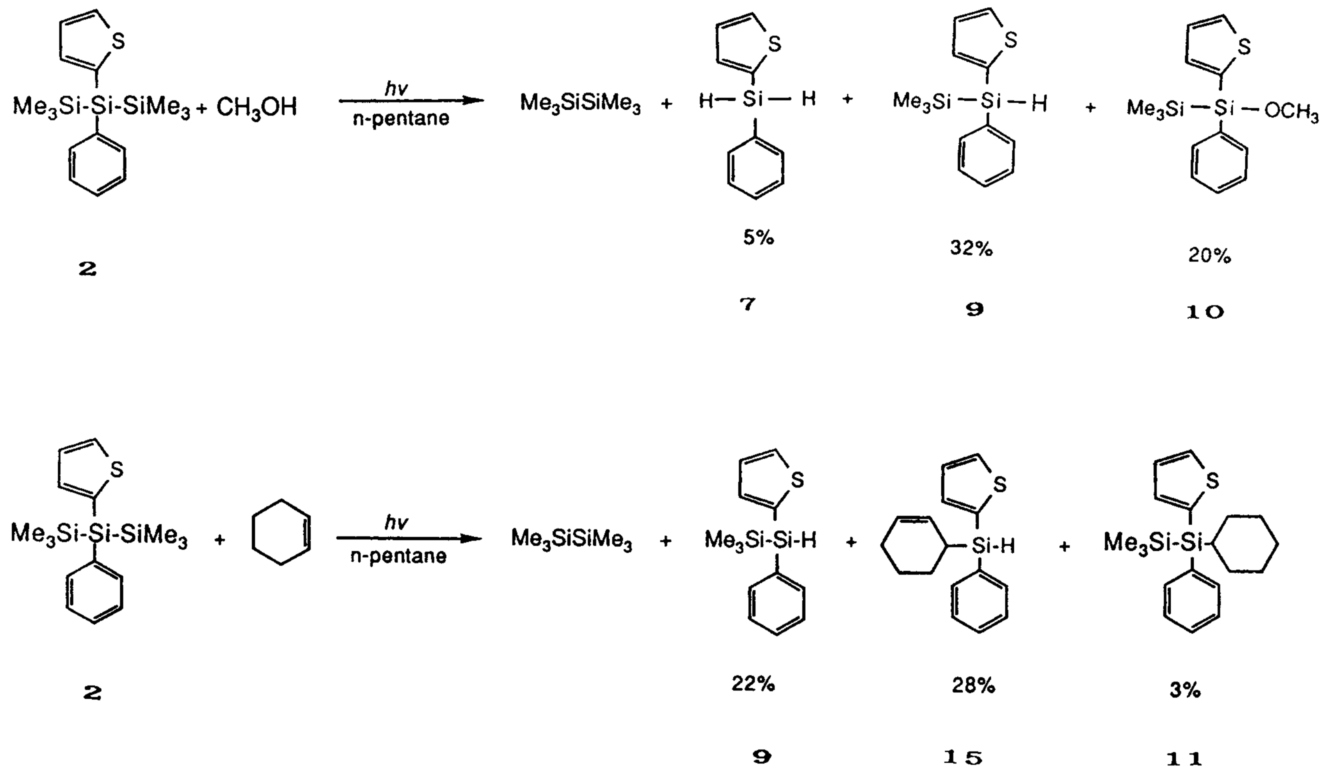
m/z (%): 146 (M^+, 7.76), 131 (14.32), 73 (M^+ −73, Me_3Si, 100).

*Phenyl-(α-Thienyl)silane $C_6H_5(C_4H_3S)SiH_2$ (**7**)*

ν_{max}: 3083 (w), 3065 (w), 3020 (w), 2940 (w), 2870 (w), 2154 (s, Si–H), 1218 (w), 856 (s) cm^{-1}. δ_H ($CDCl_3$): 5.06 (2H, s, Si–H), 7.22–7.72 (8H, m, C_6H_5 and C_4H_3S) ppm. *m/z* (%): 190 (M^+, 100), 189 (60.15), 105 (45.54).

*Phenyl-(α-Thienyl)cyclohexylsilane $C_6H_5(C_4H_3S)(C_6H_{11})SiH$ (**8**)*

m/z (%): 272 (M^+, 18.39), 189 (M^+ −83, 100), 105 (19.86).



SCHEME 5

Phenyl-(α-Thienyl)trimethylsilylsilane $Me_3SiSiH(C_6H_5)(C_3H_4S)$ (**9**)

ν_{max}: 3060 (w), 3059 (w), 2956 (w), 2903 (w), 2106 (m, Si–H), 1254 (w), 859 (m), 710 (s) cm^{-1}. δ_H ($CDCl_3$): 0.00 (9H, s, 3 × $SiMe_3$), 4.70 (H, s, Si–H), 7.01–7.44 (8H, m, C_6H_5 and C_4H_3S) ppm. *m/z* (%): 262 (M^+, 44.44), 247 (86.38), 189 (42.62), 105 (38.35), 73 ($MeSi_3$, 100).

Phenyl-(α-Thienyl)trimethylsilylmethoxysilane $Me_3SiSi(OCH_3)(C_4H_3S)C_6H_5$ (**10**)

ν_{max}: 3060 (w), 2931 (s), 2857 (w), 1214 (w), 991 (w), 817 (m), 800 (s) cm^{-1}. δ_H ($CDCl_3$): 0.22 (9H, s, $SiMe_3$), 3.56 (3H, s, OCH_3), 7.28–7.42 (8H, m, C_6H_5 and C_4H_3S). *m/z* (%): 292 (M^+, 10.04), 277 (M^+ −15, 100), 261 (3.07), 219 (68.01), 141 (7.37), 135 (13.36), 105 (18.36), 73 (28.59).

Phenyl-(α-Thienyl)trimethylsilylcyclohexylsilane $Me_3SiSi(C_4H_3S)(C_6H_5)C_6H_{11}$ (**11**)

m/z (%): 344 (M^+, 20.44), 261 (M^+ −83, 100), 189 (64.19), 135 (19.65), 105 (16.81), 73 (27.79).

Phenyl-(α-Thienyl)cyclohexylmethoxysilane $C_6H_5SiC_4H_3S(C_6H_{11})(OCH_3)$ (**12**)

m/z (%): 302 (M^+, 10.01), 219 (M^+ −83, 100), 189 (23,96), 105 (9.42).

Photolysis of **2** *in Carbon Tetrachloride*

This experiment was carried out by the same method as described previously. A mixture of 197 mg (0.59 mmol) of **2**, 5 mL of carbon tetrachloride, 2 mL of methanol, and 15 mL of pentane was irradiated for 12 hours in a water-cooled bath using a medium-pressure 500 W mercury lamp. After irradiation, the residue was analyzed by VPC and the pure products were obtained by preparative gas chromatography.

Hexamethyldisilane $Me_3SiSiMe_3$ (**3**)

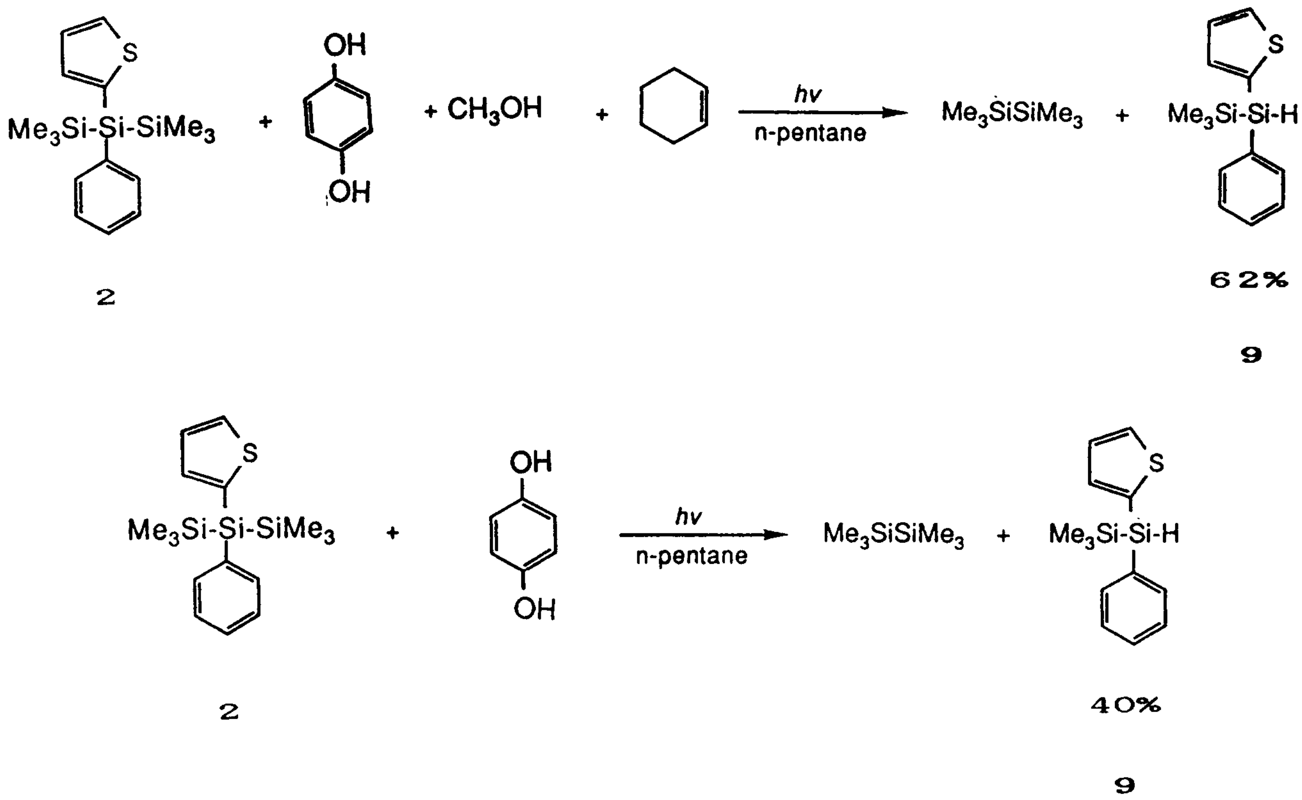
m/z (%): 146 (M^+, 7.76), 131 (14.32), 73 (M^+ −73, 100).

Hexachloroethane Cl_3CCCl_3

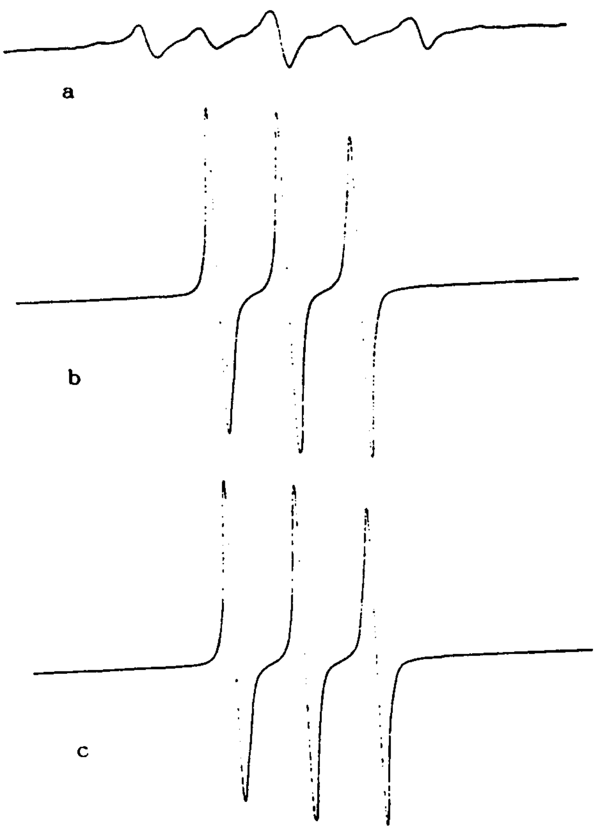
ν_{max}: 780 (s), 680 (w) cm^{-1}. *m/z* (%): 204 (4.38), 203 (22.02), 202 (66.37), 201 (100), 200 (61.32), 199 (2.52), 168 (4.65), 167 (15.23), 166 (30.12), 165 (25.24) cm^{-1}.

Phenyl-(α-Thienyl)trimethylsilylchlorosilane $Me_3SiSiCl(C_6H_5)(C_4H_3S)$ (**13**)

ν_{max}: 3609 (w), 3078 (m), 3044 (m), 3010 (w), 3000 (w), 2960 (m), 2889 (m), 1590 (w), 1498 (w), 1480 (w), 1430 (s), 1400 (s), 1322 (m), 1250 (s), 1219 (s), 1109 (s), 1080 (s), 998 (s), 858 (s), 840 (s), 740 (s),



SCHEME 6



control reaction: ESR spectrum of trapping agent

ESR spectrum of silyl radical after trapping by 2-methyl-2-nitroso-propane

spectrum subtraction of a from b

FIGURE 1 The field sweep of the spectrometer was calibrated with a pentane solution of DPPH (g = 2.0037), and the g value and coupling constant a are 2.0063 and 15.0 Gauss, respectively.

700 (s), 578 (s), 430–200 (s) cm^{-1}. δ_H (CCl_4, CH_2Cl_2 as inner standard): 0.32 (9H, s, $SiMe_3$), 7.18–7.85 (8H, m, C_6H_5 and C_4H_3S). *m/z* (%): 298 (M^+ +2, 23.45), 296 (M^+, 50.98), 281 (21.34), 189 ($C_4H_3SSiC_6H_5$, 100), 141 (11.24), 135 (19.57), 73 (37.76).

Photolysis of **2** *in Bromotrichloromethane*

The experiment was carried out by the same method as described previously. A mixture of 188 mg (0.56 mmol) of **2**, 5 mL of bromotrichloromethane, and 15 mL of pentane was irradiated for 12 hours in a water-cooled bath using a medium-pressure 500 W mercury lamp. After irradiation, the residue was analyzed by VPC and the pure products were obtained by preparative gas chromatography.

Hexamethyldisilane $Me_3SiSiMe_3$ (**3**)

m/z (%): 146 (M^+, 7.76), 131 (14.32), 73 (M^+ −73, 100).

Hexachloroethane Cl_3CCCl_3

ν_{max}: 780 (s), 680 (w) cm^{-1}. *m/z* (%): 204 (4.34), 203 (22.25), 202 (66.12), 201 (100), 200 (61.15), 199 (2.53), 168 (4.45), 167 (15.78), 166 (30.71), 165 (25.96).

Phenyl-(α-Thienyl)trimethylsilyibromosilane $Me_3SiSiBr(C_6H_5)(C_4H_3S)$ (**14**)

ν_{max}: 3623 (w), 3091 (m), 3062 (m), 3020 (w), 2982 (m), 2909 (m), 1598 (w), 1489 (w), 1441 (s), 1422 (s), 1332 (m), 1263 (s), 1232 (s), 1101 (s), 888 (s), 851 (s), 754 (s), 723 (s), 590 (s), 490–200 (s) cm^{-1}. δ_H (CCl_4, CH_2Cl_2 as inner standard): 0.29 (9H, s, $SiMe_3$), 7.08–7.66 (8H, m, C_6H_5 and C_4H_3S). *m/z* (%): 342 (M^+ +2, 11.22), 340 (M^+, 9.26), 261 (2.55), 188 ($C_4H_3SSiC_6H_5$, 100), 135 (14.51) 105 (10.34), 73 (63.91).

Photolysis of **2** *in Methanol*

The experiment was carried out by the same method as described previously. A mixture of 294 mg (0.88 mmol) of **2**, 2 mL of methanol, and 15 mL of pentane was irradiated for 12 hours in a water-cooled bath using a medium-pressure 500 W mercury lamp (Shanghai Yaming). After irradiation, the residue was analyzed by VPC and the pure products were obtained by preparative gas chromatography.

Hexamethyldisilane $Me_3SiSiMe_3$ (**3**)

m/z (%): 146 (M^+, 7.78), 131 (14.39), 73 (M^+ −73, 100).

Phenyl-(α-Thienyl)silane (**4**)

m/z (%): 190 (M^+, 100), 189 (M^+ −1, 61.15), 105 (45.94).

Phenyl-(α-Thienyl)trimethylsilylsilane (**9**)

m/z (%): 262 (M^+, 44.44), 247 (86.38), 189 (42.62), 105 (38.35), 73 (Me_3Si, 100).

Phenyl-(α-Thienyl)trimethylsilylmethoxysilane (**10**)

m/z (%): 292 (M^+, 10.56), 277 (M^+ −15, 100), 261 (3.07), 219 (68.01), 141 (7.37), 135 (13.36), 105 (18.36), 73 (28.59).

Photolysis of **2** *in Cyclohexene*

The experiment was carried out by the same method as described previously. A mixture of 196 mg (0.59 mmol) of **2**, 2 mL of cyclohexene, and 15 mL of pentane was irradiated for 12 hours in a water-cooled bath using a medium-pressure 500 W mercury lamp (Shanghai Yaming). After irradiation, the residue was analyzed by VPC and the pure products were obtained by preparative gas chromatography.

Hexamethyldisilane $Me_3SiSiMe_3$ (**3**)

m/z (%): 146 (M^+, 7.76), 131 (14.32), 73 (M^+ −73, 100).

Phenyl-(α-Thienyl)trimethylsilylsilane (**9**)

m/z (%): 262 (M^+, 44,44), 247 (86.38), 189 (42.62), 105 (38.35), 73 (Me_3Si, 100).

Phenyl-(α-Thienyl)cyclohexenylsilane $C_6H_5(C_4H_3S)SiH(C_6H_9)$ (**15**)

m/z (%): 270 (M^+, 3.04), 189 (M^+ −81, 100), 105 (22.33).

Phenyl-(α-Thienyl)trimethylsilylcyclohexylsilane (**11**)

m/z (%): 344 (M^+, 20.44), 261 (M^+ −83, 100), 189 (64.19), 135 (19.65), 105 (16.81), 73 (27.79).

Photolysis of **2** *in the Presence of Hydroquinone*

The experiment was carried out by the same method as described previously. A mixture of 175 mg (0.52 mmol) of **2**, 200 mg of hydroquinone, and 15 mL of pentane was irradiated for 12 hours in a water-cooled bath using a medium-pressure 500 W mercury lamp (Shanghai Yaming). After irradiation, the residue was analyzed by VPC and the pure products were obtained by preparative gas chromatography.

*Hexamethyldisilane $Me_3SiSiMe_3$ (**3**)*

m/z (%): 146 (M^+, 7.76), 131 (14.32), 73 (M^+ −73, 100).

*Phenyl-(α-Thienyl)trimethylsilylsilane (**9**)*

m/z (%): 262 (M^+, 44,44), 247 (86.38), 189 (42.62), 105 (38.35), 73 (Me_3Si, 100).

*Photolysis of **2** in the Presence of Cyclohexene, Methanol, and Hydroquinone*

The experiment was carried out by the usual method. A mixture of 195 mg (0.58 mmol) of **2**, 2 mL of methanol, 2 mL of cyclohexene, 100 mg of hydroquinone, and 15 mL of pentane was irradiated for 12 hours in a water-cooled bath using a medium-pressure 500 W mercury lamp. After irradiation, the residue was analyzed by VPC and the pure products were obtained by preparative gas chromatography.

*Hexamethyldisilane $Me_3SiSiMe_3$ (**3**)*

m/z (%): 146 (M^+, 7.76), 131 (14.32), 73 (M^+ −73, 100).

*Phenyl-(α-Thienyl)trimethylsilylsilane (**9**)*

m/z (%): 262 (M^+, 44,44), 247 (86.38), 189 (42.62), 105 (38.35), 73 (Me_3Si, 100).

*Photolysis of **2** and Trapping of the Silyl Radical*

A mixture of 10 mg (0.03 mmol) of **2** and 1 mL of pentane was placed in a quartz tube. The air was replaced by nitrogen (99.99%), and the mixture was irradiated with a 500 W medium pressure mercury lamp. After 1 hour irradiation, 0.2 mL of a pentane solution of 2-methyl-2-nitrosopropane was added under the N_2 atmosphere and then the ESR spectra was recorded.

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[13] G. B. Watts, U. K. Ingold, *J. Am. Chem. Soc.*, *94*, 1972, 491. The rate constant of bimolecular self-reaction of trimethylsilyl radical is 5.5×10^{-9} M^{-1} s^{-1}, and the g value and coupling constant a of the ESR signal for trimethylsilyl radical are 2.0031 and 6.3 Gauss, respectively.