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Reactions of Hydrazines and Ureas with Silenes

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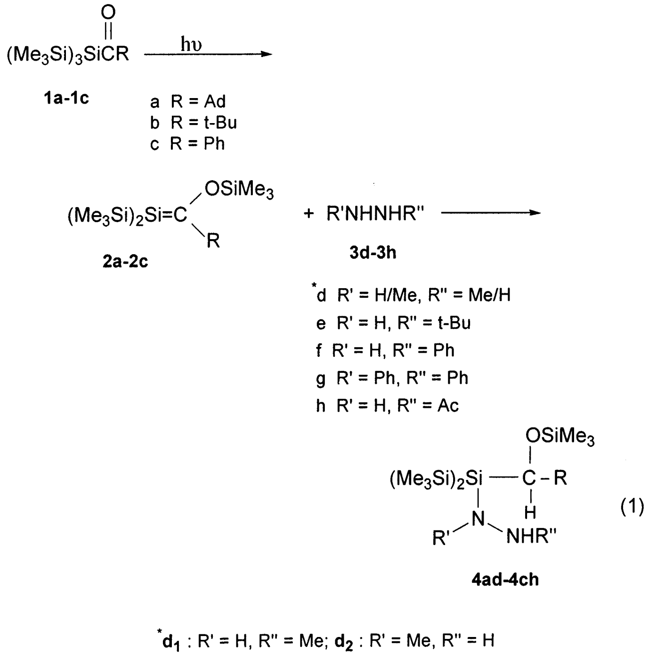
Hydrazines and ureas add cleanly in the dark to the silicon−carbon double bonds of silenes of the family $(Me_3Si)_2Si{=}C(OSiMe_3)R$ to give the adducts $(Me_3Si)_2(R'NNR'')SiCH(OSiMe_3)R$ and the heterocyclic 1-sila-3,4-diaza-3-oxocyclopentane products, respectively. However, hydrazines and ureas do not add in the dark to polysilylacylsilanes $(Me_3Si)_3SiCOR$. Mechanisms accounting for the results are proposed.

The addition reactions of silenes with a variety of reagents have been described by different workers and have been summarized in several recent reviews.[1] A thorough search of the literature indicated the complete absence of publications on the reactions of silenes with hydrazines and ureas. However, Wiberg has produced data in a paper and review article about the reactions and the relative reactivity of some amines with the silene $Me_2Si{=}C(SiMe_3)_2$,[2,3] which was generated from a precursor at about 100 °C in the presence of the amine, which trapped it as the expected adduct involving Si−N bond formation. The reaction of amines with silenes generated at relatively short UV wavelengths has also been recently mentioned.[4] Brook has recently reported on the reactions of the amines with the silenes of the family $(Me_3Si)_2Si{=}C(OSiMe_3)R$, generated photochemically at room or lower temperatures using long-wavelength radiation.[5]

In the present work, the reactions of the silenes $(Me_3Si)_2Si{=}C(OSiMe_3)R$ (R = Ad (**2a**), *t*-Bu (**2b**), Ph (**2c**)) with hydrazines and ureas were examined to ascertain their reactivity and behavior toward addition to the silicon−carbon double bond under milder conditions. No reaction was found to occur between the parent acylsilanes $(Me_3Si)_3Si{-}COR$ (R = Ad (**1a**), *t*-Bu (**1b**), Ph (**1c**)) and various hydrazines (**3d**−**h**) and ureas (**5i**−**n**) in the dark over several days. However, photolysis of a mixture of the acylsilane and hydrazine in benzene with 360 nm radiation and longer wavelengths (conditions where the acylsilane isomerizes to the silene as a result of a 1,3-silyl shift from silicon to oxygen) led to the formation of the addition products **4ad**−**4ch** of the hydrazine to the silicon−carbon double bond in quantitative yields, as confirmed by NMR and MS spectroscopy and illustrated in eq 1. The regiochemistry of these reactions with hydrazines $RNHNH_2$ (R = Ph, *t*-Bu) was established on the basis of the NMR and IR spectral analysis of the addition products obtained. The compounds **4af**, **4ae**, **4bf**, and **4cf** all show two different single N−H resonances in their ^{1}HNMR spectra and not one single resonance due to an NH_2 group. Moreover, these compounds do not give rise to two N−H stretching bands in their infrared spectra in the 3500−3300 cm^{-1} region characteristic of an NH_2 group; instead, they all exhibit only one N−H band in this region. These results suggest that the monosubstituted hydrazines add to the silenes only at the nonsubstituted N atom, which can be accounted for by the steric and resonance effects of the substituents on these hydrazines. This regioselectivity has previously been reported on the reactions of the monosubstituted hydrazines with halogenosilanes.[6] However, the formation of two regioisomers **4bd$_1$** and **4bd$_2$** in the ratio 35:25 suggests that both of the N atoms in methylhydrazine are eligible for nucleophilic addition to silenes.



That the ureas **5i**−**n** readily undergo similar reactions with silenes **2a**−**c** to produce the heterocyclic 1-sila-2,4-diaza-3-oxocyclopentanes **7ai**−**7cn**, in essentially quantitative yields, was confirmed by NMR, IR, and MS

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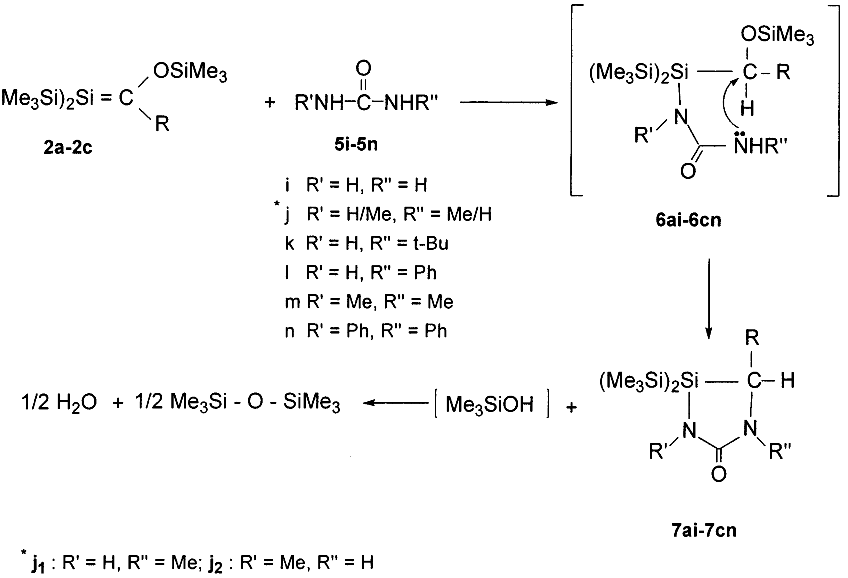
Scheme 1. Proposed Mechanism for Formation of 1-Sila-2,4-diaza-3-oxocyclopentane Products



spectroscopy. This can probably occur through the intermediate adducts **6ai**–**6cn**, which undergo rapid cyclization to 1-sila-2,4-diaza-3-oxocyclopentanes **7ai**–**7cn** as a result of intramolecular nucleophilic attack by the second amino group on the trimethylsiloxy-substituted carbon atom; this results in the release of trimethylsilanol, which undergoes a rapid condensation to hexamethyldisiloxane. The application of the nuclear Overhauser effect (NOE) helped to simplify the interpretation of the NMR spectra of the heterocyclic products and provided valuable information on the relative spatial locations of the substituents bonded to N atoms in the products **7ak**, **7al**, **7bl**, and **7ck** and also distinguished between the regioisomers **7aj$_1$** and **7aj$_2$**, produced in a ratio of 70:30, as determined by the relative integral ratio of N–Me signals of their mixed ^{1}H NMR spectrum. In an NOE measurement, continuous irradiation at the AdC–H signal position (δ 3.45) of the compound **7ai** caused enhancement of the signal intensity at δ 6.35 for the N-4 proton but not for the N-2 proton at δ 7.64. However, when for each compound **7ak**, **7al**, **7bl**, and **7ck** the C-5 proton was continuously irradiated, no enhancement in intensity of the N–H signal was detected. These results are consistent with the fact that the monosubstituted ureas RNHC(O)NH$_2$ (R = Ph, t-Bu), like their hydrazine derivatives, add to the silenes only at the nonsubstituted N atom. Here again, the steric and resonance effects are probably responsible for such regioselectivity. However, the two regioisomers **7aj$_1$** and **7aj$_2$** produced in the case of methylurea were distinguished from one another by using the NOE technique. When, in an NOE measurement, the AdC–H proton signal at δ 3.42 was continuously irradiated, only the N–Me group at δ 2.87 exhibited an enhancement in intensity with no change in the intensity of the N–H signal at δ 7.75. Conversely, in a separate NOE measurement, continuous irradiation of the AdC–H proton signal at δ 3.48 caused an enhancement of the N–H signal at δ 6.32 with no detectable change in the intensity of the N–Me signal at δ 2.75. According to these results, we can assign the resonances at δ 2.87 and 7.75, respectively, to N–Me and N–H protons of one regioisomer (**7aj$_1$**) and the resonances at δ 2.75 and 6.32, respectively, to N–Me and N–H protons of the other regioisomer (**7aj$_2$**). Moreover, to assign the signals to specific methyl groups both in the ^{1}H and the ^{13}C NMR spectra of the compound **7bm**, in an NOE measurement, the *t*-BuC–H proton signal at δ 3.29 was continuously irradiated, which caused an enhancement in the intensity of the signal at δ 2.78 with no detectable change in the intensity of the N–Me proton signal at δ 2.95. In a separate NOE measurement, irradiation of N–Me protons at δ 2.78 intensified the ^{13}C resonance signal of the N–Me group at δ 35.83 with no change in the intensity of the ^{13}C signal of the N–Me group at δ 34.62. Consequently, we can assign, in compound **7bm**, the resonances at δ 2.95 in ^{1}H NMR and δ 34.62 in ^{13}C NMR spectra to the N-2 methyl group and the resonances at δ 2.78 in ^{1}H NMR and δ 35.83 in ^{13}C NMR spectra to the N-4 methyl group. A probable mechanism for this reaction is indicated in Scheme 1. We have previously reported on the formation of 5-methylene-1-sila-2,4-diaza-3-oxocyclopentanes in reactions between silaaziridines and aromatic isocyanates under photolytic conditions.[7]

We have also found that, when silenes, preformed by photolysis of the parent acylsilanes, were treated with hydrazines and ureas in the dark, a rapid, clean, and almost quantitative addition takes place to give the expected adducts, **4ad**–**4ch**, and the heterocyclic products, **7ai**–**7cn**, respectively. The products all were viscous oils, and all attempts to crystallize them from different solvents failed.

Thus, it is clear that hydrazines and ureas can readily add to the silicon–carbon double bond in the dark, almost certainly initiated by nucleophilic attack of the less crowded nitrogen lone pair on silicon, followed by migration of the proton from the same nitrogen to the electron-rich carbon atom of the original double bond. In the case of ureas, a rapid intramolecular nucleophilic attack by the second amino group on the carbon atom of the original double bond follows, leading to the

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formation of the heterocyclic 1-sila-2,4-diaza-3-oxocyclopentanes and the release of trimethylsilanol. This mechanism has been established from the addition of alcohols to the silicon−carbon double bond.[8] Wiberg in his rate studies has suggested that the rate of addition of amines is more rapid than that of the related alcohols. In our studies it is clear that when a mixture of acylsilane and hydrazine or urea was photolyzed, the rate of isomerization of acylsilane to silene was probably the rate-determining step.

Experimental Section

Nomenclature. New compounds are characterized by a number and two letters, the letters relating to the acylsilanes (**a**−**c**), hydrazines (**d**−**h**), and ureas (**i**−**n**) employed as reagents.

General Procedures. All experiments were performed in oven-dried glassware under a nitrogen atmosphere using standard inert-atmosphere and vacuum-line techniques. Diethyl ether was distilled from benzophenone ketyl; hexane and benzene were dried over lithium aluminum hydride prior to distillation.

NMR spectra were run at room temperature on either a JEOL JNM-LA 300 or Gemini 200 spectrometer in C_6D_6 unless otherwise stated. Where necessary, APT[9] and DEPT[10] pulse sequences were used in obtaining ^{13}C NMR spectra, and all ^{29}Si NMR spectra were run in the DEPT mode. IR spectra were recorded using a Perkin-Elmer 283 spectrophotometer. Samples were prepared as solutions in a NaCl cell. Mass spectra were obtained on a VG70-2505 mass spectrometer. The data are reported in mass-to-charge units (*m/z*) followed by their relative intensities in parentheses. Elemental analyses were performed by Iran Polymer and Petrochemical Institute, P. O. Box 14965/115, Tehran, Iran. Melting points are uncorrected. Acylpolysilanes were prepared according to published procedures.[11,12]

General Experimental Procedure. Photolysis of polysilanes was carried out under dry nitrogen and employed equimolar amounts of the hydrazine and the urea relative to acylsilane. Irradiations at 360 nm and longer wavelengths used three 100 W PAR 38 clear mercury spot lamps (Blak-Ray ANSI code H44GS) to illuminate NMR tubes positioned in the intense central beam about 12 in. from the lamps and cooled by a double-walled jacket containing running water. At the end of the experiment, the volatile compounds and solvent were removed by pumping under reduced pressure. A typical experiment involving a polysilylacylsilane follows.

Photolysis of Adamantoyltris(trimethylsilyl)silane (1a) with Phenylhydrazine (3f). A solution of 0.082 g (0.2 mmol) of the acylsilane **1a** and 0.02 mL (0.2 mmol) of phenylhydrazine (**3f**) in 1 mL of C_6D_6 was irradiated under nitrogen for 8 h. Removal of the remaining phenylhydrazine and C_6D_6 under reduced pressure followed by chromatography on silica gel using hexane/ether (50:50) gave the colorless oily adduct **4af** (75% yield). Attempts to crystallize it from a variety of solvents failed. ^1H NMR: δ 0.23, 0.28, 0.32 (each 9H, s, $SiMe_3$), 1.25 (1H, br s, NHSi), 1.58−1.92 (15H, m, Ad), 3.57 (1H, s, CHOSi), 3.72 (1H, br s, NHPh), 6.80−7.75 (5H, m, Ph). ^{13}C NMR: δ 1.95, 2.02 ($SiMe_3$), 2.28 ($OSiMe_3$), 29.14 (Ad *C*H), 37.05 (Ad *C*H_2), 39.00 (C quat Ad), 40.94 (Ad *C*H_2), 80.72 (*C*HOSi), 126.42, 126.75, 127.12 (Ph CH), 142.67 (Ph ipso C). ^{29}Si NMR: δ −20.42, −19.25 ($SiMe_3$), −13.28 ($Si(SiMe_3)_2$), 12.45 ($OSiMe_3$). IR: 3485, 3035, 2920, 2895, 1610, 1564, 1500, 1457, 1285, 1263, 1154, 1092, 905, 895, 756, 708 cm^{-1}. MS (EI): *m/z* (relative intensity) 503 $(M - Me)^+$ (5), 446 $(M + H - SiMe_3)^+$ (48), 412 $(M + H - C_6H_7N_2)^+$ (12), 339 $(412 - SiMe_3)^+$ (8), 225 $((Me_3Si)_2Si(C_6H_5N_2H_2))^+$ (100), 237 $((Me_3SiO)CH(C_{10}H_{15}))^+$ (15), 191 $((Me_3Si)(Me_3SiO)SiH)^+$ (18), 135 $(C_{10}H_{15})^+$ (22), 73 $(Me_3Si)^+$ (65). HRMS for $(M - Me)^+ = C_{25}H_{47}N_2OSi_4$: *m/z* calcd, 503.2753; found, 503.2754. Anal. Calcd for $C_{26}H_{50}N_2OSi_4$: C, 60.23; H, 9.65; N, 5.40. Found: C, 60.18; H, 9.45; N, 5.48.

It was found that when a solution of 0.041 g (0.1 mmol) of the acylsilane **1a** and 0.01 mL (0.1 mmol) of hydrazine **3f** in 0.5 mL of C_6D_6 was stored in the dark at room temperature under nitrogen for 72 h, no reaction occurred and only starting materials were observed by ^1H, ^{13}C, and ^{29}Si NMR spectra.

Reaction of Adamantylsilene (2a) with Phenylhydrazine (3f) in the Dark. A solution of 0.041 g (0.1 mmol) of acylsilane **1a** in 0.5 mL of C_6D_6 was irradiated under nitrogen for 20 h to yield the silene **2a** quantitatively, as shown by NMR spectroscopy. To this solution was added 0.01 mL (0.1 mmol) of phenylhydrazine, and the solution was stored in the dark for 8 h at room temperature. NMR spectroscopy indicated that all the silene had been converted to the hydrazine adduct **4af**, which had NMR spectra as described above.

Photolysis of Adamantoyltris(trimethylsilyl)silane (1a) with *tert*-Butylhydrazine (3e). Following the procedure given above, a pale yellow solution was obtained. Concentration under reduced pressure followed by chromatography on silica gel using hexane/ether (70:30) afforded the colorless oily adduct **4ae** (70% yield). ^1H NMR: δ 0.21, 0.27, 0.30 (each 9H, s, $SiMe_3$), 1.05 (1H, br s, NHSi), 1.22 (9H, s, CMe_3), 1.60−1.85 (15H, br m, Ad), 3.25 (1H, br s, $NHCMe_3$), 3.52 (1H, s, CHOSi). ^{13}C NMR: δ 1.92, 1.98 ($SiMe_3$), 2.18 ($OSiMe_3$), 29.06 (Ad *C*H), 32.62 (C*Me*$_3$), 37.04 (Ad *C*H_2), 38.63 (C quat Ad), 40.10 (Ad *C*H_2), 52.30 (*C*Me_3), 80.57 (*C*HOSi). ^{29}Si NMR: δ −21.82, −20.21 ($SiMe_3$), −14.62 ($Si(SiMe_3)_2$), 12.25 ($OSiMe_3$). IR: 3375, 2900, 2885, 1423, 1412, 1276, 1268, 1173, 1085, 918, 823, 792, 715 cm^{-1}. MS (EI): *m/z* (relative intensity) 483 $(M - Me)^+$ (12), 426 $(M + H - SiMe_3)^+$ (35), 427 $(M + H - C_4H_{10}N)^+$ (18), 412 $(M + H - C_4H_{11}N_2)^+$ (8), 364 $(M + H - Ad)^+$ (4), 261 $((Me_3Si)_2Si(C_4H_9N_2H_2))^+$ (100), 339 $(412 - SiMe_3)^+$ (4), 237 $((Me_3SiO)CH(C_{10}H_{15}))^+$ (22), 191 $((Me_3Si)(Me_3SiO)SiH)^+$ (12), 135 $(C_{10}H_{15})^+$ (32), 73 $(Me_3Si)^+$ (58). HRMS for $(M - Me)^+ = C_{23}H_{51}N_2OSi_4$: *m/z* calcd, 483.3065; found, 483.3068. Anal. Calcd for $C_{24}H_{54}N_2OSi_4$: C, 57.83; H, 10.84; N, 5.62. Found: C, 57.78; H, 10.54; N, 5.72.

Photolysis of Adamantoyltris(trimethylsilyl)silane (1a) with 1,2-diphenylhydrazine (3g). Photolysis of a solution of 0.082 g (0.2 mmol) of acylsilane (**1a**) and 0.037 g (0.2 mmol) of diphenulhydrazine (**3g**) in 1 mL of C_6D_6 followed by removal of the solvent under reduced pressure gave a pale yellow oily residue. Chromatography on silica gel using hexane/ether (70:30) gave the colorless oily adduct **4ag** (65% yield). ^1H NMR: δ 0.24, 0.32, 0.35 (each 9H, s, $SiMe_3$), 1.6−1.98 (15H, br m, Ad), 3.65 (1H, s, CHOSi), 4.15 (1H, br s, NHPh), 6.5−8.3 (10H, m, Ph). ^{13}C NMR: δ 1.98, 2.04 ($SiMe_3$), 2.32 ($OSiMe_3$), 29.14 (Ad *C*H), 37.25 (Ad *C*H_2), 39.12 (C quat Ad), 40.54 (Ad *C*H_2), 80.75 (*C*HOSi), 126.42, 126.68, 126.92, 127.15, 127.38, 127.58 (Ph *C*H), 142.58, 143.63 (Ph ipso C). ^{29}Si NMR: δ −20.12, −18.90 ($SiMe_3$), −12.62 ($Si(SiMe_3)_2$), 12.82 ($OSiMe_3$). IR: 3438, 3018, 2968, 2887, 1610, 1554, 1500, 1468, 1443, 1408, 1273, 1260, 1149, 1112, 1985, 907, 768, 712 cm^{-1}. MS (EI): *m/z* (relative intensity) 579 $(M - Me)^+$ (14), 522 $(M + H - SiMe_3)^+$ (28), 503 $(M + H - C_6H_6N)^+$ (9), 412 $(M + H - C_{12}H_{11}N_2)^+$ (4), 357 $((Me_3Si)_2Si(C_{12}H_{11}N_2))^+$ (100), 339 $(412 - SiMe_3)^+$ (6), 237 $((Me_3SiO)CH(C_{10}H_{15}))^+$ (36), 191 $((Me_3Si)(Me_3SiO)SiH)^+$ (16), 135 $(C_{10}H_{15})^+$ (25), 77 $(C_6H_5)^+$ (14), 73 $(Me_3Si)^+$ (67). HRMS for $(M - Me)^+ = C_{31}H_{51}N_2OSi_4$: *m/z* calcd, 579.3065; found, 579.3062. Anal. Calcd for $C_{32}H_{54}N_2OSi_4$: C, 64.65; H, 9.09; N, 4.71. Found: C, 64.62; H, 9.05; N, 4.85.

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Photolysis of Pivaloyltris(trimethylsilyl)silane (1b) with Phenylhydrazine (3f). Following the procedure described above, the adduct **4bf** was produced as a colorless oil in 65% yield. ^{1}H NMR: δ 0.21, 0.26, 0.30 (9H, s, Me_3Si), 1.05 (9H, s, C*Me*$_3$), 1.26 (1H, br s, NHSi), 3.62 (1H, s, CHOSi), 3.68 (1H, br s, NHPh), 6.5−7.8 (5H, m, Ph). ^{13}C NMR: δ 1.92,1.96 (Me_3Si), 2.12 ($OSiMe_3$), 28.58 (C*Me*$_3$), 36.20 (*C*Me$_3$), 80.25 (CHOSi), 126.52, 126.95, 127.05 (Ph *C*H), 143.08 (C ipso Ph). ^{29}Si NMR: δ −20.45, −19.32 (Me_3Si), −13.15 ($Si(SiMe_3)_2$), 12.47 ($OSiMe_3$). IR: 3465, 3132, 2980, 2874, 1608, 1545, 1497, 1463, 1409, 1283, 1262, 1149, 1118, 1085, 910, 854, 793, 705 cm^{-1}. MS (EI): *m*/*z* (relative intensity) 426 $(M + H - Me)^+$ (6), 368 $(M + H - SiMe_3)^+$ (52), 348 $(M - C_6H_6N)^+$ (12), 334 $(M + H - C_6H_7N_2)^+$ (6), 225 $((Me_3Si)_2Si(C_6H_5N_2H_2))^+$ (100), 159 $((Me_3SiO)CH(C_4H_9))^+$ (10), 191 $((Me_3Si)(Me_3SiO)SiH)^+$ (23), 107 $(C_6H_5N_2H_2)^+$ (16), 92 $(C_6H_5NH)^+$ (14), 77 $(C_6H_5)^+$ (19), 73 $(Me_3Si)^+$ (68). HRMS for $(M - Me)^+ = C_{19}H_{41}N_2OSi_4$: *m*/*z* calcd, 425.2285; found, 425.2287. Anal. Calcd for $C_{20}H_{44}N_2OSi_4$: C, 54.54; H, 10.00; N, 6.36. Found: C, 54.32; H, 9.94; N, 6.48.

When a solution of 0.066 g (0.20 mmol) of acylsilane **1b** and 0.02 mL (0.20 mmol) of phenylhydrazine (**3f**) in 1 mL of C_6D_6 was stored under nitrogen in the dark for 2 weeks at room temperature, no reaction was observed to occur and only starting materials were detected by ^{1}H, ^{13}C, and ^{29}Si NMR spectra. When a similar solution was refluxed for 12 h, only starting materials were observed by NMR spectroscopy; after 48 h of refluxing the acylsilane had decomposed.

Photolysis of Pivaloyltris(trimethylsilyl)silane (1b) with Methylhydrazine (3d). Following a procedure similar to that described above, a viscous oil was obtained, which was shown by NMR spectroscopy to be a mixture of two isomeric adducts. This mixture was chromatographed on silica gel using hexane/ether (30:70) to separate into two isomeric adducts: $(Me_3Si)_2Si(NHNHMe)CH(OSiMe_3)$(*t*-Bu) (**4bd$_1$**) and $(Me_3Si)_2Si(NMeNH_2)CH(OSiMe_3)$(*t*-Bu) (**4bd$_2$**) in 35% (40 mg) and 25% (19 mg) yields, respectively. These adducts were characterized as follows.

4bd$_1$. ^{1}H NMR: δ 0.18, 0.20, 0.26 (each 9H, s, Me_3Si), 1.02 (9H, s, CMe_3), 1.12 (1H, br s, NHSi), 2.58 (3H, s, NMe), 3.25 (1H, br s, NHMe), 3.48 (1H, s, CHOSi). ^{13}C NMR: δ 1.86, 1.92 (Me_3Si), 2.18 ($OSiMe_3$), 28.53 (C*Me*$_3$), 35.83 (NMe), 36.65 (*C*Me$_3$), 80.32 (*C*HOSi). ^{29}Si NMR: δ −21.35, −20.62 (Me_3Si), −14.80 ($Si(SiMe_3)_2$), 12.15 ($OSiMe_3$). IR: 3420, 2958, 2870, 1435, 1418, 1275, 1258, 1168, 1163, 1090, 915, 825, 793 cm^{-1}. MS (EI): *m*/*z* (relative intensity) 364 $(M + H - Me)^+$ (9), 308 $(M + H - SiMe_3)^+$ (28), 349 $(M + H - CH_3NH)^+$ (10), 334 $(M + H - CH_3N_2H_2)^+$ (5), 322 $(M + H - CMe_3)^+$ (3), 219 $((Me_3Si)_2Si(CH_3N_2H_2))^+$ (100), 159 $((Me_3SiO)CH(C_4H_9))^+$ (21), 191 $((Me_3Si)(Me_3SiO)SiH)^+$ (16), 73 $(Me_3Si)^+$ (48). HRMS for $(M - Me)^+ = C_{14}H_{39}N_2OSi_4$: *m*/*z* calcd, 363.2129; found, 363.2132. Anal. Calcd for $C_{15}H_{42}N_2OSi_4$: C, 47.62; H, 11.11; N, 7.41. Found: C, 47.48; H, 11.18; N, 7.52.

4bd$_2$. ^{1}H NMR: δ 0.16, 0.18, 0.25 (each 9H, s, Me_3Si), 1.04 (9H, s, CMe_3), 3.18 (3H, s, NMe), 3.45 (1H, s, CHOSi), 3.72 (2H, br s, NH_2). ^{13}C NMR: δ 1.84, 1.89 (Me_3Si), 2.16 ($OSiMe_3$), 28.95 (C*Me*$_3$), 36.62 (*C*Me$_3$), 39.56 (NMe), 80.28 (*C*HOSi). ^{29}Si NMR: δ −21.38, −20.85 (Me_3Si), −14.95 ($Si(SiMe_3)_2$), 12.13 ($OSiMe_3$). IR: 3465, 3380, 2930, 2875, 1634, 1428, 1293, 1262, 1165, 1110, 1038, 920, 845, 787 cm^{-1}. MS (EI): *m*/*z* (relative intensity) 364 $(M + H - Me)^+$ (6), 308 $(M + H - SiMe_3)^+$ (18), 363 $(M + H - NH_2)^+$ (22), 334 $(M + H - CH_3N_2H_2)^+$ (4), 219 $((Me_3Si)_2Si(CH_3N_2H_2))^+$ (100), 159 $((Me_3SiO)CH(C_4H_9))^+$ (32), 191 $((Me_3Si)(Me_3SiO)SiH)^+$ (12), 73 $(Me_3Si)^+$ (54). HRMS for $(M - Me)^+ = C_{14}H_{39}N_2OSi_4$: *m*/*z* calcd, 363.2129; found, 363.2138. Anal. Calcd for $C_{15}H_{42}N_2OSi_4$: C, 47.62; H, 11.11; N, 7.41. Found: C, 47.56; H, 11.04; N, 7.85.

Photolysis of Pivaloyltris(trimethylsilyl)silane (1b) with Acetylhydrazine (3h). A solution of 0.081 g (0.24 mmol) of **1b** and 0.018 g (0.24 mmol) of **3h** in 1 mL of C_6D_6 was photolyzed for 18 h. Removal of the solvent gave a pale yellow oil, which was chromatographed on silica gel using hexane/ether (30:70) to give the colorless oily adduct **4bh** (64% yield). ^{1}H NMR: δ 0.23, 0.28, 0.32 (each 9H, s, Me_3Si), 1.08 (9H, s, CMe_3), 2.32 (1H, br s, NHSi), 2.68 (3H, s, MeCO), 3.65 (1H, s, CHOSi), 7.64 (1H, br s, NHAc). ^{13}C NMR: δ 1.96, 1.98 (Me_3Si), 2.18 ($OSiMe_3$), 15.83 (*Me*CO), 29.03 (C*Me*$_3$), 36.82 (*C*Me$_3$), 81.12 (*C*HOSi), 170.54 (*C*═O). ^{29}Si NMR: δ −20.25, −19.18 (Me_3Si), −12.56 ($Si(SiMe_3)_2$), 13.12 ($OSiMe_3$). IR: 3448, 2935, 2868, 1672, 1473, 1408, 1267, 1253, 1159, 1143, 1075, 918, 825, 795, 698, 650 cm^{-1}. MS (EI): *m*/*z* (relative intensity) 406 M^+ (<1), 391 $(M - Me)^+$ (3), 363 $(391 - CO)^+$ (45), 334 $(M + H - Me_3Si)^+$ (7), 205 $((Me_3Si)_2SiN_2H_3)^+$ (26), 191 $((Me_3Si)(Me_3SiO)SiH)^+$ (15), 159 $(Me_3SiO)CH(C_4H_9)^+$ (12), 73 $(Me_3Si)^+$ (100), 57 $(C_4H_9)^+$ (23). HRMS for **4bh**, $C_{16}H_{42}N_2O_2Si_4$: *m*/*z* calcd, 406.2312; found, 406.2315. Anal. Calcd for $C_{16}H_{42}N_2O_2Si_4$: C, 47.29; H, 10.34; N, 6.48. Found: C, 47.43; H, 10.58; N, 6.75.

Photolysis of Benzoyltris(trimethylsilyl)silane (1c) with Phenylhydrazine (3f). Following the general procedure described above, the adduct **4cf** was isolated (80% yield) as a colorless oil. ^{1}H NMR: δ 0.24, 0.28, 0.35 (each 9H, s, Me_3Si), 1.24 (1H, br s, NHSi), 3.28 (1H, s, CHOSi), 3.82 (1H, br s, NHPh), 6.80−7.32 (10H, m, Ph). ^{13}C NMR: δ 1.96, 2.08 (Me_3Si), 2.32 ($OSiMe_3$), 81.62 (*C*HOSi), 124.58, 125.32, 126.34, 126.85, 127.50, 128.12 (Ph CH), 139.95, 146.81 (Ph ipso C). ^{29}Si NMR: δ −20.38, −19.23 (Me_3Si), −13.16 ($Si(SiMe_3)_2$), 12.82 ($OSiMe_3$). IR: 3438, 3210, 2986, 2794, 1628, 1600, 1532, 1500, 1475, 1419, 1287, 1223, 1158, 1108, 1085, 908, 834, 763, 716 cm^{-1}. MS (EI): *m*/*z* (relative intensity) 461 $(M + H)^+$ (<1), 445 $(M - Me)^+$ (3), 388 $(M + H - Me_3Si)^+$ (38), 354 $(M + H - C_6H_5N_2H_2)^+$ (8), 281 $(354 - Me_3Si)^+$ (54), 179 $((Me_3SiO)CH(C_6H_5))^+$ (18), 191 $((Me_3Si)(Me_3SiO)SiH)^+$ (22), 77 $(C_6H_5)^+$ (16), 73 $(Me_3Si)^+$ (100). HRMS for $(M + H)^+ = C_{22}H_{41}N_2OSi_4$: *m*/*z* calcd, 461.2285; found, 461.2289. Anal. Calcd for $C_{22}H_{40}N_2OSi_4$: C, 57.39; H, 8.69; N, 6.09. Found: C, 57.62; H, 8.59; N, 6.23.

Photolysis of Adamantoyltris(trimethylsilyl)silane (1a) with Urea (5i). A solution of 0.0082 g (0.2 mmol) of acylsilane **1a** and 0.012 g (0.2 mmol) of urea (**5i**) in 1 mL of C_6D_6 was irradiated under nitrogen for 3 days, by which time all the reagents had been converted to the silacyclic product **7ai** and trimethylsilanol essentially quantitatively, as shown by NMR spectroscopy. Removal of solvent under reduced pressure gave a viscous oil, which was chromatographed on alumina using hexane/ether (70:30) to give the oily silacyclic product **7ai** (82% yield). Attempts to crystallize the material from a variety of solvents failed. ^{1}H NMR: δ 0.31, 0.38 (each 9H, s, Me_3Si), 1.40−1.85 (15H, m, Ad), 3.45 (1H, s, CHAd), 6.35 (1H, br s, HNCAd), 7.64 (1H, br s, NHSi). ^{13}C NMR: δ 0.32, 1.38 (Me_3Si), 29.38 (Ad *C*H), 36.76, 39.75 (C quat Ad), 40.65 (Ad *C*H$_2$), 64.60 (*C*HAd), 162.65 (*C*═O). ^{29}Si NMR: δ −19.48, −15.72 (Me_3Si), −9.56 (ring Si). IR: 3462, 3348, 2954, 2780, 1671, 1645, 1425, 1286, 1265, 1133, 1085, 898, 700, 670 cm^{-1}. MS (EI): *m*/*z* (relative intensity) 380 M^+ (21), 308 $(M + H - Me_3Si)^+$ (65), 246 $(M + H - Ad)^+$ (14), 172 $(246 - Me_3Si)^+$ (8), 73 $(Me_3Si)^+$ (100). HRMS for **7ai**, $C_{18}H_{36}N_2OSi_4$: *m*/*z* calcd, 408.1895; found, 408.2000. Anal. Calcd for $C_{18}H_{36}N_2OSi_4$: C, 56.84; H, 9.47; N, 7.37. Found: C, 56.89; H, 9.75; N, 7.45.

When a solution of 0.041 g (0.1 mmol) of acylsilane **1a** and 0.006 g (0.1 mmol) of urea (**5i**) in 0.5 mL of C_6D_6 was stored in the dark at room temperature under nitrogen for 5 days, no reaction occurred and only starting materials were observed by ^{1}H, ^{13}C, and ^{29}Si NMR spectra.

Addition of Urea (5i) to the Adamantylsilene 2a in the Dark. A solution of 0.041 g (0.1 mmol) of acylsilane **1a** in 0.5 mL of C_6D_6 was irradiated under nitrogen for 18 h to yield the equilibrium mixture of **1a** (minor) and its silene isomer **2a** (major), as monitored by NMR spectroscopy. To this solution was added 0.006 g (0.1 mmol) of urea (**5i**), and the mixture was kept in the dark for 24 h at room temperature. NMR spectroscopy indicated that all the silene had been converted to the silacyclic product **7ai**, which had NMR spectra as described above. In addition, the NMR spectra had signals which were attributed to trimethylsilanol produced in the reaction.

Photolysis of Adamantoyltris(trimethylsilyl)silane (1a) with *tert*-Butylurea (5k). Photolysis of 0.082 g (0.2 mmol) of acylsilane **1a** and 0.024 g (0.2 mmol) of *tert*-butylurea (**5k**) in 1 mL of C_6D_6 for 4 days gave a mixture of **7ak** and trimethylsilanol, as shown by NMR spectroscopy. After removal of the solvent under reduced pressure, the oily residue was chromatographed on alumina using hexane/ether (50:50) to yield the colorless oily silacyclic product **7ak** (58% yield). ^{1}H NMR: δ 0.32, 0.36 (each 9H, s, Me_3Si), 1.38−1.82 (15H, m, Ad), 1.42 (9H, s, C*Me*$_3$), 3.17 (1H, s, *C*HAd), 7.52 (1H, br s, NHSi). ^{13}C NMR: δ 0.31, 1.34 (Me_3Si), 28.71, 29.85 (Ad *C*H and C*Me*$_3$), 36.24, 40.62 (Ad *C*H$_2$), 40.05 (Ad quat C), 60.62 (N*C*Me$_3$), 67.52 (*C*HAd), 163.24 (*C*═O). ^{29}Si NMR: δ −18.62, −14.68 (Me_3Si), −8.98 (ring Si). IR: 3458, 2895, 2778, 1673, 1632, 1417, 1289, 1268, 1134, 1154, 890, 732, 677 cm^{-1}. MS (EI): *m*/*z* (relative intensity) 436 M$^+$ (15), 422 (M + H − Me)$^+$ (28), 380 (M − C_4H_8)$^+$ (23), 364 (M + H − Me_3Si)$^+$ (38), 349 (422 − Me_3Si)$^+$ (12), 307 (380 − Me_3Si)$^+$ (38), 302 (M + H − Ad)$^+$ (6), 283 (13), 73 (Me_3Si)$^+$ (100). Anal. Calcd for $C_{22}H_{44}N_2OSi_3$: C, 60.55; H, 10.09; N, 6.42. Found: C, 60.72; H, 10.16; N, 6.58.

Photolysis of Adamantoyltris(trimethylsilyl)silane (1a) with Phenylurea (5l). Following the procedure described above, the silacyclic product **7al** was formed together with trimethylsilanol, as shown by NMR spectroscopy. Removal of the solvent under reduced pressure followed by chromatography on silica gel using hexane/ether (40:60) gave the silacyclic product **7al** as a colorless oil in 65% yield. ^{1}H NMR: δ 0.34, 0.38 (each 9H, s, Me_3Si), 1.50−2.03 (15H, m, Ad), 3.86 (1H, s, CHAd), 6.5−7.8 (5H, m, Ph), 7.95 (1H, br s, HNSi). ^{13}C NMR: δ 0.35, 1.38 (Me_3Si), 28.65 (Ad *C*H), 36.38, 40.52 (Ad *C*H$_2$), 40.28 (Ad quat C), 73.68 (*C*HAd), 124.85, 128.42, 128.80 (Ph CH), 142.48 (Ph ipso C), 163.48 (*C*═O). ^{29}Si NMR: δ −18.23, −14.55 (Me_3Si), −8.75 (ring Si). IR: 3468, 3240, 2945, 2875, 1678, 1610, 1576, 1515, 1432, 1273, 1267, 1168, 1086, 908, 765, 714 cm^{-1}. MS (EI): *m*/*z* (relative intensity) 456 M$^+$ (18), 384 (M + H − Me_3Si)$^+$ (56), 381 (M − C_6H_3)$^+$ (15), 322 (M + H − Ad)$^+$ (8), 295 (16), 248 (M − Me_3Si − Ad)$^+$ (12), 73 (Me_3Si)$^+$ (100). Anal. Calcd for $C_{24}H_{40}N_2OSi_3$: C, 63.16; H, 8.77; N, 6.14. Found: C, 63.32; H, 8.69; N, 6.18.

Photolysis of Adamantoyltris(trimethylsilyl)silane (1a) with Methylurea (5j). A solution of 0.090 g (0.27 mmol) of acylsilane **1a** and 0.02 g (0.27 mmol) of methylurea (**5j**) in 1 mL of C_6D_6 was irradiated under nitrogen for 4 days, during which time the reagents were reacted almost quantitatively and a mixture of the two isomeric silacyclic products **7aj$_1$** and **7aj$_2$** (in the ratio of 70:30, as determined by the relative integral ratio of N−Me signals of the ^{1}H NMR spectrum of the reaction mixture) together with trimethylsilanol were produced, as indicated by NMR spectroscopy. Attempts to separate the isomers **7aj$_1$** and **7aj$_2$** by chromatography on alumina using a variety of solvents failed. ^{1}H NMR: δ 0.29, 0.31, 0.34, 0.36 (each 9H, s, Me_3Si), 1.40−1.85 (30H, m, Ad), 2.75 (3H, s, MeNSi), 2.87 (3H, s, MeNCAd), 3.42, 3.48 (each 1H, s, CHAd), 6.32 (1H, s, HNCAd), 7.75 (1H, br s, HNSi). ^{13}C NMR: δ 0.42, 0.45, 1.31, 1.38 (Me_3Si), 28.75, 28.81 (Ad *C*H), 34.62 (MeNSi), 35.83 (*Me*NCAd), 36.18, 36.23, 40.55, 40.60 (Ad *C*H$_2$), 40.12. 40.26 (Ad quat C), 64.48, 64.76 (*C*HAd), 163.26, 163.45 (*C*═O). ^{29}Si NMR: δ −18.58, −18.39, −14.65, −14.44 (Me_3Si), −8.79, −8.54 (ring Si). IR: 3458, 3398, 2976, 2874, 1678, 1675, 1435, 1387, 1280, 1265, 1108, 906, 827, 668 cm^{-1}. MS (EI): *m*/*z* (relative intensity) 394 M$^+$ (18), 379 (M − Me)$^+$ (28), 322 (M + H − Me_3Si)$^+$ (48), 245 (379 + H − Ad)$^+$ (12), 189 (22), 174 (8), 73 (Me_3Si)$^+$ (100). Anal. Calcd for $C_{19}H_{38}N_2OSi_3$: C, 57.87; H, 9.64; N, 7.11. Found: C, 57.72; H, 9.75; N, 7.38.

Photolysis of Pivaloyltris(trimethylsilyl)silane (1b) with Urea (5i). A solution of 0.083 g (0.25 mmol) of acylsilane **1b** and 0.015 g (0.25 mmol) of urea (**5i**) in 1 mL of C_6D_6 was irradiated under nitrogen for 3 days, during which time all the reagents were almost quantitatively converted to the silacyclic product **7bi** and trimethylsilanol, as shown by NMR spectroscopy. After concentration of the reaction mixture at reduced pressure, the residue was chromatographed on alumina using hexane/ether (80:20) to afford the silacyclic product as a colorless oil. ^{1}H NMR: δ 0.30, 0.36 (each 9H, s, Me_3Si), 0.95 (9H, s, C*Me*$_3$), 3.32 (1H, s, *C*HCMe$_3$), 6.48 (1H, br s, NHCCMe$_3$), 7.42 (1H, br s, NHSi). ^{13}C NMR: δ 0.68, 1.39 (Me_3Si), 28.95 (C*Me*$_3$), 36.45 (*C*Me$_3$), 78.38 (*C*HCMe$_3$), 162.85 (*C*═O). ^{29}Si NMR: δ −19.52, −15.49 (Me_3Si), −9.82 (ring Si). IR: 3456, 3379, 2984, 2863, 1675, 1625, 1460, 1285, 1232, 1183, 1105, 895, 732 cm^{-1}. MS (EI): *m*/*z* (relative intensity) 303 M$^+$ (18), 289 (M + H − Me)$^+$ (8), 247 (M − C_4H_8)$^+$ (32), 231 (M + H − Me_3Si)$^+$ (55), 174 (247 − Me_3Si)$^+$ (6), 73 (Me_3Si)$^+$ (100). HRMS calcd for **7bi**, $C_{12}H_{30}N_2OSi_3$: *m*/*z* calcd, 302.1658; found, 302.1661. Anal. Calcd for $C_{12}H_{30}N_2OSi_3$: C, 47.68; H, 9.93; N, 9.27. Found: C, 47.75; H, 10.23; N, 9.48.

Photolysis of Pivaloyltris(trimethylsilyl)silane (1b) with 1,3-Dimethylurea (5m). Following the procedure described above, a yellow oily solution containing **7bm** and trimethylsilanol was produced, as confirmed by NMR spectroscopy. Concentration under reduced pressure followed by chromatography on alumina using hexane/ether (70:30) gave the colorless oily product **7bm** in 65% yield. ^{1}H NMR: δ 0.32, 0.37 (each 9H, s, Me_3Si), 0.93 (9H, s, CMe$_3$), 2.78 (3H, s, *Me*NCCMe$_3$), 2.95 (3H, s, MeNSi), 3.29 (1H, s, CHCMe$_3$). ^{13}C NMR: δ 0.65, 1.36 (Me_3Si), 28.82 (C*Me*$_3$), 33.74 (*Me*NSi), 35.62 (*Me*NCCMe$_3$), 36.34 (*C*Me$_3$), 78.32 (*C*HCMe$_3$), 163.25 (*C*═O). ^{29}Si NMR: δ −19.48, −15.44 (Me_3Si), −9.85 (ring Si). IR: 2975, 2834, 1673, 1579, 1463, 1257, 1212, 1180, 1087, 845, 745 cm^{-1}. MS (EI): *m*/*z* (relative intensity) 330 M$^+$ (23), 316 (M + H − Me)$^+$ (14), 274 (M − C_4H_8)$^+$ (18), 258 (M + H − Me_3Si)$^+$ (62), 201 (274 − Me_3Si)$^+$ (8), 73 (Me_3Si)$^+$ (100). Anal. Calcd for $C_{14}H_{34}N_2OSi_3$: C, 50.91; H, 10.30; N, 8.48. Found: C, 51.13; H, 10.38; N, 8.63.

Photolysis of Pivaloyltris(trimethylsilyl)silane (1b) with Phenylurea (5l). A solution of 0.083 g (0.25 mmol) of acylsilane **1b** and 0.034 g (0.25 mmol) of phenylurea (**5l**) in 1 mL of C_6D_6 was photolyzed under nitrogen for 5 days, at which time the reagents were converted to the silacyclic product **7bl** and trimethylsilanol. The resulting yellow solution was concentrated under reduced pressure, and the oily residue was chromatographed on alumina using hexane/ether (50:50) to yield the silacyclic product **7bl** (50% yield) as a colorless oil. ^{1}H NMR: δ 0.35, 0.38 (each 9H, s, Me_3Si), 0.98 (9H, s, CMe$_3$), 3.68 (1H, s, CHCMe$_3$), 6.3−7.6 (5H, m, Ph), 7.92 (1H, br s, NHSi). ^{13}C NMR: δ 0.65, 1.38 (Me_3Si), 27.36 (C*Me*$_3$), 34.28 (*C*Me$_3$), 78.95 (*C*HCMe$_3$), 124.63, 128.34, 128.95 (Ph CH), 143.12 (Ph ipso C), 163.32 (*C*═O). ^{29}Si NMR: δ −18.35, −14.52 (Me_3Si), −8.72 (ring Si). IR: 3478, 3184, 2972, 2795, 1677, 1630, 1605, 1500, 1452, 1289, 1237, 1160, 1123, 898, 763, 744 cm^{-1}. MS (EI): *m*/*z* (relative intensity) 378 M$^+$ (16), 364 (M + H − Me)$^+$ (10), 322 (M − C_4H_8)$^+$ (22), 306 (M + H − Me_3Si)$^+$ (47), 249 (322 − Me_3Si)$^+$ (8), 73 (Me_3Si)$^+$ (100). Anal. Calcd for $C_{18}H_{34}N_2OSi_3$: C, 57.14; H, 8.99; N, 7.41. Found: C, 57.28; H, 9.21; N, 7.48.

Photolysis of Benzoyltris(trimethylsilyl)silane (1c) with *tert*-Butylurea (5k). A solution of 0.070 g (0.2 mmol) of acylsilane **1c** and 0.024 g (0.2 mmol) of *tert*-butylurea (**5k**) in 1 mL of C_6D_6 was photolyzed under nitrogen at room temperature for 5 days. The resulting yellow solution contained the expected silacyclic product **7ck** together with trimethylsilanol, as shown by its NMR spectra. The yellow oily residue remaining after the removal of the solvent at reduced pressure was chromatographed on alumina using hexane/ether (80:20) to give the colorless oily product **7ck** (55% yield). ^{1}H NMR: δ 0.32, 0.35 (each 9H, s, Me_3Si), 1.43 (9H, s, CMe$_3$), 3.28 (1H, s, CHPh), 6.35−7.60 (6H, m, Ph and NHSi). ^{13}C NMR: δ 0.33, 1.32 (Me_3Si), 31.28 (C*Me*$_3$), 59.90 (*C*Me$_3$), 63.43

(*C*HPh), 126.72, 127.23, 127.64 (Ph CH), 143.80 (Ph ipso C), 162.95 (*C*═O). ^{29}Si NMR: δ −18.28, −14.75 (Me_3Si), −9.25 (ring Si). IR: 3457, 2885, 2764, 1674, 1638, 1407, 1275, 1258, 1109, 1052, 970, 782, 654 cm^{-1}. MS (EI): *m*/*z* (relative intensity) 378 M$^+$ (20), 364 $(M + H - Me)^+$ (19), 322 $(M - C_4H_8)^+$ (8), 306 $(M + H - Me_3Si)^+$ (42), 215 (3), 73 $(Me_3Si)^+$ (100). HRMS for **7ck**, $C_{18}H_{34}N_2OSi_3$: *m*/*z* calcd, 378.1970; found, 378.1973. Anal. Calcd for $C_{18}H_{34}N_2OSi_3$: C, 57.14; H, 8.99; N, 7.41. Found: C, 57.36; H, 9.18; N, 7.48.

Photolysis of Benzoyltris(trimethylsilyl)silane (1c) with 1,3-Diphenylurea (5n). Following the procedure described above, the silacyclic product **7cn** was obtained as a colorless oil. ^{1}H NMR: δ 0.36, 0.40 (each 9H, s, Me_3Si), 3.90 (1H, s, CHPh), 6.30−7.85 (15H, m, Ph). ^{13}C NMR: δ 0.36, 1.42 (Me_3Si), 68.75 (*C*HPh), 124.45, 124.85, 126.72, 127.23, 127.64, 127.95, 128.36, 128.45, 128.78 (Ph CH), 142.48, 143.30, 143.80 (Ph ipso C), 164.08 (*C*═O). ^{29}Si NMR: δ −18.32, −14.58 (Me_3Si), −9.38 (ring Si). IR: 3145, 3082, 2872, 2787, 1682, 1608, 1524, 1490, 1398, 1269, 1278, 1153, 1129, 915, 784, 735 cm^{-1}. MS (EI): *m*/*z* (relative intensity) 474 M$^+$ (24), 402 $(M + H - Me_3Si)^+$ (38), 398 $(M - C_6H_4)^+$ (12), 325 $(M - Me_3Si - C_6H_4)^+$ (16), 90 $(C_7H_6)^+$ (22), 73 $(Me_3Si)^+$ (100). Anal. Calcd for $C_{26}H_{34}N_2OSi_3$: C, 65.82; H, 7.17; N, 5.91. Found: C, 66.08; 7.24; N, 5.97.

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