Organometallics 1985, 4, 623–629

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Registry No. $FcC{\equiv}CH$, 1271-47-2; $Hg(OAc)_2$, 1600-27-7; $(FcC{\equiv}C)_2Hg$, 94619-64-4; $FcCOCH_3$, 1271-55-2; $FcC{\equiv}CI$, 94598-10-4; $FcC{\equiv}CHgOHgOAc$, 94598-11-5; $FcC(OAc){=}CH_2$, 60732-82-3; $FcC(OCH_3){=}CH_2$, 94598-12-6; $PhC{\equiv}CH$, 536-74-3; $PhC{\equiv}CI$, 932-88-7; ICH_2COPh, 4636-16-2; $AcOHgCH_2COPh$, 5653-24-7; $PhCOCH_3$, 98-86-2; $PhC(OCH_3){=}CH_2$, 4747-13-1; $PhC(OCH_3){=}CHI$, 94598-14-8; $PHC{\equiv}CC{\equiv}CPh$, 886-66-8; $PhC(OH)(OCH_3)CH_2I$, 94598-15-9; $FcCOCH_2I$, 54804-01-2; $FcCOCH_2HgOHgOCOCH_3$, 94598-13-7.

Silyliron Carbonyl Complexes in Organic Synthesis: Selective Conversion of Nitriles into *N*,*N*-Bis(silyl) Enamines

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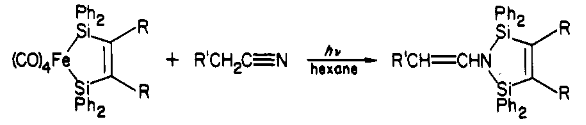
The readily available chelated bis(silyl)iron complex $(CO)_4\overline{FeSi(CH_3)_2\text{-}o\text{-}C_6H_4Si}(CH_3)_2$ reacts photochemically with nitriles, RCH_2CN, to give *N*,*N*-bis(silyl) enamines $RCH{=}CH\overline{NSi(CH_3)_2\text{-}o\text{-}C_6H_4Si}(CH_3)_2$. The scope of this new transformation of nitriles into enamines has been studied. Aliphatic and benzylic nitriles give high yields of a *Z* and *E* mixture of enamines. With dinitriles, the reaction occurs only with the cyano which has an α-hydrogen atom. Halonitriles, methoxyacetonitrile, and ethyl cyanoacetate also undergo a selective reaction affording good yields of functional enamines. In contrast, ketonitriles require protection of the carbonyl group. The reaction of allylic cyanides gives rise to *N*,*N*-bis(silyl) dienamines and to new $\{\eta^4$-[*N*,*N*-bis(silyl)amino]dienyl$\}$iron carbonyl complexes. The *N*,*N*-bis(silyl) enamines obtained give aldehydes upon hydrolysis with dilute acid. They are stable in the presence of electrophiles and nucleophiles and can be used as protected aldehydes. The reaction of the iron complex with nitriles that involve a transfer of the silyl ligand from the iron to the nitrogen atom and migration of one hydrogen atom α to the cyano group is interpreted as proceeding via an intermediate iron carbonyl carbene complex.

Introduction

Organosilicon compounds now constitute a well recognized class of reagents and intermediates in organic synthesis.[1] Our search for new silyl reagents for organic synthesis led us to study the possibilities offered by organosilyl transition-metal complexes.[2] The peculiar behavior of the silyl ligand has allowed to find new reactions affording new access to organometallic compounds[3,4] and novel chemical transformations of some organic functional groups.[5,7] This paper gives a detailed account of our studies of the reactions of a chelated bis(silyl)iron carbonyl complex with nitriles.

A preliminary study on the reactivity of bis(silyl)iron carbonyl complexes revealed that their photochemical reactions with nitriles produce *N*,*N*-bis(silyl) enamines[4a] (eq 1). The silyliron complexes are stoichiometric reagents for the conversion of nitriles into silylenamines. We report here that a readily available iron carbonyl complex can achieve this new chemical transformation of nitriles. The scope of the reaction has been examined. It provides easy access to a variety of *N*,*N*-bis(silyl) enamines[8-10] which were recently shown to be precursors of substituted 2-aza-1,3-butadienes.[11] A portion of this study has already been communicated.[6]



(1)

R = Et, R' = H, 84%; R = Et, R' = Ph, 75%, *E*/*Z* = 45/55; R = R' = Me, 71%, *E*/*Z* = 42/58

Results

A readily available chelated bis(silyl)iron carbonyl complex is 1 first reported by Fink.[12] It is obtained in two steps from *o*-dibromo- or *o*-dichlorobenzene according to a slight modification of the method described by Fink. 1,2-Bis(dimethylsilyl)benzene was easily prepared by use

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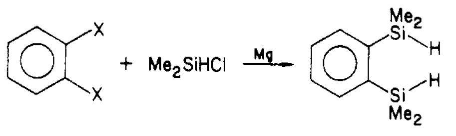
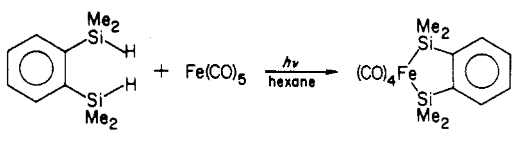
of the silylation method of Calas and Dunogues[13] (eq 2). The iron complex 1 then was obtained by reaction of the hydrosilane with iron pentacarbonyl. It was isolated as colorless, air-stable crystals in 60% average yield (eq 3).

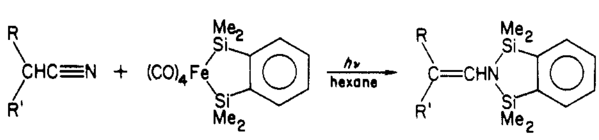

(2)

X=Cl, Mg/HMPT, yield 40%, X=Br, Mg/THF, yield 78%


(3)

1

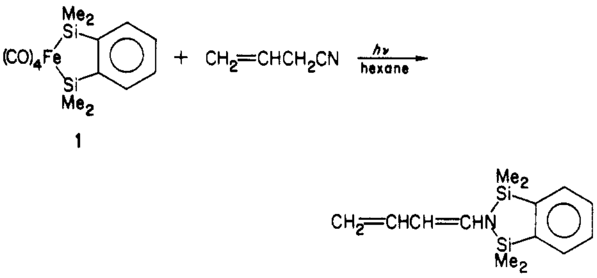
We have examined the reactivity of complex 1 toward nitriles. Exposing hexane solutions of equimolar amounts of a nitrile and complex 1 to UV irradiation for several hours afforded disilylated enamines in good yields (eq 4). The results are presented in Table I.


(4)

1 2-21

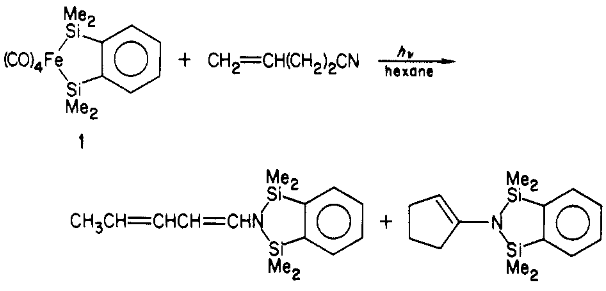
Aliphatic nitriles and benzylic nitriles (entries 1–6) gave 70–95% yields of bis(silyl) enamines as a mixture of *E* and *Z* isomers. Some variations in the *E*:*Z* ratio were observed as a result of a cis–trans isomerization under the reaction conditions.[9b] The reaction of (trimethylsilyl)acetonitrile (entry 7) gave a mixture of three isomeric enamines. The major isomers were the expected *E* and *Z* aldehyde enamines. Formation of an acylsilane enamine[8e,10] in low yield due to the migration of the trimethylsilyl group also was observed. The reaction of nitriles thus appeared quite general and allowed easy access to a variety of *N*,*N*-bis(silyl) enamines. The wide applicability of the procedure is illustrated by the conversion of functional nitriles into the corresponding functional enamines. In the case of dinitriles, the expected cyanoenamines were obtained (entries 8–11). Malononitrile gave a (cyanovinyl)amine; succinonitrile and adiponitrile gave the corresponding cyano enamine with a good selectivity. Also of interest is the reaction of α-cyanotoluonitrile (entry 11) in which only the cyano group having an α-hydrogen atom undergoes the reaction. Nitriles containing other functional groups also were selectively converted to functional enamines. The reactions of halonitriles yielded the halo enamines (entries 12–14). A low yield of enamine was, however, obtained in the case of chloroacetonitrile due to uncomplete reaction. Here the reaction appeared to be much slower. Methoxyacetonitrile, in contrast, gave a good yield of the methoxy enamine (entry 15). The reaction of 3-(trimethylsilyl)oxypropionitrile (entry 16) led to the propenylamine by loss of the (trimethylsilyl)oxy group. Such a cleavage of a silyl ether was also observed with a silyliron complex.[7a] The use of aminopropionitrile allowed isolation of an amino enamine (entry 17). Loss of the acetate substituent was observed in the reaction of 3-acetoxypropionitrile (entry 19), but ethyl cyanoacetate reacted to give the enamino ester (entry 18). Finally in the case of keto nitriles, good yields were obtained only for a protected carbonyl group (entries 20, 21).

From these various experiments, it appears that the conversion of nitriles to *N*,*N*-bis(silyl)enamines by use of the silyliron reagent 1 shows good selectivity; different functional groups are tolerated. With the object of synthesizing dienamines, we also studied the reactions of olefinic nitriles. The reaction of allyl cyanide (eq 5) gave the expected dienic amine 22, although in moderate yield. Surprisingly the reaction of 4-cyanobut-1-ene led to a mixture of two enamines (eq 6). A linear conjugated dienamine 23 was isolated in 30% yield. Its formation probably is due to isomerization of the initially formed unconjugated olefinic enamine. Such an isomerization of olefinic silylamines has been observed in the presence of iron carbonyl as catalyst.[9b] Besides the linear dienamine, a cyclic enamine 24 also was isolated in 15% yield. The reactions of other olefinic nitriles such as cyclohex-1-enylacetonitrile or the cyano enamine prepared form succinonitrile, gave the dienamine iron carbonyl complexes (eq 7 and 8). The iron moiety, which was not recovered in


1

(5)

22, yield 30%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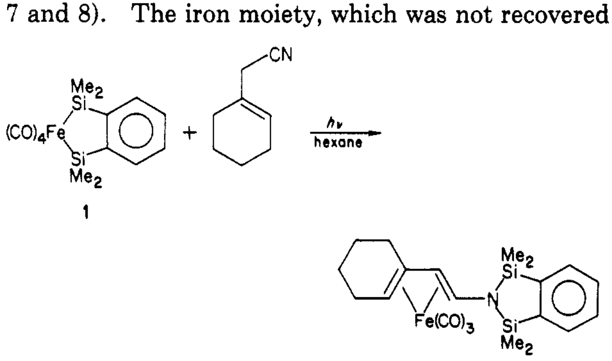
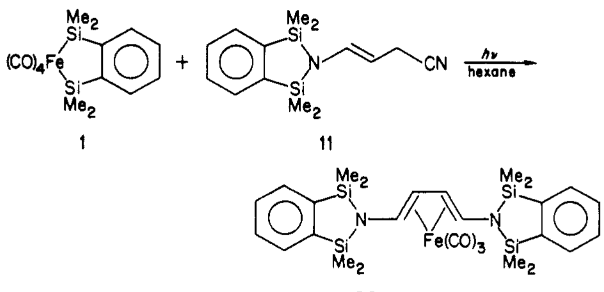
(6)

23, yield 30% 24, yield 15%


1

(7)

25, yield 30%


1 11

(8)

26, yield 70%

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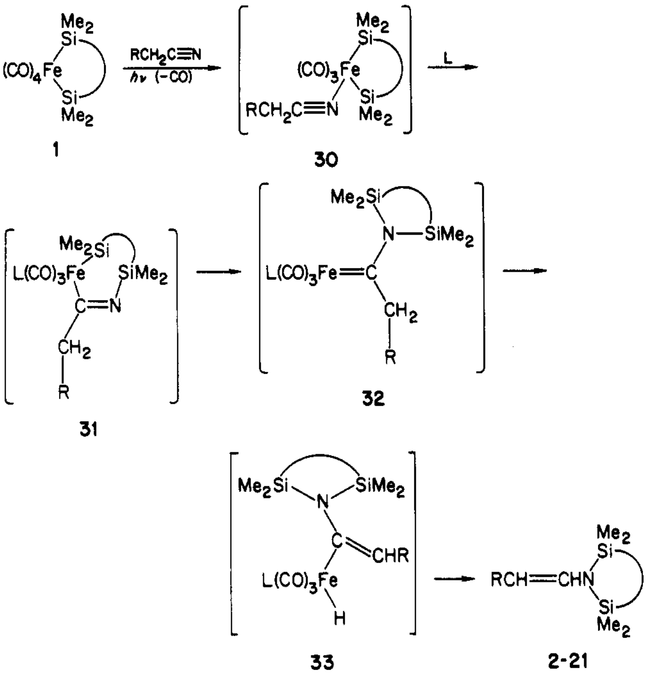
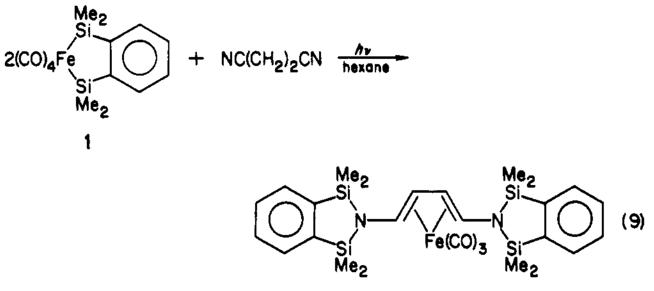
Scheme I. Possible Mechanism for the Conversion of Nitriles into *N*,*N*-Disilylated Enamines



L=CO, $RCH_2C{\equiv}N$

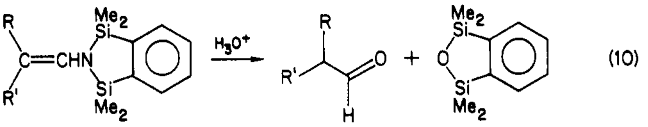
the previous cases, retained the dienic product in its coordination sphere, and the iron carbonyl complexes **25** and **26** were isolated, thus providing an interesting route to new functional (diene)iron carbonyl complexes. The (diamino diene)iron complex **26** also was obtained in a one-step reaction from succinonitrile by the use of 2 mol of the iron reagent **1** (eq 9).



26, yield 50%

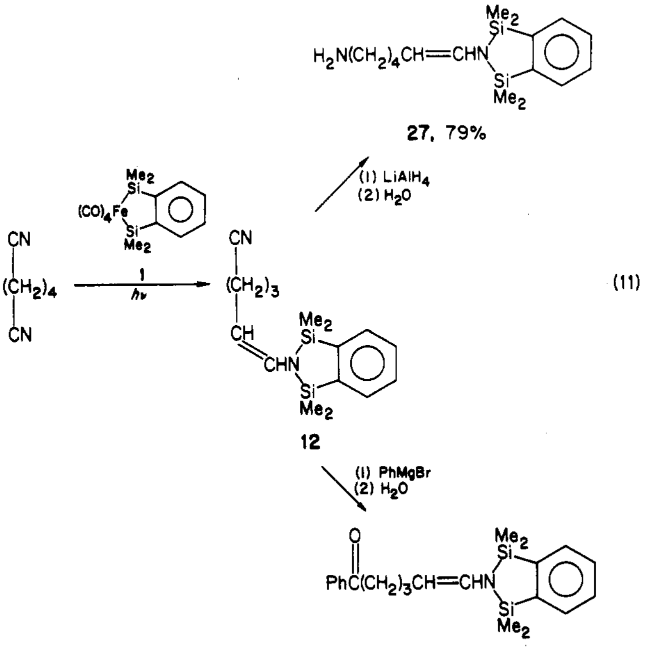
The photochemical reaction of complex **1** with nitriles offers a quite general route to *N*,*N*-bis(silyl)enamines for which few general methods have been reported.[8-10] We also examined the reactivity of these new enamines. *N*,*N*-dialkyl enamines are nucleophilic reagents and react with a large variety of electrophiles with carbon–carbon bond formation.[14] In contrast, the *N*,*N*-disilylated enamines appeared to be weak nucleophiles. They are quite stable and did not hydrolyze under neutral conditions. However, hydrolysis with dilute HCl led to the corresponding aldehydes which were isolated as their (2,4-dinitrophenyl)hydrazone derivatives (eq 10).

The propenylamine **3**, obtained from propionitrile, failed to react with benzoyl chloride, benzyl bromide, or activated olefins such as ethyl acrylate or acrylonitrile, under the reaction conditions used for carbon enamines.[14] The bis(silyl) enamines also appeared to be quite stable toward



R, R' (yield of pure recrystallized (2,4-dinitrophenyl)hydrazone): H, H (65%) H, Me (82%); H, Ph (79%); H, $NC(CH_2)_3$- (79%); H, EtOOC (73%); Me, Me (68%); Ph, Ph (84%)

nucleophiles. No cleavage of the nitrogen–silicon bond was observed with nucleophiles such as $LiAlH_4$ and Grignard or organolithium reagents. This remarkable stability is illustrated by some reactions of functional enamines. The conversion of one cyano group to an enamine in adiponitrile can be achieved, and nucleophilic reactions can then be performed on the remaining cyano group (eq 11).



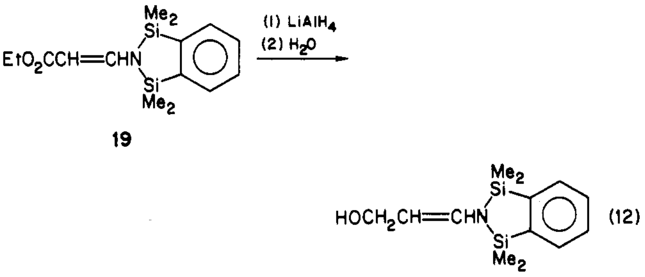
27, 79%

12

28, 43%

The cyano enamine **12** was reduced to the amino enamine **27** by $LiAlH_4$. Similarly, phenylmagnesium bromide reacted with the nitrile function of **12** to give the keto enamine **28**. In both cases mild hydrolysis did not affect the enamine function.

The lithium aluminum hydride reduction of the enamino ester **20** to the corresponding enamino alcohol **31** also was achieved (eq 12).



19

29, 39%

Discussion and Mechanism

By use of the chelated iron complex **1** a wide range of enamines can be prepared. It is noteworthy that a selective transformation of the cyano group was obtained in most cases. The photochemical reaction of complex **1** thus constitutes a general route to *N*,*N*-bis(silyl) enamines from nitriles. Moreover, these enamines exhibit a different reactivity compared to the *N*,*N*-dialkyl analogues. They are quite inert toward electrophiles and nucleophiles.

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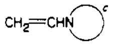
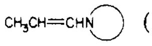
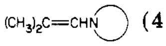
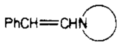
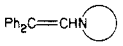
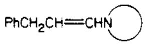
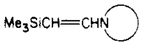
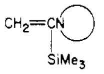
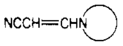
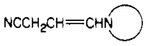
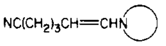
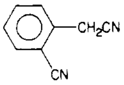
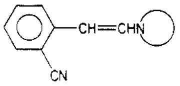
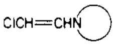
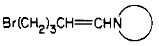
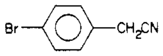
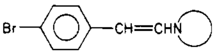
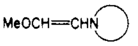
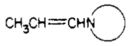
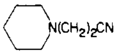
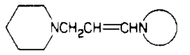
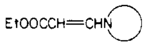
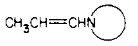
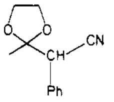
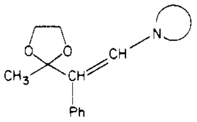
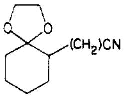
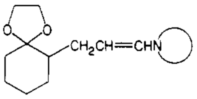
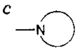
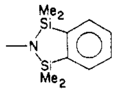
[14] (a) Cook, A. G. "Enamines: Synthesis, Structure and Reactions"; Marcel Dekker: New York, 1969. (b) Hickmott, P. W. *Tetrahedron* **1982**, *38*, 1975. (c) *Ibid.* **1982**, *38*, 3363.

Table I. Conversion of Nitriles into *N*,*N*-Bis(silyl) Enamines[a]

entry	nitrile	reactn time, h	enamine	*E*/*Z*[b]	yield, %
1	CH_3CN	30	 (2)		70
2	CH_3CH_2CN	30	 (3)	65/35	85
3	$(CH_3)_2CHCN$	30	 (4)		80
4	$PhCH_2CN$	21	 (5)	55/45	95
5	Ph_2CHCN	20	 (6)		78
6	$PhCH_2CH_2CN$	30	 (7)	62/38	78
7	Me_3SiCH_2CN	30	 76 (8)  24 (9)	89/11	51
8	$NCCH_2CN$	40	 (10)	87/13	50
9	$NC(CH_2)_2CN$	37	 (11)	65/35	50
10	$NC(CH_2)_4CN$	30	 (12)	18/82	70
11		30	 (13)	50/50	50
12	$ClCH_2CN$	60	 (14)	65/35	30
13	$Br(CH_2)_4CN$	50	 (15)	*d*	70
14		60	 (16)	25/75	63
15	$MeOCH_2CN$	43	 (17)	40/60	70
16	$Me_3SiO(CH_2)_2CN$	50	 (3)	65/35	45
17		63	 (18)	0/100	40
18	$EtOOCCH_2CN$	6	 (19)	100/0	83
19	$CH_3COO(CH_2)_2CN$	80	 (3)	65/35	40
20		76	 (20)	*e*	60
21		30	 (21)	56/44	75

[a] Reaction were carried out by irradiating a hexane solution of a 1:1 mixture of nitrile and complex 1 in a quartz reaction vessel with a high-pressure 450-W mercury lamp. [b] *E*/*Z* ratio was determined by NMR spectroscopy at 100 MHz. [c]  stands for  [d] *E*/*Z* ratio was not determined. [e] NMR spectroscopy indicated the presence of two isomers in a 70/30 ratio; *E* and *Z* configurations have not been assigned.

However, acid hydrolysis to aldehydes occurs readily. The iron complex 1 thus is a reagent for the conversion of nitriles to aldehydes via bis(silyl) enamines. Such a conversion, owing to its selectivity in the case of functional nitriles, is of special interest. Relatively few selective methods for the conversion of nitriles to aldehydes are available.[15] On the other hand, owing to their remarkable stability toward both electrophiles and nucleophiles, *N*,*N*-bis(silyl) enamines can be used as masked aldehydes. Moreover, we recently reported that bis(silyl) enamines are interesting precursors of substituted 2-aza-1,3 butadiene.[11]

From a mechanistic point of view the conversion of nitriles to *N*,*N*-bis(silyl) enamines by iron complex 1 seems to occur via a transfer of the silyl ligand from the iron atom to the nitrogen atom and a migration of one hydrogen atom α to the cyano group of the nitrile to the carbon α to the nitrogen atom. This is confirmed by the reaction of (trimethylsilyl)acetonitrile (Table I, entry 7) from which

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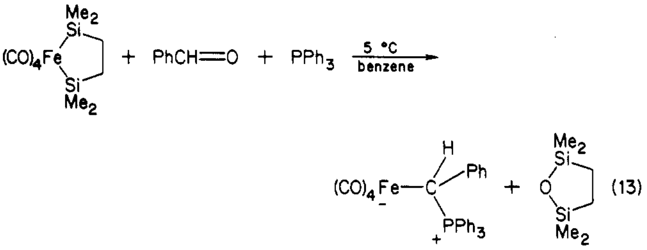
[15] Fry, J. L.; Ott, R. A. *J. Org. Chem.* **1981**, *46*, 602 and references cited therein.

enamines **8** and **9** were obtained. Enamines **8** corresponds to the migration of the hydrogen atom, whereas enamine **9** corresponds to a competing migration of the trimethylsilyl group. We have not studied the mechanism of the reaction of complex **1** with nitriles. However, a proposal is shown in Scheme I.

In the first step, we suppose that a photolabilization of one carbon monoxide ligand allows the coordination of a molecule of nitrile to the iron atom. This displacement of a carbon monoxide ligand, in a bis(silyl)iron carbonyl complex occurs easily under UV irradiation.[4] We propose that insertion of the cyano group into one of the iron–silicon bond then occurs, leading to intermediate **31**. The migration of the silicon atom to the nitrogen atom then affords a carbene complex **32**. From this intermediate a σ-vinyl complex, **33**, can be obtained via β-elimination. The *N*,*N*-bis(silyl) enamine then is formed in a reductive elimination step. The intervention of an iron carbene complex **34**[16] is supported by the isolation of iron carbene complexes in a related reaction reported by Gladysz and co-workers.[3d] The reaction of benzaldehyde with a chelated bis(silyl)iron carbonyl complex was proposed to occur by way of an insertion into a silicon–iron bond followed by elimination of a cyclic disiloxane giving a benzylidene complex which was trapped by triphenylphosphine as an ylide complex (eq 13). Moreover, an equilibrium between carbene and σ-alkenyl complexes similar to the conversion of **32** to **33** has been envisaged already as a mechanism for the initial carbene formation in olefin metathesis over fixed molybdenum catalysts.[17]

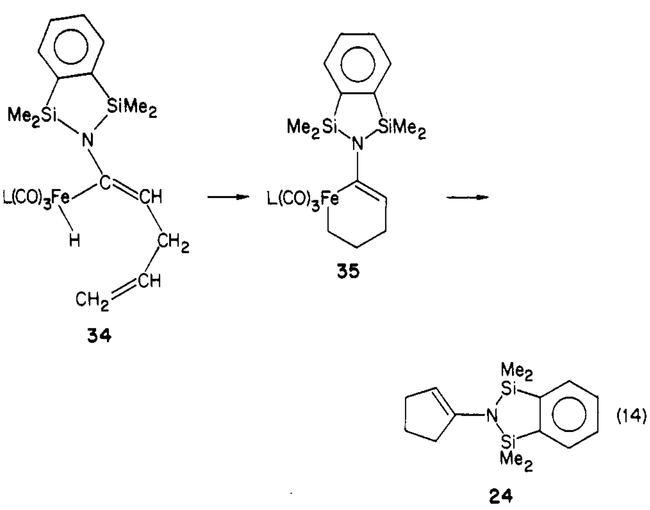


Usually the iron moiety was not recovered with the exception of some reactions of allylic nitriles in which (η^4-dienyl)iron tricarbonyl complexes **25** and **26** were produced. The iron carbonyl unit liberated in the reductive elimination step in this case remained coordinated to the dienamine.

The formation of the cyclopentanone enamine **24** in the reaction of 4-cyano-butene-1 with complex **1** (eq 6) also can be explained by the mechanism presented in Scheme I. Its formation may be due to intramolecular addition of the intermediate iron hydride complex **34**. The ferracyclohexene **35** thus formed can liberate the cyclic enamine by reductive elimination (eq 14).

Conclusion

Silyl transition-metal complexes represent a new class of potentially useful reagents for organic synthesis. This study has provided a facile conversion of nitriles into new *N*,*N*-bis(silyl)enamines by use of a silyliron carbonyl complex as a stoichiometric reagent. Bis(silyl) enamines exhibit a reactivity which differs from that of the well-known carbon enamines. Our continuing studies of the reactivity and of the uses of these new silylamino reagents in organic synthesis will be the subject of further reports.



34 35 24 (14)

Experimental Section

General Remarks. All reactions were carried out under an inert atmosphere. Hexane was distilled from sodium and saturated with nitrogen before use. Hexamethylphosphotriamide (HMPT) was distilled from sodium under reduced pressure. Nitriles were obtained from common commercial sources. Infrared spectra were recorded on a Perkin-Elmer 298 spectrophotometer in the form indicated. The ^{1}H NMR spectra were measured on a Varian EM 360 or EM 390 spectrometer. Chemical shifts (δ) are relative to Me_4Si. The mass spectra were obtained on a JEOL JMS D100 apparatus. Elemental analyses were carried out by the Service Central de Microanalyse du CNRS. All photochemical reactions were performed at room temperature, with use of a Hanovia 450-W high-pressure mercury immersion lamp in a quartz reaction vessel.

Preparation of 1,2-Bis(dimethylsilyl)benzene. From *o*-Dichlorobenzene. The compound was obtained following a procedure described by Calas and co-workers[13c] for the polytrimethylsilylation of halobenzenes. To a mixture containing 11.2 g (0.47 mol) of magnesium and 44 g (0.47 mol) of *o*-dichlorobenzene dissolved in 140 mL of HMPT was added dropwise a solution of 27.5 g of dimethylchlorosilane in 50 mL of HMPT. After the addition was complete, the mixture was heated to 80 °C for 48 h. The reaction mixture then was cooled to room temperature and poured into 500 mL of cold water. After extraction with ether, the organic layer was washed three times with water and then dried over $MgSO_4$. Evaporation of ether and distillation of the residue gave 17.6 g, bp 95–100 °C at 23 torr (yield 40%), of the known 1,2-bis(dimethylsilyl)benzene.[13b]

From *o*-Dibromobenzene. The procedure reported by Fink[13b] was used without modification.

Preparation of Bis(silyl)iron Carbonyl Complex 1. A solution containng 12 mL (90 mmol) of iron pentacarbonyl and 17.6 g (90 mmol) of 1,2-bis(dimethylsilyl)benzene in 600 mL of dry degassed hexane was irradiated for 15 h with an immersed 450-W high-pressure mercury lamp in a quartz reaction vessel. The solvent then was evaporated under reduced pressure, and to the brown residue was added 40 mL of degassed *n*-pentane. After filtration, the pentane solution was allowed to stand at −78 °C for 48 h. The brownish crystals obtained were collected, washed with *n*-pentane (cooled to −78 °C), and dried in vacuo. The solid obtained was recrystallized first from *n*-pentane and then from acetonitrile, affording 19.6 g (yield 60%) of off-white crystals: mp 46–47 °C (lit.[12] mp 46–47 °C); IR (CCl_4, cm^{-1}) ν(CO) 2070, 1990, 1950; NMR (CCl_4, δ) 0.7 (s, 12 H), 7.35 (m, 4 H).

The procedure has been scaled up successfully starting with 0.2 mol of 1,2-bis(dimethylsilyl)benzene. About 40 g of complex **1** are routinely obtained in yields ranging from 50 to 70%.

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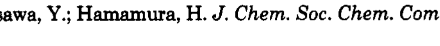
[16] An alternate mechanism without the intervention of the carbene complex **32** has been suggested by one of the reviewers. A pathway involving a 1,2-heterodiene complex formed by a β-elimination reaction in the intermediate complex **31** can also be considered.



[17] Iwasawa, Y.; Hamamura, H. *J. Chem. Soc. Chem. Commun.* **1983**, 130.

Reactions of Complex 1 with Nitriles. General Procedure. Complex **1** (3.6 g, 10 mmol) and 10 mmol of the desired nitrile were dissolved in 200 mL of dry degassed hexane. The solution then was irradiated at room temperature using an immersed high-pressure 450-W mercury lamp in a quartz reaction vessel. The course of the reaction was monitored by IR spectroscopy of aliquots until disappearance of the absorption at 2070 cm^{-1} due to complex **1** and concomitant appearance of the new ν(C=C) absorption between 1600 and 1670 cm^{-1} due to the formation of the enamine. The reaction mixture then was filtered and the solvent removed under reduced pressure. The enamines **2–26** were isolated by distillation or crystallization from pentane.

Reaction with Acetonitrile. The reaction mixture was exposed to UV light for 30 h. Distillation of the residue after removal of the solvent afforded 1.63 g (yield 70%) of enamine **2**: bp 95 °C (18 torr); IR (CCl_4, cm^{-1}) ν(C=C) 1610; NMR (CCl_4, δ) 0.3 (s, 12 H), 4.2 (m, 2 H), 6.4 (m, 1 H), 7.45 (m, 4 H); mass spectrum, parent ion M$^+$ centered at m/e 233. Anal. Calcd for $C_{12}H_{19}NSi_2$: C, 61.74; H, 8.20; Si, 24.06. Found: C, 61.98; H, 8.15; Si, 23.80.

Reaction with Propionitrile. After 30 h of UV irradiation, 2.1 g (yield 85%) of enamine **3** was isolated by distillation: E/Z = 65/35; bp 115–120 °C (20 torr); IR (CCl_4, cm^{-1}) ν(C=C) 1645; NMR (CCl_4, δ) 0.25 (s, 0.35 × 12 H), 0.35 (s, 0.65 × 12 H), 1.7 (d, 3 H), 4.8 (qd, 0.65 × 1 H, J_E = 13 Hz), 5.1 (qd, 0.35 × 1 H, J_Z = 7 Hz), 6.1 (m, 1 H), 7.4 (m, 4 H); mass spectrum, parent ion M$^+$ centered at m/e 247. Anal. Calcd for $C_{13}H_{21}NSi_2$: C, 63.09; H, 8.55; N, 5.66. Found: C, 62.92; H, 8.61; N, 5.63.

Reaction with Isobutyronitrile. After 30 h of irradiation, 2.09 g (yield 80%) of enamine **4** was isolated by distillation: bp 100 °C (0.5 torr); IR (CCl_4, cm^{-1}) ν(C=C) 1660; NMR (CCl_4, δ) 0.3 (s, 12 H), 1.65, 1.7 (2s, 6 H), 5.8 (s, 1 H), 7.5 (m, 4 H); mass spectrum, parent ion M$^+$ centered at m/e 261. Anal. Calcd for $C_{14}H_{23}NSi_2$: C, 64.30; H, 8.87; N, 5.36. Found: C, 64.50; H, 8.75; N, 5.49.

Reaction with Phenylacetonitrile. After 21 h of irradiation, 3.7 g (yield 95%) of enamine **5** were crystallized as a 55/45 mixture of E and Z isomers: mp 106–108 °C; IR (CCl_4, cm^{-1}) ν(C=C) 1625; NMR (CCl_4, δ) 0.1 (s, 0.55 × 12 H), 0.4 (s, 0.45 × 12 H), 5.7 (d, 0.55 × 1 H, J_E = 14 H_Z), 5.9 (d, 0.45 × 1 H, J_Z = 7 Hz), 6.1 (d, 0.45 × 1 H, J_Z = 7 Hz), 6.8 (d, 0.55 × 1 H, J_E = 14 Hz), 7.4 (m, 9 H); mass spectrum, parent ion M$^+$ centered at m/e 309. Anal. Calcd for $C_{18}H_{23}NSi_2$: C, 69.84; H, 7.49; N, 4.52; Si, 18.25. Found: C, 69.99; H, 7.61; N, 43.4; Si, 18.10.

Reaction with Diphenylacetonitrile. After 20 h of irradiation, 3.0 g (yield 78%) of enamine **6** were isolated by crystallization from hexane: mp 144–146 °C; IR (CCl_4, cm^{-1}) ν(C=C) 1605; NMR (CCl_4, δ) 0.1 (s, 12 H), 6.5 (s, 1 H), 7.3 (m, 14 H); mass spectrum, parent ion M$^+$ centered at m/e 385. Anal. Calcd for $C_{24}H_{27}NSi_2$: C, 74.75; H, 7.06; N, 3.63. Found: C, 74.61; H, 7.08; N, 3.48.

Reaction with 3-Phenylpropionitrile. After 30 h of irradiation, 2.5 g (yield 78%) of enamine **7** was obtained by distillation: mp 143–148 °C (0.1 torr); E/Z = 62/38; IR (CCl_4, cm^{-1}) ν(C=C) 1643; NMR (CCl_4, δ) 0.45 (s, 0.38 × 12 H), 0.5 (s, 0.62 × 12 H), 3.5 (d, 0.62 × 2 H, J = 7 Hz), 3.6 (d, 0.38 × 2 H, J = 7 Hz), 5.3 (m, 1H), 6.25 (d, 0.38 × 1 H, J_Z = 7 Hz), 6.35 (d, 0.62 × 1 H, J_E = 14 Hz), 7.6 (m, 9 H); mass spectrum, parent ion M$^+$ centered at m/e 323. Anal. Calcd for $C_{19}H_{25}NSi_2$: C, 59.26; H, 8.03; N, 5.32. Found: C, 59.04; H, 8.22; N, 5.20.

Reaction with (Trimethylsilyl)acetonitrile. After 31 h of irradiation, 1.56 g (yield 51%) of a 76/24 mixture of enamines **8** and **9** was obtained by distillation. Enamines **8** and **9** were not separated: bp 95–103 °C (0.05 torr); IR (CCl_4, cm^{-1}) ν(C=C) 1585; NMR (CCl_4, δ), enamine **8** (E/Z 89/11), 0.2 (m, 21 H), 4.7 (d, 0.11 × 1 H, J_E = 17 Hz), 5.2 (d, 0.89 × 1 H, J_Z = 11 Hz), 6.6 (d, 0.11 × 1 H, J_E = 17 Hz), 6.9 (d, 0.89 × 1 H, J_Z = 11 Hz), 7.55 (m, 4 H); enamine **9**, 0.2 (m, 21 H), 5.4 (d, 1 H, J = 2 Hz), 5.6 (d, 1 H, J = 2 Hz), 7.55 (m, 4 H); mass spectrum, parent ion M$^+$ centered at m/e 305. Anal. Calcd for $C_{15}H_{27}NSi_3$: C, 58.95; H, 8.90; N, 4.58. Found: C, 59.0, H, 8.97; N, 4.30.

Reaction with Malononitrile. After 40 h of irradiation, 1.29 g (yield 50%) of enamine **10** (E/Z = 87/13), was isolated by distillation and crystallized from hexane: bp 150 °C (0.5 torr); mp 144–145 °C; IR (CCl_4, cm^{-1}) ν(C=C) 1602; NMR (CCl_4, δ) 0.3 (s, 0.13 × 12 H), 0.4 (s, 0.87 × 12 H), 4.2 (d, 0.13 × 1 H, J_Z = 9 Hz), 4.4 (d, 0.87 × 1 H, J_E = 14 Hz), 6.6 (d, 0.13 × 1 H, J_Z = 9 Hz), 7.1 (d, 0.87 × 1 H, J_E = 14 Hz), 7.4 (m, 4 H); mass spectrum, parent ion M$^+$ centered at m/e 258. Anal. Calcd for $C_{13}H_{18}N_2Si_2$: C, 60.41; H, 7.02; N, 10.84. Found: C, 60.67; H, 7.10; N, 10.84.

Reaction with Succinonitrile. After 37 h of irradiation, 1.36 g of enamine **11** (E/Z = 65/35) was isolated by crystallization: mp 109–111 °C; IR (CCl_4, cm^{-1}) ν(C=C) 1645; NMR (CCl_4, δ), 0.4 (s, 0.65 × 12 H), 0.6 (s, 0.35 × 12 H), 3.2 (d, 0.65 × 2 H, J = 6 Hz), 3.3. (d, 0.35 × 2 H, J = 7 Hz), 4.8 (td, 0.65 × 1 H, J_E = 14 Hz), 5.4 (td, 0.35 × 1H, J_Z = 8 Hz), 6.4 (d, 0.35 × 1 H, J_Z = 8 Hz), 6.6 (d, 0.65 × 1 H, J_E = 14 Hz), 7.6 (m, 4 H); mass spectrum, parent ion M$^+$ centered at m/e 272. Anal. Calcd for $C_{14}H_{20}N_2Si_2$: C, 61.71; H, 7.40; N, 10.25. Found: C, 61.52; H, 7.32; N, 10.09.

Reaction with Adiponitrile. After 30 h of irradiation, 2.1 g (yield 70%) of enamine **12** (E/Z = 18/82) was isolated by distillation: bp 140–150 °C (0.01 torr); mp 64–65 °C; IR (CCl_4, cm^{-1}) ν(C=C) 1645; NMR (CCl_4, δ) 0.3 (s, 0.18 × 12 H), 0.4 (s, 0.82 × 12 H), 1.5 (m, 2 H), 2.1 (m, 4 H), 4.6 (td, 0.18 × 1 H, J_E = 14 Hz), 4.9 (td, 0.82 × 1 H, J_Z = 7.5 Hz), 5.9 (d, 0.82 × 1 H, J_E = 14 Hz), 6.1 (d, 0.18 × 1 H, J_Z = 7.5 Hz), 7.4 (m, 4 H); mass spectrum, parent ion M$^+$ centered at m/e 300. Anal. Calcd for $C_{16}H_{24}N_2Si_2$: C, 63.94; H, 8.05; N, 9.32. Found: C, 63.97; H, 8.05; N, 9.13.

Reaction with α-Cyanotoluonitrile. After 30 h of irradiation, 1.67 g (yield 50%) of enamine **13** (E/Z = 50/50) was isolated: IR (CCl_4, cm^{-1}) ν(C=C) 1620; NMR (CCl_4, δ) 0.2 (s, 0.5 × 12 H), 0.3 (s, 0.5 × 12 H), 6.05 (d, 0.5 × 1 H, J_E = 12 Hz), 6.3 (d, 0.5 × 1 H, J_Z = 9 Hz), 6.5 (d, 0.5 × 1 H, J_Z = 9 Hz), 7.5 (m, 8 H), 7.9 (d, 0.5 × 1 H, J_E = (12 Hz); mass spectrum, parent ion M$^+$ centered at m/e 334. Anal. Calcd for $C_{19}H_{22}N_2Si_2$: C, 68.21; H, 6.63. Found: C, 68.02; H, 6.55.

Reaction with Chloroacetonitrile. After 60 h of irradiation, 0.8 g (yield 30%) of enamine **14** (E/Z = 65/35) was isolated by distillation (1.2 g of iron complex starting material **1** was also crystallized out of the reaction mixture before distillation): bp 85 °C (3 torr); IR (CCl_4, cm^{-1}) ν(C=C) 1625; NMR (CCl_4, δ) 0.4 (s, 0.65) × 12 H), 0.45 (s, 0.35 × 12 H), 5.2 (d, 0.35 × 1 H, J_Z = 6 Hz), 5.5 (d, 0.65 × 1 H, J_E = 12 Hz), 6.3 (d, 0.35 × 1 H, J_Z = 6 Hz), 6.6 (d, 0.65 × 1 H, J_E = 12 Hz), 7.5 (m, 4 H); mass spectrum, parent ion M$^+$ centered at m/e 267 (^{35}Cl). Anal. Calcd for $C_{12}H_{18}ClNSi_2$: C, 29.18; H, 4.93. Found: C, 39.35; H, 5.07.

Reaction with 5-Bromovaleronitrile. After 50 h of irradiation, 2.46 g (yield 70%) of enamine **15** was isolated by distillation: bp 126–128 °C (0.03 torr); mp 57–59 °C; IR (CCl_4, cm^{-1}) ν(C=C) 1645; NMR (CCl_4, δ) 0.2 (s, 12 H), 1.7 (m, 6 H), 4.8 (m, 1 H), 6.0 (m, 1 H), 7.4 (m, 4 H); mass spectrum, parent ion M$^+$(^{79}Br) centered at m/e 353. Anal. Calcd for $C_{15}H_{24}BrNSi_2$: C, 50.83; H, 6.83. Found: C, 50.60; H, 6.71.

Reaction with *p*-(Bromophenyl)acetonitrile After 60 h of irradiation, 2.4 g (yield 62%) of enamine **16** (E/Z) = 25/75) was isolated by crystallization from hexane: mp 57–59 °C; IR (CCl_4, cm^{-1}) ν(C=C) 1627; NMR (CCl_4, δ) 0.1 (s, 0.75 × 12 H), 0.4 (s, 0.25 × 12 H), 5.8 (d, 0.75 × 1H, J_Z = 9 Hz), 6.3 (d, 0.75 × 1 H, J_Z = 9 Hz), 6.9 (d, 0.25 × 1 H, J_E = 13 Hz), 7.1 (d, 0.25 × 1 H, J_E = 13 Hz), 7.3 (m, 8 H); mass spectrum, parent ion M$^+$(^{79}Br) centered at m/e 387. Anal. Calcd for $C_{18}H_{22}BrNSi_2$: C, 55.65; H, 5.71; N, 3.61. Found: C, 55.95; H, 5.79; N, 3.52.

Reaction with Methoxyacetonitrile. After 43 h of irradiation, 1.84 g (yield 70%) of enamine **17** (E/Z = 40/60) was isolated by distillation: bp 125–130 °C (10 torr) IR (CCl_4, cm^{-1}) ν(C=C) 1650; NMR (CCl_4, δ) 0.3 (s, 12 H), 3.4 (s, 0.4 × 3 H), 3.5 (s, 0.6 × 3 H), 5.1 (d, 0.6 × 1H, J_Z = 6 Hz), 5.4 (d, 0.6 × 1 H, J_Z = 6 Hz), 5.8 (d, 0.4 × 1 H, J_E = 11 Hz), 6.3 (d, 0.4 × 1 H, J_E = 11 Hz), 7.45 (m, 4 H); mass spectrum, parent ion M$^+$ centered at m/e 263. Anal. Calcd for $C_{13}H_{21}NOSi_2$: C, 59.26; H, 8.03; N, 5.32. Found: C, 59.43; H, 8.08; N, 5.22.

Reaction with 3-((Trimethylsilyl)oxy)propionitrile. After 50 h of irradiation, 1.10 g (yield 45%) of enamine **3** (E/Z = 65/35), having characteristics identical with that obtained from propionitrile, was isolated.

Reaction with 3-Piperidinopropionitrile. After 63 h of irradiation, 1.32 g (yield 40%) of enamine **18** (Z) was isolated by distillation: bp 136 °C (0.05 torr); IR (CCl_4, cm^{-1}) ν(C=C) 1635; NMR (CCl_4, δ) 0.4 (s, 12 H), 1.5 (m, 6 H), 2.4 (m, 4 H), 3.0 (q, 2 H), 5.2 (td, 1 H), 6.1 (d, 1 H, J_Z = 8 Hz), 7.4 (m, 4 H). Anal. Calcd for $C_{18}H_{30}N_2Si_2$: C, 65.39; H, 9.15. Found: C, 65.50; H, 9.20.

Reaction with Ethyl Cyanoacetate. After 6 h of irradiation, 2.53 g (yield 83%) of enamine **19** (*E*) were obtained by distillation: bp 110–120 °C (3 torr); mp 58–59 °C; IR (CCl_4, cm^{-1}) ν(C=C) 1680, 1601; NMR (CCl_4, δ) 0.3 (s, 12 H), 1.2 (t, 3 H, J = 5 Hz), 4.0 (q, 2 H, J = 5 Hz), 5.0 (d, 1 H, J_E = 15 Hz), 7.4 (m, 4 H), 7.5 (d, 1 H, J_E = 15 Hz); mass spectrum, parent ion M^+ centered at *m/e* 305. Anal. Calcd for $C_{15}H_{23}NO_2Si_2$: C, 58.97; H, 7.59; N, 4.58; Si, 18.38. Found: C, 58.75; H, 7.53; N, 4.58; Si, 18.1.

Reaction with 2-Cyanoethyl Acetate. After 80 h of irradiation, 1.0 g (yield 40%) of enamine **3** (E/Z = 65/35), having characteristics identical with that obtained from propionitrile was isolated.

Reaction with 3,3-(Ethylenedioxy)-2-phenylbutyronitrile. After 76 h of irradiation, 2.77 g (yield 70%) of enamine **20** was isolated by distillation: bp 150–155 °C (0.01 torr); mp 64–66 °C; IR (CCl_4, cm^{-1}) ν(C=C) 1625; NMR (CCl_4, δ) 0.1 (s, 0.7 × 12 H), 0.4 (s, 0.3 × 12 H), 1.3 (s, 0.7 × 3 H), 1.7 (s, 0.3 × 3 H), 3.9 (s, 4 H), 6.0 (s, 0.3 × 1 H), 6.5 (0.7 × 1 H), 7.3 (m, 4 H); mass spectrum, parent ion M^+ centered at *m/e* 395. Anal. Calcd for $C_{22}H_{29}NO_2Si_2$: C, 66.79; H, 7.39; N, 3.54. Found: C, 66.72; H, 7.49; N, 3.43.

Reaction with 3-(2,2-(Ethylenedioxy)cyclohexyl)propionitrile. After 30 h of irradiation, 2.9 g (yield 75%) of enamine **21** (E/Z = 56/44) was isolated by distillation: bp 150–160 °C (0.5 torr); IR (CCl_4, cm^{-1}) ν(C=C) 1640; NMR (CCl_4, δ) 0.3 (s, 0.44 × 12 H), 0.4 (s, 0.56 × 12 H), 0.9–2.4 (m, 11 H), 3.75 (s, 0.44 × 4 H), 3.8 (s, 0.56, 4 H), 4.6 (td, 0.56 × 1 H, J = 7.5 Hz, J_E = 13 Hz), 4.9 (td, 0.44 × 1 H, J = 7 Hz, J_Z = 6 Hz), 5.9 (d, 0.44 × 1 H, J_z = 6 Hz), 6.0 (d, 0.56 × 1 H, J_E = 13 Hz), 7.3 (m, 4 H); mass spectrum, parent ion M^+ centered at *m/e* 387. Anal. Calcd for $C_{21}H_{23}NO_2Si_2$: C, 65.06; H, 8.58; N, 3.61. Found: C, 65.06; H, 8.69; N, 3.42.

Reaction with Allyl Cyanide. After 40 h of irradiation, 0.78 g (yield 30%) of enamine **22** was isolated by distillation: bp 90–100 °C (2 torr). IR (CCl_4, cm^{-1}) ν(C=C) 1630; NMR (CCl_4, δ) 0.3 (s, 12 H), 4.7–7.1 (m, 5 H), 7.5 (m, 4 H); mass spectrum, parent ion M^+ centered at *m/e* 259. Anal. Calcd for $C_{14}H_{21}NSi_2$: C, 64.80; H, 8.16. Found: C, 65.01; H, 8.20.

Reaction with 4-Cyanobut-1-ene. After 96 h of irradiation, 0.82 g (yield 30%) of enamine **23** was isolated by distillation: bp 110–120 °C (0.1 torr); IR (CCl_4, cm^{-1}) ν(C=C) 1625; NMR (CCl_4, δ) 0.4 (s, 12 H), 1.8 (d, 3 H, J = 7 Hz), 5.4–6.8 (m, 4 H), 7.5 (m, 4 H); mass spectrum, parent ion M^+ centered at *m/e* 273. Anal. Calcd for $C_{15}H_{23}NSi_2$: C, 65.87; H, 8.48. Found: C, 66.03; H, 8.52. From the reaction mixture was also crystallized 0.41 g (yield 15%) of the cyclic enamine **24**: mp 83–85 °C; IR (CCl_4, cm^{-1}) ν(C=C) 1620; NMR (CCl_4, δ) 0.3 (s, 12 H), 2.2 (m, 6 H), 4.7 (t, 1 H, J = 2 Hz), 7.4 (m, 4 H); mass spectrum, parent ion M^+ centered at *m/e* 273. Anal. Calcd for $C_{15}H_{23}NSi_2$: C, 65.87; H, 8.48; N, 5.12; Si, 20.54. Found: C, 65.64; H, 8.25; N, 5.00; Si, 20.20.

Reaction with Cyclohex-1-enylacetonitrile. After 44 h of irradiation, 1.36 g (yield 30%) of dienamine iron complex **25** was isolated by crystallization from hexane: mp 114–116 °C; IR (CCl_4, cm^{-1}) ν(CO) 2030, 1920; NMR (CCl_4, δ) 0.5 (s, 12 H), 0.8–2.4 (m, 9 H), 3.0 (d, 1 H, J = 13 Hz), 5.3 (d, 1 H), 7.6 (m, 4 H); mass spectrum, parent ion M^+ centered at *m/e* 453 and successive loss of three carbon monoxide ligands and one iron atom. Anal. Calcd for $C_{21}H_{27}NO_3Si_2Fe$: C, 55.62; H, 6.00; N, 3.09; Fe, 12.32. Found: C, 55.47; H, 5.87; N, 3.06; Fe, 11.95.

Reaction with Cyano Enamine 11. After 60 h of irradiation, 2.11 g (yield 70%) of dienamine iron carbonyl complex **26** was isolated by crystallization from hexane: mp 176–177 °C; IR (CCl_4, cm^{-1}) ν(CO) 2030, 1955, 1945; NMR (CCl_4, δ) 0.5 (s, 24 H), 2.9 (m, 2 H), 5.3 (m, 2 H), 7.4 (m, 8 H); mass spectrum, parent ion M^+ centered at *m/e* 604 and successive loss of three carbon monoxide ligands and one iron atom. Anal. Calcd for $C_{27}H_{36}N_2O_3Si_4Fe$: C, 53.62; H, 6.00; N, 4.63; Fe, 9.23. Found: C, 53.73; H, 6.27; N, 4.40; Fe, 8.96. The same compound was also obtained under the same reaction conditions starting from 3.6 g (0.01 mol) of iron complex **1** and 0.4 g (0.005 mol) of succinonitrile. In this case, crystallization afforded 1.5 g (yield 50%) of dienamine iron carbonyl complex **26**.

Hydrolysis of *N*,*N*-Bis(silyl) Enamines. Hydrolysis of enamine **3** to propanal was achieved as follows: 4.9 g (0.02 mol) of enamine **3** in 10 mL of xylene was stirred with 30 mL of a 1 N HCl aqueous solution for 2 h. The reaction mixture then was neutralized by addition of a saturated solution of $NaHCO_3$. After extraction and drying, 0.74 g (yield 64%) of propanal was distilled out of the crude organic solution. The hydrolysis of other enamines was performed on a smaller scale, and the aldehydes produced were isolated as (2,4-dinitrophenyl)hydrazone derivatives and identified by comparison with authentic samples and literature data.[18] The enamine (1 mmol) was added to 20 mL of a 0.14 M acidic solution of 2,4-dinitrophenylhydrazine. After filtration the precipitate was recrystallized twice from ethanol. Enamine **2** gave 0.146 g (yield 65%) of acetaldehyde 2,4-DNP, mp 144–146 °C. Enamine **3**: 0.195 g (yield 82%) of propionaldehyde 2,4-DNP, mp 145–147 °C. Enamine **4**: 0.171 g (yield 68%); mp 182 °C. Enamine **5**: 0.237 g (yield 79%); mp 110 °C. Enamine **6**: 0.316 g (yield 84%); mp 148–149 °C. Enamine **12**: 0.23 g (yield 79%); mp 91–93 °C. Enamine **19**: 0.216 g (yield 73%); mp 74–76 °C. After hydrolysis of the enamines, formation of the benzo[*c*]oxadisilole (eq 10) was observed. The compound was identified by comparison with an authentic sample:[13b] mp 35–37 °C (lit[13b] mp 38 °C); NMR (CCl_4, δ) 0.3 (s, 12 H), 7.4 (m, 4 H).

Reaction of $LiAlH_4$ with Enamine 12. To a suspension of 0.2 g (5.3 mmol) of $LiAlH_4$ in 40 mL of diethyl ether was added dropwise a solution containing 1.0 g (3.3 mmol) of enamine **12** in 20 mL of ether. After the addition was complete, the reaction mixture was stirred for 1 h and cooled at 0 °C. Hydrolysis was effected by slow addition of 25 mL of an aqueous potassium–sodium tartrate solution. After extraction with ether and drying over sodium carbonate, the solvent was evaporated under reduced pressure. The residue was dissolved in 10 mL of pentane and cooled at −78 °C. The solid that deposited was discarded, and evaporation of pentane solution left 0.80 g (yield 79%) of enamine **27** (E/Z = 20/80) as an oil: IR (CCl_4, cm^{-1}) ν(NH) 3270, ν(C=C) 1625; NMR (CCl_4, δ) 0.4 (0.2 × 12 H), 0.5 (0.8 × 12 H), 1.2 (m, 2 H), 1.3–2.9 (m, 8 H), 5.0 (m, 1 H), 6.0 (d, 0.8 × 1 H, J_Z = 8 Hz), 6.2 (d, 0.2 × 1H, J_E = 13 Hz), 7.4 (m, 4 H); mass spectrum, parent ion M^+ centered at *m/e* 304. Anal. Calcd for $C_{16}H_{28}N_2Si_2$: C, 63.09; H, 9.27. Found: C, 63.21; H, 9.35.

Reaction of Phenylmagnesium Bromide with Enamine 12. To 5.25 mL of a 0.73 M ether solution (3.8 mmol) of phenylmagnesium bromide was added dropwise a solution containing 1.0 g (3.3 mmol) of enamine **12** in 10 mL of ether. The mixture was refluxed for 16 h. After being cooled to 0 °C, the mixture was hydrolyzed with a 2 M aqueous ammonium chloride solution, extracted with ether, and dried over sodium sulfate. After evaporation of the solvent, the residue was dissolved in 10 mL of pentane and cooled to −78 °C. The solid that deposited was filtered off, and the filtrate was concentrated and cooled at −78 °C. After a new formation of a solid residue, evaporation of the solution gave 0.54 g (yield 43%) of enamine **28** (E/Z = 20/80) as an oil: IR (CCl_4, cm^{-1}) ν(C=O) 1680, ν(C=C) 1640; NMR (CCl_4, δ) 0.2 (s, 0.8 × 12 H), 0.3 (s, 0.2 × 12 H), 1.3–2.6 (m, 4 H), 2.9 (t, 2 H, J = 7 Hz), 5.0 (m, 1 H), 6.0 (d, 0.8 × 1 H, J_Z = 7 Hz), 6.1 (d, 0.2 × 1 H, J_E = 15 Hz), 7.1–8.1 (m, 9 H); mass spectrum, parent ion M^+ centered at *m/e* 379. Anal. Calcd for $C_{22}H_{29}NOSi_2$: C, 69.09; H, 7.70. Found: C, 69.32; H, 7.85.

Reaction of $LiAlH_4$ with Enamine 19. To a suspension of 0.1 g (2.6 mmol) of $LiAlH_4$ in 10 mL of diethyl ether was added dropwise a solution containing 1.4 g (4.6 mmol) of enamine **19** in 20 mL of ether. The mixture was stirred for 1 h at room temperature. Then it was hydrolyzed by addition of an aqueous potassium–sodium tartrate solution. After extraction with ether and drying over sodium sulfate, the solvent was removed under reduced pressure giving an oil which was crystallized from *n*-heptane. After recrystallization, 0.47 g (yield 39%) of enamine **29** (*E*) was obtained: mp 55–57 °C; IR (CCl_4, cm^{-1}) ν(OH) 3600, 3370, ν(C=C) 1660; NMR (CCl_4, ν) 0.5 (s, 12 H), 1.8 (s, 1 H), 4.0 (d, 2 H, J = 6.5 Hz), 5.0 (dt, 1 H), 6.3 (d, 1 H, J_E = 13.5 Hz), 7.4 (m, 4 H); mass spectrum, parent ion M^+ centered at *m/e* 263. Anal. Calcd for $C_{13}H_{21}NOSi_2$: C, 59.26; H, 8.03. Found: C, 59.04; H, 8.32.

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(18) "Tables for Identification of Organic Compounds", supplement to Handbook of Chemistry and Physics; CRC Press: Cleveland, 1960.