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Dehydrogenative Silylation of Amines and Hydrosilylation of Imines Catalyzed by Ytterbium−Imine Complexes

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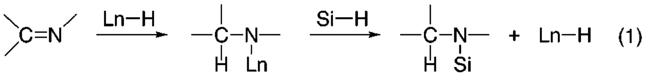
Dehydrogenative silylation of primary and secondary amines with triphenylsilane was catalyzed by ytterbium−imine complexes, [Yb(η^2-Ph$_2$CNAr)(hmpa)$_n$], to give aminosilanes in good yields. In the reaction with diphenyl- and phenylsilanes, diaminosilanes were formed as major products. Whereas *n*- and *sec*-alkylamines were readily silylated, *tert*-alkylamines and aromatic amines exhibited lower reactivities. Moreover, hydrosilylation of imines has been achieved by using phenylsilane and the imine complexes (Ar = Ph, C$_6$H$_4$F-4), giving rise to mono- and diaminosilanes. The two reactions were in agreement as regards the product selectivities and yields.

Introduction

Synthetic reactions based on lanthanide reagents have been extensively explored in the last two decades because of their unique properties.[1] Particularly, olefin transformations catalyzed by organolathanides such as hydrogenation[2] and hydrosilylation[3] have attracted much attention, because they have been proven to be comparable or, in some cases, superior to those catalyzed by late transition metals. However, the catalytic reaction of carbonyl and imino compounds instead of olefins has been rarely investigated. Recently, Marks et al. reported the first example of lanthanide-catalyzed imine hydrogenation, in which acyclic imines were reduced under more vigorous conditions than for olefin hydrogenation.[4] It has also been demonstrated that stoichiometric reaction of imine with lanthanocene hydrides proceeds via C−H activation rather than imine insertion to the Ln−H bond.[4,5]

Catalysts used for these olefin transformations have been limited to trivalent lanthanocene complexes, which are rather difficult for handling and large-scale synthesis. Previously, we reported that divalent Yb−imine complexes [Yb(η^2-Ph$_2$CNAr)(hmpa)$_n$] (**1**) were easily prepared from Yb metal and diarylimines in THF and HMPA.[6] The complexes **1** were found to catalyze irreversible isomerization[7] and dehydrogenative silylation of terminal alkynes.[8] For the synthetic application of the imine complexes **1**, we studied their catalyst activity on the hydrosilylation of imines and found that the reaction took place efficiently.

As in olefin hydrosilylation with organolathanides,[3] imine hydrosilylation would involve also two key steps: insertion of imine to the Ln−H bond and silanolysis of the resulting Ln−N bond (eq 1). The second step is very rare[9] and seems to be difficult, compared to the reaction of olefin. Thus, we studied at first a catalytic dehydrogenative silylation of amines with the imine complexes **1** to confirm the second step.[10] Next, our effort was addressed to generation of the Ln−H species from the complexes **1** for the first step and then to imine hydrosilylation. We describe herein these results.



Results and Discussion

As a model reaction, dehydrogenative silylation of amylamine (**2a**) was tested with various hydrosilanes and imine complexes **1** (Table 1). When triphenylsilane and amylamine (1:1) were successively added to a solution of **1a** (3 mol %) in THF, generated in situ from Yb metal and diphenymethylideneaniline in the presence of 4 equiv of HMPA,[11] *N*-triphenylsilylamylamine (**3a**) was produced in 61% yield, along with vigorous hydrogen evolu-

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(1) (a) Edelmann, F. T. In *Topics in Current Chemistry 179*; Herrmann, W. A., Ed.; Springer-Verlag: Berlin, 1996; pp 247−276. (b) Imamoto, T. *Lanthanides in Organic Synthesis*; Academic Press: London, 1994. (c) Molander, G. A. *Chem. Rev.* **1992**, *92*, 29−68. (d) Kagan, H. B.; Namy, J. L. *Tetrahedron* **1986**, *42*, 6573−6614.

(2) (a) Jeske, G.; Lauke, H.; Mauermann, H.; Schumann, H.; Marks, T. J. *J. Am. Chem. Soc.* **1985**, *107*, 8111−8118. (b) Molander, G. A.; Hoberg, J. O. *J. Org. Chem.* **1992**, *57*, 3266−3268. (c) Conticello, V. P.; Brard, L.; Giardello, M. A.; Tsuji, Y.; Sabat, M.; Stern, C. L.; Marks, T. J. *J. Am. Chem. Soc.* **1992**, *114*, 2761−2762. (d) Giardello, M. A.; Conticello, V. P., Brard, L.; Gagne, M. R.; Marks, T. J. *J. Am. Chem. Soc.* **1994**, *116*, 10241−10254. (e) Haar, C. M.; Stern, C. L.; Marks, T. J. *Organometallics* **1996**, *15*, 1765−1784.

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(4) Obora, Y.; Ohta, T.; Stern, C. L.; Marks, T. J. *J. Am. Chem. Soc.* **1997**, *119*, 3745−3755.

(5) Radu, N. S.; Buchwald, S. L.; Scott, B.; Burns, C. J. *Organometallics* **1996**, *15*, 3913−3915.

(6) Makioka, Y.; Taniguchi, Y.; Fujiwara, Y.; Takaki, K.; Hou, Z.; Wakatsuki, Y. *Organometallics* **1996**, *15*, 5476−5478.

(7) Makioka, Y.; Saiki, A.; Takaki, K.; Taniguchi, Y.; Kitamura, T.; Fujiwara, Y. *Chem. Lett.* **1997**, 27−28.

(8) Takaki, K.; Kurioka, M.; Kamata, T.; Takehira, K.; Makioka, Y.; Fujiwara, Y. *J. Org. Chem.* **1998**, *63*, 9265−9269.

(9) Opposite transposition (Ln−N + Si−H → Ln−Si + N−H) was proposed to account for the role of phenylsilane accelerator in lanthanocene-catalyzed imine hydrogenation by Marks et al. Simultaneously, they documented the formation of a small amount of hydrosilylation product (<10%); see: ref 4.

(10) Dehydrogenative silylation of amines catalyzed by lithium amides has been well-known; see: Kraus, C. A.; Nelson, W. K. *J. Am. Chem. Soc.* **1934**, *56*, 195−202.

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tion (run 1). Catalyst activity of **1** was dependent on the substituents on the imine nitrogen. Thus, the complexes **1** with electron-withdrawing groups generally gave better yields of **3a** than those with electron-donating ones (runs 1 and 2 vs 4−6). However, 2,6-xylyl catalyst **1c** showed the highest activity among the imine complexes tested, suggesting that a steric factor can also play an important role in the reaction (run 3). With respect to the reactivity of hydrosilanes, those with a phenyl substituent gave the aminosilanes **3** in over 90% yields (runs 3, 7, and 8), whereas trialkyl and trialkoxysilanes decreased the yield to about 20% (runs 9 and 10).

Dehydrogenative silylation of various primary and secondary amines with Ph_3SiH was studied by using the imine complex **1c** (Table 2). Of the primary amines, *n*- and *sec*-alkylamines **2a** and **2b** were readily converted to **3a** and **3f** in 93% and 97% yields, respectively (runs 1 and 2). Although reaction of *tert*-alkylamine **2c** was very slow (run 3), the product **3g** was formed in 81% yield by increasing the catalyst amount (10 mol %) and reaction time (run 4). Secondary amines **2e**−**g** were similarly silylated in good yields (runs 6−8). In contrast, aniline was converted to the aminosilane **3h** in lower yield (56%) than alkylamines (run 5), and no reaction took place with *N*-methylaniline (run 9).

Dehydrogenative silylation of amines could be performed with diphenylsilane and phenylsilane, giving rise to aminosilanes and diaminosilanes (eqs 2 and 3). In the reaction of amylamine (**2a**) with diphenylsilane, when the ratio of **2a** to Ph_2SiH_2 was unity, aminosilane **4a** and diaminosilane **5a** were obtained in 18% and 35% yields, respectively. Use of 2 equiv of **2a** resulted in the selective formation of **5a**. *N*-Benzylamylamine (**2i**) was silylated with phenylsilane (3 equiv) in the presence of the imine complex **1a** to afford aminosilane **7b** in 80% yield, together with a minor diaminosilane **8b**.

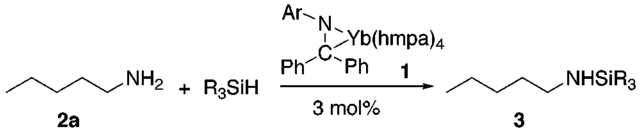
$$\underset{\textbf{2a}}{^{n}C_5H_{11}NH_2} + Ph_2SiH_2 \xrightarrow[\text{THF, rt, 2 h}]{\textbf{1c}\text{ (3 mol\%)}} \underset{\textbf{4a}}{^{n}C_5H_{11}NH\text{-}SiHPh_2} + \underset{\textbf{5a}}{(^{n}C_5H_{11}NH)_2SiPh_2} \qquad (2)$$

	4a	**5a**
2a / Ph_2SiH_2 = 1 / 1	18%	35%
= 2 / 1	–	82%

$$\underset{\textbf{2i}}{BnNH^{n}C_5H_{11}} + 3\ PhSiH_3 \xrightarrow[\text{THF, rt, 20 h}]{\textbf{1a}\text{ (5 mol\%)}} \underset{\textbf{7b}\ 80\%}{^{n}C_5H_{11}N(Bn)\text{-}SiH_2Ph} + \underset{\textbf{8b}\ 5\%}{[^{n}C_5H_{11}N(Bn)]_2SiHPh} \qquad (3)$$

The dehydrogenative silylation of amine would be explained as follows (Scheme 1). The imine complex **1** reacts with amine to yield bisamide complex **A**. Silanolysis of the Yb−N bond of **A** affords the aminosilane and hydride complex **B**.[9] Then, bisamide **A** is regenerated by the reaction of **B** with amine, along with hydrogen evolution. Of the two amide moieties of **A**, only one amide derived from the amine would be silylated; the other from the imine complex **1** remains unchanged, which is deduced from the fact that *N*-methylaniline was not silylated at all (run 9, Table 2).[12] Moreover, if another bisamide, $[(R'_2N)_2Yb]$, is formed by the double silanolysis of **A** or by further reaction of **A** with R'_2NH and works as a real intermediate, all imine complexes **1** bearing different imine ligands should exhibit similar catalyst activity.

Table 1. Dehydrogenative Silylation of Amylamine[a]

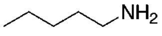
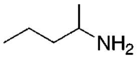
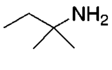
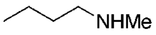
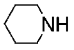


run	imine complex (Ar)		hydrosilane	product	yield (%)[b]
1	**1a**	Ph	Ph_3SiH	**3a**	61
2	**1b**	C_6H_4F-4		**3a**	52
3	**1c**	$C_6H_3(Me)_2$-2,6		**3a**	93 (92)
4	**1d**	$C_6H_3(^{i}Pr)_2$-2,6		**3a**	9
5	**1e**	C_6H_4OMe-4		**3a**	28
6	**1f**	H		**3a**	trace
7	**1c**		Ph_2MeSiH	**3b**	(90)
8	**1c**		$PhMe_2SiH$	**3c**	(93)
9[c]	**1c**		Et_3SiH	**3d**	(21)
10[c]	**1c**		$(EtO)_3SiH$	**3e**	27

[a] Conditions: **2a** / R_3SiH = 1 / 1, THF, rt, 3 h. [b] GC yield (isolated yield). [c] Carried out for 15 h.

Table 2. Dehydrogenative Silylation of Various Amines with Ph_3SiH[a]

$$\underset{\textbf{2}}{R^1R^2NH} + Ph_3SiH \xrightarrow{\textbf{1c}} \underset{\textbf{3}}{R^1R^2NSiPh_3}$$

run	amine		**1c** (mol%)	time (h)	product	yield (%)[b]
1	**2a**		3	2	**3a**	93
2	**2b**		3	2	**3f**	97
3	**2c**		3	20	**3g**	16
4			10	192	**3g**	81
5	**2d**	$PhNH_2$	10	20	**3h**	56
6	**2e**		10	5	**3i**	95
7	**2f**	BnNHMe	10	2	**3j**	89
8	**2g**		10	2	**3k**	79
9	**2h**	PhNHMe	10	20	no reaction	

[a] Conditions: **2** / Ph_3SiH = 1 / 1, THF, rt. [b] GC yield.

After it was confirmed that silanolysis of lanthanide amide, i.e., the second step of imine hydrosilylation (eq 1), took place readily, we tried to generate a hydride species from the imine complex **1** for the first step.

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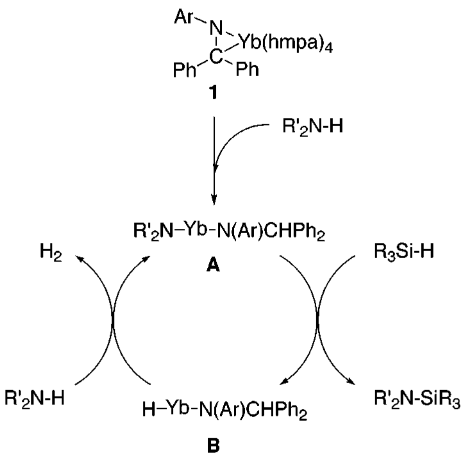
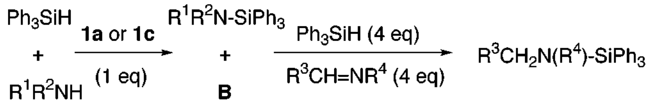
(11) Although the accurate number of the coordinated HMPA is unknown, equivalents of this additive used for generation of **1** are tentatively shown as a coordination number. The product **3a** was formed in very low yields (<5%) in the absence of HMPA.

(12) No aminosilanes derived from the imine ligands were detected in any reactions.

Scheme 1



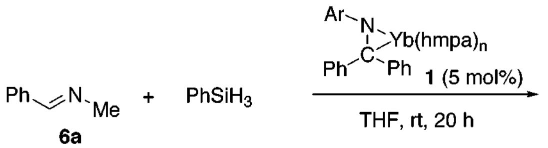
Scheme 2



Although **1** can be regarded as a sort of lanthanide carbyl complex, no reaction occurred under atmospheric hydrogen. Accordingly, we expected that hydride complex **B** generated in the dehydrogenative silylation of secondary amines could be utilized to initiate the reaction as shown in Scheme 2. Stoichiometric reaction of triphenylsilane with secondary amines by using **1a** or **1c** gave aminosilanes quantitatively, suggesting a generation of **B**. In fact, this reaction mixture reduced adamantanone to 2-adamantanol in good yields. However, addition of a 4-fold excess of hydrosilane and aldimines to the mixture did not produce any hydrosilylation or reduction products.

Next, we intended to generate the hydride species directly from the imine complexes **1** and hydrosilane. Fortunately, it was found that phenylsilane was oligomerized with hydrogen evolution in the presence of **1**.[13] It has been reported that dehydrogenative oligomerization of the silane catalyzed by lanthanocene complexes proceeds through a hydride and silyl species.[14] Therefore, similar intermediates could be generated in the present system, and if the origomerization is suppressed, the hydride species can be available for the imine hydrosilylation.

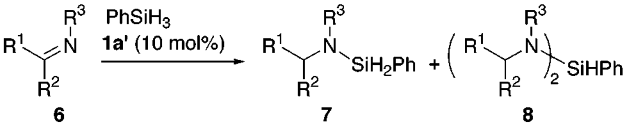
Reaction of benzylidenemethylamine (**6a**) with an equimolar amount of phenylsilane in the presence of **1a** (5 mol %) gave aminosilane **7a** and diaminosilane **8a** in 7% and 65% yields based on **6a** (7% and 32% yields based on the silane), respectively, along with a very small amount of the oligomer (eq 4). Of course, no reaction took place in the absence of **1a** (THF–HMPA, rt ~ refluxing temperature). Substitution of **1a** with **1b** caused an increase of the oligomer, though the products **7a** and **8a** were obtained in similar yields. The imine complex **1c**, the most active catalyst in the dehydrogenative silylation of amines, did not promote any reaction. In addition, triphenylsilane and diphenysilane could not be used for the present hydrosilylation, probably because a hydride species was not generated in the reaction mixture.[15] The amount of HMPA in the complex **1a** affected the product selectivity and yields.[11] When 2 equiv of the additive was used ($n = 2$), **7a** and **8a** were formed in 28% and 20% yields, respectively, whereas the reaction with **1a′** ($n = 6$) gave the products in 5% and 75% yields, respectively.

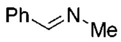
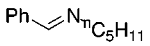
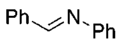
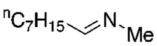
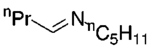
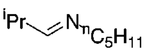
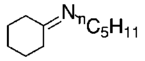
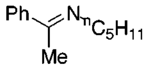
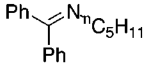


6a

$$\underset{\mathbf{7a}}{PhCH_2N(Me)\text{-}SiH_2Ph} + \underset{\mathbf{8a}}{[PhCH_2N(Me)]_2SiHPh} \quad (4)$$

Table 3. Hydrosilylation of Imines with PhSiH$_3$[a]



run	imine		product and yield (%)[b] 7		8	
1	**6a**		**7a**	2	**8a**	95
2	**6b**		**7b**	52	**8b**	35
3	**6c**		**7c**	15	**8c**	47
4	**6d**		**7d**	3	**8d**	89
5	**6e**		**7e**	21	**8e**	64
6	**6f**		**7f**	28	**8f**	43
7	**6g**		**7g**	56	**8g**	0
8	**6h**		**7h**	3	**8h**	17
9	**6i**		no reaction			

[a] Conditions: **6** / PhSiH$_3$ = 1 / 1, THF, rt, 20 h. **1a'**= [Yb(η^2-Ph$_2$CNPh)(hmpa)$_6$]. [b] GC yield based on **6**.

Hydrosilylation of aldimines and ketimines with phenylsilane was investigated by using the imine complex **1a′** (Table 3). Aldimines **6a–f** were converted to aminosilanes **7** and diaminosilanes **8** in good yields (runs 1–6). The diaminosilanes **8** were selectively obtained from *N*-methylimines (runs 1 and 4), whereas the ratio of **7** to **8** was increased in the reaction of *N*-pentylimines (runs 2, 5, and 6). Ketimines **6g–i** were less reactive than aldimines (runs 7–9). Although hydrosilylation of ketimine **6g** produced aminosilane **7g** in 56% yield as the sole product, little or no product was obtained in the reaction of **6h** and **6i**.

For comparison of the hydrosilylation of imines with the dehydrogenative silylation of amines, amines corresponding to the imines listed in Table 3 were treated with

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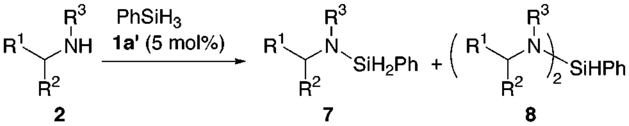
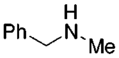
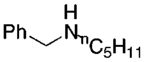
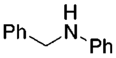
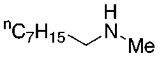
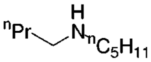
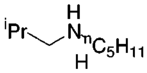
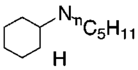
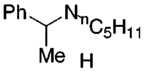
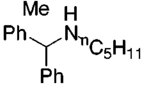
[13] For example, treatment of PhSiH$_3$ with **1b′**, [Yb(η^2-Ph$_2$CNC$_6$H$_4$F-4)(hmpa)$_6$], (10 mol %, rt, 17 h) produced polyphenylsilanes with vigorous hydrogen evolution, from which oligomer (M_W = 2966, M_W/M_N = 1.1) was isolated in 29% yield by aqueous workup, followed by short-column chromatography on silica gel using hexanes–ethyl acetate (1/1).

[14] Forsyth, C. M.; Nolan, S. P.; Marks, T. J. *Organometallics* **1991**, *10*, 2543–2545.

[15] Oligomerization and hydrogen evolution were not observed on treatment of the two silanes with **1**.

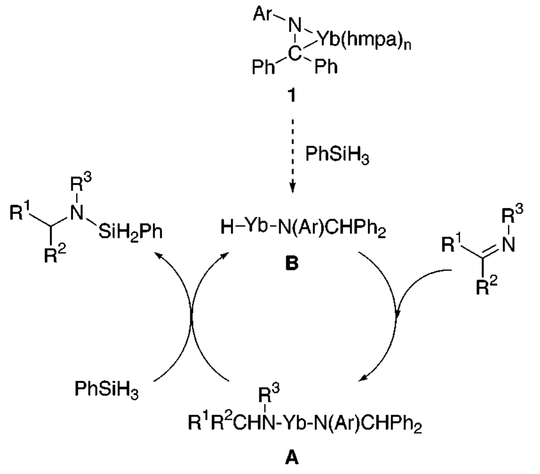
Table 4. Dehydrogenative Silylation of Amines with $PhSiH_3$[a]



run		amine	product and yield (%)[b] 7		8	
1	**2f**		**7a**	1	**8a**	93
2	**2i**		**7b**	2	**8b**	86
3	**2j**		**7c**	15	**8c**	47
4	**2k**		**7d**	tr	**8d**	83
5	**2l**		**7e**	34	**8e**	57
6	**2m**		**7f**	87	**8f**	tr
7	**2n**		**7g**	38	**8g**	0
8	**2o**		**7h**	7	**8h**	11
9	**2p**		no reaction			

[a] Conditions: **2** / $PhSiH_3$ = 1 / 1, THF, 20 h, -35 °C (runs 1-5) or rt (runs 6-9). **1a'**= $[Yb(\eta^2\text{-}Ph_2CNPh)(hmpa)_6]$. [b] GC yield based on **2**.

Scheme 3



equimolar amounts of phenylsilanes in the presence of the imine complex **1a′** (5 mol %), and these results are summarized in Table 4. As can be seen in Tables 3 and 4, the two reactions are in good agreement as regards product selectivities and yields, except for runs 2 and 6. These results indicate that the two reactions proceed through the same intermediates.

A possible catalytic cycle for the imine hydrosilylation is outlined in Scheme 3. The cycle is essentially similar to Scheme 1 proposed for the dehydrogenative silylation of amines, in that they both include bisamide **A** and hydride **B** as common intermediates. The only difference between Schemes 1 and 3 is the process for the formation of bisamide **A**. In the latter, insertion of imine to hydride species **B**, which is generated in situ by the reaction of the imine complex **1** with phenylsilane, affords bisamide **A**. Subsequent silanolysis of one Yb−N bond of **A** with phenylsilane yields aminosilane and **B**, as described in Scheme 1. The aminosilanes, particularly those derived from the less crowded imine, would be more reactive than phenylsilane in the second step, probably because metal coordination at their nitrogen atom facilitates this process. Therefore, diaminosilanes could be obtained as the major product in the most reactions. Decreased reactivity of ketimines would be attributed to the lower efficiency in the silanolysis step, as substituents on the amide moieties are secondary hydrocarbons. In fact, dehydrogenative silylation of **2n**−**p** gave yields of the products lower than those of the amines corresponding to aldimines (runs 7−9 vs 1−6, Table 4). In Scheme 3, insertion of imine to the Ln−H bond may compete with a metalation reaction, which follows by the reaction with another molecule of imine to yield coupling products.[4,5] However, this metalation process could be negligible in the present system, because the hydrosilylation proceeded effectively, at least for aldimines, and neither C-silylated imine nor a coupling product was detected.

In summary, catalytic dehydrogenative silylation of amines and hydrosilylation of imines have been achieved by using Yb−imine complexes. The two reactions proceed through common intermediates and thus exhibit similar product selectivities and yields. Although the present reactions include restrictions on the hydrosilanes and substrates, they provide a potentially useful method for the preparation of aminosilanes and diaminosilanes because readily available nonlanthanocene catalysts can be utilized under mild conditions.

Experimental Sections

General. ^1H and ^{13}C NMR spectra were recorded at 270 and 67 MHz, respectively. IR spectra were taken on a FT-IR spectrophotometer. Mass spectra were obtained at 70 eV on a GC−MS apparatus. Microanalyses were performed at our analytical laboratory. All reactions were carried out under argon. THF was distilled from sodium/benzophenone ketyl immediately prior to use. HMPA was distilled from CaH_2 and stored over molecular sieves. Ytterbium metal (40 mesh) was washed with anhydrous hexane under argon and dried in vacuo. Imines **6** were prepared from the corresponding amines and aldehydes or ketones by the conventional procedure. Amines **2i** and **2k**−**p** were prepared by the reduction of imines **6b** and **6d**−**i** with $LiAlH_4$. All other materials were commercially available and were used after distillation.

General Procedure for Dehydrogenative Silylation of Amines. Ytterbium metal (12.9 mg, 0.075 mmol) and diphenylmethylidene-2,6-xylylamine (21.4 mg, 0.075 mmol) were placed in a Schlenk tube. HMPA (53 μL, 0.3 mmol) and THF (1 mL) were added to the mixture. After addition of MeI (0.2 μL) to activate the metal, the mixture was stirred for 4 h at room temperature to yield a homogeneous reddish-black solution of the Yb−imine complex **1c**. Hydrosilane (2.5 mmol) and amine (2.5 mmol) were successively added to the solution, and the mixture was stirred at room temperature for the appropriate time as indicated in Tables 1 and 2. The reaction mixture was directly passed through a short alumina column or subjected to Kugelrohr distillation without an aqueous workup. In the reaction with $PhSiH_3$ (Table 4), a solution of the silane and amine in THF (1 mL) was added to the imine complex **1a′** to avoid oligomerization of the silane.

General Procedure for Hydrosilylation of Imines. The imine complex **1a′** was prepared from Yb metal (34.6 mg, 0.2 mmol), diphenylmethylideneaniline (51.5 mg, 0.2 mmol), and

HMPA (209 μL, 1.2 mmol) in THF (1 mL) as above. A solution of phenylsilane (216 mg, 2 mmol) and imine (2 mmol) in THF (1 mL) was added to the mixture, and stirring was continued for 20 h at room temperature with monitoring by GC. The products were isolated as amines on hydrolysis, as described bellow.

Identification of Products and Determination of Their Yields. Aminosilanes **3**, **4**, and **7** and diaminosilanes **5** and **8**, products of the dehydrogenative silylation of amines and hydrosilylation of imines, are quite unstable and readily hydrolyze to the corresponding amines and silanol or siloxane. Thus, their complete purification through aqueous workup could not be done. However, of the products, aminotriphenylsilanes **3a** and **3f**−**k** were relatively stable and could be directly chromatographed on neutral alumina (ca. 70 mm × 15 mm i.d.) with anhydrous hexanes−ethyl acetate (20/1) eluent to give almost pure samples, which were identified by ^{1}H and ^{13}C NMR. Their GC yield was determined by using an internal standard such as eicosane. Aminosilanes **3b**−**d** were isolated by Kugelrohr distillation directly from the reaction mixture, which contained a small amount of impurities such as HMPA and siloxanes, judged by ^{1}H and ^{13}C NMR spectra. The product **3e** was highly unstable and thus detected only by GC−MS of the mixture; yield was determined by GC using the relative intensity of **3a**.

Reaction mixtures of the hydrosilylation and dehydrogenative silylation with $PhSiH_3$ (Tables 3 and 4) were very labile on workup similar to the above. The two reactions gave the same products **7** and **8** monitored by GC analysis, wherein **7** was gradually changed to **8** as the reaction proceeded. On hydrolysis of the mixture, **7** and **8** disappeared immediately, and the corresponding amine was formed, which was identified with an authentic sample by GC. Product yields were determined on the basis of the ratio of GC peak areas of the starting substrates **6** or **2** and products **7** and **8**, assuming that all relative intensities were equal. This calculation agreed fairly well with a quantity of amine **2** obtained by the hydrolysis of the mixture of the imine hydrosilylation.

Supporting Information Available: Characterization data (^{1}H and ^{13}C NMR and MS spectra) for products **3** and **5a**. This material is available free of charge via the Internet at http://pubs.acs.org.

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