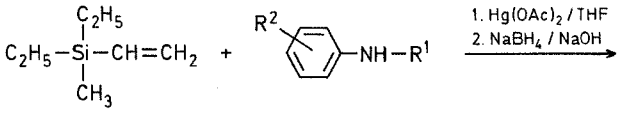
Synthesis of Substituted 2-Aminoalkylsilanes via Aminomercuration-Demercuration of Alkenylsilanes

J. BARLUENGA*, C. JIMÉNEZ, C. NÁJERA, M. YUS

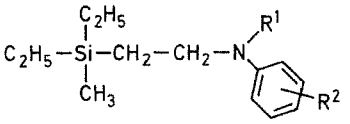
Departamento de Química Orgánica, Facultad de Ciencias, Universidad de Oviedo, Oviedo, Spain

The aminomercuration-demercuration reaction of functionalized unsaturated systems represents a convenient method for the regiospecific one-pot synthesis of nitrogen-containing 1,2-bifunctional compounds[1]. On the other hand, most of the methods described for the preparation of 2-aminoalkylsilanes[2,3] are rather complicated and are not generally applicable. We therefore investigated the preparation of 2-aminoalkylsilanes via aminomercuration-demercuration of alkenylsilanes.

We found that the reaction of diethylmethylvinylsilane[4] **(1)** with primary or secondary arenamines **(2)**[5] in tetrahydrofuran in the presence of mercury(II) acetate at room temperature followed by reduction with sodium borohydride in the basified reaction medium leads to the formation of 2-aminoethyldiethylmethylsilanes **(3)**.



1 **2**



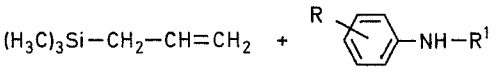
3

R^1 = H, CH_3

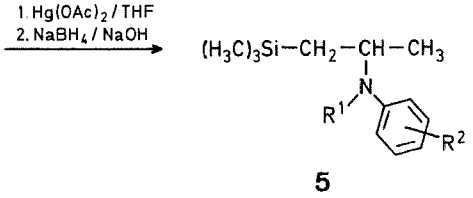
R^2 = H, 4-Cl

The ability of a C—Si bond to stabilize an adjacent carbenium ion[6] favors the formation of the anti-Markovnikov[7] addition product in the mercuration process.

Aminomercuration-demercuration of allyltrimethylsilane[8] **(4)** under the same conditions affords 2-aminopropyltrimethylsilanes **(5)** in accord with the Markovnikov addition rule[7].



4 **2**



5

R^1 = H, CH_3

R^2 = H, 4-CH_3

The formation of compounds **5** in this reaction is in contrast to the oxymercuration-demercuration of allyltrimethylsilane **(4)**[9] which yields C—Si cleavage products instead of the addition product 1-trimethylsilyl-2-propanol.

The aminomercuration of diallyldimethylsilane[8] **(6)** followed by reduction with sodium metal and hydrolysis affords the mono- and diamination products **7** and **8**. 1-Aza-4-silacyclohexanes **(9)** have in no case been obtained from primary arenamines (**2**, $R^1 = H$)[10].

The predominant formation of either products **7** or **8** depends on the reaction conditions. When the reduction with sodium is carried out immediately after aminomercuration is complete the monoamines **7**, formed via deaminomercuration[11], are the main products. When the solvents are removed after the aminomercuration step and the resultant organomercury compound is reduced with sodium/aniline in tetrahydrofuran the diamines **8** are the main products. Compounds **7** and **8** can be easily separated by vacuum distillation. When secondary aromatic amines (**2**, $R^1 \neq H$) are used compounds **7** are obtained exclusively.

1-Aza-4-silacyclohexanes **(9)** are prepared by intramolecular aminomercuration of the 2-aminopropyldimethylallylsilanes **7**

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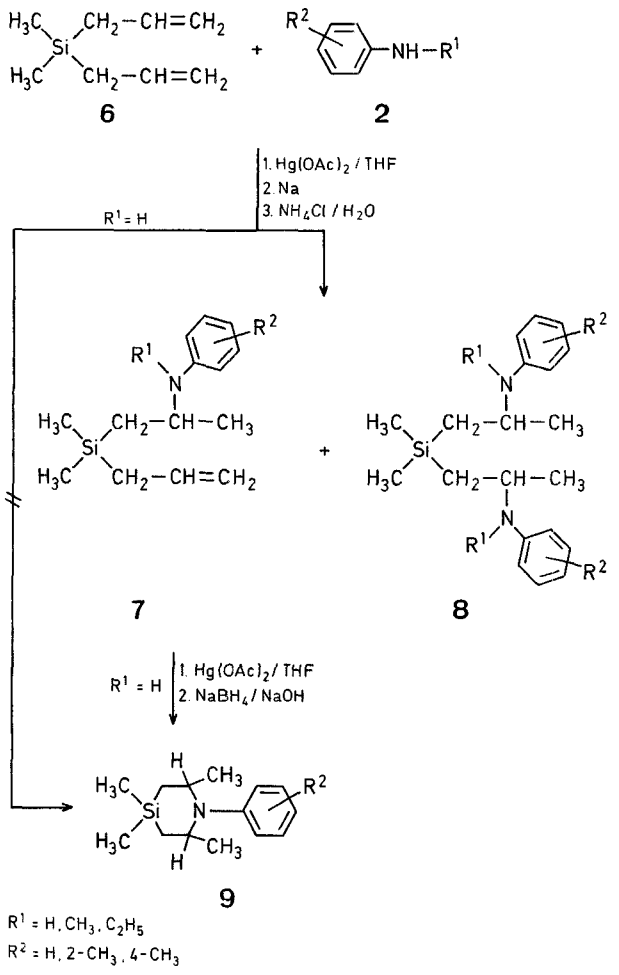

6 2 7 8 9

followed by reduction with sodium borohydride in alkaline medium. Compounds **9** are separated from the accompanying starting material **7** by conversion of **7** into the corresponding acetamide by treatment of the ethereal solution of the product mixture with acetyl chloride. Compounds **9** are isolated as mixtures of *cis*- and *trans*-isomers[10,11].

Diethylmethylvinylsilane (**1**) was prepared by reaction of commercially available dichloro-(methyl)-vinylsilane (Aldrich) with ethylmagnesium bromide and hydrolysis; b.p. 117–118°C (Ref.[4], b.p. 118°C).

Allyltrimethylsilane (**4**) was prepared according to Ref.[5]; yield: 85%; b.p. 84–85°C (Ref.[6], b.p. 84–85.5°C).

Diallyldimethylsilane (**6**) was prepared according to Ref.[5]; yield: 80%; b.p. 134–135°C (Ref.[6], b.p. 134–135°C).

N-[2-(Diethylmethylsilyl)-ethyl]-aniline (3a); Typical Procedure for Compounds 3 and 5:

Mercury(II) acetate (4.78 g, 15 mmol) is added to a stirred solution of diethylmethylvinylsilane (**1**; 1.95 g, 15 mmol, purity ≥98%) and aniline (10 ml) in tetrahydrofuran (50 ml). Stirring is continued for 3 h at room temperature; then, 0.5 normal aqueous sodium hydroxide (75 ml) and a solution of sodium borohydride (0.38 g, 10 mmol) in 2.5 normal aqueous sodium hydroxide (10 ml) are added. After 15 min, the mixture is extracted with ether (2 × 50 ml) and the organic layer is washed with water (50 ml) and dried with sodium sulfate. The solvent is removed in vacuo and the residue is distilled in vacuo; yield: 2.2 g (65%); b.p. 80–83°C/0.001 torr.

$C_{13}H_{23}NSi$	calc.	C 70.52	H 10.47	N 6.33
(221.4)	found	70.44	10.42	6.28

N-[1-Methyl-2-(allyldimethylsilyl)-ethyl]-aniline (7a); Typical Procedure:

To a stirred solution of diallyldimethylsilane (**6**; 1.4 g, 10 mmol) and aniline (10 ml) in tetrahydrofuran (50 ml), mercury(II) acetate (6.36 g, 20 mmol) is added and stirring is continued for 10 h. Then, a dispersion of sodium (3.2 g; ~140 mmol) in toluene is added under an argon atmosphere. After 4 d, the reaction mixture is cooled to 0°C and hy-

Table 1. Preparation of 2-Aminoalkylsilanes **3, 5, 7, 8, 9**

Product	Starting silane	R^1	R^2	Mercuration time	Reducing agent	Reduction time	Yield [%] Hg[a]	Yield [%] Product[a]	b.p./torr [°C]	Molecular formula[b] or Lit. b.p./torr [°C]
3a	**1**	H	H	3 h	$NaBH_4$	15 min	85	65	80–83°/0.001	$C_{13}H_{23}NSi$ (221.4)
3b	**1**	H	4-Cl	1 d	$NaBH_4$	2 d	90	72	98–100°/0.001	$C_{13}H_{22}ClNSi$ (255.9)
3c	**1**	CH_3	H	2 h	$NaBH_4$	30 min	80	55	78–81°/0.001	$C_{14}H_{25}NSi$ (235.4)
5a	**4**	H	H	3 h	$NaBH_4$	16 h	85	75	50–53°/0.001	50–52°/0.001[3]
5b	**4**	H	4-CH_3	11 h	$NaBH_4$	1 h	86	50	58–61°/0.001	$C_{13}H_{23}NSi$ (221.4)
5c	**4**	CH_3	H	15 min	$NaBH_4$	1 h	80	56	82–85°/0.1	$C_{13}H_{23}NSi$ (221.4)
7a	**6**	H	H	10 h	Na[c]	4 d	99	40 (17)[d]	83–86°/0.001	$C_{14}H_{23}NSi$ (233.4)
7b	**6**	H	2-CH_3	20 h	Na[c]	9 d	96	30 (10)[d]	82–85°/0.001	$C_{15}H_{25}NSi$ (247.5)
7c	**6**	H	4-CH_3	8 h	Na[c]	3 d	97	45 (3)[d]	78–80°/0.001	$C_{15}H_{25}NSi$ (247.5)
7d	**6**	CH_3	H	6 h	Na[c]	2 d	93	42 (< 1)[d]	80–83°/0.001	$C_{15}H_{25}NSi$ (247.5)
7e	**6**	C_2H_5	H	1 h	Na[c]	3 d	65	40 (< 1)[d]	77–80°/0.001	$C_{16}H_{27}NSi$ (261.5)
8a	**6**	H	H	10 h	Na[c]	3 d	95	44 (12)[f]	115–118°/0.001	$C_{20}H_{30}N_2Si$ (326.6)
8b	**6**	H	2-CH_3	20 h	Na[c]	5 d	92	30 (8)[f]	118–120°/0.001	$C_{22}H_{34}N_2Si$ (354.6)
8c	**6**	H	4-CH_3	8 h	Na[c]	5 d	91	47 (11)[f]	120–122°/0.001	$C_{22}H_{34}N_2Si$ (354.6)
9a[g]	**7a**	—	H	9 h	$NaBH_4$	3 d	82	32 (61)[h]	75–80°/0.001	$C_{14}H_{23}NSi$ (233.4)
9b[i]	**7b**	—	2-CH_3	1 d	$NaBH_4$	4 h	65	25 (56)[h]	76–81°/0.001	$C_{15}H_{25}NSi$ (247.5)
9c[j]	**7c**	—	4-CH_3	5 h	$NaBH_4$	45 min	75	22 (72)[h]	70–75°/0.001	$C_{15}H_{25}NSi$ (247.5)

[a] Based on starting silane (**1**, **4**, **6**, **7**); determined by G.L.C. analysis.

[b] The microanalyses were in good agreement with the calculated values: C, ±0.13; H, ±0.12; N, ±0.10.

[c] Immediate reduction of the organomercury compound in the reaction mixture.

[d] The yield of **8** is given in parentheses.

[e] The solvent is first evaporated and the organomercury compound then reduced with sodium/aniline in tetrahydrofuran.

[f] The yield of **7** is given in parentheses.

[g] *cis*/*trans* Ratio: 58/42 (G.L.C. analysis).

[h] The yield of starting material **7** is given in parentheses (G.L.C. analysis).

[i] *cis*/*trans* Ratio: 30/70 (G.L.C. analysis).

[j] *cis*/*trans* Ratio: 53/47 (G.L.C. analysis).

Table 2. Spectral Data of Compounds **3, 5, 7, 8, 9**

Product	I.R.[a] (film) ν [cm^{-1}] N—H	C—Si	^{1}H-N.M.R. (CCl_4/TMS)[b] δ [ppm]	^{13}C-N.M.R. (CCl_4)[b] δ [ppm][c]
3a	3400	810	0.05 (s, 3H, H_3C—Si); 0.6 (m, 4H, C—CH_2—Si); 0.8 (t, 2H, J=7.5 Hz, N—CH_2—CH_2—Si); 0.9 (t, 6H, J=6 Hz, C—CH_3); 3.1 (m, 2H, CH_2—N); 3.4 (broad s, 1H, NH); 6.4–7.3 (m, 5H_{arom})[d]	−6.9 (q, H_3C—Si); 4.4 (t, H_3C—CH_2Si); 6.4 (q, C—CH_3); 13.7 (t, N—CH_2—CH_2—Si); 39.0 (t, CH_2—N); 111.9, 116.2, 128.1, 147.4 (C_{arom})
3b	3400	820	0.05 (s, 3H, H_3C—Si); 0.6 (m, 4H, C—CH_2—Si); 0.85 (m, 2H, N—CH_2—CH_2—Si); 0.95 (t, 6H, J=6 Hz, C—CH_3); 3.1 (m, 2H, CH_2—N); 3.5 (broad s, 1H, NH); 6.3–7.3 (m, 4H_{arom})[d]	−6.9 (q, H_3C—Si); 4.3 (t, H_3C—CH_2Si); 6.3 (q, C—CH_3); 13.4 (t, N—CH_2—CH_2—Si); 39.8 (t, CH_2—N); 113.5, 115.5, 128.0, 145.2 (C_{arom})
3c	—	800	0.05 (s, 3H, H_3C—Si); 0.6 (m, 4H, C—CH_2—Si); 0.85 (m, 2H, N—CH_2—CH_2—Si); 0.95 (t, 6H, J=6 Hz, C—CH_3); 2.85 (s, 3H, H_3C—N); 3.35 (m, 2H, CH_2—N); 6.3–7.3 (m, 5H_{arom})	−7.1 (q, H_3C—Si); 4.3 (t, H_3C—CH_2—Si); 6.4 (q, C—CH_3); 9.2 (t, N—CH_2—CH_2—Si); 36.2 (t, CH_2—N); 47.4 (t, H_3C—N); 112.1, 115.5, 128.0, 148.2 (C_{arom})
5a	3400	860	0.05 (s, 9H, H_3C—Si); 0.8 (2d, 2H, J=6 Hz, CH_2); 1.1 (d, 3H, J=6 Hz, C—CH_3); 3.1 (broad s, 1H, NH); 3.5 (m, 1H, CH); 6.3–7.2 (m, 5H_{arom})	−1.8 (q, H_3C—Si); 22.8 (q, C—CH_3); 25.3 (t, CH_2); 44.9 (d, CH); 112.3, 115.8, 128.1, 146.3 (C_{arom})
5b	3400	850	0.05 (s, 9H, H_3C—Si); 0.8 (2d, 2H, J=6 Hz, CH_2); 1.2 (d, 3H, J=6 Hz, C—CH_3); 2.2 (s, 3H, Ar—CH_3); 3.0 (broad s, 1H, NH); 3.5 (m, 1H, CH); 6.2–7.0 (m, 4H_{arom})	−1.7 (q, H_3C—Si); 19.3 (q, Ar—CH_3); 23.1 (q, C—CH_3); 25.6 (t, CH_2); 45.4 (d, CH); 112.7, 124.5, 128.7, 144.2 (C_{arom})
5c	—	860	0.05 (s, 9H, H_3C—Si); 0.8 (2d, 2H, J=6 Hz, CH_2); 1.15 (d, 3H, J=6 Hz, C—CH_3); 2.6 (s, 3H, N—CH_3); 4.0 (m, 1H, CH); 6.4–7.2 (m, 5H_{arom})	−1.8 (q, H_3C—Si); 19.1 (q, C—CH_3); 22.3 (t, CH_2); 29.0 (q, N—CH_3); 49.9 (d, CH); 112.9, 116.0, 128.1, 149.3 (C_{arom})
7a	3400	830	0.05 (s, 6H, H_3C—Si); 0.85 (2d, 2H, J=6 Hz, C—CH_2—Si); 1.2 (d, 3H, J=6 Hz, C—CH_3); 1.5 (d, 2H, J=8 Hz, CH_2—C=); 3.1 (broad s, 1H, NH); 3.6 (m, 1H, CH—N); 4.8 (m, 2H, H_2C=); 5.7 (m, 1H, CH=); 6.4–7.3 (m, 5H_{arom})	−3.7 (q, H_3C—Si); 23.0, 23.2, 23.8 (2×CH_2—Si, C—CH_3); 45.4 (d, CH—N); 112.3 (t, H_2C=); 112.5, 116.2, 128.3, 146.4 (C_{arom}); 133.8 (d, CH=)
7b	3400	850	0.05 (s, 6H, H_3C—Si); 0.8 (2d, 2H, J=6 Hz, C—CH_2—Si); 1.15 (d, 3H, J=6 Hz, C—CH_3); 1.5 (d, 2H, J=8 Hz, CH_2—C=); 2.0 (s, 3H, Ar—CH_3); 3.0 (broad s, 1H, NH); 3.6 (m, 1H, CH—N); 4.75 (m, 2H, H_2C=); 5.65 (m, 1H, CH=); 6.2–7.1 (m, 4H_{arom})	−3.6 (q, H_3C—Si); 16.5 (q, Ar—CH_3); 23.0, 23.4, 24.1 (2×CH_2—Si, C—CH_3); 44.8 (d, CH—N); 109.7, 115.9, 120.5, 126.1, 129.3, 144.1 (C_{arom}); 112.3 (t, H_2C=); 133.6 (d, CH=)
7c	3400	840	0.05 (s, 6H, H_3C—Si); 0.8 (2d, 2H, J=6 Hz, C—CH_2—Si); 1.15 (d, 3H, J=6 Hz, C—CH_3); 1.5 (d, 2H, J=8 Hz, CH_2—C=); 2.2 (s, 3H, Ar—CH_3); 2.95 (broad s, 1H, NH); 3.6 (m, 1H, CH—N); 4.75 (m, 2H, H_2C=); 5.6 (m, 1H, CH=); 6.15–7.0 (m, 4H_{arom})	−3.7 (q, H_3C—Si); 19.4 (q, Ar—CH_3); 23.0, 23.3, 24.0 (2×CH_2—Si, C—CH_3); 45.4 (d, CH—N); 109.3 (t, H_2C=); 112.8, 124.8, 128.8, 144.2 (C_{arom}); 133.8 (d, CH=)
7d	—	850	0.05 (s, 6H, H_3C—Si); 0.9 (2d, 2H, J=6 Hz, C—CH_2—Si); 1.15 (d, 3H, J=6 Hz, C—CH_3); 1.5 (d, 2H, J=8 Hz, CH_2—C=); 2.6 (s, 3H, N—CH_3); 4.1 (m, 1H, CH—N); 4.8 (m, 2H, CH_2=); 5.65 (m, 1H, CH=); 6.45–7.3 (m, 5H_{arom})	−3.9 (q, H_3C—Si); 19.0, 20.6, 22.7 (2×CH_2—Si, C—CH_3); 29.1 (q, N—CH_3); 49.8 (d, CH—N); 112.4 (t, CH_2=); 112.9, 116.2, 128.1, 149.2 (C_{arom}); 133.6 (d, CH=)
7e	—	850	0.05 (s, 6H, H_3C—Si); 0.7–1.3 (m with d at 1.15, 8H, J=6 Hz, CH—CH_3, CH_2—CH_3, C—CH_2—Si); 1.5 (d, 2H, J=8 Hz, CH_2—C=); 3.1 (m, 2H, CH_2—N); 3.95 (m, 1H, CH—N); 4.8 (m, 2H, H_2C=); 5.65 (m, 1H, CH=); 6.3–7.3 (m, 5H_{arom})	−3.7 (q, H_3C—Si); 3.7 (q, CH_2—CH_3); 20.5, 22.0, 28.8 (2×CH_2—Si, CH—CH_3); 36.7 (t, CH_2—N); 50.1 (d, CH—N); 112.4 (t, H_2C=); 113.6, 115.9, 128.1, 147.3 (C_{arom}); 133.5 (d, CH=)
8a	3330	860	0.05 (s, 6H, H_3C—Si); 0.8 (d, 4H, J=6 Hz, CH_2—Si); 1.1 (d, 6H, J=6 Hz, C—CH_3); 2.8 (broad s, 2H, NH); 3.6 (m, 2H, CH); 6.3–7.2 (m, 10H_{arom})	−2.4 (q, H_3C—Si); 23.3 (q, C—CH_3); 24.8 (t, CH_2—Si); 45.1 (d, CH); 112.5, 116.1, 128.3, 146.4 (C_{arom})
8b	3400	850	0.05 (s, 6H, H_3C—Si); 0.8 (2d, 4H, J=6 Hz, CH_2—Si); 1.1 (d, 6H, J=6 Hz, C—CH_3); 1.9 (s, 6H, Ar—CH_3); 3.0 (broad s, 2H, NH); 3.6 (m, 2H, CH); 6.2–7.0 (m, 8H_{arom})	−2.2 (q, H_3C—Si); 16.4 (q, Ar—CH_3); 23.5 (q, C—CH_3); 25.1 (t, CH_2—Si); 44.8 (d, CH); 109.7, 115.8, 120.5, 126.2, 129.3, 144.1 (C_{arom})
8c	3330	830	0.05 (s, 6H, H_3C—Si); 0.8 (2d, 4H, J=6 Hz, CH_2—Si); 1.1 (d, 6H, J=6 Hz, C—CH_3); 2.15 (s, 6H, Ar—CH_3); 3.1 (broad s, 2H, NH); 3.5 (m, 2H, CH); 6.1–7.0 (m, 8H_{arom})	−2.3 (q, H_3C—Si); 19.5 (q, Ar—CH_3); 23.4 (q, C—CH_3); 24.9 (t, CH_2—Si); 45.4 (d, CH); 112.8, 124.7, 128.8, 144.1 (C_{arom})
9a	—	850	0.05 (s, 6H, CH_3—Si); 0.6–1.3 (m with 2d at 0.85 and 1.1, 10H, J=6 Hz, CH_2—Si, 2×C—CH_3); 3.5–4.4 (m, 2H, CH); 6.2–7.2 (m, 5H_{arom})	—[e]
9b	—	850	0.05 (s, 6H, H_3C—Si); 0.5–1.2 (m with 2d at 0.85 and 1.1, 10H, J=6 Hz, CH_2—Si, 2×C—CH_3); 2.1 (s, 3H, Ar—CH_3); 3.1–3.7 (m, 2H, CH); 7.2 (m, 4H_{arom})	—[e]
9c	—	850	0.05 (s, 6H, H_3C—Si); 0.5–1.2 (m with 2d at 0.85 and 1.0, 10H, J=6 Hz, CH_2—Si, 2×C—CH_3); 2.1 (s, 3H, Ar—CH_3); 3.1–4.2 (m, 2H, CH); 6.8 (m, 4H_{arom})	—[e]

[a] Recorded in a Pye Unicam SP-1000 I.R. spectrometer.
[b] Recorded in a Varian FT-80 spectrometer with a D_2O capillary when CCl_4 is used as solvent.
[c] Referred to the solvent CCl_4 (or $CDCl_3$). Multiplicity was assigned by "off" resonance experiments.
[d] In $CDCl_3$ solution.
[e] Very complicated spectra.

drolyzed with methanol (20 ml) and 10% aqueous ammonium chloride (20 ml). The organic layer is extracted with ether (2 × 50 ml), washed with water (50 ml), and dried with sodium sulfate. The solvents are removed under reduced pressure (15 torr) and the residue is distilled in vacuo; yield: 0.9 g (40%); b.p. 83–86 °C/0.001 torr.

$C_{14}H_{23}NSi$	calc.	C 72.04	H 9.93	N 6.00
(233.4)	found	71.97	9.88	5.95

***N,N'*-Diphenyl-4,4-dimethyl-4-silaheptane-2,6-diamine (8a); Typical Procedure:**

After the mercuration process as described above for the preparation of compound **7a**, the solvents are removed under reduced pressure (15 and then 0.001 torr). The resultant oil is dissolved in tetrahydrofuran (50 ml) and aniline (10 ml) and a dispersion of sodium (3.2 g, ~140 mmol) in toluene is added under an argon atmosphere. After 3 d, the mixture is hydrolyzed and extracted as above. The residue is distilled in vacuo; yield: 1.4 g (44%); b.p. 115–118 °C/0.001 torr.

$C_{20}H_{30}N_2Si$	calc.	C 73.56	H 9.26	N 8.58
(326.6)	found	73.53	9.21	8.51

***cis*- and *trans*-1-Phenyl-2,4,4,6-tetramethyl-1-aza-4-silacyclohexane (9a); Typical Procedure:**

To a stirred solution of *N*-[1-methyl-2-(allyldimethylsilyl)-ethyl]-aniline (**7a**; 1.2 g, 5 mmol) in tetrahydrofuran (30 ml), mercury(II) acetate (1.6 g, 5 mmol) is added and stirring is continued for 9 h. Then, 0.5 normal aqueous sodium hydroxide (25 ml) and, thereafter, a solution of sodium borohydride (0.19 g, 5 mmol) in 2.5 normal aqueous sodium hydroxide (5 ml) are added. After 3 d, the mixture is extracted with ether (2 × 25 ml) and the organic layer washed with water (25 ml) and dried with sodium sulfate. A solution of acetyl chloride (0.7 ml, 10 mmol) in ether (10 ml) is added dropwise to the stirred ethereal solution containing **7a** and **9a**. The mixture is hydrolyzed with water (10 ml), the organic layer washed with aqueous sodium hydrogen carbonate (20 ml), and evaporated at 15 torr to give **7a** as the corresponding acetamide. The aqueous layer is alkalinized with an 10% aqueous solution of sodium hydroxide, extracted with ether (2 × 10 ml), dried with sodium sulfate, evaporated, and the residue distilled; yield of **9a**: 0.4 g (32%); b.p. 75–80 °C/0.001 torr.

$C_{14}H_{23}NSi$	calc.	C 72.04	H 9.93	N 6.00
(233.4)	found	71.93	9.81	5.90

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* Address for correspondence.

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