Peculiarities of the reaction of alkali metal bis(trimethylsilyl)amides with halobenzenes*

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Lithium and sodium bis(trimethylsilyl)amides react with fluoro-, bromo-, and chlorobenzenes in THF or toluene to give a mixture of *N*,*N*-bis(trimethylsilyl)aniline and *N*,2-bis(trimethylsilyl)aniline. The latter compound is resulted from 1,3-shift of the trimethylsilyl group from nitrogen to *ortho*-carbon atom of the benzene ring. Effects of the solvent, halogen, and alkali metal nature as well as the reaction conditions on the ratio of isomers were examined. Reaction of iodobenzene with sodium bis(trimethylsilyl)amide in THF produces *N*,*N*-bis(trimethylsilyl)aniline and 2-iodo-*N*,*N*-bis(trimethylsilyl)aniline, while in toluene a mixture of three products, two indicated above and *N*,*N*-bis(trimethylsilyl)benzylamine, was obtained.

Key words: dehydrobenzene, alkali metal bis(trimethylsilyl)amides, halobenzenes, trimethylsilyl group shift.

In recent years, the interest in aryne chemistry shows fast increase due to unique reactivity of arynes and their promising applications in organic synthesis.[1—3] To date, a number of methods for the aryne generation are known,[4] which have both advantages and certain limitations. Therefore, the aryne generation is intensively studied.

Earlier, we have found[5] that bromobenzene reacts with sodium bis(trimethylsilyl)amide (**1**) to give a mixture of *N*,*N*-bis(trimethylsilyl)aniline (**2**) and *N*,2-bis(trimethylsilyl)aniline (**3**), which resulted from the trimethylsilyl group 1,3-shift from the nitrogen atom to the *ortho*-carbon of the benzene ring. The reaction proceeds *via* dehydrobenzene intermediate. Subsequent nucleophilic addition of hexamethyldisilazane to dehydrobenzene could be accompanied by the trimethylsilyl group migration. Intermediate formation of dehydrobenzene is confirmed by the obtaining *N*,*N*-diethylaniline in the reaction carried out in triethylamine.[6]

Reaction of halobenzenes with alkali metal bis(trimethylsilyl)amides is of interest in two aspects. First, this reaction is a new route for the aryne generation. Second, since the 1,3-shift of trimethylsilyl group from nitrogen to carbon is rather scarcely studied (we found the only example[7] described by Yoshida *et al.*), it is of interest to reveal the effects of different factors on rearrangement accompanying the reaction of aryl halides with alkali metal silylamides. These findings will allow rational synthesis of various types of aromatic silicon-nitrogen compounds. In the present work, we studied the effect of the nature of the halogen atom in halobenzenes (PhHal) on structure and compositions of the products arising from their reactions with lithium and sodium bis(trimethylsilyl)amides.

It has been noted earlier[8] that the halogen nature does not affect the yield and ratio of the products in the reactions of arynes generated by treatment of aryl halides with strong bases. Moreover, the extensive structural and mechanistic studies of transformations of 2-halo derivatives of aryllithium into the corresponding arynes[9] show substantial effects of the nature of both solvent and the leaving halogen atom on regioselectivity of these reactions.

We found that the reactions of bromo- (**4**), chloro- (**5**), and fluorobenzenes (**6**) with sodium and lithium bis(trimethylsilyl)amides (**1** and **7**, respectively) in all cases afford a mixture of compounds **2** and **3** (GC and NMR data). The ratio of products **2** and **3** is predetermined by the structure of the started reagents and the reaction conditions but it is not affected by the degree of conversion.

The data given in Table 1 show that the isomeric ratio is virtually independent on reaction temperature and is determined by the solvent polarity and the nature of the halogen atom in halobenzene PhHal **4—6**. At 100 °C, the content of the rearrangement product formed in THF is decreased in the following order **4** > **5** > **6**, while in less polar toluene, the order is reversed.

Examining the effect of the molar reagent ratio on the composition of the products formed in the reaction of **1** with halobenzenes **4—6** revealed that content of product **2** decreases upon an increase in the amount of halobenzene

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Table 1. Effects of the solvent nature, halogen, and reaction temperature on the ratio of products **2** and **3** formed in the reaction of $(Me_3Si)_2NNa$ (**1**) with halobenzenes **4**—**6**[a]

PhHal	Ratio **2** : **3** (yield (%))		
	Toluene, 100 °C	Toluene, 130 °C	THF, 100 °C
4	3.69 : 1 (56)	3.33 : 1 (53)	0.74 : 1 (58)
5	2.72 : 1 (46)	4.1 : 1 (48)	1.15 : 1 (76)
6	1.7 : 1 (49)	1.5 : 1 (53)	50 : 1 (45)[b]

[a] Reaction time is 3 h.

[b] Reaction time is 48 h.

Table 2. Effect of the reagent ratio on the ratio of products **2** and **3** formed in the reaction of $(Me_3Si)_2NNa$ (**1**) with halobenzenes **4** and **5***

Ratio PhHal : **1**	Ratio **2** : **3** (yield (%))		
	Toluene, 130 °C		THF, 100 °C
	4	**5**	**4**
1 : 2	12.1 : 1 (66)	7.3 : 1 (63)	8.9 : 1 (54)
1 : 1	3.9 : 1 (60)	4.2 : 1 (57)	0.85 : 1 (61)
2 : 1	2.6 : 1 (67)	2.8 : 1 (60)	0.65 : 1 (65)

* Reaction time is 3 h.

relative to sodium bis(trimethylsilyl)amide **1** (Table 2). Similar antibate dependence was also observed by varying both solvents and halogen atom in halobenzene. A decrease in the amount of halobenzenes **4**—**6** in the reaction mixture always results in a significant increase in the content of product **2** (see Table 2).

Table 3 summarizes the effects of the addition of hexamethyldisilazane **8** in two-fold excess relative to the reagents.

From Table 3, it is clearly seen that addition of hexamethyldisilazane **8** to the reaction mixture leads to a noticeable increase in the content of product **2**, when the reaction is carried out in toluene (*cf*. Table 1). However, in more polar THF the effect of additives of $(Me_3Si)_2NH$ **8** is less pronounced. Reaction of sodium bis(trimethylsilyl)amide **1** with bromobenzene **4** in hexamethyldisilazane **8** affords a mixture of products **2** and **3** in a 4 : 1 ratio in 45% total yield.

Reaction of halobenzenes **4**—**6** with lithium bis(trimethylsilyl)amide $(Me_3Si)_2NLi$ (**7**) also produces a mixture of products **2** and **3**, however their ratio is noticeably different from one obtained in the reactions involving compound **1** (Table 4).

A comparison of the data given in Tables 1 and 4 revealed that the content of the rearrangement product **3** formed in the reactions of halobenzenes with lithium amide **7** in THF increases in the order **4** < **5** < **6**, while in the similar reactions involving $(Me_3Si)NNa$ **1** a ratio of products **2** : **3** changes in the reversed order.

Hartwig and co-workers have studied Pd-catalyzed regioselective synthesis of arylamines *via* the reaction of aryl chlorides and aryl bromides with lithium bis(trimethylsilyl)amide (**7**).[10] For instance, they have examined catalyst-free reactions of some aryl bromides with lithium amide **7** in toluene: migration of the trimethylsilyl group to the aromatic ring did not occur, but the yields of arylamines were relatively low. These data are in agreement with the results obtained by us (see Table 4) since the trimethylsilyl group shift is observed only in the reactions of compounds **7** and **6**.

Thus, several factors enable formation of rearrangement product **3**. The content of product **3** is higher when using sodium bis(trimethylsilyl)amide **1** but not its lithium analog **7** and decreases in the following order of halobenzenes **4** > **5** > **6**. An increase in both the solvent polarity and mole fraction of halobenzenes relative to the corresponding amide also favors formation of product **3**.

We obtained unexpected results by studying the reaction of iodobenzene (**9**) with sodium bis(trimethylsilyl)amide **1** in THF. The reaction produces a mixture of two products: compound **2** and 2-iodo-*N*,*N*-bis(trimethylsilyl)aniline (**10**) (Scheme 1) with a ratio **2** : **10** = 1 : 6 in 57% total yield.

Table 3. Effects of additives of hexamethyldisilazane **8** on the ratio of products **2** and **3** formed in the reaction of $(Me_3Si)_2NNa$ (**1**) with halobenzenes **4**—**6**[a,b]

PhHal	Ratio **2** : **3** (yield (%))	
	Toluene, 130 °C	THF, 100 °C
4	5.8 : 1 (69)	0.56 : 1 (66)
5	10.5 : 1 (67)	1.8 : 1 (73)
6	5.4 : 1 (51)	40 : 1 (48)[c]

[a] Reagent ratio is **8** : **1** : PhHal = 2 : 1 : 1.

[b] Reaction time is 3 h.

[c] Reaction time is 48 h.

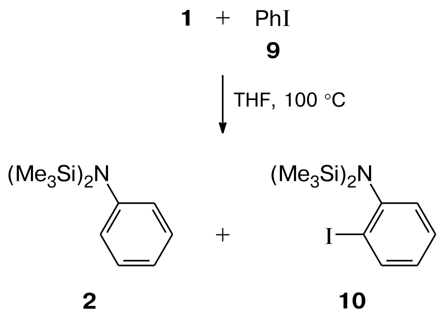
Table 4. Effects of the solvent and halogen nature on the ratio of products **2** and **3** formed in the reaction of lithium bis(trimethylsilyl)amide (**7**) with halobenzenes **4**—**6**[a]

PhHal	Ratio **2** : **3** (yield (%))	
	Toluene, 130 °C	THF, 100 °C
4	1 : 0 (45)[b]	4.65 : 1 (25)
5	1 : 0 (16)	3.77 : 1 (21)
6	2.1 : 1 (85)	3 : 1 (44)

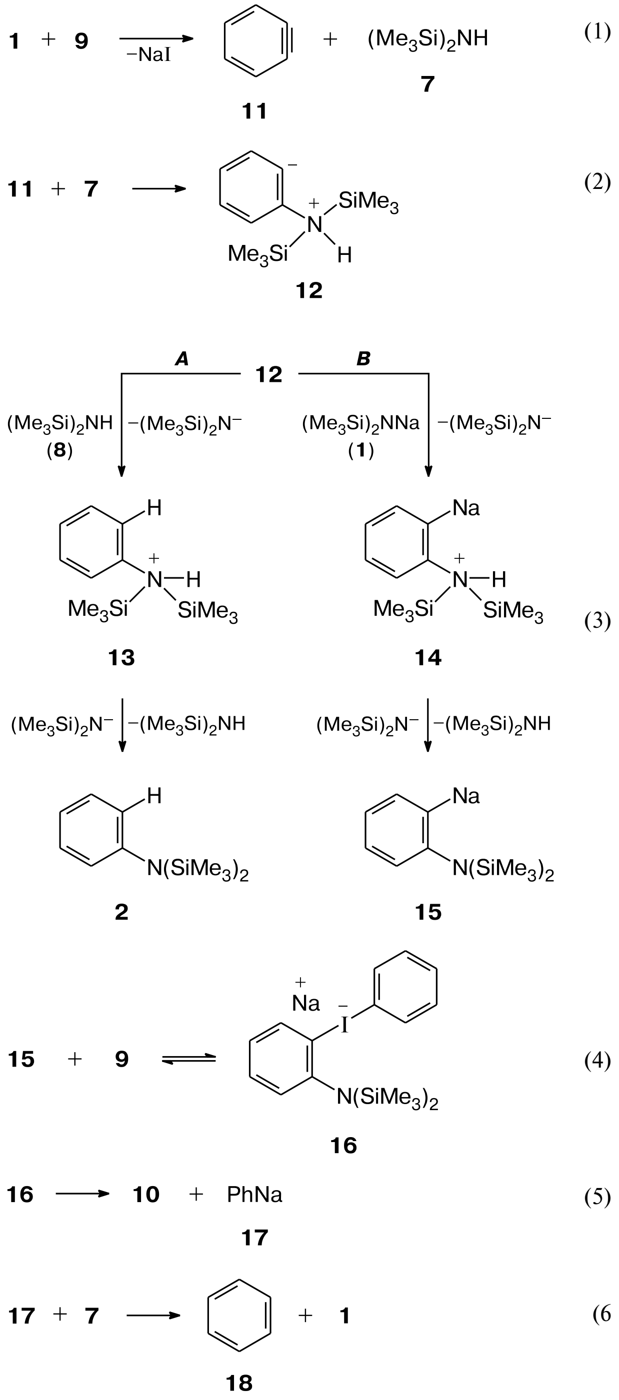
[a] Reaction time is 16 h.

[b] Reaction time is 24 h.

Scheme 1



Scheme 2



Mass-spectrometry confirms the formation of compound **10** exhibiting a peak for the molecular ion $[M]^+$ with *m/z* 363 and an intense peak for the fragment ion $[M - Me - I]^+$ with *m/z* 221. ^{1}H NMR spectrum contains additional singlet signal of the trimethylsilyl group at δ 0.12. ^{13}C NMR spectrum exhibits characteristic signal at δ 95.39 attributed to the C atom bonded to the iodine atom.

The reaction mechanism is shown on Scheme 2.

Similar mechanism involving simultaneous reactions proceeding *via* dehydrobenzene **11** and the corresponding iodine-ate complex (hypervalent iodine complex) was suggested by Durst and co-workers[11] upon studying the reaction of iodobenzene **9** with lithium tetramethylpiperidine in THF at –40 °C leading to a mixture of *N*-(2-iodophenyl)-2,2,6,6-tetramethylpiperidine and *N*-phenyl-2,2,6,6-tetramethylpiperidine.

Iodobenzene **9** reacts with $(Me_3Si)_2NNa$ **1** in hexamethyldisilazane **8** at 130 °C to give a mixture of three products **2**, **3**, and **10** in a ratio of 10 : 3.4 : 1 in 54% total yield. Formation of compound **3** and a dramatic decrease in the content of compound **10** can be due to the poor solubility of $(Me_3Si)_2NNa$ (**1**) in hexamethyldisilazane **8**. This leads to a significant decrease in concentration of intermediate dehydrobenzene **11** thus enabling the trimethylsilyl group 1,3-shift from nitrogen to C(2) atom of the benzene ring in zwitterion **12** (Scheme 3).

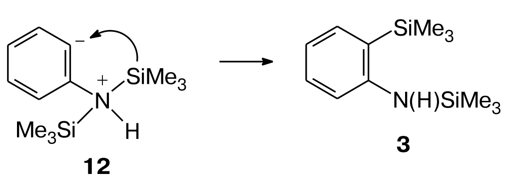
Simultaneously, low concentration of $(Me_3Si)_2NNa$ **1** results in a decrease in the rate of the reaction of zwitterion **12** following the ***B*** pathway (see Scheme 2, reaction (3)) to give a C(2)-metallated intermediate **15**. This, in turn, decreases the possibility of the iodine-ate complex to form and, consequently, formation of compound **10** is less probable.

Reaction of iodobenzene **9** with $(Me_3Si)_2NNa$ **1** in toluene proceeds unexpectedly affording three products, **2**, **10**, and *N*,*N*-bis(trimethylsilyl)benzylamine (**19**), in the ratio of 5.9 : 1 : 1 in 60% total yield (Scheme 4). This reaction also produces benzene (**18**), which is confirmed by GC using an internal standard.

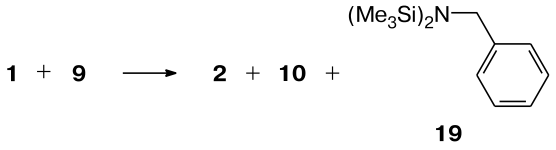
Formation of amine **19** is evidenced by the presence in mass spectrum of a molecular ion peak with *m/z* 251 $[M]^+$ and a fragment ion peak with *m/z* 162 $[M - Bn]^+$; ^{1}H NMR spectrum contains the singlet signal of the Me_3Si group at δ 0.08. ^{1}H and ^{13}C NMR spectra reveal the signals of the methylene group at δ 4.09 and 48.46, respectively.

A plausible mechanism of formation of product **19** is shown on Scheme 5. Iodine-ate complex **16** decomposes eliminating phenyl sodium (**17**) (see Scheme 2, reaction (5)), which metallates toluene (**20**) to give benzyl

Scheme 3



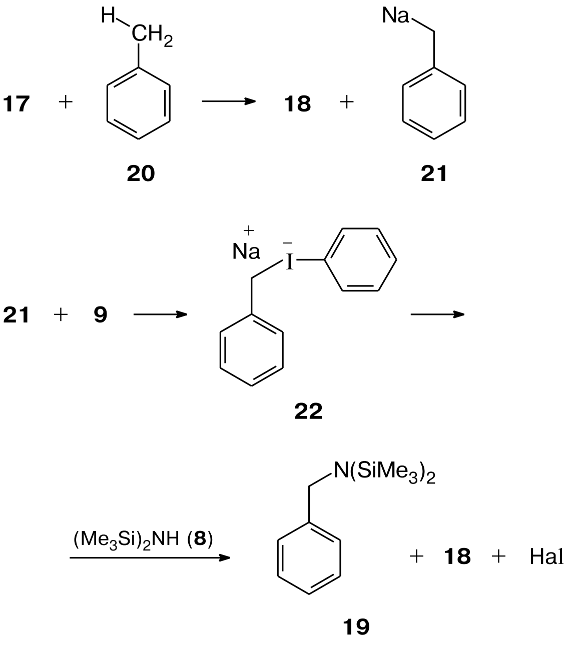
Scheme 4



Conditions: toluene, 100 °C.

sodium (**21**)[12] capable of producing the corresponding complex **22**.[13] Decomposition of complex **22** induced by hexamethyldisilazane **8** leads to amine **19**, benzene **18**, and sodium iodide.

Scheme 5



Thus, the reaction of sodium bis(trimethylsilyl)amide **1** with iodobenzene **9** differs sharply from the similar reaction with halobenzenes **4**—**6** (Hal = Br, Cl, F), which is due apparently to the formation of the corresponding iodine-ate complexes upon the reaction. This reaction can be regarded as a relatively convenient synthetic procedure towards *o*-iodoaniline and its *N*,*N*-disilyl derivative.

Experimental

NMR spectra were recorded on a Bruker DPX-400 instrument (working frequencies are 400.13 MHz (^{1}H), 100.58 MHz (^{13}C), and 79.46 MHz (^{29}Si)) in $CDCl_3$ using Me_4Si as an internal standard.

Mass spectrometry (EI, 70 eV) was performed with a Shimadzu GCMS-QP2010 Ultra instrument (column HP-1MS, 30000×0.25 mm) equipped with a quadruple mass filter, an ion source temperature of 250 °C, working mass range of 35—500 Da.

The compositions of the reaction mixtures were analyzed by GC performed on a Tsvet-500 gas chromatograph equipped with a thermal conductivity detector and a glass columns 3 m×4 mm filled with 10% polymethylsiloxane-1000 (PMS-1000) on Inerton-Super 0.125—0.150 mm using helium as a carrier gas.

Lithium bis(trimethylsilyl)amide (**7**) was synthesized by the known procedure.[14]

Yields and ratios of the products were determined as earlier described.[15]

Sodium bis(trimethylsilyl)amide (1). To a mixture of sodium (11.50 g, 500 mmol) and hexamethyldisilazane **8** (80.70 g, 500 mmol), 1,4-dioxane (100 mL) was added. A fine suspension of sodium was obtained by refluxing the above mixture with magnetic stirring, then styrene (26.0 g, 250 mmol) was added dropwise and the stirred reaction mixture was refluxed for 1 h. Cooling the reaction mixture resulted in crystallization of sodium bis(trimethylsilyl)amide **1** as large needles. Solvent was removed, product was washed with hexane, collected by filtration, and dried *in vacuo* (1 Torr) at 100 °C for 3 h to give the title product in the yield of 82.5 g (90%).

Reaction of sodium bis(trimethylsilyl)amide (1) with bromobenzene (4). To a solution of sodium bis(trimethylsilyl)amide **1** (49.5 g, 270 mmol) in toluene (250 mL), bromobenzene **4** (42.4 g, 270 mmol) was added dropwise at room temperature. Heating the reaction mixture to reflux resulted in precipitation. The reaction mixture was refluxed for 4 h. Removal of the solvent and subsequent vacuum distillation of the residue afforded *N*,*N*-bis(trimethylsilyl)aniline **2** (7.2 g) and *N*,2-(trimethylsilyl)aniline **3** (2.9 g).

***N*,*N*-Bis(trimethylsilyl)aniline (2).** B.p. 50 °C (1 Torr), n_D^{19} 1.4851 (*cf.* Ref. 16: b.p. 40—41 °C (0.3 Torr), n_D^{120} 1.4846). ^{1}H NMR, δ: 0.05 (s, 18 H, Me_3Si); 6.88 (dd, 2 H, H(2), H(6), *J* = 7.7 Hz, *J* = 1.1 Hz); 7.03 (tt, 1 H, H(4), *J* = 7.3 Hz, *J* = 1.1 Hz); 7.18 (dd, 2 H, H(3), H(5), *J* = 7.7 Hz, *J* = 7.3 Hz). ^{13}C NMR, δ: 2.1 (Me_3SiN); 123.6 (C(4)); 128.4 (C(3), C(5)); 130.23 (C(2), C(6)); 148.0 (C(1)). ^{29}Si NMR, δ: 4.5. MS, *m/z* (I_{rel} (%)): 237 $[M]^+$ (25), 222 $[M - 15]^+$ (100), 206 (20), 150 $[Me_3SiPh]^+$ (10), 73 $[Me_3Si]^+$ (15).

***N*,2-Bis(trimethylsilyl)aniline (3).** B.p. 85 °C (2 Torr), n_D^{19} 1.5049. ^{1}H NMR, δ: 0.27 (s, 9 H, Me_3SiN); 0.31 (s, 9 H, Me_3SiC); 3.58 (br.s, 1 H, NH); 6.71 (d, 1 H, H(6), *J* = 8.0 Hz); 6.73 (ddd, 1 H, H(4), *J* = 8.0 Hz, *J* = 7.2 Hz, *J* = 0.7 Hz); 7.18 (ddd, 1 H, H(5), *J* = 8.0 Hz, *J* = 7.2 Hz, *J* = 1.5 Hz); 7.28 (dd, 1 H, H(3), *J* = 7.2 Hz, *J* = 1.5 Hz). ^{13}C NMR, δ: –0.6 (Me_3SiC); 0.2 (Me_3SiN); 115.2 (C(6)); 117.6 (C(4)); 125.1 (C(2)); 130.4 (C(5)); 135.3 (C(3)); 152.4 (C(1)). ^{29}Si NMR, δ: 2.6 (Me_3SiN); –6.3 (Me_3SiC). MS, *m/z* (I_{rel} (%)): 237 $[M]^+$ (100), 222 $[M - 15]^+$ (80), 206 (25), 164 (10), 150 $[Me_3SiPh]^+$ (15), 73 $[Me_3Si]^+$ (80). Found (%): C, 60.72; H, 9.80; N, 5.88; Si, 23.60. $C_{12}H_{23}NSi_2$. Calculated (%): C, 60.69; H, 9.76; N, 5.90; Si, 23.65.

Reaction of sodium bis(trimethylsilyl)amide (1) with iodobenzene (9) in THF. To a solution of sodium bis(trimethylsilyl)amide **1** (18.3 g, 100 mmol) in THF (90 mL), iodobenzene **9** (20.4 g, 100 mmol) was added dropwise at room temperature. Heating the reaction mixture to reflux resulted in precipitation. The reaction mixture was refluxed for 4 h. Vacuum distillation afforded a mixture of *N*,*N*-bis(trimethylsilyl)aniline **2** and 2-iodo-*N*,*N*-bis(trimethylsilyl)aniline with b.p. 48—100 °C (1 Torr), yield 6.1 g, ratio **2** : **10** = 3.6 : 1. Compound **10** (0.75 g) was isolated by vacuum distillation.

2-Iodo-*N*,*N*-bis(trimethylsilyl)aniline (10). Oily liquid, b.p. 105 °C (1 Torr). ^{1}H NMR, δ: 0.12 (s, 18 H, Me_3Si); 6.76 (td, 1 H, H(4), J = 7.6 Hz, J = 1.6 Hz); 6.89 (dd, 1 H, H(6), J = 7.8 Hz, J = 1.5 Hz); 7.21 (td, 1 H, H(5), J = 7.5 Hz, J = 1.5 Hz); 7.82 (dd, 1 H, H(3), J = 7.9 Hz, J = 1.5 Hz). ^{13}C NMR, δ: 2.1 (Me_3SiN); 95.4 (C(2)); 122.7 (C(4)); 122.8 (C(6)); 126.8 (C(5)); 140.1 (C(3)); 151.0 (C(1)). ^{29}Si NMR, δ: 6.4. MS, m/z (I_{rel} (%)): 363 $[M]^+$ (9), 348 $[M - Me]^+$ (55), 221 $[M - Me - I]^+$ (100), 206 $[M - 2\ Me - I]^+$ (43), 73 $[Me_3Si]^+$ (23). Found (%): C, 39.71; H, 6.13; I, 34.85; N, 3.80; Si, 15.51. $C_{12}H_{22}INSi_2$. Calculated (%): C, 39.66; H, 6.10; I, 34.92; N, 3.85; Si, 15.46.

Reaction of sodium bis(trimethylsilyl)amide (1) with iodobenzene 9 in hexamethyldisilazane (7). A dry ampule flushed with dry argon was charged with compound **1** (3.2 g, 17.4 mmol), iodobenzene **9** (3.56 g, 17.4 mmol), and hexamethyldisilazane (**7**) (15 mL). After freeze—pump—thaw degassing, the ampule was sealed. The reaction was carried out at 130 °C for 4 h. Vacuum distillation afforded a mixture of compounds **2**, **3**, and **10** with b.p. 53—106 °C (1 Torr), yield 1.9 g, ratio **2** : **3** : **10** = 15 : 1.7 : 1.

Reaction of sodium bis(trimethylsilyl)amide (1) with iodobenzene (9) in toluene. To a solution of compound **1** (18.3 g, 100 mmol) in toluene (100 mL), iodobenzene **9** (20.4 g, 100 mmol) was added dropwise at room temperature. Heating the reaction mixture to reflux resulted in precipitation. The reaction mixture was refluxed for 4 h. Vacuum distillation afforded a mixture of compounds **2**, **10**, and *N*,*N*-bis(trimethylsilyl)benzylamine (**19**) with b.p. 45—110 °C (1 Torr), yield 9.2 g, ratio **2** : **10** : **19** = 7 : 2 : 4.3. Compound **19** was also synthesized by counter synthesis as earlier described.[17] NMR and mass spectra of compound **19** coincide with those of an authentic sample.

***N*-Benzyl-*N*,*N*-bis(trimethylsilyl)amine (19).** ^{1}H NMR, δ: 0.08 (s, 18 H, Me_3Si); 4.09 (s, 2 H, CH_2); 7.17 (m, 1 H, C(4)); 7.26 (m, 4 H). ^{13}C NMR, δ: 1.9 (Me_3Si); 48.5 (CH_2); 125.8 (C(3), C(5)); 126.0 (C(2), C(6)); 127.8 (C(4)); 143.6 (C(1)). ^{29}Si NMR, δ: 7.2. MS, m/z (I_{rel} (%)): 251 $[M]^+$ (16), 236 $[M - Me]^+$ (100), 174 $[M - C_6H_5]^+$ (32), 162 $[M - CH_2 - C_6H_5]^+$ (100), 148 $[M - Me - CH_2 - C_6H_5]^+$ (33), 135 $[M - 2\ Me - CH_2 - C_6H_5]^+$ (45), 73 $[Me_3Si]^+$ (67).

The main results were obtained on the material and technical base of the Baikal Analytical Center for Collective Use of the Siberian Branch of the Russian Academy of Sciences.

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