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Silylamines in organic synthesis: preparation and some reactions of *N,C*-dilithiosilylamines *

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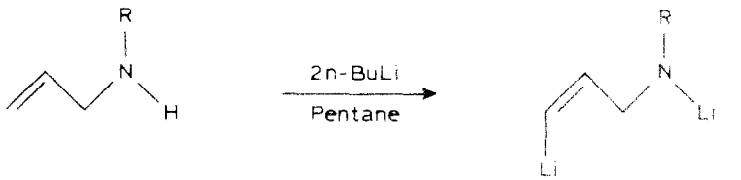
Abstract

The dimetallation of some olefinic and aromatic *N*-trimethylsilylamines has been examined. Treatment with a two molar proportion of n-butyllithium gives organodimetallic reagents, which react with trimethylchlorosilane to give *N,N,C*-tris(trimethylsilyl)amines in 45 to 65% yields. The dianonic reagent obtained from (trimethylsilyl)(allyl)amine reacts with benzoyl chloride, *N,N*-dimethylformamide, ethyl-2-furylcarboxylate, and benzil to give substituted pyrroles and pyridines.

Introduction

Heteroatom-facilitated metallation of organic compounds is a useful tool in organic syntheses and provides an effective route to heterocyclic compounds [1]. Our current interest in the synthetic uses of compounds containing silicon nitrogen bonds [2–4] led us to study the preparation and reactions of *N,C*-dilithiated reagents derived from silylamines. Compounds of this type have been obtained from the allylamines **1**. Treatment with n-butyllithium gave metallation at both the nitrogen atom and the sp^2 carbon atom, producing the dilithio reagents **2** [5–6] (eq. 1).



(1)

(**1**: R = t-Bu, $SiMe_3$) (**2a**: R = t-Bu, **2b**: R = $SiMe_3$)

* This work was presented at the 14th workshop Conference Hoechst see ref 4

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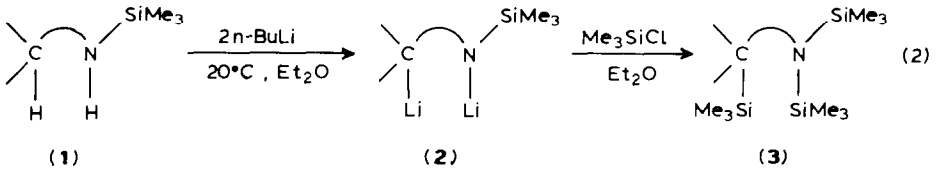
Reactions of carbon electrophiles with such 1,4-organodimetallic reagents lead to nitrogen heterocyclic compounds by formation of carbon–carbon and carbon–nitrogen bonds. We report here the initial results of a study of the preparation and use of dianionic reagents derived from silylamines.

Results and discussion

We examined the dilithiation of various *N*-trimethylsilyl olefinic and aromatic amines (eq. 2).



(1) (2) (3)

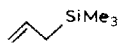
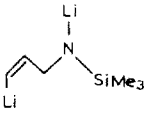
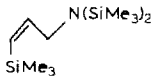
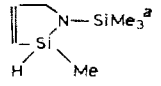
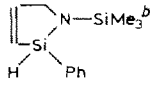
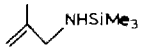
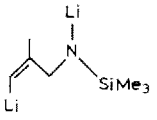
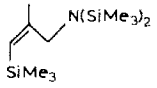
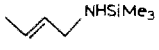
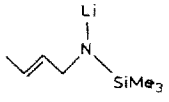
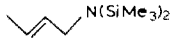
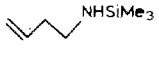
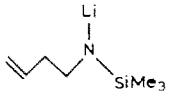
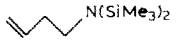
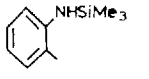
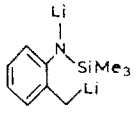
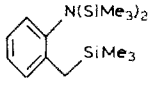
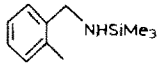
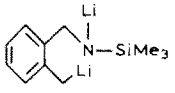
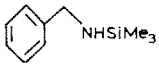
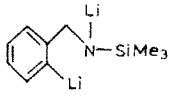
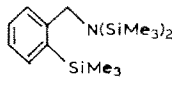
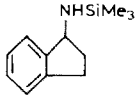
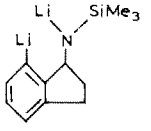
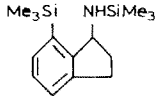
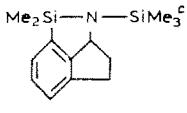
Treatment of ethereal solutions of silylamines **1** with a two molar proportion of n-butyllithium in ether gave yellow to orange solutions of the *N,C*-dilithio reagent **2**, which upon reaction with chlorosilanes gives the *N,N,C*-tris(trimethysilyl) derivatives, **3**. The results are presented in Table 1.

The dilithiation of *N*-trimethylsilylallylamine of ref. 6 followed by treatment with Me_3SiCl gave (*Z*)-$(Me_3Si)_2NCH_2CH{=}CHSiMe_3$ in 60% yield (entry 1). Similarly reactions with $MeHSiCl_2$ or $PhHSiCl_2$ gave 2-silapyrrolines. We then tried to extend the dilithiation reaction to other olefinic silylamines and *N,C*-dilithiation was also observed in the reaction of *N*-trimethylsilyl(α-methallyl)amine (entry 2). Formation of the more stable allylic dianion was not observed; under the conditions used, metallation occurred at the vinylic carbon atom. Complexation of the initially formed lithium amide with the lithiating agent is probably responsible for an enhanced kinetic acidity of the *syn*-vinyl proton of the methylene group, a similar explanation was suggested for the related dimetallation of α-methallyl alcohol [7]. Abstraction of the more acidic allylic proton to give the thermodynamically more stable allyl dianion was observed in the lithiation of α-methallyl alcohol in ether [8], whereas in hexane there was substantial lithiation (ca. 50%) at the vinylic carbon atom [7]. In contrast, no *C*-lithiation occurred in the case of *N*-(trimethylsilyl)crotyl or homoallylamines (entries 3, 4) and only formation of the lithium amide was observed. Metallation at the benzylic position occurred in the case of silylamines derived from *ortho*-toluidine and *ortho*-methylbenzylamine, giving a 1,4- and a 1,5-dilithio reagent, respectively (entries 5, 6); silylation reaction with Me_3SiCl then gave the corresponding bis(trimethylsilyl)aminobenzylsilanes in 54 and 65% yields. *ortho*-Metallation at the aromatic ring was also observed in the case of benzylamine and indanylamine (entries 7, 8). It is evident from the data in Table 1 that a variety of organodimetallic reagents can be obtained by dimetallation of monosilylamines. The first metallation, at the nitrogen atom giving a lithium amide, favors *C*-metallation in a second step.

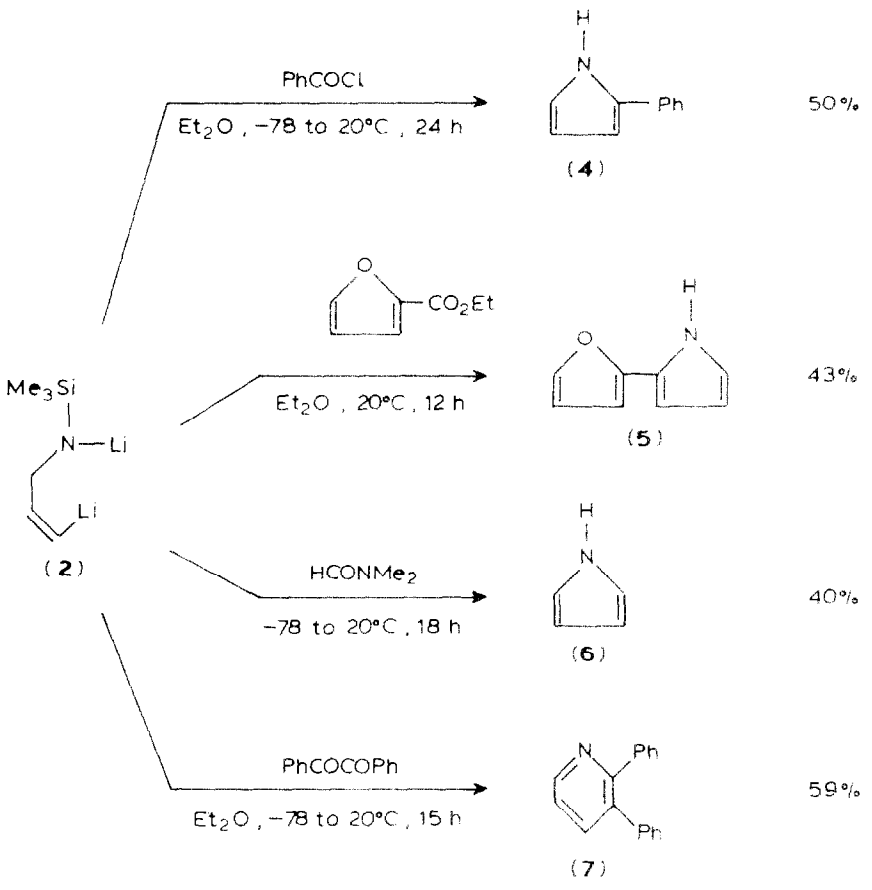
Dianionic reagents are of potential interest for the synthesis of heterocyclic compounds. We examined some reations with carbon electrophiles of the *N,C*-dilithio reagent **2b** derived from *N*-trimethylsilylallylamine. The results are outlined in Scheme 1.

Table 1

Metallation of silylamines **1**

Entry	Silylamine **1**	Amine metallated **2**	Product after silylation **3**	Yield %
1				60
				45
				42
2				50
3				63
4				44
5				54
6				65
7				45
8				50
				55

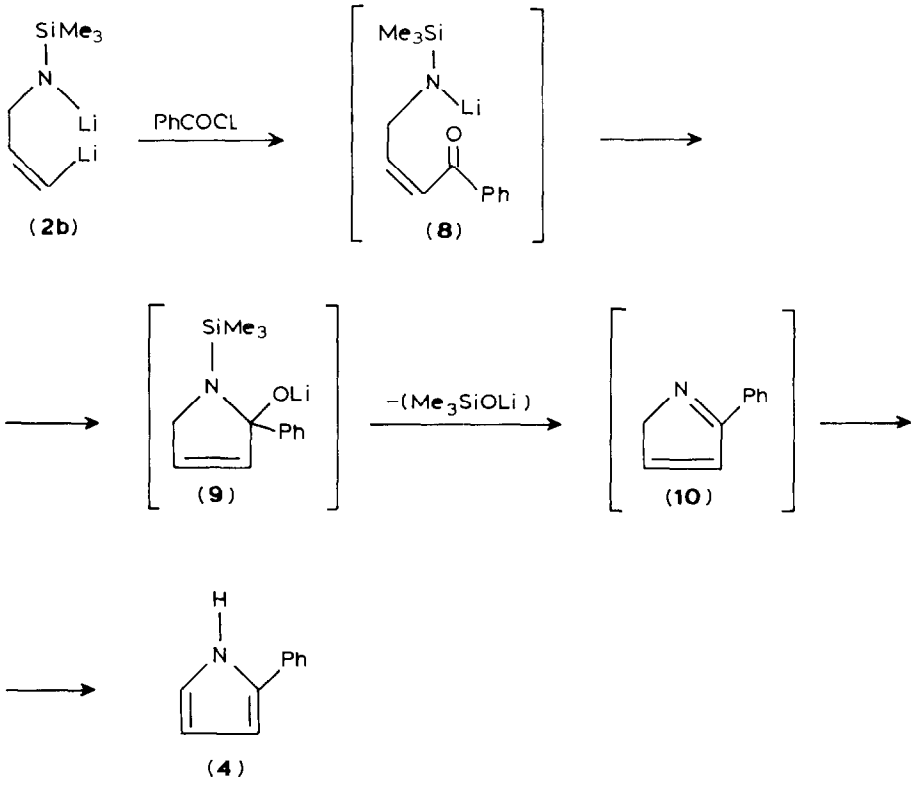
[a] The dilithio reagent was quenched with $MeSiHCl_2$. [b] The dilithio reagent was quenched with $PhSiHCl_2$. [c] The dilithio reagent was quenched with Me_2SiCl_2.



Scheme 1

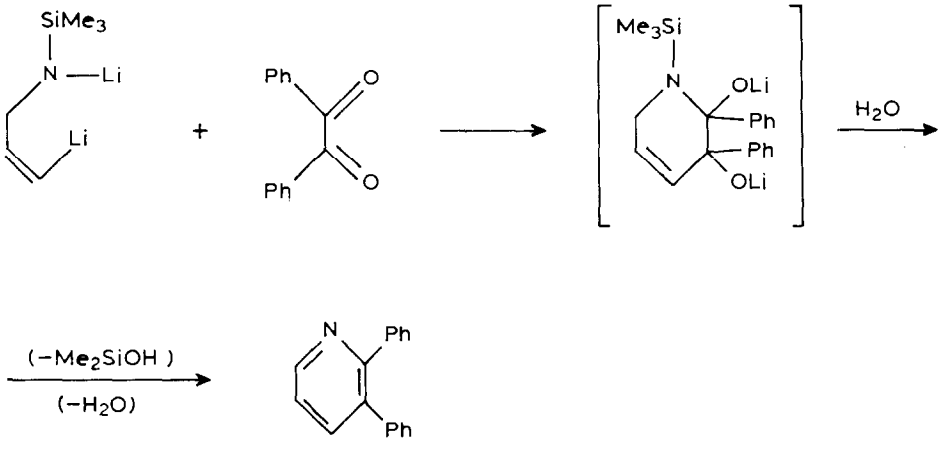
Reaction with benzoyl chloride followed by hydrolysis of the reaction mixture gave 2-phenylpyrrole, which was isolated in 50% yield. The formation of the pyrrole ring can be accounted for in terms of a process involving initial formation of a carbon–carbon bond by reaction of the vinyllithium centre with the electrophilic reagent (Scheme 2). The carbonyl bond intermediate enone **8** undergoes an intramolecular reaction with the lithium amide to give a *N*-trimethylsilyl-Δ_2-pyrroline (**9**), which then undergoes spontaneous elimination of trimethylsilanolate to give a carbon–carbon double bond [9]. The cyclic intermediate **10** thus formed then tautomerizes to 2-phenylpyrrole (**4**). Similarly the reaction of dilithio reagent **2b** with ethyl-2-furyl carboxylate gave 2-furylpyrrole (**5**). Reaction of dimethylformamide also produced a pyrrole species, **6**. Formation of 6-membered ring heterocycles is also possible: thus 2,3-diphenylpyridine (**7**) was obtained by reaction of **2b** with benzil. In this case the reaction of the dilithio reagent took place at the two carbonyl bonds, and the pyridine ring is then formed by two elimination reactions (Scheme 3).

Use of the dilithio reagents of type **2** provides a straightforward synthesis of pyrrole and pyridine ring structures; thus Smith and Visnick [10] obtained sub-



Scheme 2

stituted indoles by use of the reaction of a dilithio reagent with various esters. However, such *N,C*-dilithio reagents are quite basic, and their use is limited to non-enolisable reactants. For example we did observe no cyclisation upon treatment



Scheme 3

of **2b** with diacetyl. Despite this limitation due to the strongly basic character of the reagent, synthesis of nitrogen heterocycles by this method can give rise to various structures owing to the variety of the *N,C*-dilithio reagents which are available.

Experimental

General remarks. All reactions were carried out under an inert atmosphere. 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, ppm) 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. Satisfactory C, H, N analyses ($\pm$0.3%) were obtained for the new compounds. The monosilylamines **1** (Table 1) were prepared by silylation of the corresponding primary amines by standard procedures [11,12].

Lithiation and silylation reactions of N-monosilylamines

N-Trimethylsilylallylamine. To a stirring solution of trimethylsilylallylamine (2.58 g, 0.02 mol) in 100 ml of anhydrous ether at 0 °C under nitrogen were added 48.0 ml (0.041 mol) of 0.85 *M* n-butyllithium in ether. After 15 min stirring the mixture was allowed to warm to room temperature and, then stirred for 24 h, to give a yellow-green solution of the dilithio compound **2b**.

(i) Trimethylchlorosilane (4.35 g, 0.04 mol) in 15 ml of anhydrous ether was added at 0 °C to the above mixture. After 12 h stirring at room temperature the solution was filtered and the filtrate hydrolysed with saturated aqueous ammonium chloride. The aqueous layer was washed twice with ether, and the ethereal extracts were combined, washed with water, and dried over sodium sulfate. Distillation then gave 3.27 g (60%) of (*Z*)-3-bis(trimethylsilylamino)-1-trimethylsilylpropene (b.p. 124 °C, 20 mmHg). NMR (CCl_4): 0.10 (27H, S), 3.50 (2H, center of dd; J 5 Hz, J' 2 Hz), 5.30 (1H, center of dt; J 14 Hz, J' 2 Hz), 6.10 (1H, center of dt; J 14 Hz, J' 5 Hz); IR (CCl_4 cm^{-1}): ν (C–C) 1595 cm^{-1}; mass spectrum, *m/e* 273 (M^+).

(ii) Methyldichlorosilane (23 g, 0.2 mol) in 200 ml of ether was added at −20 °C to the dilithiated compound **2b**, prepared as above from 25.8 g, (0.2 mol) of *N*-trimethylsilylallylamine. The mixture was warmed to room temperature and stirred overnight. The salts were filtered off and the filtrate evaporated. The residue was subjected to trap-to-trap distillation (room temperature, 0.1 mmHg) into a trap cooled in liquid nitrogen. The oil obtained was then distilled to give Δ^3-2-methyl-1-trimethylsilyl-2-silapyrroline (15.4 g, 45%). NMR (CCl_4): 0.0 (9H, s); 0.1 (3 H, d); 3.65 (2, H, m); 5.0 (1H, m); 5.9 (1H, td); 6.8 (1H, td). IR (CCl_4, cm^{-1}): ν(Si–H) 2110, ν(C=C) 1570.

(iii) Similarly reaction with phenyldichlorosilane (35.4 g, 0.2 mol) gave 19.6 g (42%) of Δ^3-2-phenyl-1-trimethylsilyl-2-silapyrroline, (b.p. 130–150 °C, 20 mmHg. NMR (CCl_4): 0.0 (9H, s); 3.9 (2H, m); 5.5 (1H, m); 6.05 (1H, td); 7.1 (1H, td); 7.4 (3H, m); 7.6 (2H, m). IR (CCl_4, cm^{-1}): ν(Si–H) 2110, ν(C=C) 1570.

N-Trimethylsilyl-2-methylallylamine. A mixture of 3.24 g (0.023 mol) of 3-trimethylsilylamino-2-methylpropene and 100 ml of anhydrous ether was cooled to −78 °C under nitrogen atmosphere and 33.5 ml (0.05 mol) of 1.5 *M* n-buthyllithium in ether were added. The mixture was allowed to warm slowly to room

temperature which caused no change in colour. After 24 h stirring the pale yellow mixture was cooled to 0 °C and trimethylchlorosilane (5.43 g, 0.05 mol) in 20 ml of anhydrous ether was added. After 12 h stirring the ether was removed by rotary evaporation, pentane was added, and the salts filtered off. The filtrate was concentrated and the residue distilled to give 3.3 g (50%) of 3-bis(trimethylsilyl)amino-2-methyl-1-trimethylsilylpropene (b.p. 114 °C, 20 mmHg). NMR (CCl_4): 0.10 (27H, S), 1.82 3H, broad s), 3.65 (2H, broad s), 5.22 (1H, broad s); IR (CCl_4 cm^{-1}): ν(C=C) 1605; mass spectrum *m/e* (% rel. intensity) 287 (15), 272 (15), 174 (100), 147 (70), 73 (70).

N-Trimethylsilylcrotylamine. A solution of trimethylsilylcrotylamine (1.0 g, 0.007 mol) in 100 ml of anhydrous ether under nitrogen was cooled to −78 °C and 12 ml (0.014 mol) of 1.2 *M* n-butyllithium in ether were added. The mixture was allowed to warm slowly to room temperature and stirred for 48 h, after which, no colour had developed. The mixture was cooled to 0 °C and treated with trimethylchlorosilane (1.52 g, 0.014 mol) then allowed to warm to room temperature with stirring during 5 h. Dilution with 100 ml of pentane was followed by filtration and the filtrate was concentrated on a rotary evaporator. The residue was distilled to give 0.95 g (63%) of bis-silylated crotylamine (b.p. 96 °C, 20 mmHg). NMR (CCl_4): 0.10 (18H, s), 1.66 (3H, m), 3.40 (2H, m), 5.37 (2H, m); IR (CCl_4, cm^{-1}): ν(C=C) 1665 (weak); mass spectrum, *m/e* (% rel. intensity) 215 (7), 200 (100), 146 (34), 73 (90).

(N-Trimethylsilyl)(2-vinylethyl)lamine. To a stirred solution of the amine (2.245 g, 0.0158 mol) in 100 ml of anhydrous ether at 0 °C under nitrogen were added 32 ml (0.032 mol) of 1.0 *M* n-butyllithium in ether. After 30 min stirring at 0 °C the mixture was allowed to warm to room temperature, and stirring continued for 48 h. The pale gray mixture was cooled to 0 °C and treated with trimethylchlorosilane (3.44 g, 0.032 mol), then stirred 24 h. Following filtration of the salts and concentration by rotary evaporation, distillation gave 1.5 g (44%) of bis(trimethylsilylbutenyl)amine (b.p. 90 °C, 20 mmHg). NMR (CCl_4): 0.05 (18H, s), 2.15 (2H, m), 2.85 (2H, m), 4.70–5.20 (2H, m), 5.40–6.00 (1H, m); IR (CCl_4 cm^{-1}): ν(C=C) 1638; mass spectrum *m/e* (% rel. intensity) 215 (2), 200 (20), 164 (95), 147 (70), 73 (100).

ortho-N-Trimethylsilyltolylamine. To a stirred solution of the monosilylamine (3.58 mg, 0.02 mol) in 100 ml of anhydrous ether was slowly added 26 ml (0.039 mol) of 1.5 *M* n-butyllithium in ether. The mixture was stirred at room temperature for 48 h, then cooled to 0 °C. The reddish reaction mixture was then treated with trimethylchlorosilane (4.32 g, 0.04 mol) and stirred for 15 h at room temperature. Following filtration of the salts and concentration by rotary evaporation, distillation gave 3.4 g (54%) of *ortho-N,N*-bis(trimethylsilyl)trimethylsilylmethyltoluidine (b.p. 170 °C, 20 mmHg), Hg), NMR (CCl_4): 0.0 (27H, s), 1.7 (2H, s), 6.7 (4H, m). Treatment of the *N*-silylated amine with 2*N* aq. HCl, washing of the aqueous phase with ether, neutralisation with aq. Na_2CO_3 and extraction with ether, allowed isolation of *ortho*-trimethylsilylmethyltoluidine. NMR (CCl_4): 0.0 (9H, s), 1.7 (2H, s) 3.3 (2H, s), 6.7 (4H, m); IR (CCl^4, cm^{-1}): ν(N–H) 3410.

(ortho-Methyl)(N-trimethylsilyl)benzylamine. To a stirred solution of the amine (3.8 g, 0.02 mol) in 100 ml of ether was added slowly 26 ml (0.039 mol) of 1.5 *M* n-butyllithium in ether. After 24 h stirring at room temperature the mixture was orange red. Trimethylchlorosilane (4.3 g, 0.04 mol) in 50 ml of ether was added and the mixture stirred for 15 h. After filtration, and concentration of the filtrate, the

residue was distilled to give 4.4 g (65%) of *ortho-N,N*-bis(trimethylsilyl)(trimethylsilylmethyl)benzylamine (b.p. 90 °C, 0.5 mmHg). NMR (CCl_4): 0.0 (27H, s), 1.8 (2H, s), 3.7 (2H, s), 7.0 (4H, m). Aqueous HCl treatment as in the previous case afforded the free amine NMR (CCl_4): 0.0 (9H, s), 1.8 (2H, s), 3.2 (2H, s), 3.7 (2H, s), 7.0 (4H, m). IR (CCl_4, cm^{-1}): ν(N–H) 3400.

N-Trimethylsilylbenzylamine. To a stirred solution of amine (3.6 g, 0.02 mol) in 100 ml of ether was slowly added 26 ml (0.039 mol) of 1.5 *M* n-butyllithium in ether. The mixture was stirred for 48 h at room temperature then trimethylchlorosilane (4.3 g, 0.04 mol) in 50 ml of ether was added and the mixture stirred for 24 h. Filtration and concentration of the solution followed by distillation of the residue gave 2.9 g (45%) of *ortho-N,N*-bis(trimethylsilyl)trimethylsilylbenzylamine (b.p. 110 °C, 20 mmHg). NMR (CCl_4): 0.0 (18H, s), 0.2 (9H, s), 4.2 (2H, s), 7.1 (4H, broad s). Aqueous HCl treatment as in the previous case gave the primary amine. NMR (CCl_4): 0.2 (9H, s), 3.3 (2H, broad s), 3.7 (2H, s), 7.0 (4H, m). IR (CCl_4, cm^{-1}): ν(N-H) 3410.

1-Trimethylsilylaminoindane. Silylation with trimethylchlorosilane. A solution of 1-trimethylsilylaminoindane (1.65 g, 0.008 mol) in 100 ml of anhydrous ether was cooled to 0 °C under nitrogen and 16 ml (0.016 mol) of 1.0 *M* n-butyllithium in ether was added. The mixture was allowed to warm to room temperature then stirred for 36 h. The yellow solution was then cooled to 0 °C and trimethylchlorosilane (1.74 g, 0.016 mol) was added. The mixture was allowed to warm to room temperature and stirred for 1 day. The salts were filtered off and the filtrate concentrated by rotary evaporation. Distillation of the residue gave 1.1 g (50%) of 8-trimethylsilyl-1-trimethylsilylaminoindane (b.p. 88–92 °C, 0.05 mmHg). NMR (CCl_4): 0.04(9H, s), 0.33 (9H, s), 1.74–3.54 (5H, m), 4.54 (1H, m), 7.24 (3H, m). Mass spectrum *m/e* (%rel. intensity) 277 (2), 276 (1), 262 (1), 204 (5) 119 (100), 73 (20). Hydrolysis of the product with 1 *N* aqueous HCl gave 8-trimethylsilyl-1-aminoindane. Mass spectrum *m/e* (%rel. intensity) 205 (1), 204 (5), 203 (3), 189 (40), 187 (60), 173 (100), 132 (33), 74 (28), 73 (28).

Silylation with dimethyldichlorosilane. To a stirred solution of 2.00 g (0.010 mol) of 1-trimethylsilylaminoindane in 100 ml of ether at 0 °C were added 13.3 ml (0.020 mol) of 1.47 *M* n-butyllithium in ether. The mixture was stirred for 30 min at 0 °C then allowed to warm to room temperature and stirred for 36 h. The solution was diluted with anhydrous ether to a total volume of 500 ml, cooled to 0 °C, and treated with 1.29 g (0.01 mol) of dimethyldichlorosilane in 10 ml of ether. After 12 h stirring the salts were filtered off and the filtrate concentrated. The residue was distilled to give 1.40 g (55%) of cyclic aminosilane (b.p. 90 °C, 0.02 mmHg). NMR (CCl_4): 0.17 (9H, s), 0.28 (3H, s), 0.40 (3H, s), 1.0–3.18 (4H, m), 4.55 (1H, dd, J 10 Hz, J' 5 Hz), 7.02 (3H, m); mass spectrum *m/e* (% rel. intensity) 261(90), 260(100), 246(40), 119(100), 117(100), 73(80).

*Reactions of N,C-dilithio-N-trimethylsilyl-allylamine (**2b**)*

Preparatioin of 2-phenylpyrrole. The dilithio reagent **2b**, prepared as described above from 0.02 mol of *N*-trimethylsilylallylamine and 0.04 mol of n-butyllithium. It was treated at −78 °C with 2.8 g (0.02 mol) of benzoyl chloride and the mixture was allowed to warm to room temperature then stirred for 24 h. It was then hydrolysed with 200 ml of a 2*N* aqueous HCl and the aqueous layer was separated out, extracted twice with 50 ml of ether. The extracts were combined and dried over

sodium sulfate, and the solvent was evaporated. The residue was transferred to a silica gel column and elution with 1/9 ether/hexane gave 1.3 g (50%) of 2-phenylpyrrole with properties identical with those of an authentic sample, m.p. 129°C. NMR ($CDCl_3$): 6.35 (1H, m); 6.6 (1H, m); 6.75 (1H, m); 7.4 (5H, m); 8.1 (1H, m). IR ($CHCl_3$, cm^{-1}): ν(N–H) 3480; ν(C = C) 1610, 1590.

Preparation of 2-furylpyrrole. A solution of the dilithio compound **2b** (0.02 mol) prepared in the usual way was stirred for 24 h, then diluted with 400 ml of anhydrous ether, cooled to −78°C, and treated with ethyl-2-furylcarboxylate (2.8 g, 0.02 mol). The mixture was allowed to warm to room temperature, stirred for 12 h, then hydrolyzed with saturated NH_4Cl. The organic layer was separated washed with water, dried (Na_2SO_4), and concentrated. The residue was transferred to a base-washed alumina column, and elution with hexane gave a mixture of the 2-furylpyrrole and the starting ester (NMR yield of product; 1.15 g, 43%). The product was isolated by preparative GLC (SE30 column, 120°C). NMR (CCl_4) δ 6.32 (4H, m), 6.72 (1H, m), 7.34 (1H, m); IR (CCl_4, cm^{-1}) ν(N–H) 3488, mass spectrum m/e (% rel. intensity) 133 (30), 104 (20), 27 (100).

Preparation of pyrrole. The dilithio reagent **2b** (0.02 mol), prepared in the usual way was diluted with 500 ml of anhydrous ether under nitrogen and the solution was cooled to −78°C and dimethylformamide (1.46 g, 0.02 mol) in 30 ml of anhydrous ether was added. Stirring was continued for 30 min at −78°C and then for 18 h at room temperature. The mixture was added to 10 ml of 15% aqueous sodium hydroxide and then filtered. The salts were washed with ether and the washings added to the organic layer from the filtrate. The extract was dried (Na_2SO_4) and concentrated by rotary evaporation at room temperature. The residue was subjected to column chromatography (silica gel eluted with hexane) to afford 0.67 g (50%) of pyrrole, with spectra identical with those of an authentic sample.

Preparation of 2,3-diphenylpyridine. A solution of 4.2 g (0.02 mol) of benzil in 100 ml of ether was added to a solution of dilithio reagent **2b** prepared from 0.02 mol of allylamine at −78°C. The mixture was allowed to warm to room temperature then stirred for 15 h and hydrolysed with 200 ml of 2*N* aqueous HCl. The aqueous phase was washed twice with 50 ml of ether then neutralized with saturated aqueous sodium carbonate and extracted with three portions of 50 ml of ether. The combined ether extracts were dried over sodium carbonate and concentrated. Distillation of the residue afforded 2.7 g (59%) of 2,3-diphenylpyridine (b.p. 179–181°C, 0.3 mmHg) which solidified (m.p. 61–63°C). NMR ($CDCl_3$): 7.3 (11H, m); 7.8 (1H, dd); 8.75 (1H, dd). IR ($CHCl_3$, cm^{-1}): ν(C=C) 1610, 1590, 1570. Mass spectrum $m/e = 231$ (M^+).

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