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Alkoxy(alkyl)silylalkyl derivatives of nitrogen-containing heterocycles

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Abstract. The published data on the synthesis, properties and transformations of alkoxy(alkyl)silylalkyl derivatives of nitrogen-containing heterocycles of the general formula Het$(CH_2)_n SiX_3$ are surveyed and systematised. Data on the biological activities and applications of these compounds are presented. The bibliography includes 255 references.

I. Introduction

Considerable interest in organosilicon compounds containing a heterocyclic fragment in a hydrocarbon chain linked to the silicon atom is due to their valuable properties. These compounds served as the basis for the preparation of a large series of adsorbents, immobilised catalysts, synthetic intermediates, medicinal drugs, pesticides and other substances endowed with specific biological activities.

To date, a great number of publications are devoted to organosilicon derivatives in which the silicon atom is directly linked to a heterocyclic substituent (*e.g.,* see, Refs 1 – 7). However, the publications which describe carbofunctional organosilicon compounds with a silicon atom attached to the heterocyclic fragment through a hydrocarbon bridge are still few in number. Among those, special mention should be made of the review [8] which describes the compounds containing a heterocyclic substituent with one nitrogen atom (the studies surveyed therein had been published before 1970) and two more recent reviews [9, 10] concerned with the synthesis of organosilicon derivatives of pyridine and quinoline.

The present review gives a systematic and critical analysis of the data published after 1970 concerning heterylalkylsilanes containing at least one nitrogen atom in their heterocycles.

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II. Synthesis

1. Organometallic synthesis

One of the methods for preparing alkoxy(alkyl)silylalkyl derivatives of nitrogen-containing heterocycles in which the silicon atom is linked to the nitrogen or the carbon atom through a hydrocarbon bridge, $-(CH_2)_n-$, where $n = 1, 3$, is based on the reaction of organolithium, -sodium, -potassium or -magnesium derivatives of the corresponding heterocycles with silicon-substituted (chloroalkyl)silanes:[11 – 70]

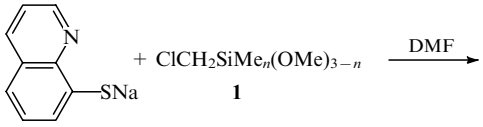
$$R^1M + Cl(CH_2)_nSiR^2_m(OR^3)_{3-m} \longrightarrow$$
$$\longrightarrow R^1(CH_2)_nSiR^2_m(OR^3)_{3-m} + MCl$$

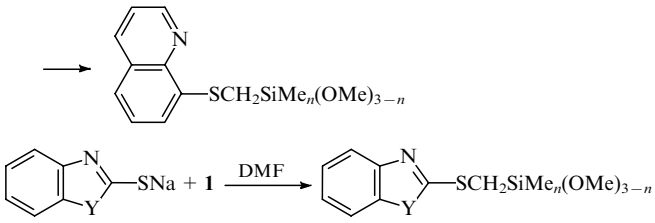
R^1 = *N*- or *C*-heteryl; R^2, R^3 = Me, Et; M = Li, Na, K, MgBr, ZnCl; $n = 1, 3$; $m = 0 – 3$.

Thus silicon-substituted *N*-heterylalkylsilanes were obtained from the corresponding alkoxy(alkyl)chloroalkylsilanes and *N*-potassium derivatives of phthalimide,[11] imidazole, 1,2,4-triazole,[12, 13] benzothiazinone,[14] piperidine, perhydroazepine, morpholine [15] and purine [16] in boiling benzene or toluene. The use of Bu_4NBr as a phase transfer catalyst permits one to reduce the reaction time.[12]

The reaction of *C*- or *N*-sodium derivatives of quinoline,[17, 18] 2-pyrrolidinone,[19] substituted 1,2,4-triazole,[20 – 31] imidazole,[22, 23, 26, 30 – 35] uracil, thymine and adenine,[36] pyridone,[37] pyrazine [38] and benzotriazole [39] with $Cl(CH_2)_nSiR^2_m(OR^3)_{3-m}$ (where $n = 1, 3$ and $m = 0 – 3$) in DMF gave the corresponding *C*- or *N*-substituted heterylalkylsilanes.

The silicon-substituted (heterylthiomethyl)silanes $HetSCH_2.SiMe_n(OMe)_{3-n}$, where $n = 0$ or 1, were synthesised in 65% – 86% yields by the reaction of *S*-sodium derivatives of 8-mercaptoquinoline, 2-mercaptobenzooxazole, 2-mercaptobenzothiazole and 2-mercaptobenzoimidazole with trimethoxy-(chloromethyl)- and dimethoxymethyl(chloromethyl)silanes (**1**) in a polar aprotic solvent (DMF, DMSO) for 2 – 3 h at 80 – 90 °C.[40 – 42]





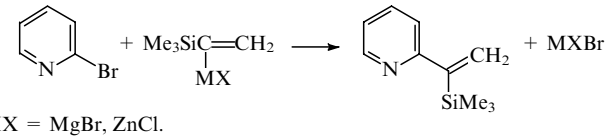
n = 0, 1; Y = NH, O, S.

The synthesis of trimethylsilylalkyl-substituted pyrrole,[43,44] piperidine,[45] pyridine,[46–49] 2-pyridone,[50] pyrrolidine,[51,52] pyrazole, imidazole, benzoimidazole,[53] quinoline,[54–56] 3,4-dimethylisoxazole,[57,58] 2-mercaptobenzothiazole,[59,60] benzothiazole,[61] indole,[62] tetrazole,[63] pyrimidine[64] and carbazole[65] makes wide use of *C*-, *N*- or *S*-lithium derivatives of these heterocycles. The latter are obtained by the reaction of the corresponding heterocyclic compounds with RLi (R = Me, Bu^n, Bu^t, Ph, NPr^i_2) in THF or hexane. Thus the 2-ethylpyridine carbanion prepared by the reaction of ethylpyridine with butyllithium (THF, −70 °C) readily reacts with *tert*-butyldimethylchlorosilane. In this case, the silyl group enters into the α-position of the alkyl substituent.[47]

The reactions of 2-, 3- and 4-methylquinoline[54] with lithium diisopropylamide and then with trimethylchlorosilane give rise to 2-, 3- and 4-trimethylsilylmethyl derivatives (in this case, 3-substituted derivatives are formed with the lowest yield). Metallation of 2-methyl-6-(2,5-dimethylpyrrolo)pyridine (THF–hexane, −25 °C) with lithium diisopropylamide and subsequent reaction with trimethylchlorosilane gave 2-trimethylsilylmethyl-6-(2,5-dimethylpyrrolo)pyridine in 77% yield.[44] Lithium derivative of 2-methylbenzothiazole reacts with trimethylchlorosilane analogously.[59,60]

The reaction of halogen-substituted pyridines and quinolines with trimethylsilylmethylmagnesium chloride or -zinc chloride in ether in the presence of a catalytic amount of $Cl_2Ni[Ph_2P(CH_2)_3PPh_2]$ afforded 2- and 3-trimethylsilylmethylpyridines (72% and 35% yields, respectively), 2,6-bis(trimethylsilylmethyl)pyridine (yield 69%) and trimethylsilylmethylquinoline (yield 44%).[66]

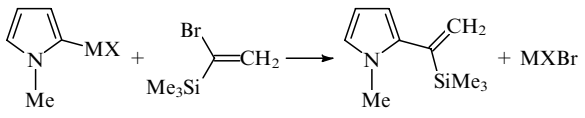
The reaction of 2-bromopyridine with (1-trimethylsilylvinyl)zinc chloride or -magnesium bromide in the presence of a palladium–phosphine complex results in 2-(1-trimethylsilylvinyl)pyridine.[67,68]



MX = MgBr, ZnCl.

2-(3-Trimethylsilylbut-3-enyl)pyridine was obtained unexpectedly when a fourfold excess of 2-bromopyridine was used.

(2-Trimethylsilylvinyl)magnesium bromide or -zinc chloride was used in the synthesis of the corresponding organosilicon derivatives of pyrrole and indole.[67] Thus trimethyl(α-bromovinyl)silane reacts with 1-methyl-2-pyrrolyl- and 1-methyl-2-indolylmagnesium bromide or -zinc chloride in the presence of a catalytic amount of $Cl_2Pd[Ph_2P(CH_2)_4PPh_2]$ to give the corresponding 1-methyl-2-trimethylsilylvinyl derivatives of these heterocycles in 74%–87% yields.[67] For example:



MX = MgBr, ZnCl.

The reaction of 6-methylpyridine with methyl chloroformate and trimethylsilylethynylmagnesium bromide gives exclusively 2-trimethylsilylethynyl-6-methyl-*N*-methoxycarbonyl-1,2-dihydropyridine (yield 79%).[69]

The reaction of 7-chloro-4-(4-iodopyrazolo)quinoline with CuI and $Me_3SiC{\equiv}CH$ in the presence of $Cl_2Pd(PPh_3)_2$ gives 7-chloro-4-(4-trimethylsilylethynylpyrazolo)quinoline.[70]

2. Amination reactions

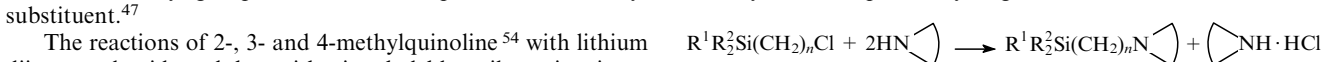
The following methods are also used in the synthesis of alkoxy(alkyl)silylalkyl derivatives of nitrogen-containing heterocycles:

1. Reaction of the corresponding derivatives of (halogenoalkyl)silanes $X(CH_2)_nSiR^1_m(OR^2)_{3-m}$ with compounds containing an endocyclic (>NH) group.

2. Reaction of halogen-substituted nitrogenous heterocycles with organosilicon derivatives containing a $(CH_2)_nNH_2$ group at the Si atom.

3. Attachment of substituted nitrogenous heterocycles containing endocyclic (>NH) groups with an active hydrogen atom to unsaturated organosilicon compounds.

Organosilicon derivatives of morpholine,[71] perhydroazepine,[71–73] piperazine,[71,73,74] piperidine,[71,72,74,75] pyrrolidine[71–73,76] and imidazole[77,78] were obtained by heating trialkyl(chloroalkyl)- or trialkoxy(chloroalkyl)-silanes with an excess of the corresponding N-heterocycle which acts simultaneously as an acceptor of hydrogen chloride:



$R^1R^2_2Si$ = Me_3Si, Et_3Si, $Me(EtO)_2Si$, $(EtO)_3Si$, $Me(Me_3SiO)_2Si$, $(Me_3SiO)_3Si$; n = 1, 3;

Hereinafter  is a nitrogen-containing heterocyclic base.

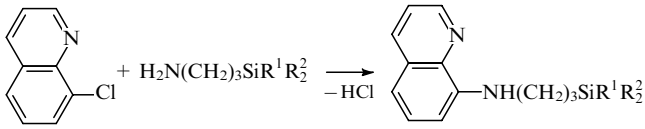
This reaction is carried out in a boiling non-polar solvent (benzene, hexane, toluene). Trialkoxy(chloromethyl)silanes react faster than the corresponding trialkyl derivatives.

The synthesis of silicon-substituted *N*-silylmethyllactams,[79–81] 1-trialkylsilylmethyl(propyl)piperazine[82] and 1-trimethylsilylmethyl-1,2,4-triazole[83–85] was carried out in the presence of anhydrous potassium carbonate or sodium carbonate.

Siliconalkylation of pyrrole, carbazole, pyrazole, imidazole, benzoimidazole, 1,2,4-triazole, 1,2,3-benzotriazole and tetrazole with trialkyl(3-halogenopropyl)silanes gave the corresponding *N*-(3-trialkylsilylpropyl)azoles in 80%–90% yields.[86,87] The reaction was carried out in a biphasic system (benzene–60% aqueous KOH) with boiling for 4–6 h in the presence of 5 mol. % of tetrabutylammonium hydrogen sulfate as a phase transfer catalyst. Attempts to obtain these compounds by hydrosilylation of *N*-allyl derivatives of the corresponding azoles failed.[86] The reaction of 4-amino-1,2,4-triazole with trimethyl(halogeno-methyl)silanes in DMSO (8 h, 80 °C) afforded 4-amino-1-trimethylsilylmethyl-1,2,4-triazolium halides. 4-Amino-3-trimethylsilylmethyl-1,2,4-triazole is formed when boiling nitromethane is used as a solvent.[88]

Triethylamine was used as a hydrogen chloride acceptor in the synthesis of *N*-(trialkylsilylalkyl)- and *N*-(trialkoxysilylalkyl) derivatives of piperazine,[89] 3,5-dimethylpyrazole,[90] 1,2,3,4-tetrahydro(iso)quinoline,[91–93] pyrrolidone[94] and other lactams.[95] This reaction is carried out in boiling toluene or xylene. *N,N′*-Bis(triethoxysilylalkyl)piperazines were obtained by heating triethoxy(chloroalkyl)silane with piperazine in a 2 : 1 molar ratio.[89]

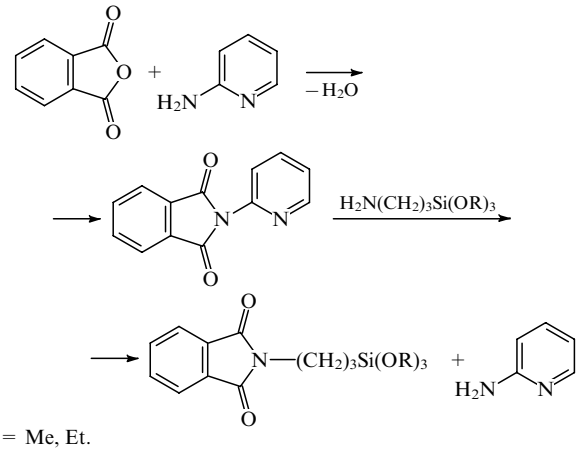
Heating of quinoline-2- and -4-carbonyl chlorides,[92] 9-chloroacridine[96] and chloroquinolines[91,97–99] with (3-aminopropyl)-trialkylsilanes gives the corresponding 3-(trialkylsilyl)propylamino derivatives of these heterocycles.



$R^1R^2_2Si$ = Et_3Si, $MeBu^n_2Si$, Bu^iMe_2Si.

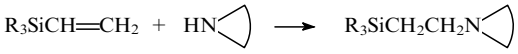
Their isolation is usually simplified when diglyme is used as a solvent and triethylamine is used as a hydrogen chloride acceptor.

A two-step procedure for the synthesis of *N*-[3-(trialkoxysilyl)propyl]phthalimide has been developed. The first step consisted in condensation of phthalic anhydride with 2-aminopyridine to give the corresponding imide; its interaction with (3-aminopropyl)trialkoxysilane (toluene, 10–15 h) resulted in 3-(trialkoxysilyl)propylphthalimide with good yield.[100]



R = Me, Et.

Heterocycles containing an endocyclic NH group are smoothly and regioselectively added to unsaturated organosilicon compounds containing a $SiCH=CH_2$ or $SiCH_2CH=CH_2$ fragment in the presence of alkali metals (Li, Na, K or their amides). In both cases, the adducts formed contain a nitrogen atom in the β-position relative to the silicon atom. The addition of pyrrolidine,[71] perhydroazepine,[71] piperazine,[82] 3,5-dimethylpyrazole, imidazole, benzoimidazole,[90,101] 3(5)-methylpyrazole,[102] aziridine,[103] piperidine[71,104,105] and azetidine[106] to vinyltrialkyl- and vinyltrialkoxy-silanes proceeds rather easily even at 20–75 °C. The adducts are formed in 70%–85% yields.

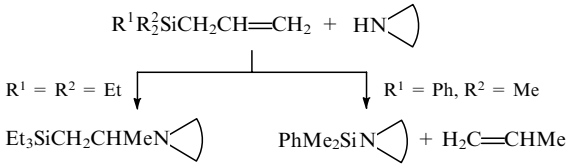


R = Alk, AlkO.

The rate of addition of nitrogenous heterocycles to silicon-substituted vinylsilanes depends substantially on the nature of the solvent. Thus the yields of the addition products of aziridine and piperidine to triethylvinylsilane in 2 h under identical conditions were 39% and 72% in the absence of a solvent, 20% and 15% in n-heptane and 55% and 92% in THF.[103]

The nature of the substituents at the silicon atom has also a profound effect on the reactivity of the original silicon-substituted vinylsilanes. Thus the yield of 2-(aziridinoethyl)triethylsilane formed in the reaction of triethylvinylsilane with aziridine is 90% (7 h, 100 °C). 2-(Aziridinoethyl)triethoxysilane, the addition product of aziridine to triethoxyvinylsilane, was obtained in 69% yield in 10 h at 100 °C. The rate of catalysed addition of *N*-heterocycles to silicon-substituted vinylsilanes $R^1R^2_2SiCH=CH_2$ increases in the following order depending on the substituents R^1 and R^2 at the silicon atom: $(OEt)Me_2 < Et_3 < Me_3 < Me_2Ph < MePh_2$. This was ascribed to an increase in the $p_\pi - d_\pi$ interaction of the vinyl group with the silicon atom which resulted in electron density displacement toward the α-carbon.[103] At present, this effect is interpreted as σ–π conjugation, *i.e.*, the interaction of the C=C bond π-orbital with the Si–C bond σ-orbitals. The decrease in the rate of addition of *N*-heterocycles to triethoxyvinylsilane was attributed to the consumption of the catalyst upon cleavage of the Si–OR bond.[103]

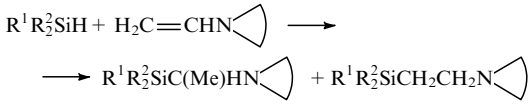
As the double bond in compounds of the $R^1R^2_2Si(CH_2)_nCH=CH_2$ series, where $n = 0$, 1 and 2 becomes more distant from the silicon atom, the rate of addition of *N*-heterocycles decreases. In the case of $n = 1$, β-elimination often accompanies the addition reaction.



The yield of the addition product of aziridine to allyltriethylsilane is 35%. However, aziridine is not added to allyldimethylphenylsilane, the β-degradation product, *viz*., azidirinyldimethylphenylsilane (10% yield), and propene are formed instead.[105]

3. Hydrosilylation reactions

Hydrosilylation of heterylalkenes usually gives a mixture of two isomers the yields and ratio of which depend on the structure of each of the reagents and the catalyst as well as on reaction conditions (solvent, temperature).



R^1, R^2 = Alk, AlkO, Cl.

Trichlorosilane is added to 2-vinyl-, 4-vinyl- and 2,6-dimethyl-4-vinylpyridines in the absence of a catalyst to give the corresponding pyridylethyltrichlorosilanes.[8] It is assumed that the role of a catalyst is played by the heterocyclic base itself. Labile yellow complexes $Cl_3SiH \cdot nB$ ($n = 3$ for 4-vinylpyridine; $n = 4$ for 2-vinylpyridine) are intermediate products of this reaction.

Triorganyl- and diorganyl-chlorosilanes are added to the same vinylpyridines only in the presence of catalysts. Thus the corresponding β-adducts are formed in the presence of an $Me_2NCH_2CH_2NMe_2–Cu_2Cl_2$ complex, while a mixture of α- and β-adducts with the predominance of the former is formed in H_2PtCl_6-catalysed hydrosilylation of 2-vinylpyridine.[107] In the absence of a catalyst, triethylsilane does not react with 2- and 4-vinylpyridines, whereas in the presence of H_2PtCl_6 it is added to 4-vinylpyridine and 2-methyl-5-vinylpyridine to give solely β-adducts (in 46% and 36% yields, respectively).

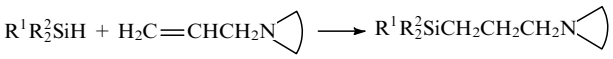
Hydrosilylation of 2-vinylquinoline catalysed by H_2PtCl_6 also results in a mixture of α- and β-adducts. Thus ethyldichlorosilane is added to 2-vinylquinoline to give predominantly the β-adduct (75% yield); trialkylsilanes give a mixture of regioisomers with the predominance of the α-adduct (10 h, 120–160 °C, overall yield 65%), while triethoxysilane gives a mixture of α- and β-adducts in a 1:1 ratio. The use of non-polar aprotic solvents (xylene, dioxane, diglyme) has no effect on the ratio of isomeric adducts but permits one to increase the total yield from 55% to 70%.[108] As for polar solvents (pyridine, quinoline), these favour the formation of the β-isomer; the total yield of the adducts decreases to 40% under these conditions. No hydrosilylation takes place in DMF.

2-(1-Triethoxysilylethyl)pyrrolidone[109–111] and 2-(1-triethoxysilylethoxy)perhydroazepine[112,113] were obtained by the reaction of *N*-vinylpyrrolidone and *N*-vinylcaprolactam with triethoxysilane. This reaction was catalysed by rhodium or palladium complexes, rhodium dicarbonyl acetylacetonate being the most efficient catalyst.[111,112] With this catalyst, the reaction was selective and gave only the α-adduct in 73% yield.

Hydrosilylation of 2- and 2,3-substituted 1-vinylpyrroles and 1-vinyl-4,5,6,7-tetrahydroindole with triethylsilane catalysed by H_2PtCl_6 and $ClRh(PPh_3)_3$ (4–10 h, 80–100 °C) was highly regioselective and resulted exclusively in the β-adducts in 55%–80% yields.[114–118] The use of polar aprotic solvents (THF, acetone) and increase in the reaction time allow one to increase the yield of the adduct up to 90%. No hydrosilylation of

these 1-vinylpyrroles with alkyldichloro-, trichloro- and trialkoxysilanes occurred.

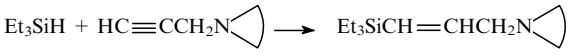
Hydrosilylation of *N*-allyl pyrrolidine, -piperidine,[71, 119] -*N*′-methylpiperazine,[120] -morpholine [120, 121] and -benzotriazole [122] with triorganylsilanes catalysed by 0.1 M H_2PtCl_6 resulted in *N*-(3-triorganylsilylpropyl)derivatives of the corresponding heterocycles.



$R^1 = Me$, $R^2 = Et$, Pr, Bu, C_5H_{11}, Ph, Cl;
$R^1 = Et$, $R^2 = Et$, Pr, Bu;
$R^1 = R^2 = Pr$, Bu, EtO.

No individual reaction products could be obtained upon hydrosilylation of *N*-allylcarbazole with ethyldichlorosilane.[123]

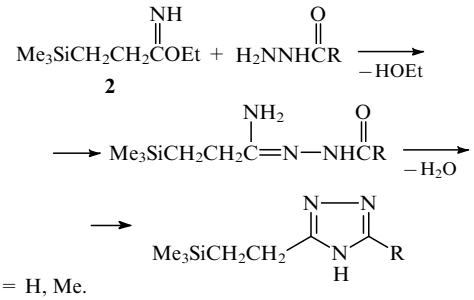
Hydrosilylation of *N*-propargyl-substituted pyrrolidine, piperidine, perhydroazepine and morpholine with triethylsilane in the presence of H_2PtCl_6 gives the γ-adducts with a *trans*-structure.[71, 124, 125]



The yields of the hydrosilylation products formed upon 10-h boiling of the reaction mixture vary from 25% to 53%. Substitution of silanes with more electronegative substituents for triethylsilane favours the formation of the α-adduct.

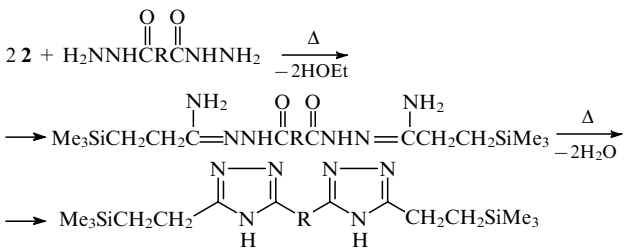
4. Cyclisation reactions

The reaction of carbohydrazides with ethyl β-trimethylsilylpropionimidate (**2**) yields 3-alkyl-5-(2-trimethylsilylethyl)-1,2,4-triazoles.[126] Trimethylsilylethylamidrazones are the primary products of this reaction; their intramolecular cyclisation results in the corresponding organosilicon derivatives of 1,2,4-triazole upon heating *in vacuo*.



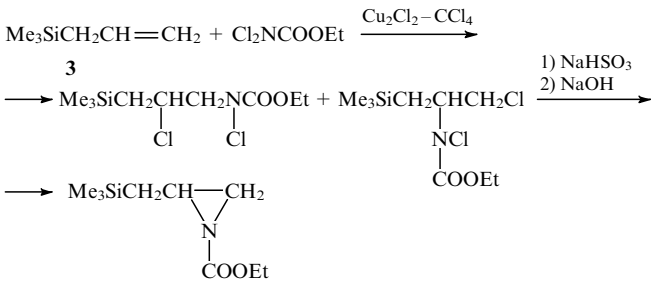
R = H, Me.

Dicarboxylic acid dihydrazides are converted into 3,3′-R-di[5-(2-trimethylsilyl)ethyl]-1,2,4-triazoles less easily (only upon heating to 200 °C).

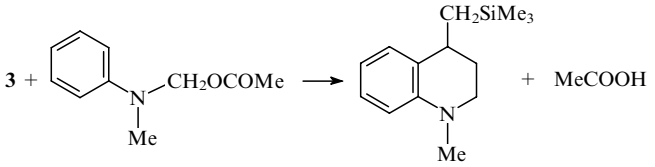


R = CH_2, $(CH_2)_4$, C_6H_4.

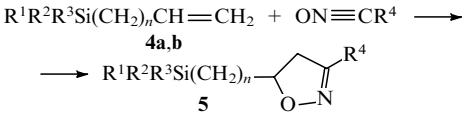
The addition of ethyl *N,N*-dichlorocarbamate to allyltrimethylsilane (3) with subsequent reduction with sodium hydrogensulfite and dehydrochlorination affords ethyl-*N*-chloro-*N*-(2-chloro-3-trimethylsilylpropyl)carbamate and ethyl-*N*-chloro-*N*-(1-chloromethyl-2-trimethylsilylethyl)carbamate. Intramolecular alkylation of the latter results in ethyl 2-(trimethylsilylmethyl)aziridine-2-carboxylate in 20% yield.[127]



The reaction of allyltrimethylsilane with $PhMeNCH_2O.C(O)Me$ in dichloromethane results ultimately in the corresponding 4-trimethylsilylmethyl derivative of 1,2,3,4-tetrahydroquinoline.[128]



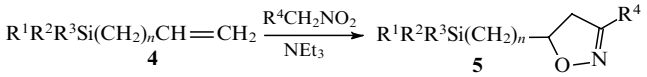
The addition of nitrile oxides to vinyl- and allyltrialkylsilanes **4** gave the corresponding organosilicon derivatives of 4,5-dihydroisoxazole **5** in 50% – 70% yields.[129, 130]



$R^1 = R^2 = R^3 = Me$ (**a**), Et (**b**); $n = 0, 1$; $R^4 = Br$, Ph, $4\text{-}C_6H_4NO_2$.

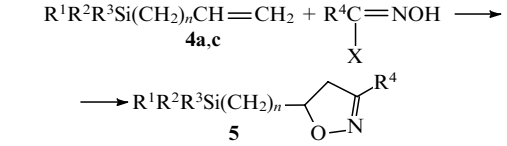
The rate of this reaction decreased upon substitution of phenyl or methoxy groups, having a negative inductive effect for methyl or ethyl groups at the silicon atom.

Vinyl- and allyltrialkylsilanes **4** react with nitroethane or 1-nitropropane in the presence of phenyl isocyanate and catalytic amounts of Et_3N in benzene to give the corresponding 3-alkyl-5-trialkylsilylisoxazolines **5** in 64% – 88% yields.[131]



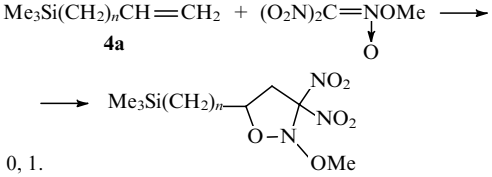
$R^1 = R^2 = R^3 = Alk$; $n = 0, 1$; $R^4 = Me$, Et.

The reaction of vinyl- and allyltrialkyl(aryl)silanes [$R^1 = R^2 = R^3 = Me$ (**4a**), Ph (**4c**)] with hydroxyamoyl halides proceeds regiospecifically and yields only one isomer.



$R^1 = R^2 = R^3 = Me$ (**a**), Ph (**c**); $n = 0, 1$; $R^4 = Ph$, COOEt, Br;
X = Cl, Br.

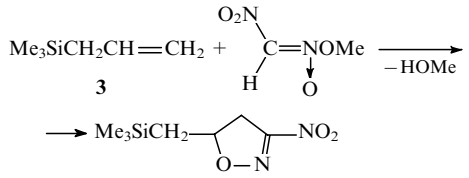
The reaction of trinitromethane *O*-methyl ester with vinyl- and allyltrimethylsilanes (**4a**) results in *N*-methoxy-3,3-dinitro-5-(trimethylsilyl)- and -5-(trimethylsilylmethyl)isoxazolidine.[130]



$n = 0, 1$.

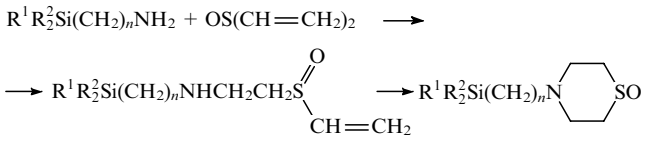
The reaction of these silanes with diethyl nitromalonate *O*-methyl ester proceeds in a similar way.[132]

The reaction of dinitromethane *O*-methyl ester with allyl(trimethyl)silane is also accompanied by the formation of a cyclic adduct. However, 3-nitro-5-(trimethylsilylmethyl)isox-azoline is formed in high yield instead of the expected product, *N*-methoxy-3-nitro-5-(trimethylsilylmethyl)isoxazolidine.



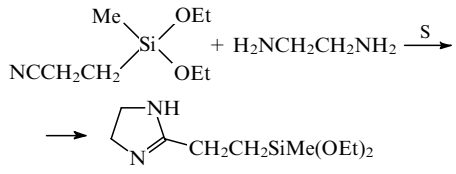
It can be inferred from these data that the cyclisation is regiospecific, *viz*., the oxygen atom always attacks the least hydrogenated carbon atom.

At 50–75 °C, triorganylsilylalkylamines react with divinyl sulfoxide to give 4-(triorganylsilylalkyl)tetrahydro-1,4-thiazine oxides (in 62%–80% yields).[133] At lower temperatures (30–50 °C), a linear monoadduct is predominantly formed.



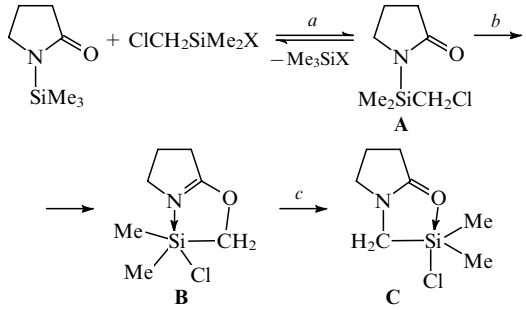
R^1 = Me: R^2 = Me, Et, Bu; R^1 = R^2 = Et, EtO; n = 1, 3.

Cyclisation of methyl(2-cyanoethyl)diethoxysilane with ethylenediamine in the presence of sulfur as a catalyst gives 2-(2-methyldiethoxysilylethyl)-4,5-dihydroimidazole.[134]



5. Transsilylation reactions

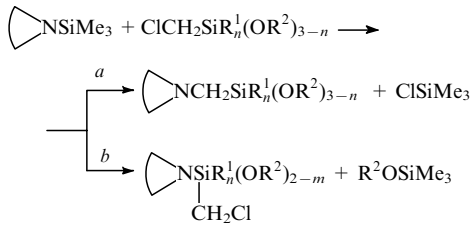
The reactions of halogeno- and alkoxydimethyl(chloro-methyl)-silanes $ClCH_2SiMe_2X$ with *N*-trimethylsilyl derivatives of 2-piperidone,[135–144] pyrrolidone,[81, 136, 138–148] caprolactam,[81, 138, 139, 141–144, 148] butyrolactam[139] and morpholinone[149] occur in three steps: (*a*) transsilylation (substitution of CH_2SiMe_2Cl for $SiMe_3$); (*b*) intramolecular silylmethylation of the oxygen with migration of the chlorine atom from the carbon atom to silicon and (*c*) *O,N*-migration of the silylmethyl group. For instance:



X = F, Cl, Br, I, OR.

Data from X-ray analysis[148] confirmed the conclusion that the silicon atom in *O*- (compound **B**) and *N*-alkylation (compound **C**) products is five-coordinate. It should be noted that *N*-trimethylsilyl derivatives of piperidone and caprolactam react with $ClCH_2SiMe_2X$ at room temperature, whereas the *N*-trimethylsilyl derivative of butyrolactam reacts with these silanes at 110–130 °C.[139] This reaction is catalysed by $AlCl_3$, $Me_3SiOSO_2CF_3$ or $PhNEt_2$. In this case, the reaction time decreases from 3 to 0.5 h but the yield of the final product does not change (32%–71%).[142]

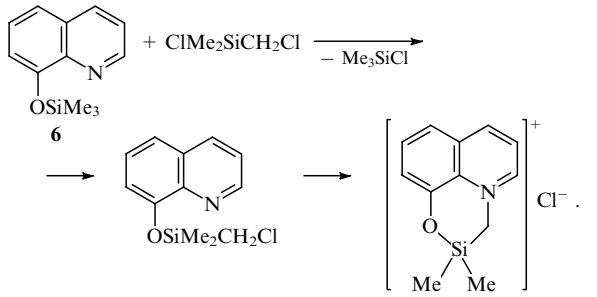
In the reaction of $ClCH_2SiR^1_n(OR^2)_{3-n}$ with *N*-trimethylsilyl-substituted 3,5-dimethylpyrazole, imidazole, benzoimidazole[90, 150, 151] and 2,2′-dipyridylamine,[152] the main *N*-silylmethylation reaction (reaction *a*) is accompanied by transsilylation (reaction *b*):



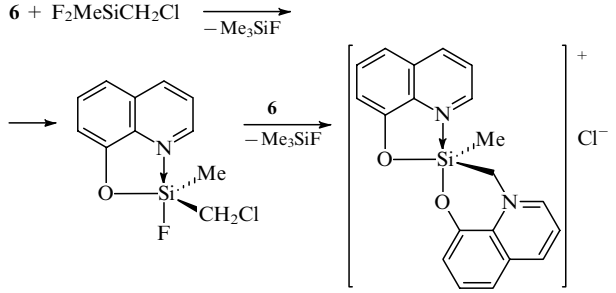
R^1, R^2 = Me, Et; n = 0–3; m = 0, 2.

Thus in the case of 3(5)-methylpyrazole, the ratio of silylmethylation and transsilylation products is 85 : 15.[102]

The reaction of 8-trimethylsiloxyquinoline (**6**) with dimethyl(chloromethyl)chlorosilane proceeds according to the following scheme:[153, 154]



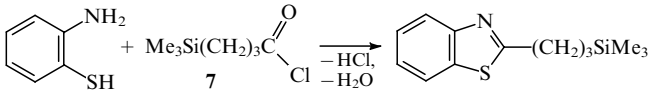
Methyl(chloromethyl)dihalogenosilanes react with 8-trimethylsiloxyquinoline to give a spirocyclic ionic derivative of five-coordinate silicon.



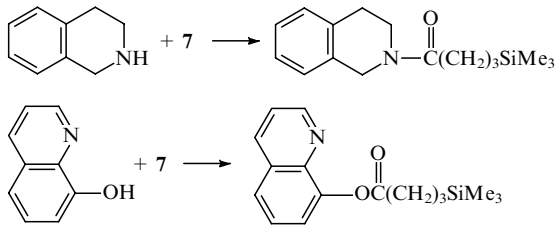
This review provides only a superficial description of transsilylation reactions resulting in heterocyclic derivatives of hypervalent silicon, because they have been surveyed in other reviews.[141, 142, 146, 148]

6. Miscellaneous reactions

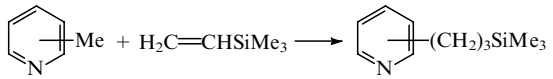
2-(3′-Trimethylsilylpropyl)benzothiazole was obtained by the reaction of 2-aminobenzenethiol with 3-trimethylsilylbutyryl chloride (**7**) in a polar solvent and in the presence of triethylamine (50–150 °C).[155]



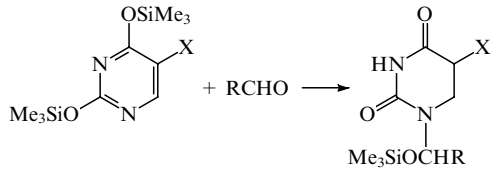
1,2,3,4-Tetrahydroquinoline, 1,2,3,4-tetrahydroisoquinoline and *N*-methylpiperazine are more reactive than 8-hydroxyquinoline with respect to the acid chloride **7** in inert solvents (ether, hexane) at 0–10 °C.[156]



Thermal homolytic addition of 2-, 3- and 4-methylpyridines to vinyltrimethylsilane in the presence of $(Bu^tO)_2$ at 350 °C gives 2-, 3- and 4-(3-trimethylsilylpropyl)pyridines in low yields (12%–28%).[157]

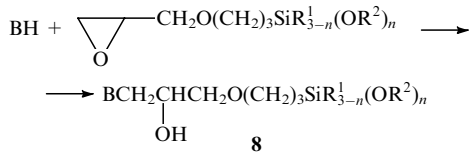


The reaction of 2,4-bis(trimethylsilyl)uracil with aromatic (benzaldehyde) and heteroaromatic (furfural, 5-nitrofurfural, furylacrolein) aldehydes gave *N*-substituted siloxymethyluracil derivatives in 16% yield.[158]



X = H, F; R = Ph, 2-C_4H_3O, 2-$C_4H_3OCH{=}CH$, 2-(5-$NO_2C_4H_3O$).

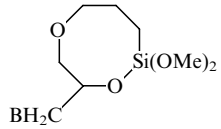
Heterocyclic compounds (3-mercapto-1,2,4-triazole,[159] 2-mercapto derivatives of 1-methylimidazole,[160] benzoimidazole, benzothiazole,[161] *N*-substituted benzoimidazoles,[162,163] benzotriazoles[164] and 3-amino-1,2,4-triazole[165]) containing active hydrogen atoms react at 120 °C with 3-glycidoxypropyl-(alkoxy)alkylsilanes to give the corresponding azoles **8**.



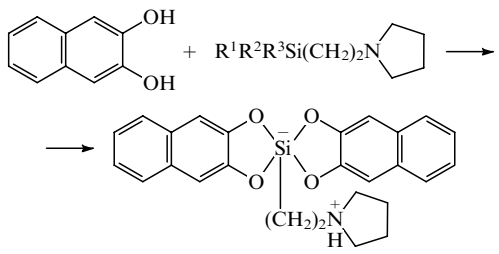
8

BH = HetSH, ; R^1, R^2 = Alk(C_1–C_3); n = 0–3.

For R^2 = Me and n = 3, a cyclic intramolecular transesterification product is formed in addition to the azole.



The zwitter-ionic spirocyclic bis(2,3-naphthylenediolato)[2-(pyrrolidino)ethyl]silicate was obtained by the reaction of 2,3-dihydroxynaphthalene with (2-pyrrolidinoethyl)silanes.[166]



R^1 = OMe, R^2 = Ph, R^3 = OMe, C_6H_{11}; R^1 = R^2 = R^3 = OMe.

In the case of silanes with R^1 = OMe, R^2 = Ph and R^3 = C_6H_{11}, the formation of the zwitter-ionic silicate (yield 81%) is accompanied by two unusual reactions involving cleavage of the Si–C bonds [Si–C(Ph) and Si–C(C_6H_{11})] under mild conditions.[166]

III. Chemical properties

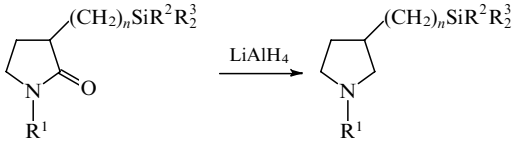
1. Reduction

Reduction of silicon-substituted heterylalkylsilanes containing reactive substituents at the silicon atom was used to obtain the corresponding derivatives having the Si–H bond. Thus the reaction of alkoxy(alkyl)(2-aziridinoethyl)silanes with lithium aluminium hydride occurs under mild conditions at equimolar ratios of the reagents.[75,167]

R^1, R^2 = H, Alk; n = 0–2; m = 0–3, 5; x = 2, 5.

The reactivity of the functional groups at the silicon atom (OR^2 and H) in the starting compounds and reaction products strongly depends on the relative remoteness of the silicon and nitrogen atoms. When the nitrogen atom is in the α-position relative to the silicon atom, the yield of the reduction products drops drastically. Thus the yields of $R^1_nH_{3-n}Si(CH_2)_mN(CH_2)_5$ are 12%–15% for m = 1, whereas for m = 3 and 5 the yields are 55%–60%.

N-substituted 3-trialkylsilylalkylpyrrolidones are reduced with lithium aluminium hydride to the corresponding pyrrolidine derivatives (yields 58%–60%).[146]



R^1 = Me: R^2 = Me, R^3 = Ph; R^2 = R^3 = Me, Et; n = 0, 1.

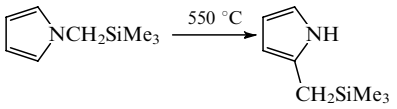
2. Desilylation

The Si–CH_2 bond in benzyltrimethylsilane and 1-(trimethylsilylmethyl)pyrrole is cleaved with alcoholic alkali; this latter reaction occurs with greater difficulty.[8]

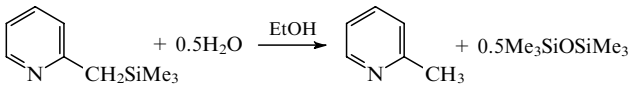


R = Ph, .

At 550 °C, 1-(trimethylsilylmethyl)pyrrole is isomerised to 2-(trimethylsilylmethyl)pyrrole in 39% yield.[8]



The Si–CH_2 bond in 2-trimethylsilylmethylpyridine is cleaved even with 95% EtOH to give 2-methylpyridine and hexamethyldisiloxane (82%).

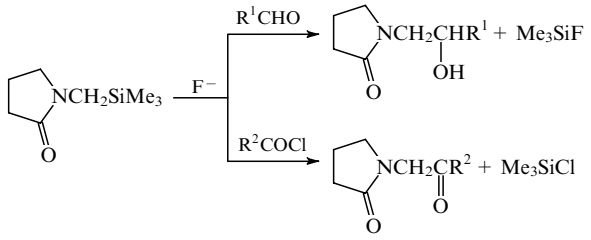


Apparently, this reaction is initiated by the water present in the alcohol and proceeds autocatalytically and in contrast with 2-trimethylsilylpyridine it needs prolonged (48 h) heating.[9] This reaction is catalysed by alkalis and acids.[168] Acetic acid can be used instead of 95% ethyl alcohol.[8]

The rate of alkaline hydrolysis of the Si–CH_2 bonds in isomeric trimethylsilylmethyl-substituted pyridines (Py) and quinolines (Q) was determined in aqueous methanol at 50 °C.

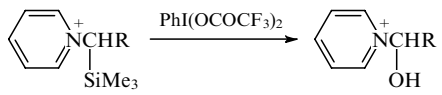
Depending on the position of the substituent (CH_2SiMe_3) in the heterocycle, the reactivity decreases in the following order (the relative reaction rates are given in parentheses): 2-QCH_2SiMe_3 (41) > 4-QCH_2SiMe_3 (37) > 4-$PyCH_2SiMe_3$ (8.9) > 2-$PyCH_2$–$SiMe_3$ (1.0) > 3-QCH_2SiMe_3 (0.161) > 3-$PyCH_2SiMe_3$ (0.03).[54] These data suggest that quinoline derivatives are desilylated faster than the corresponding pyridine derivatives.

In the presence of tetrabutylammonium fluoride in DMF or CsF in HMPA, *N*-trimethylsilylmethyl-substituted phthalimide,[11] 2-piperidone,[37] 2-mercaptobenzothiazole,[60] 2-benzothiazole,[61] 1,2,4-triazole [83, 85, 169] and 4-methylpyridine [170] are smoothly desilylated with aldehydes in THF (20–25 °C) to give the corresponding heterocyclic alcohols. Similar reactions of acid chlorides give the corresponding heterocyclic ketones.[37] For example:

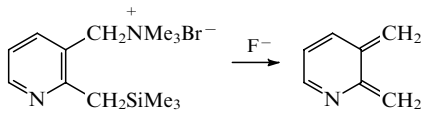


R^1 = Pr^n, Pr^i, Ph; R^2 = Pr^n, 4-MeC_6H_4, PhO.

Oxidative desilylation of trimethylsilylmethyl derivatives of pyridine and quinoline with $PhI(OCOCF_3)_2$ yields the corresponding alcohols in good yields.[171]



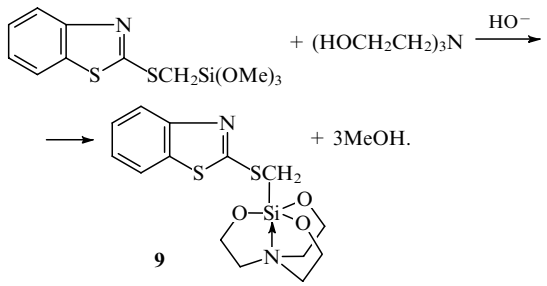
CsF-Induced desilylation of trimethyl[3-(2-trimethylsilylmethylpyridyl)methyl]ammonium bromide in acetonitrile (55 °C) affords a pyridinium analogue of *o*-quinodimethane.[172, 173]



The reaction of 2-(trimethylsilylmethyl)pyridine with perfluoro(2-methylprop-1-enylmethyl ether) and perfluoro(2-methylpent-2-ene) gave 2-[2-methoxy-3-(trifluromethyl)but-1-enyl]pyridine (yield 50%) and 2-pentafluoroethyl-3-trifluoromethyl-4*H*-quinolizin-4-one, respectively.[174]

3. Reactions with functional substituents at the silicon atom

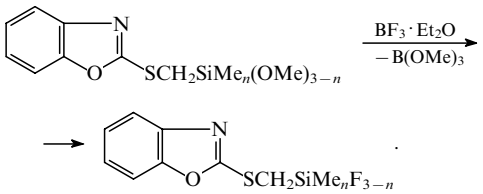
KOH-Catalysed transesterification of (heterylmethyl)trialkoxysilanes with triethanolamine results in the corresponding heterylmethylsilatranes.[40–42, 72, 89, 91, 117, 118, 133, 142, 175–178] For example:



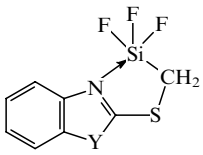
9

The molecular and crystal structures of 1-(2-benzothiazolylthiomethyl)silatrane was established by high-resolution X-ray analysis.[178]

The reactions of trimethoxysilylmethyl- and methyldimethoxysilylmethyl derivatives of pyridine, 8-mercaptoquinoline, 8-hydroxyquinoline, 2-mercaptobenzooxazole, 2-mercaptobenzothiazole, pyrrolidine and pyridine with HF, $BF_3 \cdot Et_2O$ or SbF_3 give the corresponding heterylalkyltrifluoro- and methyl(heterylalkyl)difluorosilanes in 61%–72% yields.[117, 118, 179–183] For example:

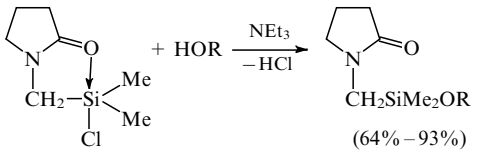


According to X-ray analysis data, the molecules of (2-benzooxazolylthiomethyl)- and (2-benzothiazolylthiomethyl)-trifluorosilanes contain an intramolecular N → Si coordination bond, which closes the five-membered ring.[117, 181]



Y = O, S.

Etherification of the corresponding chlorides in the presence of triethylamine as the HCl acceptor is the most versatile method for the synthesis of *N*-(dimethylalkoxysilylmethyl)lactams.[184, 185]

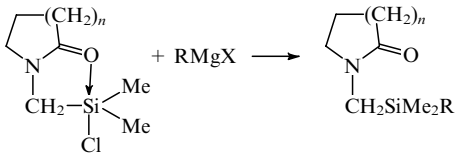


R = Me, Pr^i, $C_{10}H_{21}$, CH_2Ph.

The O → Si coordination bond is destroyed in the synthesis of alkoxy derivatives. With substitution of an RONa–PhMe for an ROH–Et_3N system, the yields of *N*-(dimethylalkoxysilylmethyl)lactams (R = Me and Pr^i) are only 46% and 38%, respectively. This is due to the cleavage of the Si–CH_2 bond, which occurs simultaneously with the formation of *N*-methyllactams.

Trialkylalkoxystannanes can also be used instead of sodium alkoxides. This method was used to prepare *N*-(dimethylalkoxysilylmethyl)-substituted pyrrolidones and 2-azepinones in 79%–96% yields.[186]

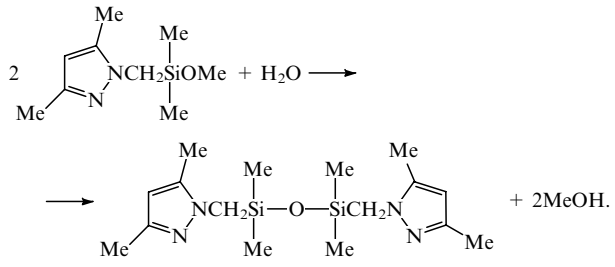
N-Chlorodimethylsilyllactams react with the Grignard reagents (MeMgI, $PhCH_2MgCl$) at the Si–Cl bond with high chemoselectivity to give the corresponding *N*-(trimethylsilylmethyl)- and *N*-(benzyldimethylsilylmethyl)-lactam derivatives.[187]



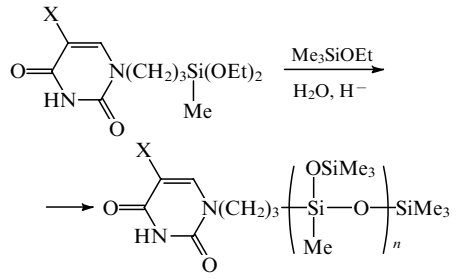
R = Me, CH_2Ph; X = Cl, I; *n* = 1–3.

4. Hydrolytic reactions

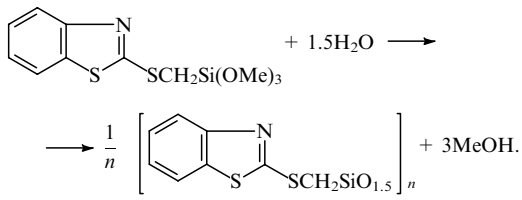
The hydrolysis of *N*-(methoxydiphenylsilylmethyl)-1,2,4-triazole,[28] *N*-(methoxydimethylsilylmethyl)-3,5-dimethylpyrazole,[90] *N*-(chlorodimethylsilylmethyl)-[188] and *N*-(alkoxydimethylsilylmethyl)lactams [189] gives the corresponding linear 1,3-bis(*N*-heterylmethyl)tetraorganyldisiloxanes in 60%–82% yields. For example:



The hydrolytic polycondensation of *N*-[3-(diethoxy)methylsilylpropyl] derivatives of uracil, thymine and adenine gives rise to the corresponding polyorganylsiloxanes.[36]

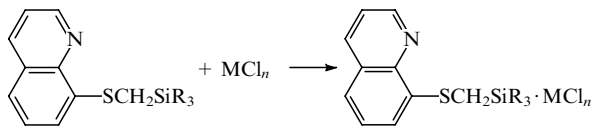


The hydrolysis of 4-(trialkoxysilylalkyl)tetrahydro-1,4-thiazine oxide,[133] 2-(2-trimethoxysilylmethylthio)benzoimidazole, -benzooxazole and -benzothiazole,[40–42, 190] as well as of (*N*-trialkoxysilylpropyl)morpholine [191] proceeds with the formation of the corresponding polyheterylalkylsilylsesquioxanes in 10%–63% yields. For example:



5. Complex formation

2-[3-(Triethylsilylpropyl)methyl]quinoline,[91] *N*-trimethylsilylmethyl-1,2,3,4-tetrahydroisoquinoline,[75, 93, 192] *N*-[3-(diphenylmethylsilyl)propyl]morpholine and piperazine [110] and *N*-[3-(trialkylsilyl)propargyl]pyrrolidine and -piperidine [124] react with a saturated solution of hydrogen chloride in ether to yield the corresponding hydrochlorides. The reaction of (8-quinolylthiomethyl)trimethylsilanes, -trimethoxysilanes and -silatrane with $CuCl_2$, $ZnCl_2$, $PtCl_2$ and $SnCl_4$ [193–195] gave various types of coordination compounds the structure of which depended on the nature of the metal and substituents at the silicon atom. Thus the reaction of (8-quinolylthiomethyl)trimethylsilane with potassium tetrachloroplatinate results in a planar-tetragonal chelate complex with $PtCl_2$.



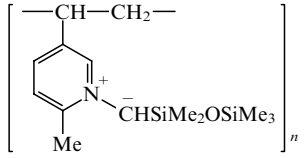
R = Me, OMe; $R_3 = (OCH_2CH_2)_3N$; $MCl_n = CuCl_2$, $ZnCl_2$, $PtCl_2$, $SnCl_4$, HCl.

IV. Biological activities and practical applications

Many of the compounds considered in this review possess highly specific biological activities which are determined by both the presence of a heterocyclic system and the silicon atom flanked with the corresponding substituents. Therefore, such compounds have attracted considerable attention in the past decade. *N*-(Triorganylsilylalkyl)imidazoles,[12, 13, 20, 22, 23, 26, 30, 31, 196, 197] and -1,2,4-triazoles,[12, 13, 20–24, 26–31, 84, 85, 196–206] 8-(trimethoxysilylpropylamino)quinoline and -5,7-dibromoquinoline,[207] as well as 2-(trimethylsilyl)methylpyridines [208] possess a broad spectrum of bactericidal and fungicidal activities. Used as fungicides (20–100 g hectare^{-1}), these compounds inhibit the growth of *Puccinia recondita, Sphaerotheca fuliginea, Erysiphe graminis* and *Podosphaera leucotricha*. Thus 1-[2-(4-bromophenyl)ethyldimethylsilylmethyl]imidazole (50–200 mg litre^{-1}) exhibits a fungicidal activity against *Erysiphe cichoracearum, Erysiphe graminis, Puccinia recondita*, *Puccinia coronata* and *Phalososphaeria nodorum*.[26]

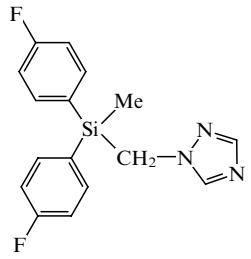
Substituted (trimethylsilyl)-3,5-bisaminomethyl-1,2,4-triazole and 2-(trimethylsilylmethoxy)pyrrolidone manifest high insecticidal activity.[94] *N*-[3-(4-Chlorophenyl)dimethylsilylpropyl]piperidine (2 and 5 g kg^{-1}) inhibits the growth of tobacco sucking discs.[15]

(4-Halogenophenyl)methyl-bis(1,2,4-triazol-1-ylmethyl)silane (Hal = F, Cl),[24] 3-[bis-(4-fluorophenyl)methylsilyl]imidazole [34] and 7-chloro-4-(4-trimethylsilylethynylpyrazolo)quinoline [70] used at a concentration of 500 mg litre^{-1} exert catalytic systemic effects against various diseases of wheat, rice, barley, oats, maize, cotton, soybean, coffee, sugar cane, apple trees, *etc*. *N*-Trialkylsilylmethyl-2-pyrrolidones and -caprolactams inhibit the growth of certain bacteria inducing diseases in vine and cucurbitaceous plants.[80] *N*-Trimethylsilylmethylbenzooxazine is an active and non-phytotoxic (for culture plants) component of herbicidal preparations which are commonly used in doses of 0.06–1.00 kg hectare^{-1} (see Ref. 209). 3,5-Diamino-6-(trimethylsilylethyl)-1,2,4-triazine is used for elimination of organisms attacking cabbage, beetroot and tobacco seedlings.[210] Poly-[2-methyl-5-vinylpyridinium 1-pentamethyldisiloxymethylide] possesses high pesticidal and growth-regulating activities.[211, 212]



4-[3-(Trimethylsilyl)propylamino]quinolines manifest not only herbicidal but also selective antimicrobial activities,[97, 99] *viz*., they efficiently suppress pathogenic fungi and exhibit moderate inhibiting action on the growth of gram-positive bacteria without any effect on gram-negative bacteria. 4-[3-(Trialkylsilyl)propylamino]-7-chloroquinolines exhibit high fungistatic activity. Incorporation of a methoxy group at position 6 of these compounds markedly increases their bacteriostatic activity.[98, 99]

Flusilazole, 1-[bis(4-fluorophenyl)methylsilylmethyl]-1,2,4-triazole, is an efficient organosilicon ergosterol-like fungicide able to suppress various biosynthetic reactions.[25, 213–231]



At present, flusilazole is one of the most widely used broad-spectrum agricultural fungicides. It is used against a complex of diseases in cereals, vine, seed fruits, sugar beet and peanut induced by asco-, basidio- and deuteromycetes (also in combination with other fungicides) in the doses of 80–240 g hectare^{-1}.

(*N*-Methylpiperazinylmethyl)phenylcyclohexylsilanol sulfomethylate exerts a potent and highly selective antimuscarinic effect and is used in experimental pharmacology for classification of various subtypes of muscarine receptors.[74]

N-(Triethylsilylpropargyl)piperidine and -pyrrolidine hydrochlorides have a neurotropic effect on animals.[124] 6-[4-(Trimethylsilyl)butyrylamino]quinoline hydrochloride manifests an antiviral activity against influenza A virus.[92] 1-(2-Chlorophenyldiphenyl]methyl-3-(dimethylphenylsilyl)methylimidazolium chloride,[77] *N*-[3-(trialkylsilyl)propyl]imidazole,[86,87] *N*-[3-(triorganylsilyl)propyl]piperazine and -morpholine,[110] *N*-[1-(trimethylsilylmethyl)]-1,2,4-triazole,[209] as well as 4-trialkylsilylpropylaminoquinoline[91,93,156] and -pyridine[232] possess high antimicrobial activities. These compounds display high fungistatic and cytotoxic activities.

4-(Triorganylsilylalkyl)tetrahydro-1,4-thiazine 1-oxides produce a potent effect on the central and peripheral nervous systems and possess a bacteriostatic activity.[133]

Weakly toxic [1-(2-oxopyrrolidino)ethyl]triethoxysilane exerts antishock and tranquillising effects.[116] Methyldibutyl-(3-piperidinopropyl)silane and 2-(3-trimethylsilylpropargyloxy)-pyrazine-6-thiocarboxamide hydrochlorides and *N*-(triethylpropargylsilylmethyl)piperidine manifest a tuberculostatic activity *in vitro* against *M. tuberculosis* and a bacteriostatic activity against *Staphylococcus aureus haemolyticus* 209.[38,233]

4-[3-(Trialkylsilyl)propylamino]quinolines, 1-(trialkylsilylpropyl)fluorouracil,[234] *N*-trimethylsilylmethyl-1,2,3,4-tetrahydroisoquinoline hydrochloride,[93] 4-quinolylaminoalkyl-, 1-(1-tetrahydroquinolylmethyl)- and 1-[1-(4-quinolylamino)propyl]-silatranes[235] manifest an antitumour activity, Ehrlich ascites tumour and leukemia L_D 5178 cells being especially sensitive to the cancerostatic effects of these compounds. (Heteryl-alkyl)trialkylsilanes are less active than the corresponding silatrane derivatives.

1-(3-Trialkylsilylpropyl)piperidine is used as an active component of a disinfectant used for the treatment of textiles.[236] 4-(2-Trimethoxysilyl)ethyl-*N*-alkylpyridinium bromide, which has the property to increase the volume and softness of textile fibres, also manifests an antimicrobial activity.[237]

(Triorganylsilyl)benzotriazole derivatives are used as additives to sun protection lotions.[112]

N-(Trimethoxysilylpropyl)maleimide[238] and 3-(triorganylsilylalkyl)-2-pyrrolidone[239] are added to polymer-based pharmaceuticals in order to improve their mechanical properties.

5-[(Trimethylsilyl)methylthio]- and 5-[(diethylmethylsilyl)-methylthio]-8-mercaptoquinolines are used as organic analytical reagents.[18,240,241]

2-(2′-Trimethoxysilylethyl)pyridine is used for the preparation of rhodium carbonyl complexes. The latter are immobilised on silica gel and used as catalysts.[242]

1-(Trialkoxysilylalkyl)-3,5-diaminotriazine,[243] -piperidine[244] and -2-(4-triazolyl)benzoimidazole[245] derivatives are used for treatment of fibre glass fabrics in order to improve their coupling with the polymeric binders. *N*-Substituted organosilicon derivatives of imidazole,[246] benzoimidazole[247] and triazine[248] are used to protect metal surfaces owing to their ability to form highly thermostable films that withstand 100 heating cycles.

2-(Trialkylsilylalkyl)benzothiazole has been patented as a binder in the production of siliceous fillers for rubber tyres.[78,249–253]

Polysilanes containing *N*-alkylpyrrole groups[254] and *N*-trimethoxysilylpropyl imidazole derivatives[255] are used in the production of conducting materials.

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