Journal of Organometallic Chemistry, 376 (1989) 249–258
Elsevier Sequoia S.A., Lausanne – Printed in The Netherlands
JOM 20062

The synthesis and structure of chiral compounds with two hypervalent silicon atoms, *Si,Si′*-substituted 1,2-bis(dimethylsilylmethyl)diacetylhydrazines

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(Received May 17th, 1989)

Abstract

The multi-stage character of the reaction of 1,2-diacetylhydrazine bis-*O,O′*-trimethylsilyl derivative with $ClCH_2SiMe_2Cl$ to give bis-(*O–Si*)-1,2-bis(dimethylchlorosilylmethyl)-1,2-diacetylhydrazine (VIII) was established from NMR spectroscopy. Methanolysis of VIII in the presence of NEt_3 gives the *Si,Si′*-dimethoxy derivative (IX). The corresponding *Si,Si′*-difluoro derivative (XI) was obtained by treatment of IX with boron trifluoride etherate.

The structure, the stereodynamic behaviour, and the exchange reactions of chiral molecules of VIII, IX and XI and their precursors have been studies by multinuclear NMR spectroscopy. Compounds VIII, IX, and XI are the first examples of substances that contain two hypervalent silicon atoms and an $XSi(C)_3O$ coordination centre.

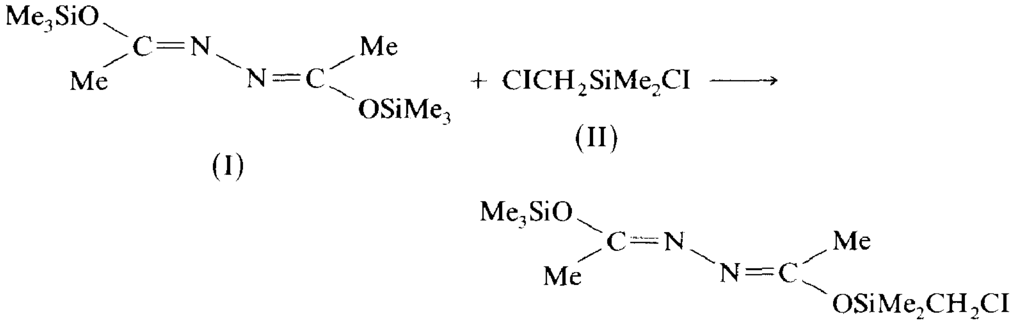
Introduction

The reaction of dimethyl(chloromethyl)chlorosilane with *N*- or *O*-trimethylsilylamides [1,2], -lactams [2,3], -ureas [4], -acetylacetamide [5], or 1,1-dimethyl-2-acylhydrazines [6] is a convenient synthetic route to the corresponding (O–Si)chelate *N*-(chlorodimethylsilyl)methyl derivatives having a $ClSi(C)_3O$ coordination centre.

The use of bis-*O,O′*-trimethylsilylated 1,2-diacetylhydrazine in a similar reaction has opened the way to the previously unknown bis(*O–Si*)chelate *Si,Si′*-disubstituted 1,2-bis-(dimethylsilylmethyl)-1,2-diacetylhydrazines [7], which are the first examples of chiral compounds bearing two (*O–Si*)chelated pentacoordinate silicon atoms in one molecule. The relevant data on the synthesis, structure, and some of the chemical conversions of these compounds and their precursors are described.

Results and discussion

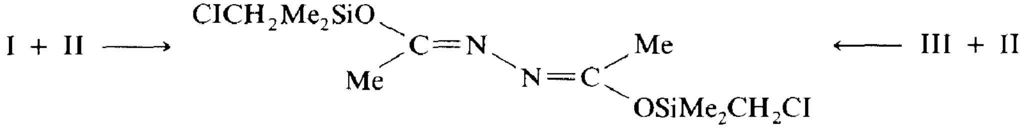
The use of NMR spectroscopy has revealed the multi-stage and peculiar character of the interaction of bis-*O,O'*-trimethylsilylated 1,2-diacetylhydrazine (I) with a two-fold excess of dimethyl(chloromethyl)chlorosilane (II). After several minutes mixing in chloroform at −25 °C the slow replacement of one of the trimethylsilyl groups in molecule I by a dimethyl(chloromethyl)silyl group takes place:



(I) (II)

(III)

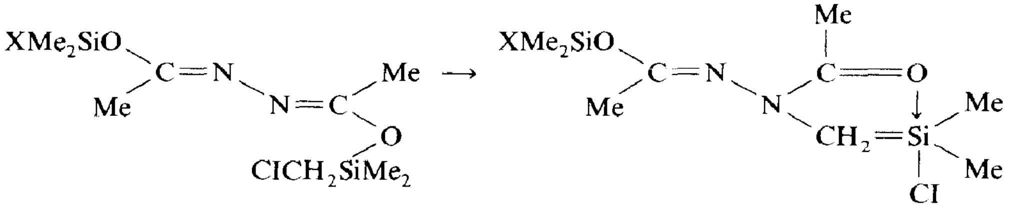
The formation of the double *trans*-silylation product (IV) occurs far more slowly (III : IV = 4 : 1) under these conditions:



(IV)

Compounds III and IV show a *trans-S-trans* configuration analogous to those of molecules I [8]. It is this configuration that predetermines the course of further conversions.

At −15 °C, compounds III and IV undergo an intramolecular rearrangement. Nucleophilic attack on the nearest nitrogen atom by the chloromethyl group results in its siliconmethylation whereas subsequent bond redistributions in the five-membered heterocycle and migration of the chlorine anion towards the silicon atom are completed with the formation of mono-(O–Si)chelate compounds V and VI.



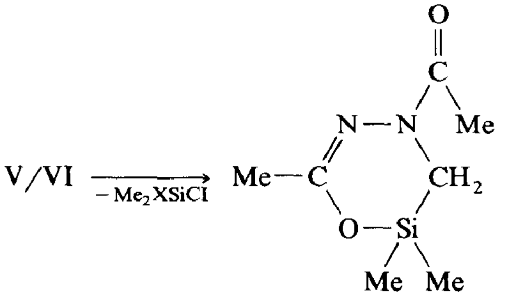
(III: X = Me;
IV: X = $ClCH_2$)

(V: X = Me;
VI: X = $ClCH_2$)

The amounts of V and VII in the reaction mixtures become noticeable after 20 min at 20 °C. At this temperature the compounds are stable for a few hours.

It is noteworthy that the second dimethyl(chloromethyl)silylimidate fragment of molecule VI does not rearrange under these conditions. Evidently, this is caused by the strong electron-withdrawing effect of the neighbouring (*O–Si*)chelate group. The latter decreases the electrophilicity of the sp^2-hybridized nitrogen atom in molecules VI and considerably hinders its siliconmethylation. As a result, the subsequent conversion of compounds V and VI proceeds in nearly the same manner.

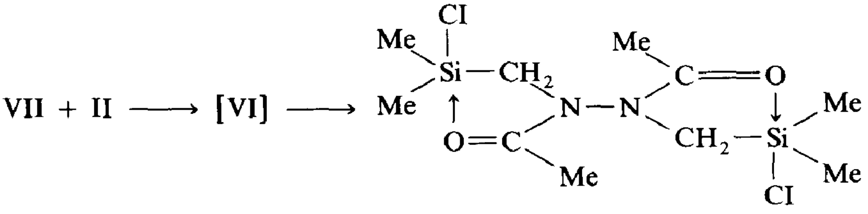
At 25 °C the intermediates V and VI undergo cyclization at the expense of elimination of trimethyl- or dimethyl(chloromethyl)chlorosilane:



(VII)

The quantitative formation of 1-sila-2-oxa-4,5-diazacyclohexene-3 (VII) requires several minutes in concentrated solutions of the reaction mixtures and half an hour in dilute solutions for completion *. The clearly defined concentration dependence of this reaction stage suggests its intermolecular characters. Compound VII can be readily isolated from the reaction mixture by removal of the solvent, the Me_3SiCl, and the $ClCH_2SiMe_2Cl$ by distillation in vacuum at 0 °C. In the absence of active reactants this compound is fairly stable. Its formation, however, is controlled by kinetic rather than thermodynamic factors.

In fact, in the reaction mixture the heterocycle VII reacts slowly with $ClCH_2SiMe_2Cl$ under a 10% conversion to give the end reaction product 1,2-bis(*O–Si*)chelate 1,2-bis(dimethylchlorosilylmethyl)-1,2-diacetylhydrazine (VIII) at room temperature during 24 h.



(VIII)

The formation of this compound, the first of the known (*O–Si*)chelate compounds having two pentacoordinate silicon atoms, also takes place either in the reaction of the preparatively isolated heterocycle VII with the equimolar amount of $ClCH_2SiMe_2Cl$ or in that of a two-fold excess of the latter with the starting I in $CDCl_3$ solution. These reactions go to completion within four days at 20 °C, and within an hour at 80 °C. We have used the latter method to obtain preparatively

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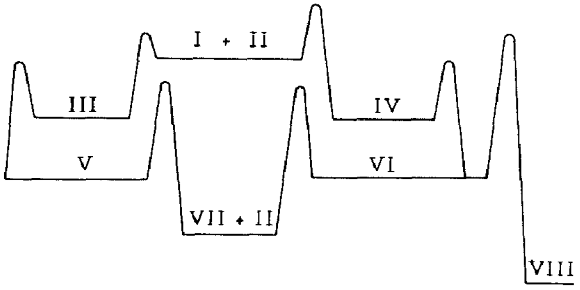
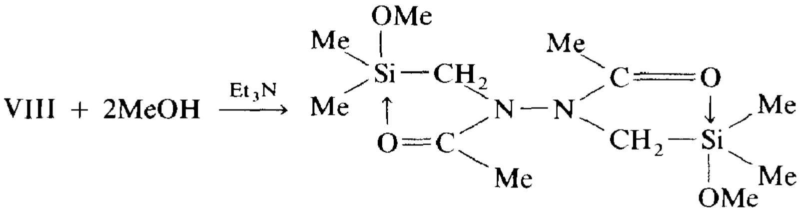
* At −15 °C a small amount of VII (~ 5%) is formed after 50 min.

bis(*O–Si*)chelate compound VIII. On the basis of the results achieved the reaction under investigation can be represented by the following qualitative scheme:

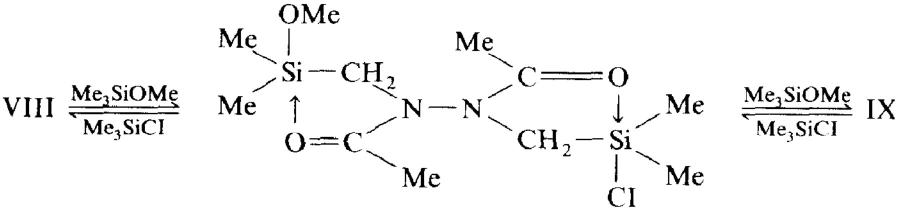


Compound VIII is a convenient starting reagent for the synthesis of a series of parent *Si,Si′*-disubstituted 1,2-bis(dimethylsilylmethyl)-1,2-diacetylhydrazines. The methanolysis of VIII in the presence of triethylamine gives the dimethoxy derivative IX in quantitative yield.



(IX)

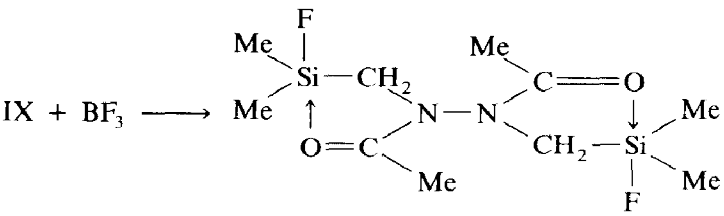
This compound is also formed by an exchange reaction of VIII with trimethylmethoxysilane. However, even with a five-fold excess of the trimethylmethoxysilane the conversion of VIII at 25 °C does not exceed 33%, and the equilibrium mixture contains the final product IX and the monomethoxylated product X (the IX : X ratio is 1 : 1).



(X)

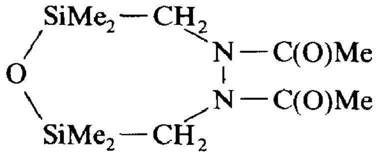
The preparative isolation of the *Si,Si′*-unsymmetrically substituted X is hindered by its ready symmetrization into VIII and IX, not only during distillation, but also when the reaction mixture is kept for a long time at room temperature.

When treated with boron trifluoride etherate, compound IX converts smoothly into the *Si,Si′*-difluoro derivative XI:



(XI)

The previously unknown Si,Si′-disubstituted 1,2-bis(dimethylsilylmethyl)-1,2-diacetylhydrazines VIII, IX, XI are white crystalline substances. They are hydrolyzed (also by atmospheric moisture) to 1,1,3,3-tetramethyl-5,6-diacetyl-1,3-disila-2-oxa-5,6-diazaheptane (XII):

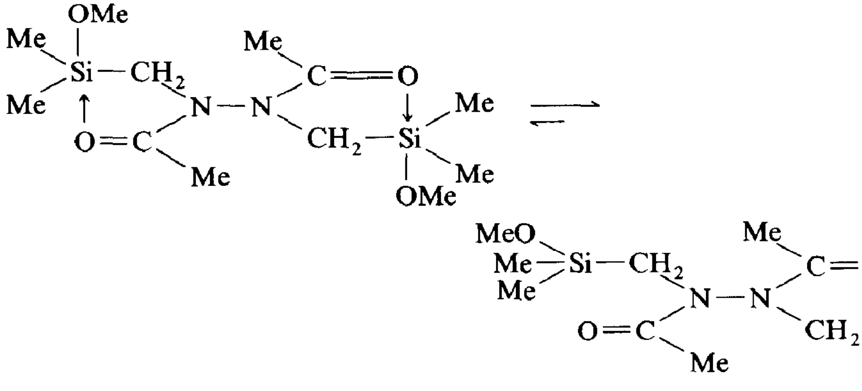


(XII)

The NMR data (Tables 1 and 2) support the suggested structure of the intermediates III–VI and the products of the reactions studied VII–XII, and allow some conclusions concerning the structural and stereodynamic features of these compounds. 1,fig t2

In the *N*-(dimethylchlorosilylmethyl)amide fragments of molecules V, VI, and VIII the sum of the electronegativities of the substituents "covalently" bonded with the Si atom is nearly the same as that for the tetracoordinate silicon compound $ClCH_2SiMe_2Cl$. Nevertheless, the corresponding ^{29}Si NMR signals in the spectra of the former compounds display a considerable upfield shift with respect to the latter (δ 24.5 ppm). These shifts provide direct evidence [9,10] for the pentacoordination by the Si atom and the (*O*–*Si*)chelate structure of the above fragments in molecules V, VI and VIII. The "coordinate" nature of the Si←O bond in hypervalent Cl–Si–O groups is confirmed by an increase in the ^{29}Si shieldings as the temperature falls. The compounds with a coordinate Si←Cl bond are characterized by an opposite dependence [6].

The ^{29}Si shieldings of methoxy- and fluorodimethylsilyl fragments of molecules IX and XI (especially those of IX are not that much higher than the values observed for the corresponding tetracoordinate silicon derivatives $ClCH_2SiMe_2X$ ($X = OMe$, $\delta = 15.9$; $X = F$, $\delta = 24.8$ ppm). This indicates a significant weakening in the coordinate Si←O bond on going from chloro- to fluoro- and methoxy-Si-substituted 1,2-bis(dimethylsilylmethyl)-1,2-diacetylhydrazines. Moreover, for molecules IX (and X) the obvious equilibrium between the (*O*–*Si*)chelate and the acyclic (without Si←O bond) forms seems to be considerably shifted toward the acyclic form.



The influence of the Si-substituent on the degree of hypervalent Si bonding is typical [9,11].

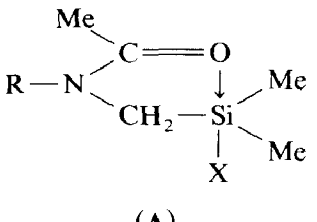
For compounds with the same coordination centre $XSi(C)_3O$ the decrease in the degree of Si←O interaction is normally accompanied by an increase in the Si

Table 1

^{13}C and ^{29}Si NMR chemical shifts of intermediates (XMe$_2'$Si'O)Me'C'=N–N=CMe(OSiMe$_2$CH$_2$Cl) (III, VI) at −25°C and (XMe$_2'$Si'O)Me'C'=NN[C(O)Me](CH$_2$SiMe$_2$Cl) (V, VI) at −15°C

Compound	X	δ(Si)		δ(C)							
		Si	Si'	MeSi	Me'Si	*Me*C	*Me'*C	ClCH$_2$	NCH$_2$	NCO	NCO
III	Me	12.9	19.5	−0.1	−0.2	16.2	15.7	29.6	–	163.8	163.2
IV	ClCH$_2$	12.4	12.4	−3.1	−3.1	15.9	15.9	29.5		163.6	163.6
V	Me	−37.3	25.5	7.3	3.1	16.6	16.8		42.6	171.1	173.8
VI	ClCH$_2$	−37.9	19.3	7.3	−0.3	16.5	17.0	28.1	44.5	170.9	174.9

shieldings [9,12]. Thus a comparison of the ^{29}Si NMR chemical shifts for compounds of type **A** (see Tables 1 and 2) reveals an unexpectedly pronounced effect by the nature of *N*-substituent R on the degree of Si–O bonding in the chelate fragment.



(**A**)

Compound	X	R
V	Cl	Me$_3$SiO(Me)C=N
VI	Cl	Me$_2$(ClCH$_2$)SiO(Me)C=N
VIII	Cl	(Me$_2$ClSiCH$_2$)MeC(O)N
X	Cl	[Me$_2$(MeO)SiCH$_2$]MeC(O)N
XIII	Cl	NMe$_2$ ($\delta_{Si} = -35.9$ [6])
IX	MeO	[Me$_2$(MeO)SiCH$_2$]MeC(O)N
X	MeO	(Me$_2$ClSiCH$_2$)MeC(O)N

The ^{29}Si NMR spectra show that in the series of Si-substituted molecules with X = Cl the Si←O interaction seems to weaken on going from the *N*-amine (XIII) and *N*-imidate compounds (V, VI) to the *N*-amide derivatives (VIII and X). The reason for this is the higher electron-withdrawing properties of the substituent R in the latter compounds. The close δ(Si) values and Si←O bond orders in the intermediates V and VI and compound XIII are also quite understandable. In normally conjugated systems the electron donating ability of the Me$_2$SiO(Me)C=N– should be lower than that of the NMe$_2$ group [13]. However, the mutual repulsion of lone pairs of electrons of the nitrogen atoms in molecules XIII precludes conjugation between the "amide" fragment and the substituent R = NMe$_2$. At the same time, the higher electronegativity of the α-atom of the substituent R in molecules V and VI can be compensated by π-electron transfer from this substituent to the "amide" fragment.

That the ^{29}Si chemical shifts of compounds **A** are susceptible to changes in the substituent R periphery is remarkable. An increase in the degree of its chelation (on going from IX to X and, especially clearly, from X to VIII) causes a noticeable decrease in the ^{29}Si screening constants and thus in the degree of Si←O bonding.

In analogy to other 1,2-substituted 1,2-diacetylhydrazines [14] the molecules VIII–XII have a high barrier to rotation (> 24 kcal/mole) about the N–N bond and the dihedral CNNC angles close to 90°. This is due to the high energy of

Table 2

^{1}H, ^{13}C and ^{29}Si NMR chemical shifts and coupling constants of compounds $(X'Me_2SiCH_2)[MeC(O)]NN[C(O)Me](CH_2SiMe_2X)$ (VIII–XI) at 25 °C

Compound	X (X′)	δ(Si)	δ(H)					^{2}J(HH)	δ(C)					
			MeSi		MeC	NCH$_2$			MeSi		MeC	NCH$_2$	OMe	CO
VIII	Cl	−8.8	0.62	0.66	2.09	2.56	3.19	15.1	5.63	6.01	17.8	39.2		175.1
	(Cl)													
IX	OMe	9.1	0.27	0.29	2.05	2.60	2.92	15.6	−1.63 [a]	−1.97 [a]	19.7	38.2	50.3	172.3
	(OMe)													
X	OMe	11.0	0.26	0.29	2.03	2.65	2.90	15.6	−1.97 [a]	−2.71 [a]	17.8	39.6	50.3	171.0
	(Cl)	−16.9	0.63	0.65	2.11	2.56	3.19	15.1	6.39 [a]	6.72 [a]	19.6	38.6		174.2
XI [b]	F	4.1	0.34	0.39	2.06	2.39	2.92	14.8	0.21	0.24	18.4	35.8		173.1
	(F)	(266.1)	(7.8)			(1.5)			(20.8)			(25.6)		

[a] At −10 °C. [b] The spin–spin couplings with ^{19}F nuclei are in parentheses.

mutual repulsion by the lone pairs of their nearly planar nitrogen atoms. As a result molecules VIII–XII are chiral. These features are confirmed by the non-equivalence of the diastereotopic geminal methylene protons as well as the Si-methyl groups in ^{1}H and ^{13}C NMR spectra. When solutions of preparatively isolated compounds VIII, IX, XI, and XIII are heated to 180 °C (in $C_6H_5NO_2$) the non-equivalence of the methylene protons and the spin–spin coupling between them (^2J 15.0 ± 0.5 Hz) remain constant.

However, the Si-methyl groups of these compounds become isochronous at 70–75 °C, the coalescence temperature and free energy of activation of the exchange process ($\ll$ 25 kcal/mole) are dependent upon the compound, its concentration, and the amount of other Si-functional derivatives in the solution. The reason for the coalescence of the Si-methyl group signals under molecular chirality retention is the ease of both the Berry type pseudorotation of pentacoordinate Si atom and intermolecular exchange of Si-functional substituents.

The latter suggestion is supported by the following observations. In the ^{1}H and ^{13}C NMR spectra of fluoro derivative XI the splitting of peaks caused by spin–spin coupling with ^{19}F nuclei disappears at temperatures only slightly above that required for coalescence of the methylsilyl group signals. In the spectra of solutions of mixtures of compounds VIII, IX, and X all the signals of dimethylchloro- and dimethyl(methoxy)silyl groups merge to give a single signal at temperatures above 75 °C. Such spectral changes can only result from an inversion of the Si atoms in a fast intermolecular exchange process of rigid Si-substituents, Cl and MeO.

From the ^{1}H and ^{13}C NMR data the diastereotopy of methyl groups at the tetrahedral silicon atoms in the heterocycle XII inert to exchange remains as high as 180 °.

Thus, bis(*O–Si*)chelate *Si,Si′*-disubstituted 1,2-bis(dimethylsilylmethyl)-1,2-diacetylhydrazines have been obtained for the first time and their schemes of formation, structure and some chemical transformations have been studied. These compounds and their derivatives with more active Si-substituents could become rather promising reagents in organic and polymer chemistry.

Experimental

^{1}H, ^{13}C and ^{29}Si NMR spectra were recorded on a JEOL FX 90Q spectrometer at 89.55, 22.49 and 17.85 MHz, respectively. ^{13}C and ^{29}Si NMR spectra were recorded with proton decoupling, the latter being obtained by use of the INEPT pulse sequence.

Chemical shifts were measured relative to internal TMS, with an accuracy of ±0.01 ppm for ^{1}H and ±0.05 ppm for ^{13}C and ^{29}Si.

Owing to the limited thermal stability of the intermediates mixing of the solutions of I and II in chloroform was performed at −70 °C. The ampoule containing the reaction mixture was placed in the thermostated NMR probe; the sample temperature was raised by 10 °C every 30 min until the signals of the new compounds appeared in the NMR spectrum. The mass spectrum was recorded on a MAT-212 chromatomass-spectrometer at an ionization voltage 70 eV.

1-Sila-2-oxa-4,5-diazacyclohexene-3 (VII)

A solution of compound I (15.0 g, 57.6 mmole) and dimethyl(chloromethyl)chlorosilane (9.0 g, 62.9 mmole) in 50 ml of dry Et_2O was stirred with a magnetic stirrer in an evacuated, glass ampoule at 20 ° C for 4 d. Low boiling point compounds were removed under reduced pressure (0.5 mmHg, 20 ° C). Recrystallization of the residue from 50 ml of hexane at −50 ° C gave 8.8 g (81.94%) of VII, m.p. 43–44 ° C (evacuated capillary). Found: C, 45.05; H, 7.55; N, 14.61; Si, 14.82. $C_7H_{14}N_2O_2Si_2$ Calc: C, 45.13; H, 7.57; N, 15.04; Si, 15.08%. Mass spectrum, m/z (intensity (%): $M^{+\cdot}$ 186(27), $[M-CO]^{+\cdot}$ 158(9), $[M-COCH_3]^{+\cdot}$ 143(43), 129(5), 116(38), 102(13), 100(11), 88(15), 75(100). NMR (δ, ppm): ^{1}H 0.35 (Me_2Si), 2.23 (MeC=O), 1.98 (MeC=N), 3.24 (CH_2); ^{13}C −1.55 (Me_2Si), 21.4 (MeC=O), 21.2 (MeC=N), 28.8 (CH_2) 171.5 (C=O) 149.4 (C=N); ^{29}Si: 18.0.

*1,2-Bis(*O–Si*)-1,2-bis(dimethylchlorosilylmethyl)-1,2-diacetylhydrazine (VIII)*

A mixture of compound I (3.10 g, 11.9 mmole), dimethyl(chloromethyl)chlorosilane (3.77 g, 26.3 mmole) and dry $CHCl_3$ (20 ml) was stirred with a magnetic stirrer in a glass evacuated ampoule for 2 h at 20 ° C and 2 h at 70–80 ° C. The solution was evaporated at a reduced pressure (0.5 mmHg). Recrystallization of the residue from 50 ml of hexane gave 3.18 g (79.7%) of compound VIII. M.p. 130 ° C (evacuated capillary). Found: C, 36.07; H, 8.48; N, 8.23; Si, 16.39; Cl, 20.98. $C_{10}H_{22}N_2O_2Si_2Cl_2$ calc: C, 35.81; H, 8.40; N, 8.35; Si, 16.75; Cl, 21.14%.

1,2-Bis(dimethylmethoxysilylmethyl)-1,2-diacetylhydrazine (IX)

A mixture of I (9.45 g, 36.2 mmole), dimethyl(chloromethylchlorosilane (10.38 g, 72.5 mmole) and dry $CHCl_3$ (60 ml) was boiled for 3 h in a glass evacuated ampoule. After cooling to 20 ° C the reaction mixture was evaporated under reduced pressure (0.5 mmHg). To the residue was added a solution of dry triethylamine (8.00 g, 79.1 mmole) and dry methanol (4.00 g, 124.8 mmole) in dry Et_2O (100 ml). The mixture was boiled for 4 h and filtered after cooling to 20 ° C. The filtrate was evaporated under reduced pressure (0.5 mmHg). The residue was boiled with 50 ml of hexane and the extract decanted (the procedure was repeated 5 times). Upon cooling to 20 ° C colourless crystals precipitated from the extract, which were separated from the mother liquor, recrystallized from hexane, and dried in vacuum (0.5 mmHg, 20 ° C). Product IX: yield 7.7 g (66.5%), m.p. 72–73 ° C (evacuated capillary). Found: C, 44.81; H, 8.72; N, 8.96; Si, 18.07. $C_{12}H_{28}N_2O_4Si_2$ Calc: C, 44.96; H, 8.80; N, 8.74; Si 17.53%.

1,2-Bis(dimethylfluorosilylmethyl)-1,2-diacetylhydrazine (XI)

To a solution of IX (1.30 g, 4.06 mmole) in dry Et_2O (10 ml) in an evacuated, glass ampoule was added a solution of $BF_3 \cdot Et_2O$ (0.38 g, 2.68 mmole) in dry Et_2O (10 ml). The mixture was stirred with a magnetic stirrer for 2 h at 20 ° C, boiled for 10 min, and then evaporated under reduced pressure (0.5 mmHg). Recrystallization of the residue from hexane gave compound XI, 0.95 g (79.0%), as colourless crystals, m.p. 84.5–85.5 ° C (evacuated capillary). Found: C, 40.71; H, 7.85; F, 13.21; N, 9.59; Si, 18.34. $C_{10}H_{22}F_2N_2O_2Si_2$ calc: C, 40.51; H, 7.48; F, 12.82; N, 9.45; Si, 18.95%.

1,1,3,3-Tetramethyl-5,6-diacetyl-1,3-disila-2-oxa-5,6-diazaheptane (XII)

A mixture of compound IX (11.0 g, 33.3 mmole), K_2CO_3 (5.0 g, 36.2 mmole), wet acetone (40 ml) and H_2O (3 ml) was stirred with a magnetic stirrer for 5 h at 20 ° C.

The reaction mixture was filtered. The filtrate was dried over K_2CO_3 and evaporated in vacuum (0.5 mmHg). Recrystallization of the residue from 60 ml of hexane gave 7.08 g (77.2%) of the crude product and subsequent sublimation (0.5 mmHg, bath temperature 100 °C) gave 6.84 g (74.6%) of product XII, m.p. 112–112.5 °C. Found: C, 43.91; H, 8.50; N, 10.27; Si, 20.30. $C_{10}H_{24}N_2Si_2O_4$ calc: C, 43.76; H, 8.08; N, 10.21; Si, 20.47. NMR, δ, ppm: ^1H 0.15 and 0.16 (Me_2Si), 2.40d and 4.08d (^2J 15.6 Hz) (CH_2), 1.99 (Me); ^{13}C 0.65 and 0.75 (Me_2Si), 20.2 (Me) 42.3 (CH_2), 171.7 (C=O); ^{29}Si: 8.5.

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