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Trimethylsilylated *N*-alkyl-substituted carbamates

III *. Structure of trimethylsilyl-*N*,*N*-diisopropyl carbamate in solid phase and in solution

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Abstract

The conformers of trimethylsilyl-*N*,*N*-diisopropyl carbamate were studied by ^{1}H NMR spectroscopy. The barriers to rotation by isopropyl groups and to the rotation around the amide bond, and the population of the conformers were determined from the coalescence temperature of the methyne signal. Single crystal X-ray diffraction studies revealed that the crystal had formed from preferred conformer in solution. The mirror symmetric conformer is stabilized by an intramolecular C–H···O hydrogen bond and by an intramolecular O···Si donor–acceptor interaction.

Introduction

The *N*-substituted trimethylsilyl carbamic acid esters are efficient silylating agents [1,2]. Thus it was of interest to find out about the intramolecular interactions of the silyl and carbamate groups with the substituents so as to evaluate the reactivity of these compounds. The temperature dependent internal rotation of acid

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* For part II see ref. 24.

amides is one of the most frequently studied phenomena since dynamic NMR (DNMR) spectroscopy has become widely used [3–8]. Literature on the conformation of carbamate and thiocarbamate esters is scarce. Although it has been found that extended delocalization [9,10] considerably affects the order of the CO–N bond.

The rotation of carbamate group in aliphatic, and in Me_3Si, Me_3Ge, Me_3Sn and Me_3Pb esters of *N,N*-dimethyl-carbamic acid was studied [11,12]. Delocalization is further expanded by incorporation of heteroatoms of the column IV into the molecule. The energetic effect of this phenomenon is revealed by ^1H DNMR studies. (In several instances splitting of the *N*-methyl signals were not observed even at low temperature [11,12]). The nature of the delocalization, however, was not clear from the NMR data alone, and the molecular geometry of carbamates could not be obtained from diffraction parameters either, since most of these compounds are liquids.

According to Sandström et al. [13,14] the methine protons of the *N,N*-diisopropylamino group are forced into the plane of the amide group and consequently their ^1H NMR shifts depend to a greater extent on the rotamer structure than on the *N*-methyl protons. Thus, diisopropyl carbamate esters are excellent model compounds for the investigation of the above-mentioned topic. Our aim was to study the effect of the empty *d*-orbital of the Si-atom on the delocalized π electron system of the carbamate group in trimethylsilyl *N,N*-diisopropyl carbamate ($i\text{-}Pr_2NCOOSiMe_3$; i-PrSiC).

The compound i-PrSiC can be prepared in crystalline form at room temperature so that diffraction measurements can be carried out.

Experimental

Trimethylsilyl-*N,N*-diisopropyl carbamate was prepared from the corresponding ammonium salt by silylation with Me_3SiCl [15]. The product was recrystallized from n-hexane to give crystals suitable for X-ray studies.

The ^1H NMR spectra were recorded on a Bruker WM-250 FT spectrometer equipped with a superconducting magnet and controlled by an Aspect 2000 com-

Table 1

^1H and ^{13}C [a] NMR chemical shifts in i-PrSiC (δ_{TMS} in ppm)

Nucleus	Solvent	$SiCH_3$ s(9H)	$CCH_3(J)$ [b] d(12H)	CH 2×m [c] (2H)
^1H	$CDCl_3$	0.29	1.20 (~5) [d]	3.60, 4.15
^1H	DMSO-d_6	0.24	1.15 (6.7)	3.70, 3.98
^1H	Toluene-d_8	0.56	1.29 (6.3)	3.74, 4.23
^{13}C [e]	$CDCl_3$	0.2	21.1	46.1

[a] Measured at 62.89 MHz in a 5 mm tube, at room temperature; lock, ^2H signal of the solvent; reference, TMS; spectrum width, 15 kHz; pulse width, 7 μs (~30° flip angle); acquisition time, 0.5 s; number of scans, 1K; computer memory, 16K. Total proton decoupling with ~3 W power; in order to improve the signal-to-noise ratio FID (signal width 1 Hz) was multiplied by Lorentzian exponential. [b] $J(CH_3, CH)$ in Hz. [c] The lines in the septet coalesced. [d] The signal is slightly broadened, thus splitting of signal can only be approximated. [e] $\delta(C{=}O)$ 154.6 ppm.

puter in $CDCl_3$, DMSO-d_6 and toluene-d_8 solutions under dry N_2. Measurements in $CDCl_3$ were carried out at room temperature, whereas those for DMSO-d_6 and toluene-d_8 solutions were carried out in the ranges 303 to 353 K and 187 to 322 K, respectively (Table 1).

X-Ray diffraction studies were carried out with an Enraf–Nonius CAD-4 diffractometer with graphite-monochromated Mo-K_α radiation at room temperature. The structure was solved by direct methods [16]. Hydrogen atoms were located from a difference-electron density synthesis. The structure was refined by complete matrix least-squares. In the last two cycles the spatial coordinates of the hydrogen atoms were also refined. Identical isotropic thermal motion coefficients were refined to hydrogens bonded to the same carbon atom. Absorption correction was not used. The atomic dispersion coefficients were taken from the literature [17]. Crystallographic data and the parameters of the intensity measurements and those of the refinement are listed in Table 3 while atomic coordinates are summarised in Table 4. The tables containing the structure factors and the anisotropic thermal motion coefficients are available on request from the authors.

Results and discussion

NMR spectroscopy

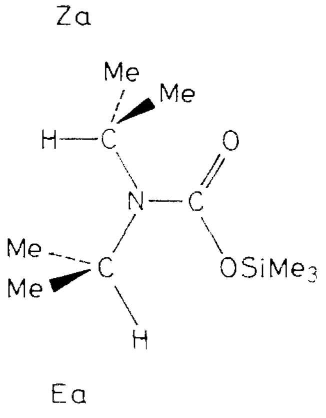
The ^1H chemical shifts of i-PrSiC are listed in Table 1. The separation of the ^1H NMR methine signals indicated that rotation around the N–C amide bond was hindered at room temperature in the solvents used. A gradual increase in temperature (both in DMSO-d_6 and toluene-d_8) led first to coalescence of the two methine signals and then the septet lines became sharper and finally they split. In DMSO-d_6: $T_c = 316$ K, $\Delta\nu = 70$ Hz, $\Delta G^{\ddagger} = 64.3$ kJ/mol. In toluene-d_6: $T_c = 321$ K, $\Delta\nu = 122$ Hz, $\Delta G^{\ddagger} = 63.9$ kJ/mol. The methyl doublet of isopropyl hydrogens was invariably sharp. The rotation barrier of trimethylsilyl *N,N*-dimethyl carbamate ($Me_2NCOOSiMe_3$) was measured under the same conditions. The data in toluene-d_8 ($T_c = 316$ K, $\Delta\nu = 17.4$ Hz, $\Delta G^{\ddagger} = 67.9$ kJ/mol) are consistent with literature data (67.3 kJ/mol in hexane [11]). The two methine signals of i-PrSiC of the same intensity split into a septet in toluene-d_8 at about 277 K, while the sharp C-methyl doublet remained coalesced on account of accidental isochrony. The methyl and methine signals broadened again with a decrease in temperature near to 237 K. The width of the methine proton signals increased to ca. 500 Hz at 217 K. The C-methyl signal also was found to have broadened. Both broad maxima separated into three distinct signals at about 187 K and further doublet and septet splitting was revealed by Lorentz-Gauss resolution enhancement.

The temperature dependence of the NMR spectra can be interpreted in terms of an equilibrium assumed for the two most favourable conformations **A** and **B** of the i-PrSiC molecule, in which the mutual steric hindrance is the least. The interconversion of **A** and **B** can occur in two ways:

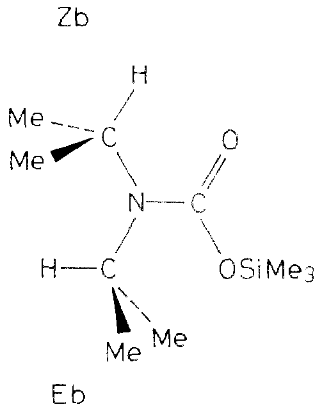
(1) Rotation of 180° around the N–C(sp^2) bond. (Exchange of positions **Za-Eb** and **Ea-Zb**.)

(2) Simultaneous rotation of 180° around the two N–C(sp^3) bonds. (Exchange of positions **Za-Zb** and **Ea-Eb**.)

Rapid rotation (compared to the NMR time scale) can occur around the N–C bonds above 320 K and the four different chemical surroundings of the i-propyl groups (**Za**, **Ab**, **Ea**, **Eb**) in the two conformers become averaged. Thus a doublet



A



B

and a septet, both of which were sharp, were observed. The rotation around the N–C(sp^2) bond was found to slow down markedly between 320 and 220 K while type (2) motion remained rapid. In this case environments **a** and **b** are averaged while those of **Z** and **E** are not. Shielding of the methine proton in the **Z** position decreases due to the anisotropic effect of the near coplanar carbonyl group: its signal is shifted downfield compared with that of the **E** methine proton. Conformers **A** and **B** are present in 1/1 ratio on account of the identical R substituents and of the rapid type (2) interchange. Type (2) rotations slow down completely below 220 K. In this case the four methine septets and the four methyl doublets attributable to the different positions would appear separately. However, the **Za**/**Zb** ratio is not 1/1 because the stability of conformer **A** is not the same as that of **B** but the **Za**/**Zb** = **Ea**/**Eb** = **A**/**B** and **Za** = **Ea** and **Zb** = **Eb** relations do hold for the signal intensities of methine and methyl protons of course. Shielding of the **Za** and **Eb** methine protons is very similar owing to their greater distance from the CO and $OSiMe_3$ groups and consequently the upfield methine septet and methyl doublet pairs are expected to be close to each other, or should even overlap. Thus the spectrum can be assigned unambiguously: CH_3(i-Pr), *d*: 0.74 (**Zb**), 0.80 (**Ea**), 1.22 (**Eb**), and 1.45 ppm (**Za**); CH, *sp*: 2.65 (**Eb** + **Za**), 4.51 (**Ea**) and 4.84 ppm (**Zb**). Conformer ratio **A**/**B** is 1/2 (as measured in the case of methine proton signals), $\Delta G^{\ddagger} = 40$ kJ/mol, $T_c = 217$ K for type (2) rotation.

The free activation energy of the rotation around the N–C(O) bond (Table 2) was found to be smaller in *O*-methyl carbamates than in acetamides owing to the interconjugation (cross-conjugation) [9,10] while the rotation barrier is larger in trimethylsilyl carbamates than in methyl esters. This fact can be explained in terms of expanded conjugation which also involves the *d*-orbital of silicon [11].

In addition to the limiting structures I and II which are characteristic of amide mesomery, structure III must also be considered in terms of methyl carbamates. Consequently the statistic weight of structure II and thus the energy barrier is smaller. In the case of trimethylsilyl carbamates the limiting structures IV and V must also be considered. The effect of structure III suppressing structure II is

Table 2

The free energy of activation $\Delta G^{\ddagger}$ of the rotation around the N–C(O) bond in some of the $R_2N–CO–R'$ molecules

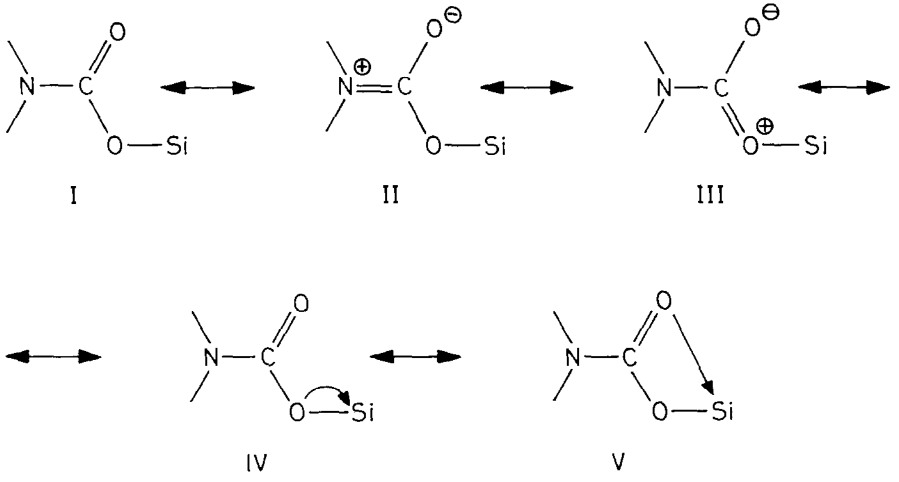
Molecule	$\Delta G^{\ddagger}$ (kJ/mol)		T_c (K)	Solvent	References
	N–C(sp^2)	N–C(sp^3)			
R = R' = Me	72.5		298	CCl_4	18, 19
R = Me; R' = $OSiMe_3$	67.9		316	toluene-d_8	
	67.5		298.2	n-hexane	11
R = Me; R' = OMe	65.8		198.2	$CDCl_3$	12
R = i-Pr; R' = Me	75.4 [a]	50.3	293	$CDCl_3/CDCl_2F$	18/13
R = i-Pr; R' = $OSiMe_3$	64.1	40.2	321	toluene-d_8	
	64.5		316	DMSO-d_6	
R = i-Pr; R' = OMe	51.1	39.8	> 248	$CDCl_2F$	14

[a] From $\Delta S^{\ddagger}$: -4.3 eu, $\Delta H^{\ddagger} = 16.7$ kcal/mol.

overcompensated by those of structures IV and V and so the energy barrier is increased.

The rotation barrier was found to vary in a wide range when R = i-Pr than when R = Me. This is due in part to the greater steric demand by isopropyl group, and also to the steric interaction of methine protons and carbonyl group. Such conformers of diisopropyl acetamide where the methine protons are not in the plane of the molecule are also stable [13] whereas in the case of diisopropyl methyl carbamate only two conformers are possible [14] which correspond structurally conformers **A** and **B** of i-PrSiC. At low temperature, the conformer ratio **A**/**B** was found to be 31/69 [14], one which is very close to our observed **A**/**B** ratio of 1/2.

It is apparent from Fig. 1, which depicts the chemical shift of the methyne protons versus temperature, that the shifts of the overlapping signals at a temperature above that of coalescence are not exactly the mathematical average of the separate signals as expected. This points to the fact that conformation- and



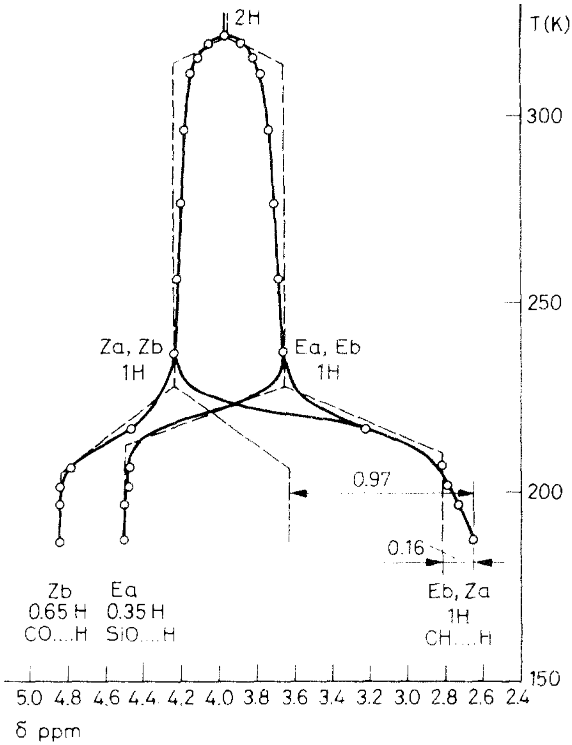


Fig. 1. Temperature dependent ^1H NMR chemical shifts of the methine protons of i-PrSiC. ——— measured data; - - - - - - expected shifts.

temperature-dependent specific solvent–solute interaction of carbamates occurs, a phenomenon shared with the amides [20]. The shielding of methine protons via ASIS [21] is increased mainly in the **Za** position and to a smaller extent in the **Eb** position [20]. (The differences between observed and calculated chemical shifts are 0.97 ppm for the **Za** and 0.16 ppm for the **Eb** positions). This 0.2 to 1.0 ppm effect can be used to find out whether there is interaction between carbamate and various aromatic solvents.

X-Ray diffraction study

The diagram of the i-PrSiC molecule is shown in Fig. 2, and selected bond lengths and bond angles are listed in Table 5.

H(1a), C(1), O(1), O(2), C(3), N, C(4), H(4), C(6), and H(6) atoms are in the mirror plane in $z = 1/4$. In spite of its apparent flexibility the molecule crystallized as a mirror symmetric conformer (C_s symmetry). This indicates that the symmetry attained by the atoms is the result of several interactions.

The atoms of the carbamate group O(1), O(2), C(3), N are arranged in a plane due to cross conjugation (cf. bond lengths N–C(3), C(3)–O(2) and C(3)–O(1) in Table 5). The O(1)–Si bond is also in the plane of the molecule owing to the weak conjugation by Si and O(1) atoms. The Si–O(1) bond length is 1.68 Å which indicates multiple bond character viz. a $(p-d)_\pi$ bond. The calculated O–Si single

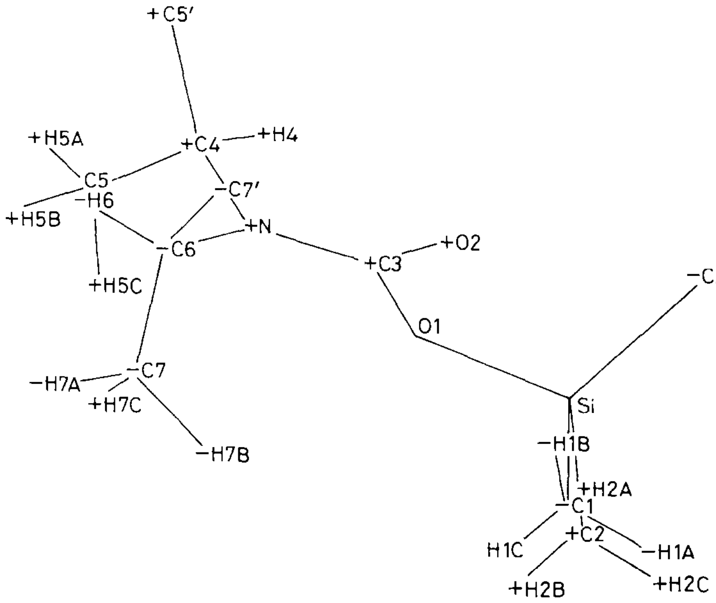


Fig. 2. Structure of i-PrSiC in crystalline phase.

bond length is 1.83 Å, while in siloxanes having strong π-bond character it ranges from 1.61 to 1.68 Å. The relatively large C(3)–O(1)–Si bond angle (122.1°) suggests that the electrons of carbamate group extend to the Si atom through O(1). The

Table 3

Crystallographic data, data collection parameters and least-squares data

Empirical formula	$C_{10}H_{23}NO_2Si$
M (amu)	217
a (Å)	11.646(4)
b (Å)	10.984(2)
c (Å)	10.861(2)
V (Å)	1389.3(1.0)
Space group (from extinction and refinement data)	*Pnam*
Z	4
D_c (g cm^{-3})	1.039
λ(Mo-$K_{\bar{\alpha}}$) (Å)	0.71073
μ(Mo-$K_{\bar{\alpha}}$) (cm^{-1})	1.45
2θ limits (°)	3–70
Scan technique	θ–2θ
Number of non-zero intensity reflections	1788
Number of reflections used for refinement (NO)	1486 [a]
Number of variables (NV)	119
R_0	0.040
R_w	0.053
R_{tot}	0.050
$[\Sigma w(\lvert F_o\rvert - \lvert F_c\rvert)^2/(NO-NV)]^{1/2}$	3.41
Size of crystal (mm)	0.05 × 0.35 × 0.25

[a] $[F_o^2 \geqslant 3.0\sigma(F_o^2)]$.

Table 4

Atomic coordinates ($\times 10^4$) are referred to hydrogen atoms ($\times 10^3$), B_{eq} are referred to hydrogens (Å) and C · · · H distances ($\times 10^2$ Å) are with e.s.d. [a]

Atom	*x/a*	*y/b*	*z/c*	B_{eq}
Si	9442.6(4)	3506.5(5)	1/4	4.29(2)
O(1)	8460(1)	4626(1)	1/4	5.10(6)
O(2)	9768(1)	6119(1)	1/4	5.55(7)
N	7860(1)	6577(1)	1/4	4.17(6)
C(1)	8531(2)	2133(2)	1/4	7.15(1)
C(2)	10326(1)	3552(2)	3902(2)	6.20(8)
C(3)	8759(2)	5814(2)	1/4	4.24(8)
C(4)	8107(2)	7894(2)	1/4	5.16(9)
C(5)	7669(2)	8504(2)	3649(2)	7.00(9)
C(6)	6644(2)	6196(2)	1/4	4.32(8)
C(7)	6302(2)	5523(2)	3659(2)	6.77(9)

Atom	*x/a*	*y/b*	*z/c*	*B*	C–H
H(1a)	894(3)	134(3)	1/4	11.5(6)	99(3)
H(1b)	808(1)	217(2)	181(2)	11.5(6)	92(2)
H(2a)	1077(1)	428(2)	392(2)	9.5(3)	95(2)
H(2b)	993(1)	360(2)	461(2)	9.5(3)	90(2)
H(2c)	1072(1)	282(2)	394(2)	9.5(3)	93(2)
H(4)	893(2)	797(2)	1/4	5.6(3)	96(2)
H(5a)	788(2)	944(2)	365(2)	10.8(6)	106(2)
H(5b)	685(1)	843(2)	367(2)	10.8(6)	96(1)
H(5c)	799(1)	805(2)	437(2)	10.8(6)	100(2)
H(6)	619(2)	697(2)	1/4	4.6(3)	100(2)
H(7a)	547(1)	545(2)	370(2)	10.7(6)	97(2)
H(7b)	665(1)	474(2)	369(2)	10.7(6)	95(2)
H(7c)	652(1)	603(2)	434(2)	10.7(6)	96(2)

[a] Definition of B_{eq}: 4/3 × trace (*BG*); *B* is the tensor of thermal motion and *G* the direct metric tensor.

dihedral angle of Si–O(1)–C(3)–O(2) (*cis* position) is 0 ° which indicates that there is a probable weak donor–acceptor interaction between the Si and O(2) atoms. This assumption is corraborated by the fact that the Si · · · O(2) distance (2.894 Å) is

Table 5

Bond lengths (Å) and bond angles (°) in i-PrSiC

Si–O(1)	1.680(1)	O(1)–C(3)	1.351(2)	N–C(4)	1.475(2)
Si–C(1)	1.845(2)	O(2)–C(3)	1.222(3)	N–C(6)	1.477(3)
Si–C(2)	1.838(2)	N–C(3)	1.341(3)	C(4)–C(5)	1.505(3)
				C(6)–C(7)	1.513(2)
O(1)–Si–C(1)	101.9(1)	C(4)–N–C(6)	117.7(2)		
O(1)–Si–C(2)	111.2(1)	O(1)–C(3)–O(2)	120.9(3)		
C(1)–Si–C(2)	110.1(1)	O(1)–C(3)–N	113.7(2)		
C(2)–Si–C(2′) [a]	111.8(2)	O(2)–C(3)–N	125.4(3)		
Si–O(1)–C(3)	122.1(2)	N–C(4)–C(5)	111.7(3)		
C(3)–N–C(4)	117.4(2)	C(5)–C(4)–C(5′)	112.0(3)		
C(3)–N–C(6)	124.9(2)	N–C(6)–C(7)	113.0(2)		
C(7)–C(6)–C(7′)	112.6(3)				

[a] The atom coordinates of C(2′), C(5′) and C(7′) are *x*, *y*, 1/2 − *z*.

considerably shorter than the sum of the van der Waals radii of the two atoms (3.4 Å).

The trigonal pyramidal configuration of the $SiMe_3$ group is distorted by the effect of the O(2) atom. The C(2) and C(2′) atoms have mirror symmetry at the plane flanking the O(2) atom. Significant deformation is observed in the angles related to the O(1)–Si bond, indicating that the *Si*-methyl groups have moved to the side opposite that of O(2) atom. Thus O(1), C(2) and C(2′) atoms have a *quasi-equatorial* position while C(1) and O(2) are *quasi-axial* and Si–C(1) bond is slightly elongated (1.845 Å), compared with the Si–C(2) bond (1.838 Å). Finally, the interaction between Si–O(2) is also suggested by the magnitude of the O(1)–C(3)–N and O(2)–C(3)–N angles indicating that the O(2)–C(3) bond bends towards the silicon atom.

NMR spectroscopy showed the ratio of the two stable conformers (**A** and **B**) to be 1/2 in solution. The overwhelming predominance in the solid phase compared to that of conformer **A** existing only in solution may be explained in terms of the existence of a weak C–H · · · O hydrogen bond. The latter is also indicated by the relatively small H(4) · · · C(2) distance as well as by the difference in the angles C(3)–N–C(6) and C(3)–N–C(4). The fact that the latter angle is considerably smaller suggests that the isopropyl group is *cis* relative to the carbonyl group, and turned towards the carbonyl oxygen. The latter interaction is maximal if H(4) lies in the mirror plane.

A comparison of the geometrical data of the $Me_3SiOCON$ group in i-PrSiC and those of the *N*,*O*-di-trimethylsilyl carbamate [22] and of trimethylsilyl-*N*-phenyl

Table 6

Comparison of the geometrical data of i-PrSiC, *N*,*O*-di-SiC and *N*-ph-SiC [a]

	i-PrSiC	*N*,*O*-di-SiC	*N*-ph-SiC	Difference
Bond lengths (Å)				
Si–O(1)	1.680	1.685	1.707	0.027
O(1)–C(3)	1.351	1.345	1.340	0.011
O(2)–C(3)	1.222	1.213	1.226	0.013
N–C(3)	1.341	1.340	1.32?	0.02?
Si–C(Me)	1.838	1.821	1.852	0.031
	1.838	1.823	1.855	0.032
	1.845	1.844	1.860	0.016
Si · · · O(2)	2.895	2.981	2.93	0.086
Bond angles (°)				
O(1)–C(3)–N	113.7	111.3	112.3	2.4
O(2)–C(3)–N	125.4	126.8	126.3	1.4
O(1)–C(3)–O(2)	120.9	121.9	121.4	1.0
Si–O(1)–C(3)	122.1	125.4	117.3	8.1
O(1)–Si–C(Me)	101.9	–	103.9	2.0
	111.2	–	108.1	3.1
	111.2	–	109.6	1.6
C(Me)–Si–C(Me)	111.8	–	113.2	1.4
	110.1	–	110.4	0.3
	110.1	–	111.1	1.0

[a] i-PrSiC = trimethylsilyl-*N*,*N*-diisopropyl carbamate; *N*,*O*-di-SiC = *N*,*O*-di(trimethylsilyl) carbamate; *N*-ph-SiC = trimethylsilyl-*N*-phenyl carbamate.

carbamate [23] is to be found in Table 6. Various substituents bound to the nitrogen only slightly affect the structure of $Me_3SiOCON$ group only. The substituent effect is mainly observed in the Si · · · O(2) distance (0.086 Å). The change in the Si–C(methyl) bond length is smaller (0.032 Å) while the Si–O(1) bond length decreases by 0.027 Å only, and that of O(1)–C(3) is affected the least (0.011 Å). The bond angles vary thus: Si–O(1)–C(3) is affected to the greatest extent (8.1°), followed by the O(1)–C(3)–N angle (2.4°), while relatively large differences are to be found in the O(1)–Si–C(methyl) angles (2 to 3.1°). The other angles vary by 1 to 1.5° only. The trimethylsilyl group was found to consist of two long, and one short, Si–C(methyl) bonds, for which there are one smaller and two larger O(1)–Si–C(Me) bond angles, while there are two C(Me)–Si–C(Me) bond angles are larger and one is smaller. The bending of the silyl groups towards the trigonal bipyramidal structure is probably due to the effect of the neighbouring O(2) atom.

Thus we conclude that a weak interaction between O(2) · · · Si atoms is encountered in all cases although cross-conjugation in trimethylsilyl carbamates is mainly extended to the Si atom through the O(1)–Si bond.

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