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14. ABSTRACT

Trimethyl(trifluoromethyl)silane, (CH3)3SiCF3, has been in situ pressure frozen in a diamond anvil cell and its structure determined at 0.90(5) GPa/296 K by single-crystal X-ray diffraction. The crystal is monoclinic, space group P21/m, with the molecules lying on crystallographic mirror planes. The CH3 and CF3 groups assume the fully staggered conformation. The 14-fold coordination scheme of the molecules is similar to those in (CH3)3SiCl polymorphs, but different from that in crystalline tetramethylsilane, (CH3)4Si (TMS). The longest Si–C bond length of 1.943(12)A˚ observed in the crystal structure has been documented. The shortest intermolecular contacts in the structure of pressure-frozen CF3Si(CH3)3 are observed between the hydrogen atoms, and those involving fluorine atoms are longer than sums of van der Waals' radii. These structural features explain the facile cleavage of Si–CF3 bond for CF3 group transfer in organic reactions.

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High-pressure freezing and crystal structure studies of $TMSCF_3$: Understanding nucleophillic -CF_3 transferring ability (Preprint)

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Abstract

Trimethyl(trifluoromethyl)silane, $(CH_3)_3SiCF_3$, has been *in-situ* pressure-frozen in a diamond anvil cell and its structure determined at 0.9 GPa/296 K by single-crystal X-ray diffraction. The crystal is monoclinic, space group $P2_1/m$ with molecules lying on the crystallographic mirror plane. The CH_3 and CF_3 groups assume the fully staggered conformation. The 14-fold coordination scheme of the molecules is similar to those in $(CH_3)_3SiCl$ polymorphs, but different from that in crystalline tetramethylsilane $(CH_3)_4Si$ (TMS). The Si-CF_3 bond is slightly longer than Si-CH_3 bond and the shortest intermolecular contacts in the structure of pressure-frozen $CF_3Si(CH_3)_3$ are observed between fluorine and hydrogen atoms. These structural features explain the facile cleavage if Si-CF_3 bond for CF_3 group transfer in organic reactions.

Introduction

Silanes are important chemical compounds, owing to their physical and chemical properties. They are widely applied, for example: in dentistry, medicine, construction technologies, synthesis, as adhesion promoters, coupling, cross-linking or dispersing agents and surface modifiers.[1] Trimethyl(trifluoromethyl)silane can be considered as a derivative of tetramethylsilane (TMS) where three hydrogen atoms of a methyl group are replaced by fluorine atoms. Generally, this kind of substitution changes physical and chemical properties of compounds considerably as a result of intermolecular interaction. Consequently, fluorinated compounds have markedly different properties than their non-fluorinated counterparts and thus find wide ranging applications from medicine to technology.[2] Trimethyl(trifluoromethyl)silane, first synthesized in 1984 by Ruppert[3] and commonly known as Ruppert's reagent, is used for syntheses of perfluorinated compounds and for introducing of a trifluoromethyl group by nucleophillic reaction.[4-8] For these reasons the structural

information about $(CH_3)_3SiCF_3$ are of particular interests and studies by gas phase electron diffraction, microwave spectroscopy and quantum chemical calculations were performed.[9] The principal interest for these studies focused on the difference between Si–CF_3 and Si–CH_3 bond lengths and in the intermolecular interactions. Vibrational-spectroscopy studies on CF_3-silanes showed that the substitution of H-atoms with F-atoms lengthens and weakens the Si–CF_3 bond,[10,11] which results from the electronegative F-atoms withdrawing the electrons from C and Si-atoms, causing electrostatic repulsion of their positive net-atomic charges, and a deficiency in the binding σ-electron pair. However, to the best of our knowledge, no solid state/crystal structure of $(CH_3)_3SiCF_3$ as a result of x-ray or neutron powder diffraction results has been reported so far. It was also established most recently, that the $Si(CH_3)_4$ (*i.e.* tetramethylsilane, TMS) molecules in the crystalline state are distorted from the fully-staggered conformation.[12] That result is consistent with previous electron-diffraction measurements for TMS vapor, and it was proposed that the twisted TMS conformation releases the intramolecular strains of interacting H-atoms.[13] Thus the aim of this study was to provide the molecular dimensions and conformation of $(CH_3)_3SiCF_3$, and also to investigate the influence of the fluorine atoms and the molecule polarization for the intermolecular interactions and crystal packing. Further, it is also important to understand why this molecule does not spontaneously decompose to form a more stable molecule, $(CH_3)_3SiF$, with an explosive formation of difluorocarbene.

Experimental

Trimethyl(trifluoromethyl)silane has been crystallized in a modified Merrill-Bassett[14] diamond anvil cell (DAC). Because of the relatively low boiling point (327-328 K),[15] the liquid and the DAC were cooled with solid CO_2 for loading the sample. It crystallized at 0.2 GPa and then the high-pressure chamber was squeezed till its whole chamber volume was filled by the polycrystalline sample. To ensure stable conditions for the diffraction measurements, pressure was increased to 0.9 GPa, and then the DAC was heated. A single crystal was grown isohorically from one seed crystal left in the melt sample at 473 K, followed by slowly cooling the sample to 296 K. During the first stage of cooling many defects appeared on the crystal surface (Fig. 1 a-b). Therefore, the crystal was kept at 333 K for 2 hours until the process of self-healing eliminated all defects observed in crystal morphology (Fig. 1c-e). After cooling the DAC to room temperature five days passed before the diffraction data were measured.

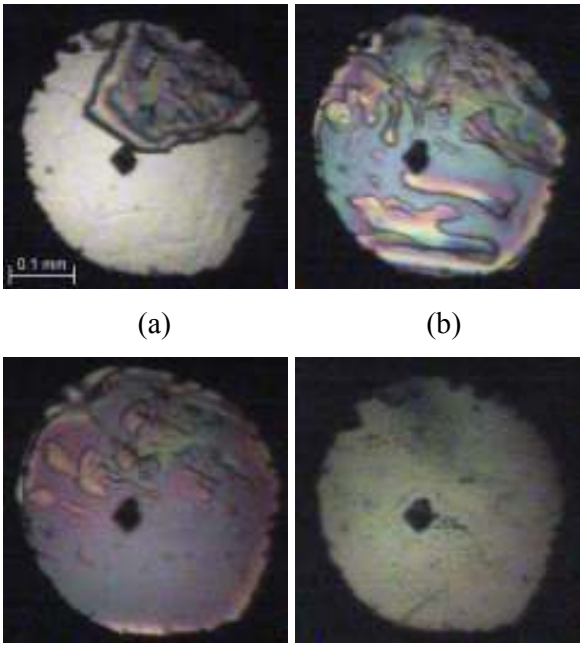


Figure 1. Isochoric-growth stages of the $(CH_3)_3SiCF_3$ single crystal: (a) distorted morphology of a small single crystal at 453 K; (b) the crystal at 333 K with its faces highly irregular; (c) the same crystal after 1 hour at 333 K; and (d) the crystal at room temperature and 0.9 GPa. The small ruby chip for pressure calibration is placed close to the centre of the DAC.

Pressure in the DAC was calibrated by ruby-fluorescence method,[16,17] using a Betsa PRL spectrometer, with an accuracy of 0.05 GPa. The single-crystal X-ray diffraction study was carried out with a KUMA KM4-CCD diffractometer. The CrysAlis version 1.171.24 software[18] was used for the data collections[19] and the preliminary reduction of the data. After correcting the intensities for the effects of DAC absorption, sample shadowing by the gasket, and the sample absorption,[20,21] the diamond reflections were eliminated. The systematic absences and statistics of reflections intensities indicated that the crystal is monoclinic in space group $P2_1/m$. The structure was solved straightforwardly by direct methods,[22] and refined by full-matrix least-squares.[23] Anisotropic temperature factors were applied for all non-hydrogen atoms. The crystal data and the structure refinement details are listed in Table 1. Structural drawings were prepared using the X-Seed interface of POV-Ray.[24,25]

Table 1. Selected crystal data and details of structure refinement for $(CH_3)_3SiCF_3$.

Formula		$(CH_3)_3SiCF_3$
Pressure (GPa)		0.90(5)
Temperature (K)		296(2)
Formula weight		142.20
Wavelength (Å)		0.71073
Crystal system		Monoclinic
Space group		$P2_1/m$
Unit cell dimensions (Å,°)	a=	5.9737(12)
	b=	9.4228(19)
	c=	6.3231(13)
	β=	105.30(3)
Volume ($Å^3$)		343.31(12)
Z		2
Calculated density (g/cm^3)		1.376
Absorption coefficient (mm^{-1})		0.301
F(000)		148
Crystal diameter/height (mm)		0.38/0.20
θ-range for data collection (°)		3.98 to 29.43
Min./max. indices h, k, l		-5/5, -12/12, -6/6
Reflect. collected/ unique (R_{int})		2543/378 (0.0733)
Completeness (%) (to θ_{max})		37.5 (to 29.43°)
Refinement method:		Full-matrix least-squares on F^2
Data/restrains/parameters		378/2/48
Goodness-of-fit on F^2		1.092
Final R_1/wR_2 ($I>2\sigma_1 I$)		0.0600/0.1391
R_1/wR_2 (all data)		0.0709/0.1464
Weighting scheme		w=1/($\sigma^2(F_o^2)$+(0.0724*P)2+0.17*P), where P=(Max(F_o2,0)+2*F_c2)/3
Largest diff. peak and hole ($e.Å^{-3}$)		0.440/-0.380

Results and discussion

Molecular geometry

In the pressure-frozen $(CH_3)_3SiCF_3$ structure atoms Si1, C1, C2, F2 and H2 of the molecule are located on a crystallographic mirror plane of space group $P2_1/m$ (Figure 2).

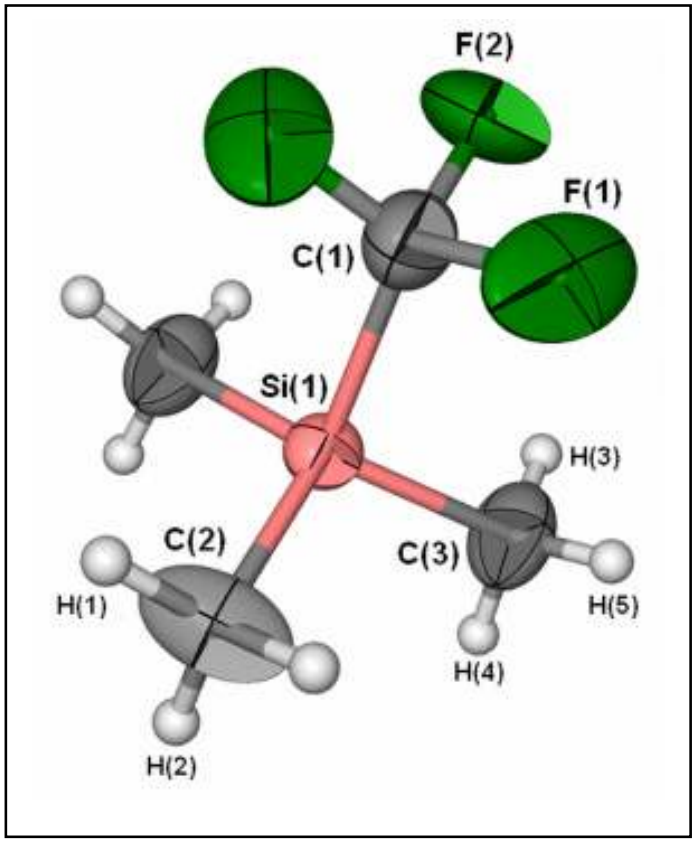


Figure 2. The molecule of $(CH_3)_3SiCF_3$ at 0.9 GPa and 296 K. Only the symmetry-independent atoms have been labeled in this drawing.

The Si-atom is in a tetrahedral environment and is bonded to one CF_3 group and three CH_3 groups. However, the C–Si–C angles are significantly distorted from the ideal tetrahedral value, and range from 102.1(7)° to 113.9(3)° (Table 2). It is noteworthy, that the valency angles C-Si-C involving atom $C(1)F_3$ are considerably smaller than the C-Si-C valency angles between the methyl groups, which rules out the possibility of intramolecular steric hindrances between F and H atoms. The lengths of three $Si–CH_3$ bonds (two of which are symmetry independent) are consistent within errors. The methyl group (C2) lying on the mirror plane is slightly longer at 1.862(11) Å compared to the one with C3 located at a general position 1.848 (6), whereas the $Si–CF_3$ bond is by nearly 0.1 Å longer at 1.943(12) Å. All these values agree

well with the electron diffraction in the gaseous phase and by theoretical calculations for the isolated molecule.[9] The largest differences between calculated and experimental dimensions are in the C–F bonds lengths and in CF_3–Si–CF_3 and F–C–F angles (Table 2). As predicted, the Si–C bond length depends on its intramolecular environment, and in the X–Si–$(CH_3)_3$ molecules (X stands for H, Cl, F, I, CH_3, CF_3) it shortens with increasing electronegativity of the X-group.[9] The theoretical studies on structures of some silanes showed that this is $(CH_3)_3SiCF_3$ where Si–C bond is the longest.[9] This is consistent with the tendency of $(CH_3)_3SiCF_3$ for the -CF_3 group donation.

The molecular conformation of $(CH_3)_3SiCF_3$ was also investigated theoretically and the potential energy barrier hindering the internal rotation of all methyl groups was calculated to be 5.71 kJ/mol.[9] The staggered positions for the CH_3 and CF_3-groups were chosen for optimizing geometry in quantum chemical calculations. In the crystal structure the ideal staggered conformation is imposed by symmetry for groups $C(1)F_3$ and $C(2)H_3$. However, the $C(3)H_3$ methyl groups are not restricted by symmetry to rotate, and a deviation of 8(4)° from the fully staggered conformation has been observed for $C(3)H_3$. This deviation of $C(3)H_3$ is small (and statistically insignificant) and may also result from the distorted tetrahedral geometry of the $(CH_3)_3SiCF_3$ molecule and its intermolecular interactions.

Table 2. Bond lengths (Å) and angles (°) for $(CH_3)_3SiCF_3$ determined by electron diffraction[9] and in this study (symmetry codes: i = *x* ,-*y*+½ ,*z*)

	Electron diffraction[9]	X-ray diffraction
Si–CH_3	1.862(3)	1.848(6) Si(1)-C(3) 1.862(11) Si(1)-C(2)
Si–CF_3	1.942(5)	1.943(12) Si(1)-C(1)
C–F	1.355(2)	1.296(9) C(1)-F(1) 1.403(9) C(1)-F(2)
CH_3–Si–CH_3[i]	Not given	111.1(5) C(3)-Si(1)-C(3)[i] 113.9(3) C(3)-Si(1)-C(2)
CF_3–Si–CH_3[i]	106.0(5)	102.1(7) C(1)-Si(1)-C(2) 107.5(3) C(1)-Si(1)-C(3)
F–C–F[i]	106.5(2)	111.8(12) F(1)-C(1)-F(1)[i] 103.6(4) F(1)-C(1)-F(2)

Intermolecular interactions and molecular packing

Despite high-pressure conditions, almost all intermolecular distances in the structure of $(CH_3)_3SiCF_3$ are longer than sums of tabulated values of van der Waals radii of the closest atoms[26]. The shortest distances between F-atoms are of 3.014(7) Å, the shortest F···H contacts are of 2.798 Å, and 2.348 Å between the closest H-atoms. The shortest H···H contacts arrange molecules into [100] chains and the molecules with the shortest F···F and F···H distances run along the [010] direction. All these contacts arrange the molecules into sheets along planes (100) in the crystal lattice (Fig. 3).

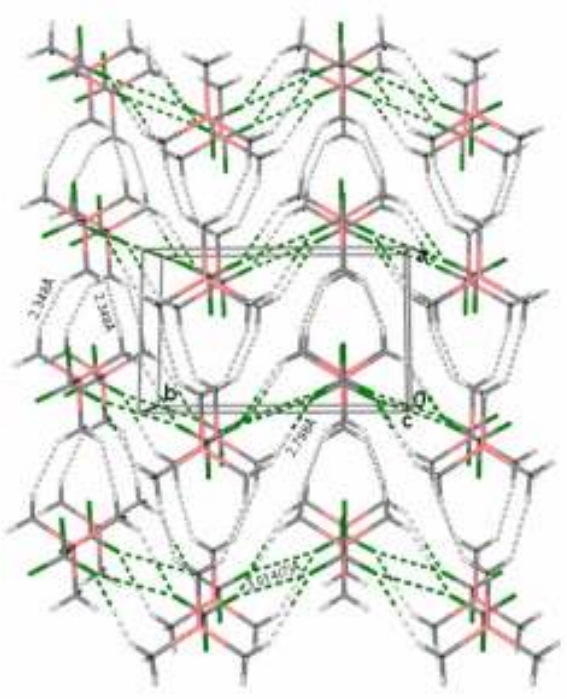


Figure 3. The molecular packing of $(CH_3)_3SiCF_3$ with the shortest H···H, F···H and F···F contacts indicated as dashed lines.

The regions and directions of intermolecular contacts in $(CH_3)_3SiCF_3$ are mapped onto the molecular Hirshfeld surface[27,28,29] in Fig. 4. It confirms that the shortest contacts in this structure involve the hydrogen atoms (red spots on the Hirshfeld surfaces), and that each CH_3 group fits between two other CH_3 groups of the neighboring molecules. Each molecule has four closest neighbors forming contacts H···H and H···F.

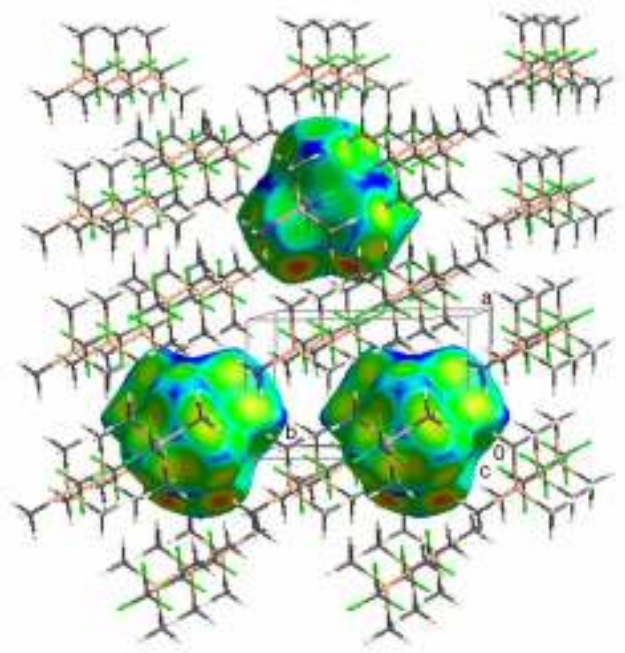
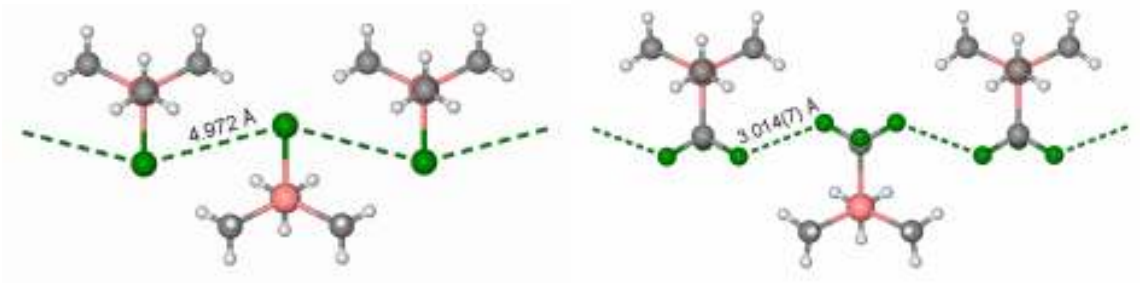


Figure 4. The intermolecular interactions represented by a color scale on the Hirshfeld surface.[27,28,29] The property mapped onto the surface is the normalized contact distance (intermolecular contacts closer than the sum of their van der Waals radii are highlighted in red on the d_{norm} surface, longer contacts are blue, and contacts around this sum are white).

With respect to the distances to the molecular centers (associated with the Si atom in this discussion), the crystal structure corresponds to the close-packing arrangement of ideal spheres, with the coordination number of 14. The Si···Si distances are 5.8896(18), 5.8896(18), 5.9737(12), 5.9737(12), 6.216(2), 6.216(2), 6.3231(13), 6.3231(13), 6.336(4), 6.336(4), 7.288(5), 7.288(5), 7.466(3) and 7.466(3) Å within this 14-fold coordination scheme. The 12-fold coordination number was indicated as favored for molecular crystals by Kitajgorodski,[30] but more recent survey based on the Cambridge Crystallographic Database revealed that the 14-fold coordination may be more frequent for molecular crystals.[31]

The $(CH_3)_3SiCF_3$ crystal is isostructural with low-temperature $(CH_3)_3SiCl$ α-phase[32] as both these structures are monoclinic, space group $P2_1/m$. The largest differences in unit-cell dimensions are, for the b parameter 1.69 Å longer and the β angle is 14.5° larger in $(CH_3)_3SiCF_3$ than in $(CH_3)_3SiCl$, the molecular packing is similar. Gajda *et al.*[33] established, that in low-temperature phase α and high-pressure phase β of $(CH_3)_3SiCl$ the molecules are arranged head to tail in the manner avoiding short Cl···Cl contacts. Likewise, there are no short contacts between CF_3 groups in the high-pressure $(CH_3)_3SiCF_3$ phase. The patterns of

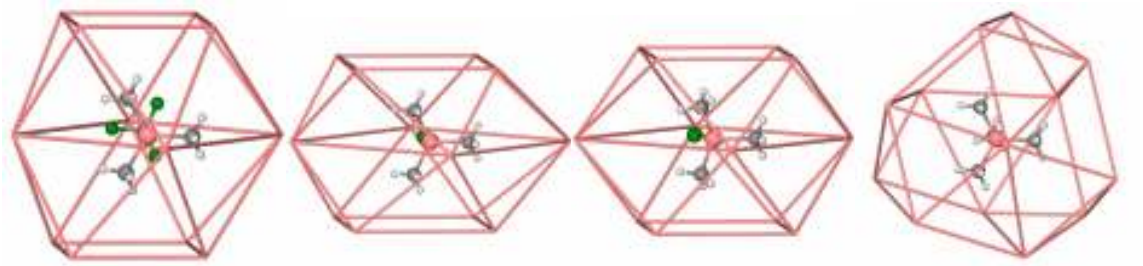
these molecules in $(CH_3)_3SiCl$ and in $(CH_3)_3SiCF_3$ crystals, where Cl···Cl and F···F contacts are the shortest, are presented in Figure 5. Owing to the molecular geometry intermolecular contacts of the F-atoms are closer and those of the Cl-atoms are more distant in $(CH_3)_3SiCF_3$ and $(CH_3)SiCl$, respectively. In this respect the behavior of the halogens, or their role in the formation of the crystal structures can be described as halophobic, in accordance with the observation of other structures of halogenated compounds by Grineva & Zorkii.[34,35]



(a) (b)

Figure 5. Patterns of molecules with the shortest Cl···Cl distance in $(CH_3)_3SiCl$–phase α (a), and the shortest F···F distance in $(CH_3)_3SiCF_3$ (b).

These repulsive halogen···halogen interactions are reflected in the shapes of coordination schemes presented in Figure 6. The molecules of $(CH_3)_3SiCF_3$ and $(CH_3)_3SiCl$ in phases α and β are similarly 14-fold coordinated. Despite similar molecular shape, the $(CH_3)_3SiCF_3$ structure is considerably different from that of $(CH_3)_4Si$, which is cubic, space group $Pa\bar{3}$, with the molecules located on C_3 axes and 13-fold coordinated[12]. The common feature of all the silanes investigated is weak intermolecular interactions.



(a) (b) (c) (d)

Figure 6. The coordination schemes of $(CH_3)_3SiCF_3$ (a); $(CH_3)_3SiCl$, phase α (b); $(CH_3)_3SiCl$, phase β (c); and $(CH_3)_4Si$ (d). The vertices of the coordination schemes represent the Si atoms closer than 8 Å to the central Si atom.

Conclusions

This study on the crystal structure of trimethyl(trifluoromethyl)silane confirmed that the Si–CF_3 bond is longer and weaker than the Si–CH_3 bonds. It has been confirmed that there are no strong intermolecular interactions in $(CH_3)_3SiCF_3$. This result supports the trifluoromethylating nature of this reagent as the CF_3 group is easily transferred without decomposing to form $(CH_3)_3SiF$ and $:CF_2$ (difluorocarbene Also, the structure of $(CH_3)_3SiCF_3$ isostructural with $(CH_3)SiCl$ in α-phase seems to confirm that the H···H interactions and the similar molecular shape are essential facts for the molecular coordination scheme, similar to those in both α- and β-phases of $(CH_3)_3SiCl$. The different coordination scheme in $(CH_3)_4Si$ may be due to the presence of an additional methyl group in this molecule. However, the common feature of all of the silanes investigated is the absence of strong intermolecular interactions.[12,32,33,36]

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