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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, crystal structure studies and Si–CF_3 bond polarizability of trimethyl(trifluoromethyl)silane

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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 0.90(5) GPa/296 K by single-crystal X-ray diffraction. The crystal is monoclinic, space group $P2_1/m$, with the molecules lying on crystallographic mirror planes. 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 longest Si–C bond length of 1.943(12) Å observed in the crystal structure has been documented. The shortest intermolecular contacts in the structure of pressure-frozen $CF_3Si(CH_3)_3$ 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–CF_3 bond for CF_3 group transfer in organic reactions.

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1. 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, fluorination strongly changes intermolecular interactions and often desired physical and chemical properties for practical applications can be obtained [2]. Trimethyl(trifluoromethyl)silane, first synthesized in 1984 by Ruppert et al. [3] is commonly known as Ruppert's reagent. It is used in the syntheses of perfluorinated compounds and for introducing a trifluoromethyl group by nucleophilic substitution [4–7]. 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 [8]. The principal interest for these studies focused on the differences between Si–CF_3 and Si–CH_3 groups dimensions and their 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 [9,10]. It is caused by the electronegative F-atoms withdrawing the electrons from C and Si-atoms, which results in 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 $(CH_3)_3SiCF_3$ determined by X-ray or neutron powder diffraction has been reported so far. It was also established most recently, that the $Si(CH_3)_4$ (i.e. TMS) molecules in the crystalline state are distorted from the fully staggered conformation [11]. That result is consistent with the previous observation by electron-diffraction measurements for TMS vapor, and it was proposed that the twisted TMS conformation releases the intramolecular strains generated by interactions between H-atoms [12]. Thus the aim of this study was to provide the dimensions and conformation of $(CH_3)_3SiCF_3$ molecule embedded in the crystal structure, and also to investigate the influence of the fluorine atoms and the molecule polarization for the intermolecular interactions and crystal packing. All this information is essential to understand why this molecule does not spontaneously decompose to form a more stable molecule, $(CH_3)_3SiF$, with an explosive formation of difluorocarbene.

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2. Results and discussion

2.1. Molecular structure

In the pressure-frozen $(CH_3)_3SiCF_3$ structure, atoms Si(1), C(1), C(2), F(2) and H(2) of the molecule are located on a crystallographic mirror plane of space group $P2_1/m$ (Fig. 1).

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 1). 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. Thus the Si–$C(1)F_3$ bond lengthening is purely due to the effect of the grouped highly electronegative fluorine atoms on electronic structure of the molecule. The lengths of three Si–CH_3 bonds (two of which are symmetry independent) are consistent within errors. The Si–$C(2)H_3$ bond to methyl group lying on the mirror plane is insignificantly longer than this to C(3) located in a general position (Table 1), whereas the Si–$C(1)F_3$ bond is by nearly 0.1 Å longer. This C–Si bond is significantly longer than any of Si–C bond length listed in the tables of molecular dimensions, even if the maximum values of Si–C in database are considered [13]. All these values agree well with the results of electron-diffraction studies in the gaseous phase and theoretical calculations for the isolated molecule [8]. The

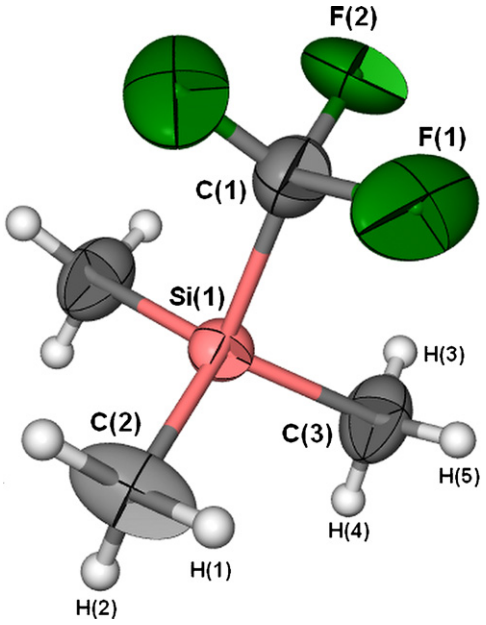

Fig. 1. The molecule of $(CH_3)_3SiCF_3$ at 0.90 GPa and 296 K, with displacement ellipsoids at 50% probability. Only the symmetry-independent atoms have been labeled in this drawing.

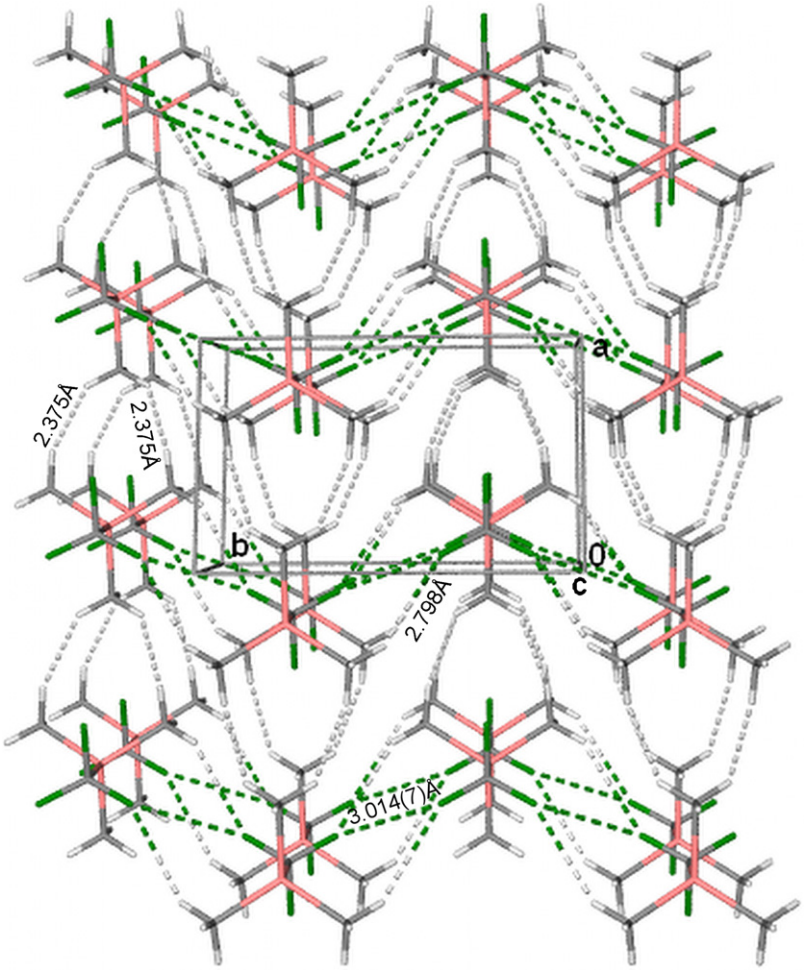

Fig. 2. The molecular packing of $(CH_3)_3SiCF_3$ with the shortest H···H, F···H and F···F contacts indicated as dashed lines.

Table 1
Bond lengths (Å) and angles (°) for $(CH_3)_3SiCF_3$ determined from single-crystal X-ray diffraction study, compared to those obtained by gas–electron diffraction (GED) averaged with microwave spectroscopy (MW), and computed by the DFT method at B3LYP/6-311++G(2df,pd) level of theory [8]

	X-ray at 0.90 GPa	GED/MW[a]	DFT[a]
Si(1)–C(3)	1.848(6)	1.862(2)	1.872
Si(1)–C(2)	1.862(11)		
Si(1)–C(1)	1.943(12)	1.941(3)	1.949
C(1)–F(1)	1.296(9)	1.356(1)	1.360
C(1)–F(2)	1.403(9)		
C(3)–Si(1)–C(3)[b]	111.1(5)	112.7[c]	112.6[c]
C(3)–Si(1)–C(2)	113.9(3)		
C(1)–Si(1)–C(2)	102.1(7)	106.2(2)	106.1
C(1)–Si(1)–C(3)	107.5(3)		
F(1)–C(1)–F(1)[b]	112(1)	106.4(2)	106.1
F(1)–C(1)–F(2)	103.6(4)		

[a] Ref. [8].
[b] Symmetry code: x, $1/2 - y$, z.
[c] Calculated from the data in Ref. [8].

largest differences between calculated and experimental dimensions are in the C–F bond lengths and in CH_3–Si–CF_3 and F–C–F angles (Table 1). 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. The theoretical studies on structures of selected silanes showed that this is $(CH_3)_3SiCF_3$ where Si–C bond is the longest [8]. 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 methyl groups was calculated to be of 5.7 kJ/mol [8]. 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.

2.2. 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 [14], but they are shorter than the distances of the strongest attraction given by Dance [15]. The shortest distances between F-atoms are of 3.014(7) Å, the shortest F···H contacts are of 2.80 and 2.38 Å between the closest H-atoms. The shortest H···H contacts arrange molecules into [1 0 0] chains and the molecules with the shortest F···F and F···H distances run along the [0 1 0] direction. Thus the molecules linked by the shortest F···F, F···H and H···H contacts lie in sheets along planes (1 0 0) in the crystal lattice (Fig. 2).

The regions and directions of intermolecular contacts in $(CH_3)_3SiCF_3$ are mapped onto the molecular Hirshfeld surface [16–18] in Fig. 3. It confirms that the shortest contacts in this

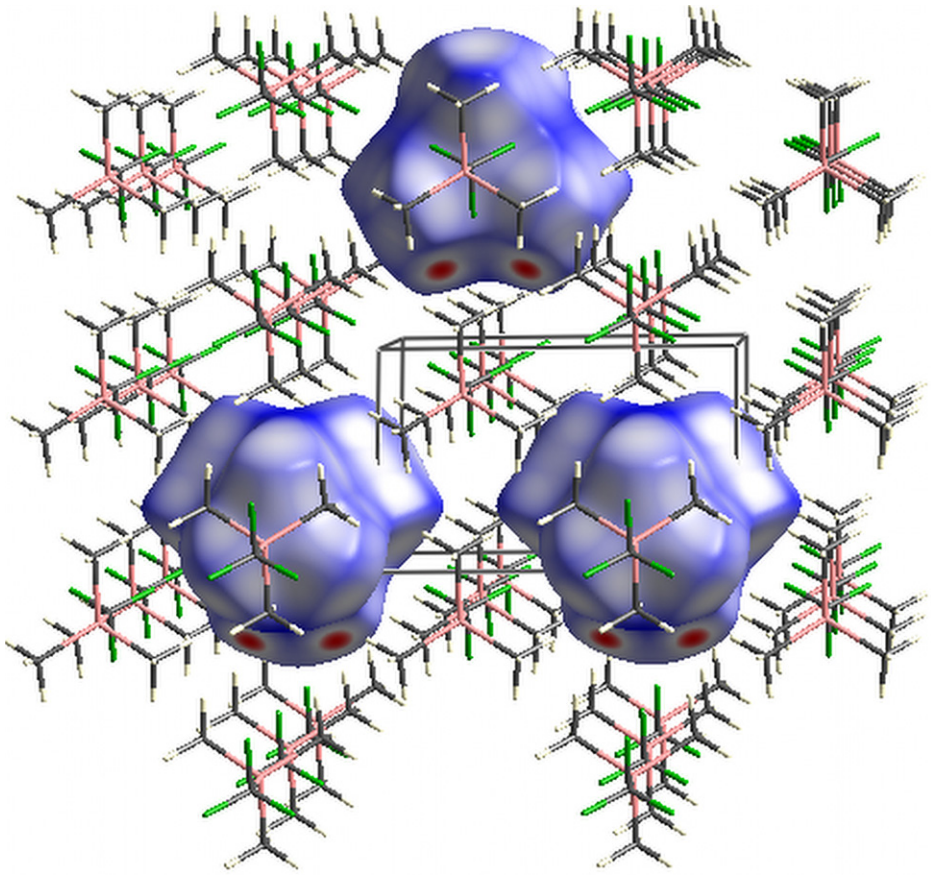

Fig. 3. Hirshfeld surfaces of $(CH_3)_3SiCF_3$ molecules decorated with the color scale depending on the intermolecular distances (normalized contact distance). The intermolecular contacts closer than the sum of their van der Waals' radii are highlighted in red on the surface, longer contacts are blue, and contacts around this sum are white. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of the article.)

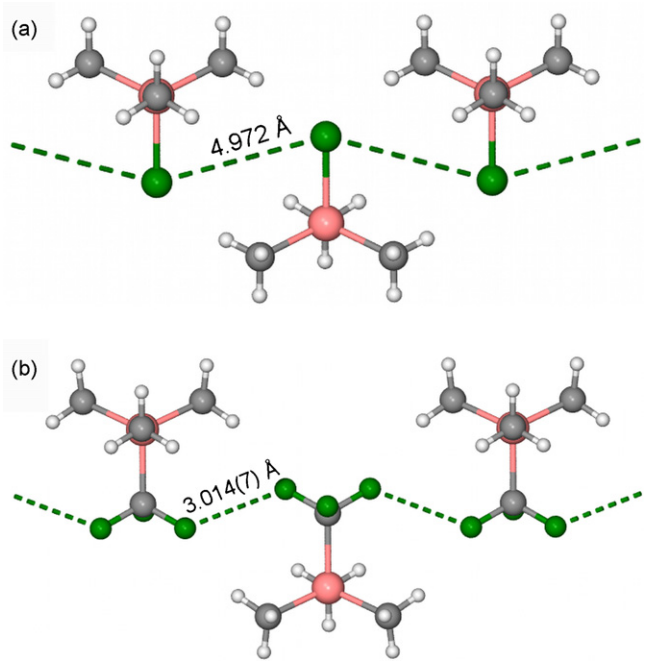

Fig. 4. Patterns of molecules with: (a) the shortest Cl···Cl distance in $(CH_3)_3SiCl$, phase α; and (b) the shortest F···F distance in $(CH_3)_3SiCF_3$.

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.

The $(CH_3)_3SiCF_3$ crystal is isostructural with low-temperature $(CH_3)_3SiCl$ phase α [19] as both these structures are monoclinic, space group $P2_1/m$. The largest differences in unit-cell dimensions are in the b parameter, 1.69 Å longer, and in the β angle, 14.5° larger in $(CH_3)_3SiCF_3$ than in $(CH_3)_3SiCl$; the molecular packing is similar. Gajda et al. [20] 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 Fig. 4. Owing to the molecular structure 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 halogens, and their role in the formation of crystal structures, can be described as halophobic, in accordance with the observation of other structures of halogenated compounds by Grineva and Zorkii [21,22].

These repulsive halogen···halogen interactions are reflected in the shapes of coordination schemes presented in Fig. 5. With respect to the distances to the molecular centers (associated with the Si atom in this discussion), the crystal structure of $(CH_3)_3SiCF_3$ corresponds to the close-packing arrangement of ideal spheres,

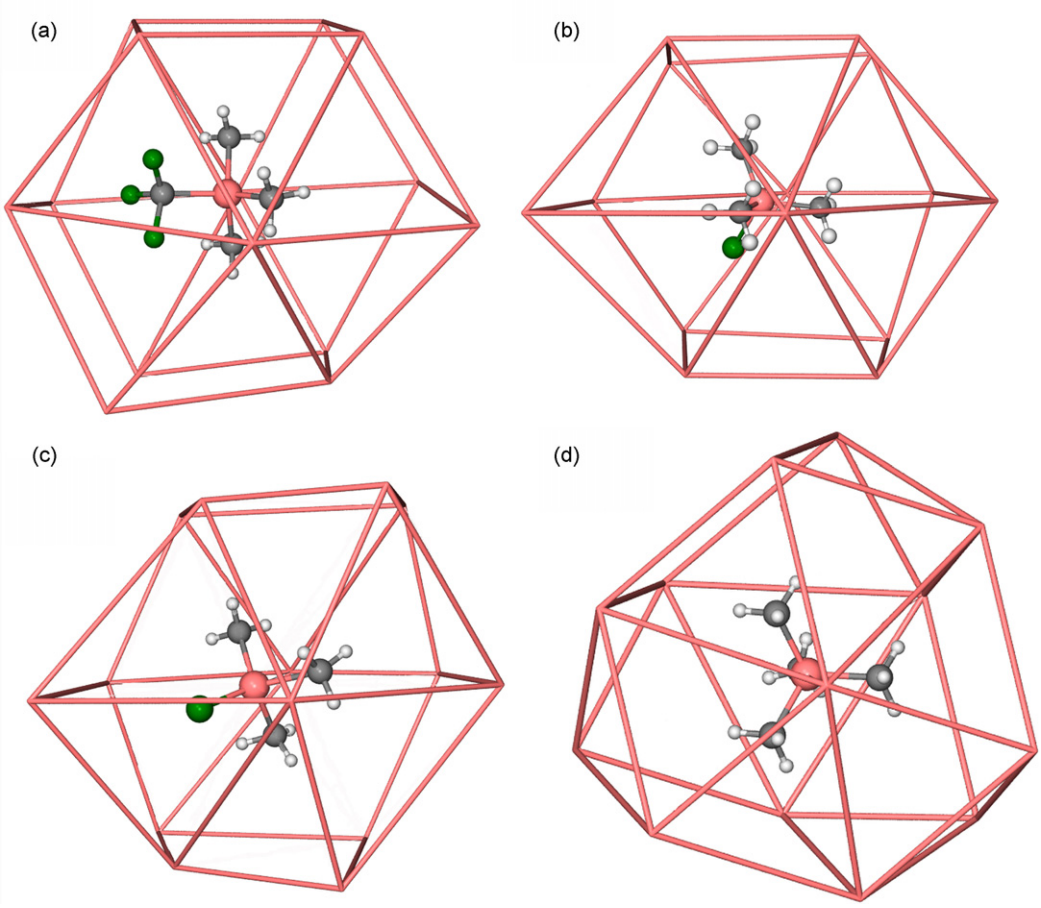

Fig. 5. The coordination schemes of: (a) $(CH_3)_3SiCF_3$; (b) $(CH_3)_3SiCl$, phase α; (c) $(CH_3)_3SiCl$, phase β; (d) $(CH_3)_4Si$. The vertices of the coordination schemes represent the Si-atoms closer than 8 Å to the central Si atom.

with the coordination number of 14. The Si···Si distances are 5.8897(18), 5.8897(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 [23] but more recent survey based on the Cambridge Crystallographic Database revealed that the 14-fold coordination may be most frequent for molecular crystals [24]. 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 [11]. The common feature of all the silanes investigated is weak intermolecular interactions.

3. 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 established 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 $(CH_3)_4Si$ is most certainly due to the considerably different dimensions and interactions of the methyl and trifluoromethyl groups. However, the common feature of all the silanes investigated is the absence of intermolecular interactions, which would indicate a contribution of electrostatic forces [11,19,20,25].

4. Experimental

4.1. Crystallization of trimethyl(trifluoromethyl)silane

Trimethyl(trifluoromethyl)silane has been crystallized in a modified Merrill–Bassett [26] diamond anvil cell (DAC). Because of the relatively low boiling point (327–328 K) [27], the liquid and the DAC were cooled with solid CO_2 for loading the sample. It crystallized at 0.20(5) GPa and then the high-pressure chamber was squeezed till its whole volume was filled by the polycrystalline sample. To ensure stable conditions for the diffraction measurements, pressure was increased to 0.90(5) GPa, and then the DAC was heated. A single crystal was grown at isochoric conditions from one seed crystal left in the melt sample at 473 K, followed by

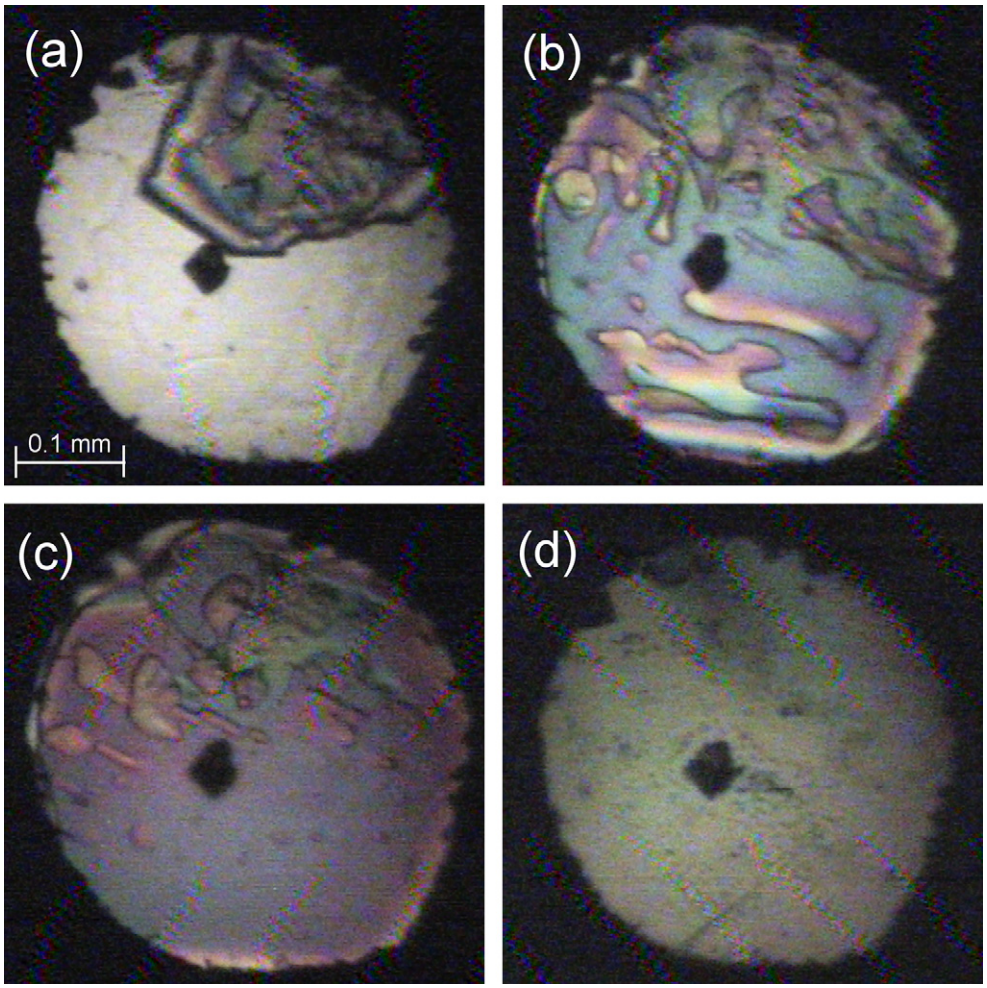

Fig. 6. Isochoric-growth stages of the $(CH_3)_3SiCF_3$ single crystal: (a) distorted morphology of a small single crystal at 453 K; (b) the crystal at 333 K with its faces highly irregular; (c) the same crystal after 1 h at 333 K; (d) the crystal at room temperature and 0.90 GPa. The small ruby chip for pressure calibration is placed close to the centre of the DAC.

Table 2
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(0 0 0)	148
Crystal diameter/height (mm)	0.38/0.20
θ-range for data collection (°)	3.98–29.43
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	1.091
Final R_1/wR_2 ($I > 2\sigma_1 I$)	0.0600/0. 0.1389
R_1/wR_2 (all data)	0.0708/0.1463
Weighting scheme	$w = 1/(\sigma^2(F_o^2) + (0.0724P)2 + 0.17P)$, where $P = (\mathrm{Max}(F_o^2,0) + 2F_c^2)/3$
Largest diff. peak and hole (e Å^{-3})	0.179/−0.136

slowly cooling the sample to 296 K. During the first stage of cooling many defects appeared on the crystal surface (Fig. 6a and b). Therefore, the crystal was kept at 333 K for 2 h until the process of self-healing eliminated all defects observed in crystal morphology (Fig. 6c and d). After cooling the DAC to room temperature 5 days passed before the diffraction data were measured.

4.2. General experimental procedures

Pressure in the DAC was calibrated by ruby-fluorescence method [28,29], 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 [30] was used for the data collections [31] and preliminary reduction of the data. After correcting the intensities for the effects of DAC absorption, sample shadowing by the gasket, and the sample absorption [32,33], the diamond reflections have been 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 [34], and refined by full-matrix least-squares [35]. Anisotropic temperature factors were applied for all non-hydrogen atoms. The H-atoms in the structures were calculated from molecular geometry (d_{C-H} = 0.97 Å) their U_{iso}'s constrained to 1.2 times U_{eq} of the carrier atoms. Unexpectedly large difference between C(1)–F(1) and C(1)–F(2) may be due to the experimental limitations resulting from the restricted access to the DAC and incomplete data in this high-pressure experiment. On the other hand atoms F(1) and F(2) differ in their intermolecular contacts, much longer for F(2), which could affect strong vibrations of these atoms and their average positions. The restricted access to the sample, limiting the completeness of reflections data to about 50% at θ_{max} = 20° and 40% at θ_{max} = 29° (Table 2), is strongly anisotropic [26,36] and the accuracy of obtained dimensions depends on the orientation of the particular molecular groups in question. The crystal data and the structure refinement details are listed in Table 2. Structural drawings were prepared using the X-Seed interface of POV-ray [37,38]. The crystal structure has been deposited in the CIF form as supplementary publications No. 687260 CCDC in the Cambridge Crystallographic Database Centre.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jfluchem.2008.07.015.

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