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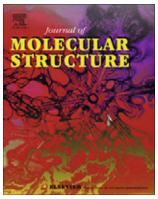

# Synthesis, characterization and structural aspects of 3-azidopropylsilatrane

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## ABSTRACT

Reaction of 3-chloropropyltriethoxysilane (**1**) with sodium azide (**2**) in the presence of phase transfer catalyst, tert-butylammonium bromide provided 3-azidopropyltriethoxysilane (**3**). This undergoes transesterification reaction with triethanolamine in the presence of KOH to form a new silatrane 3-azidopropylsilatrane (**4**). Multinuclear NMR ($^{1}$H, $^{13}$C and $^{29}$Si), IR and elemental analyses support the structure of novel silatrane **4**. $^{29}$Si NMR indicates the presence of penta-coordinated silicon atom, which is supported by X-ray studies. It revealed that Si is present in distorted trigonal bipyramidal environment with three O atoms in equatorial positions and N atom at one of the apical positions. The second axial position is occupied by long alkyl chain bearing azide moiety. The interaction of Si with N forms a trans-annular bond having 2.176 Å bond length.

## 1. Introduction

More than a century after their discovery organic azides have received renewed interest in synthetic chemistry and are becoming established as an important and versatile class of chemical compounds [1–3]. In particular, in the past two decades an increasing interest in organic azides could be observed due to their vast synthetic utility in conjunction with easy accessibility via various synthetic routes. Among others, organic azides are currently considered as powerful precursors for reactive species such as nitrenes and nitrenium ions as well as nitrogen – rich compounds such as aziridines, azirines, triazoles, triazolines and triazenes. Moreover, organic azides can be easily transformed into amines, isocyanates and other functional molecules. More recently, these compounds received an increasing interest as valuable and versatile reagents within the concept of 'Click Chemistry' [4,5]. However, alongside their huge utility in organic synthesis the potential hazardous properties of organic azides must be carefully taken into account. Many organic compounds containing azido groups have not found wide application as practical energetic materials because of their high sensitivity to heat and shock stimuli. This poor thermal and mechanical stability of many organic azides is probably the most crucial property that has prevented chemists in the past from extending their research on azido compounds. Organic and inorganic azides are energy-rich molecules that often exhibit explosive properties. Azido group is a highly energetic functional group because $N_3$ $\pi$-bond can be easily polarized, which consequently results in strong exothermic dissociation reactions with the release of molecular nitrogen and reactive nitrene groups. In general, the introduction of an azido group into an organic compound increases its energy content by approx. 290–355 kJ/mol [6,7]. This is one of the reasons, why organic azides are considered and partly used as energetic materials such as energetic polymers or high-energy–density-materials (HEDM) in explosives or propellant formulations [8,9].

Among organosilanes, 3-chloropropyltriethoxysilane is considered as one of the important precursor, especially for the synthesis of ionic liquids [10–12], chloropropylsilatrane [13], siloxanes [14] and hybrid materials [15–17]. Recently, SiMCM-41 was successfully modified by using 3-aminopropyltrimethoxysilane and 3-chloropropyltriethoxysilane to give organic group modified samples [18]. A mesoporous and amorphous silica, chemically modified by using 3-chloropropyltriethoxysilane and organic reagents, have been utilized for the adsorption of toxic metal ions such as Cr(VI) and Hg(II) from aqueous samples [19,20].

Herein, we report synthesis of novel 1-(3-azido-propyl)-2,8,9-trioxa-5-aza-1-sila-bicyclo[3.3.3]undecane with the structural characterization in solid state by X-ray crystallography, multinuclear NMR ($^{1}$H, $^{13}$C and $^{29}$Si), IR, elemental analysis and mass spectroscopy. The results obtained from X-ray crystallography are compared with theoretical studies such as DFT and HF methods.

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## 2. Experimental

### 2.1. Materials

All the syntheses were carried out under a dry nitrogen atmosphere using vacuum glassline. Organic solvents used were dried and purified according to standard procedures and stored under nitrogen. 3-Chloropropyl(triethoxy)silane (Aldrich), sodium azide (Acros organics) and triethanolamine (Merck) were used as such.

### 2.2. Instruments and measurements

Infrared spectra were routinely obtained as thin films or Nujol mulls and KBr pellet on a Perkin–Elmer RX-I FT IR Spectrophotometer. Mass spectral measurements (EI, 70 eV) were carried out on a VG Analytical (70-S) spectrometer. C, H and N analyses were obtained on a Perkin–Elmer Model 2400 CHN elemental analyzer. The solution $^{1}H$ and $^{13}C$ NMR spectra were recorded at 25 °C on a Jeol and Bruker FT NMR (AL 300 MHz and 400 MHz) spectrometer using $CDCl_3$ as the solvent. Chemical shifts in ppm were determined relative to internal $CDCl_3$ and external tetramethylsilane (TMS).

### 2.3. Synthesis of 3-azidopropyltriethoxysilane (**3**)

To a dried 100 mL single-neck round-bottom flask equipped with reflux condenser, 3-chloropropyltriethoxysilane (4 g, 16.6 mmol), sodium azide (2.16 g, 33.2 mmol) and tetrabutyl ammonium bromide (1.288 g, 4 mmol) were added in dry acetonitrile (50 mL), under nitrogen atmosphere. The reaction mixture was then brought to stirr under reflux for 18 h. After completion of the reaction, the solvent was removed under reduced pressure. The crude mixture was diluted in dry hexane and the suspension was filtered under vacuum. Solvent was removed from the resulting filtrate and the crude oil obtained was distilled under reduced pressure to give 3-azidopropyltriethoxysilane as a colorless liquid. Yield: 3.02 g, 74%. IR (Neat, KBr plates cm$^{-1}$): 2098 ($—N{=}N^{+}{=}N^{-}—$); $^{1}H$ NMR (300 MHz, $CDCl_3$): $\delta$ 0.60 (t, 2H, $CH_2Si$), 1.15 (t, 9H, $CH_3$), 1.64 (q, 2H, $CCH_2C$), 3.19 (t, 2H, $CH_2N_3$), 3.77 (q, 6H, $OCH_2$); $^{13}C$ NMR (300 MHz, $CDCl_3$) $\delta$ 7.38 ($SiCH_2$), 18.41 ($CCH_2C$), 22.45 ($CH_2N_3$), 53.54 ($NCH_2$), 58.58 ($OCH_2$).

### 2.4. Synthesis of 3-azidopropylsilatrane (**4**)

To a dried 100 mL single-neck round-bottom flask fitted with Dean Stark apparatus, 3-azidopropyltriethoxysilane (2.22 g, 8.98 mmol) and triethanolamine (1.34 g, 8.98 mmol) was added in dry benzene. The contents were refluxed for 5 h in the presence KOH in catalytic amount. Benzene was removed under vacuum and dry diethylether was added when white solid was separated out. The contents were further stirred for 1 h at room temperature. The solid was filtered under vacuum, washed twice with diethylether (2 × 10 mL) and dried. M.pt. 55–57 °C; yield: 1.95 g, 84%. Anal. Calcd for $C_9H_{18}N_4O_3Si$: C, 41.84; H, 7.02; N, 21.69; found: C, 41.10; H, 6.89; N, 21.20; IR ($CCl_4$, KBr plates, cm$^{-1}$): 2093 vs. $\nu$ ($—N{=}N^{+}{=}N^{-}—$), 583 m $\nu$ ($Si \leftarrow N$), 612 m $\delta_s$ ($SiO_3$), $\nu_s$ (Si–C), 936 s, 1087 s, 1105 vs. 1130 vs. $\nu$ (Si–O–C–C), 1180 m $\tau$ ($CH_2O$), 1278 m $\omega$ ($CH_2O$), 1352 m, 1454 m $\omega$ ($CH_2N$); $^{1}H$ NMR (400 MHz, $CDCl_3$): $\delta$ 0.35 (t, $CH_2Si$), 1.66 (q, $CCH_2C$), 2.75 (t, $NCH_2$), 3.12 (t, $CH_2N_3$), 3.70 (t, $OCH_2$); $^{13}C$ NMR (400 MHz, $CDCl_3$): $\delta$ 13.32 ($SiCH_2$), 24.78 ($CCH_2C$), 54.63 ($CH_2N_3$), 51.00 ($NCH_2$), 57.62 ($OCH_2$); $^{29}Si$ (300 MHz, $CDCl_3$); −70.24 ppm; MS: *m*/*z* (relative abundance, %): 132 (48), 150 (89), 172 (71), 174 (60), 192 (100), 216 (23), 233 (91).

### 2.5. X-ray structure determination

Single crystal X-ray diffraction data for **4** was collected on a Nonius Kappa CCD diffractometer equipped with a CCD detector and graphite-monochromatized Mo K$\alpha$ radiation ($\lambda$ = 0.71073 Å). All intensities were corrected for Lorentz and polarization. The structure was solved by direct methods using SIR97 program [21] and refined on $F^2$ by full-matrix least-square methods. Crystal data is given in Table 1 and ORTEPIII [22] view along with atomic labelling (thermal ellipsoids are drawn at 40% probability) of **4** is shown in Fig. 1. Non hydrogen atoms were refined anisotropically apart from N4 and C2, which were found disordered over two almost equivalent positions and refined isotropically. The other disordered atoms C4 and C6 were refined anisotropically with a fixed occupancy factor of 0.5 (Fig. 1). H atoms were included on calculated positions. All calculations were performed using SHELXL-97 [23], and PARST [24], as implemented in the WINGX [25] system of programs.

### 2.6. Theoretical studies

The quantum mechanical calculations were carried out using the GAUSSIAN 03 series of programs. Geometries were fully optimized at both the Restricted Hartree–Fock (RHF) and Density Functional Theory level (DFT), using Becke's three parameter hybrid exchange functional and the correlation functional of Lee, Yang, and Parr (B3LYP) with 3-21 + $G^*$ and 6-31$G^*(d)$ basis sets.

## 3. Results and discussion

### 3.1. Synthesis

3-Azidopropyltriethoxysilane can be prepared from 3-chloropropyltriethoxysilane and sodium azide by using two alternate methods, either in the presence or in the absence of catalyst. In the absence of catalyst, reaction has been carried out by refluxing the reactants in butanone for 72 h [26]. On the other hand, use of catalyst such as tetrabutylammonium bromide reduces the reaction time to 18 h [27].

In the present work, 3-azidopropyltriethoxysilane (**3**) was synthesized in high yield by nucleophilic substitution reaction of 3-chloropropyltriethoxysilane (**1**) and sodium azide (**2**) in the presence of tetrabutylammoium bromide as catalyst and acetonitrile as solvent. The compound was isolated as a colorless liquid after distillation under reduced pressure, which can be used as a precursor for the synthesis of new silatrane (**4**). Transesterification of compound (**3**) with triethanolamine in the presence of a catalytic

**Table 1**
X-ray crystal data and structure refinement of 4.

| Chemical formula | $C_9H_{18}N_4O_3Si$ |
|---|---|
| $M_r$ | 258.36 |
| Crystal system, space group | Orthorhombic, *Pna*$2_1$ |
| *a*, *b*, *c* (Å) | 13.2149 (2), 12.0619 (3), 8.1850 (3) |
| *V* (Å$^3$) | 1304.66 (6) |
| *Z* | 4 |
| Radiation type | Mo K$\alpha$ |
| $\mu$ (mm$^{-1}$) | 0.18 |
| Crystal size (mm) | 0.41 × 0.37 × 0.29 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 2700, 2700, 2279 |
| $R[F^2 > 2s(F^2)]$, $wR(F^2)$, *S* | 0.063, 0.200, 1.04 |
| No. of reflections | 2700 |
| No. of parameters | 171 |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.43, −0.50 |

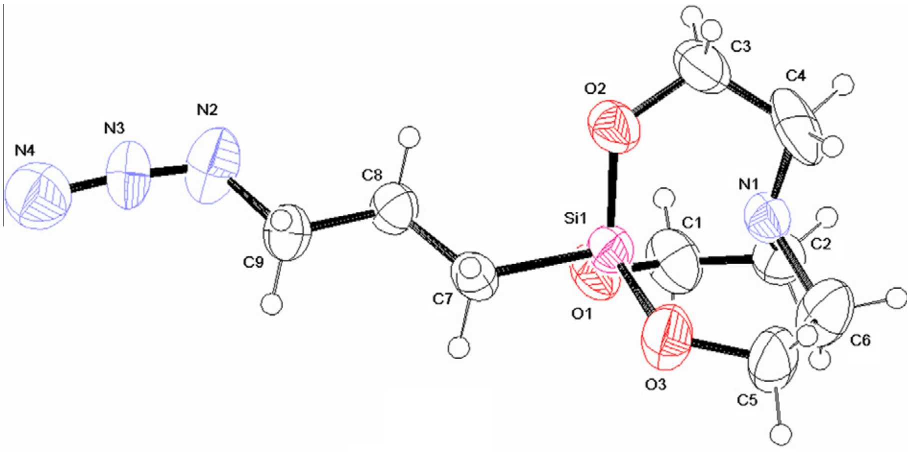


**Fig. 1.** ORTEP III view of the molecule 1. Ellipsoids are drawn at 40% probability. For the sake of clarity, only one component of the disordered part is shown.

amount of KOH resulted in the formation of 3-azidopropylsilatrane as a white solid (Scheme 1).

### 3.2. Spectroscopic data

#### 3.2.1. IR spectra

IR spectrum of compounds was recorded in the range 4000–400 cm$^{-1}$. The absorption bands were assigned on the basis of literature [28]. IR absorption frequencies are in accordance with the structure of prepared compound. The spectrum shows presence of C–H vibrations near 3000 cm$^{-1}$ for both compounds. Bands observed in this region are not affected appreciably by the nature of substituent. A very strong band is observed at 2098 cm$^{-1}$ and 2093 cm$^{-1}$ for **3** and **4**, respectively, which is the main characteristic band for the presence of azide group. Other vibrations of silatranyl moiety are observed in the region of 580–1454 cm$^{-1}$. In addition, Si–N vibration frequency, observed at 583 cm$^{-1}$, is of great interest as it indicates hypervalency of silicon.

#### 3.2.2. NMR spectra

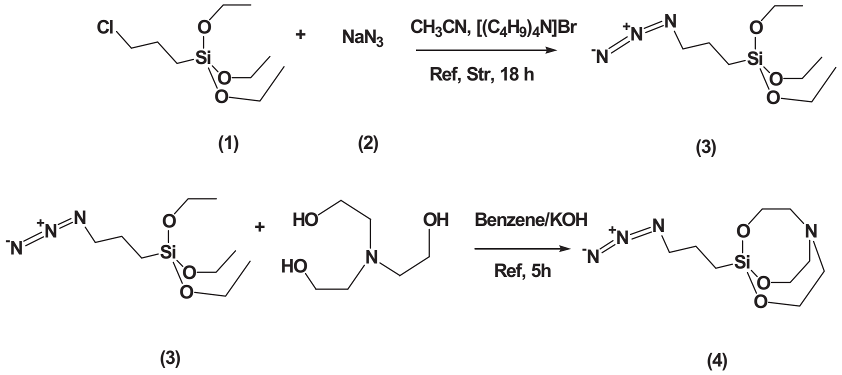
Multinuclear ($^1$H, $^{13}$C and $^{29}$Si) NMR spectra are consistent with the structure of synthesized compound. In $^1$H NMR spectrum, an upfield shift is observed for SiCH$_2$ protons of **4** as compared to **3**, which clearly indicates increase in electron density at Si due formation of Si $\leftarrow$ N dative bond. Two equivalent triplets are observed in the region 2.73–2.76 and 3.69–3.72 ppm due to NCH$_2$ and OCH$_2$, respectively, of Si(OCH$_2$CH$_2$)$_3$N moiety in **4**. All protons in the compounds have been identified and the total number of protons calculated from the integration curve is found to be equal to expected number. In $^{13}$C NMR, $\delta$ CH$_2$N$_3$ appeared at 54.63 ppm. The peaks appeared at 57.62 and 51.00 ppm are assigned to $\delta$ O**C**H$_2$ and N**C**H$_2$ in **4**. $^{29}$Si NMR shows a peak at −70.24 ppm, which clearly indicates the formation of penta-coordinated Si [29,30].

#### 3.2.3. Mass spectra

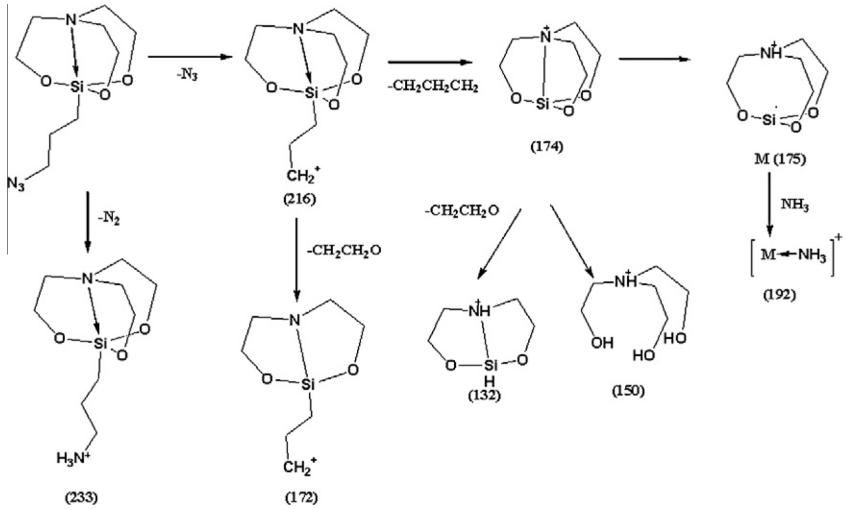
Mass spectrum of **4** shows common features of siltrane in which fragmentation proceeds through cleavage of Si–CH$_2$ bond and one arm of silatranyl ring. The molecular ion peak is not observed in the spectrum as **4** transformed predominantly to form aminopropylsilatrane ($m/e = 233$) with the evolution of N$_2$ due to its less stability. On the other hand, it loses N$_3$ to form a fragment having $m/e = 216$, which in turn loses one of the OCH$_2$CH$_2$– ring to form a bicyclic fragment with alkyl chain ($m/e = 172$). Homolytic cleavage of Si–CH$_2$ forms a silatranyl fragment with $m/e = 174$ which is generally found in all silatranes. This fragment also loses a cyclic arm to form bicyclic moiety ($m/e = 132$). Along with a very intense peak due to protonated triethanolamine ($m/e = 150$), most abundant peak at $m/e = 192$ is also observed due to the formation of adduct of silatranyl cation with NH$_3$ [31], which is also a general feature of silatrane fragmentation (Scheme 2).

### 3.3. Single crystal X-ray crystallography

Colorless crystals suitable for X-ray analysis were grown from concentrated solution of **4** in chloroform. Single crystal X-ray diffraction study has shown that the compound crystallizes in a orthorhombic crystal system (space group = P*na*2$_1$). The compound possesses usual skeleton containing Si atom in trigonal bipyramidal



**Scheme 1.** Reaction pathway for the synthesis of 3-azidopropylsilatrane.



**Scheme 2.** Mass fragmentation pattern of 3-azidopropylsilatrane.

geometry because of the coordination of tripodal triethanolamine ligand. The trianionic $N(CH_2CH_2O^-)_3$ entity act as tetradentate ligand coordinating each to Si to form five membered ring, so that transannular Si–N bond is formed. Due to this bond, Si atom achieves a coordination number of five with trigonal bipyramidal coordination sphere. Three O atoms of tetradentate ligand occupy three basal positions and one N is present at apical site. Other apical position of pyramid is occupied by alkyl chain with azide group. Si–C, Si–O and Si–N distances are 1.895(3), 1.662(2) and 2.141(7) Å, respectively, which are consistent with the mean values calculated on 52 structures retrieved from CSD (Table 2). The azide group though disordered, exhibits bond distances and angles in line both with the calculations and experimental values found in the literature.

Due to the complete absence of strong hydrogen bonding donors, the molecules are assembled in the crystal lattice by means of few weak C–H···O/N hydrogen bonds, which are listed in Table 3. Each unit is linked to four surrounding silatranes, as shown in Fig 2. The unit cell content is reported in Fig 3.

### 3.4. Theoretical studies

Density Functional Theory and Hartree–Fock calculations were performed at 3-21 + $G^*$ and 6-31 $* G(d)$ basis sets for **4** and the results are compared with crystal data. The most important geometrical parameter, N → Si distance, examined with theoretical studies (HF and DFT) is found to be significantly longer than the crystal data. However, this bond length is sensitively affected by adding the electron correlation effect at the B3LYP levels and showed an increase in the value. Similarly, experimental values of O–Si–O bond angles are found to be different from their theoretical values, whereas other parameters computed from RHF and DFT levels are almost same with the values obtained from X-ray crystallographic data (Table 2). The difference between calculated and experimental values may be a consequence of the shallow vibrational potential of Si–N bond. It may also be concluded that denser packing of molecules intensifies crystal field, which in turn leads to compression of the molecules and hence variation in geometric parameters. The

**Table 3**
Hydrogen bonding parameters (Å, °).

| D–H···A | D–H | D···A | H···A | <D–H···A |
|---|---|---|---|---|
| C9–H9a···O1[a] | 0.97 | 3.578(8) | 2.63 | 167 |
| C9–H9b···O2[b] | 0.97 | 3.622(8) | 2.65 | 174 |
| C5–H5a···N4A[b] | 0.98 | 3.39(3) | 2.61 | 137 |
| C4a–H4d···O3[c] | 0.97 | 3.30(1) | 2.48 | 142 |
| C3–H3b···N4a[d] | 0.97 | 3.52(2) | 2.64 | 151 |

[a] $1 - x, 2 - y, z - ½$.
[b] $1 - x, 2 - y, z + ½$.
[c] $x - 1/2, 3/2 - y, z$.
[d] $x - 1/2, -y + 5/2, z$.

**Table 2**
Theoretical and X-ray crystallography data of 4.

| Parameters | X-ray crystal data | DFT, B3LYP 3-21 + $G^*$ | DFT, B3LYP 6-31$G^*(d)$ | RHF, 3-21 + $G^*$ | RHF, 6-31$G^*(d)$ |
|---|---|---|---|---|---|
| N(1) → Si | 2.176 (2) | 2.582 | 2.610 | 2.698 | 2.674 |
| Si–C(7) | 1.879 (2) | 1.873 | 1.878 | 1.861 | 1.871 |
| Si–O av. | 1.656 (2) | 1.683 | 1.677 | 1.651 | 1.647 |
| N(1)–C(2,4,6) av. | 1.420 (6) | 1.481 | 1.458 | 1.463 | 1.447 |
| N(2)–N(3) | 1.118 (5) | 1.258 | 1.232 | 1.258 | 1.224 |
| N(3)–N(4) | 1.220 (1)* | 1.160 | 1.144 | 1.115 | 1.104 |
| N(4)–N(3)–N(2) | 173.1 (7) | 172.92 | 173.25 | 174.21 | 175.47 |
| C(7)–C(8)–C(9) | 112.7 (2) | 111.91 | 113.32 | 112.16 | 113.50 |
| C(8)–C(7)–Si(1) | 114.4 (2) | 114.57 | 117.67 | 114.60 | 117.80 |
| O(1)–Si(1)–O(2) | 118.8 (2) | 113.49 | 114.11 | 111.12 | 112.89 |
| O(2)–Si(1)–O(3) | 118.5 (2) | 117.80 | 116.82 | 114.56 | 111.05 |
| O(3)–Si(1)–O(1) | 117.9 (2) | 112.34 | 112.31 | 110.05 | 114.43 |
| C(2)–N(1)–C(4) | 139.0 (1)* | 117.33 | 118.06 | 118.45 | 118.72 |
| C(4)–N(1)–C(6) | 117.0 (1)* | 115.77 | 116.59 | 116.94 | 117.19 |
| C(6)–N(1)–C(2) | 115.6 (9)* | 117.44 | 118.48 | 118.48 | 119.13 |

[*] Only one set of the disordered part has been considered.

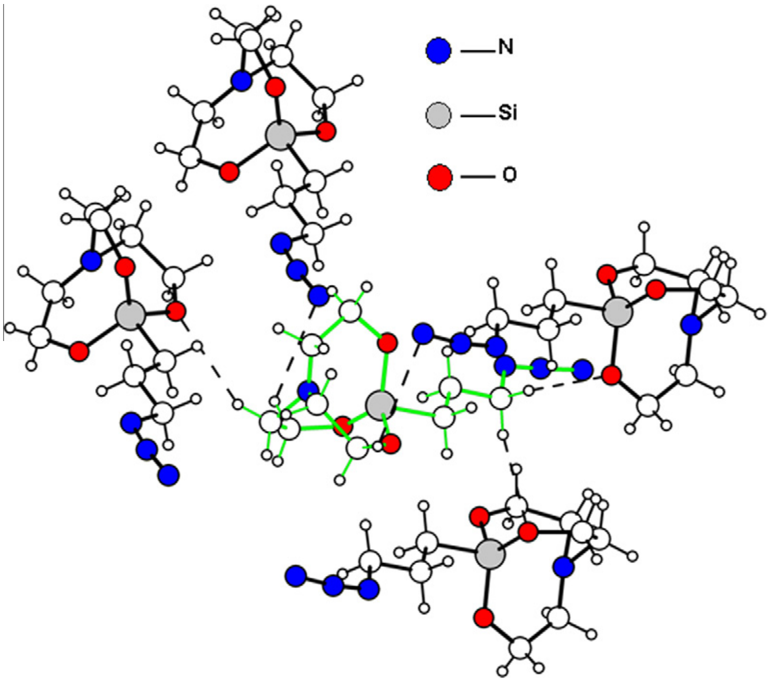


**Fig. 2.** Packing pattern around the asymmetric unit (in green). C–H···O/N hydrogen bonds are shown by broken lines. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

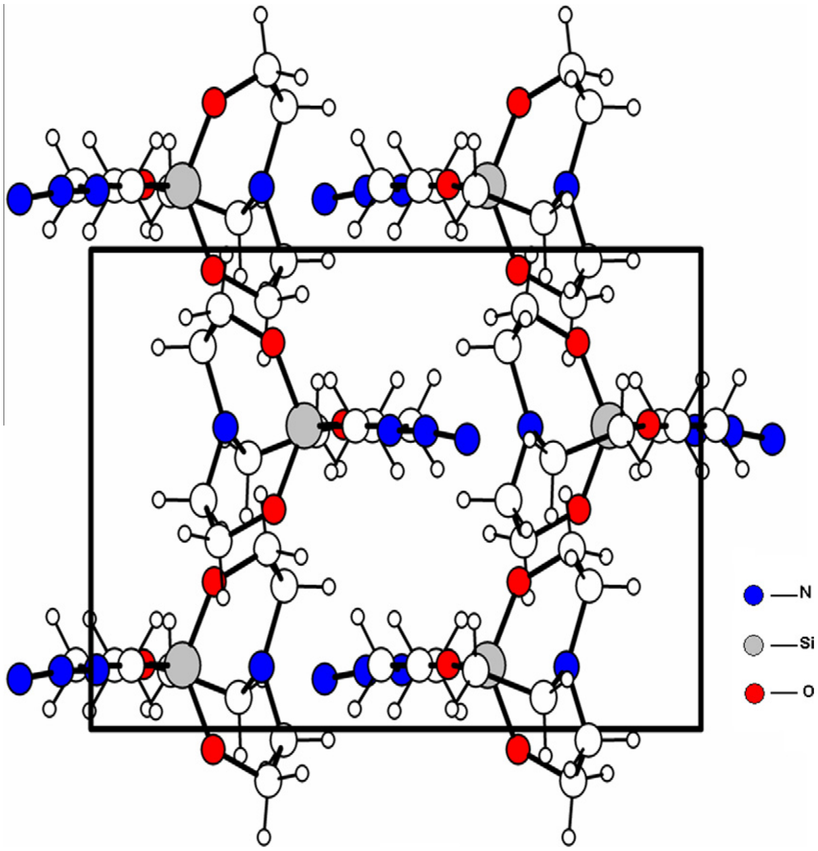


**Fig. 3.** Cell content along the '*b*' axis.

reduced N → Si bond length results from increased crystal density and packing coefficient. Other parameter responsible for the shorter Si–N bond and slight change observed in other bond lengths might be because of crystal packing forces in the solid state and hydrogen bonding involved due to presence of N and O in the molecule [28,32].

## 4. Conclusion

A novel silatrane, 3-azidopropylsilatrane, has been synthesized and characterized based on analytical, IR, multinuclear NMR ($^{1}$H, $^{13}$C and $^{29}$Si) and mass spectroscopic studies. Single crystal X-ray structure shows a penta-coordinated Si with Si–N bond distance of 2.176(2) Å and overall crystal lattice is stabilized by C–H···O/N interactions. DFT and RHF studies revealed systematically higher value of Si–N bond distance as compared to X-ray diffraction studies. The slight short bond distances calculated by theoretical studies are attributed to crystal packing forces during crystallization and weak hydrogen bonding interactions.

## 5. Supplementary material

Crystallographic data for the structural analysis of the title compound has been deposited at the Cambridge Crystallographic Data Center, 12 Union Road, Cambridge, CB2 1EZ, UK, and are available free of charge from the Director on request quoting the deposition number CCDC 784354 (fax: C44 1223 336033, e-mail: deposit@ccdc.cam.ac.uk).

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