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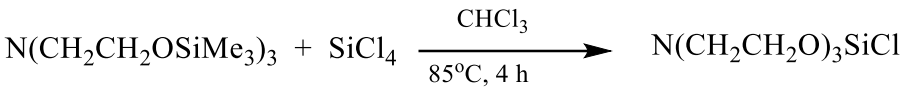
X-ray structural study of 3:1 solvate of 1-chlorosilatrane with MeCN

Boris A. Gostevsky, Alexander V. Vashchenko and Nataliya F. Lazareva

Synthesis of 1-chlorosilatrane



Tetrachlorosilane (2.28 g, 14.42 mmol), tris[2-(trimethylsilyloxy)ethyl]amine (4.56 g, 12.47 mmol) and dry $CHCl_3$ (10 ml) were placed into an Schlenk flask. This flask was evacuated, and the mixture was stirred at at 85 °C for 4 h. After cooling to room temperature, the volatiles were removed under reduced pressure. The residue was washed with chloroform (2 x 10 ml) and dried *in vacuo*. The yield of 1-chlorosilatrane as a white powder was 1.86 g (8.87 mmol, 71%). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 3.02 t (6H, NCH_2, ^3J = 5.9 Hz), 3.98 t (6H, OCH_2, ^3J = 5.9 Hz). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 51.38 (NCH_2), 58.13 (OCH_2). Crystals for X-ray analysis were obtained from acetonitrile in the form of needles. These crystals do not melt in an evacuated capillary even at 270 °C.

NMR spectra of 1-chlorosilatrane

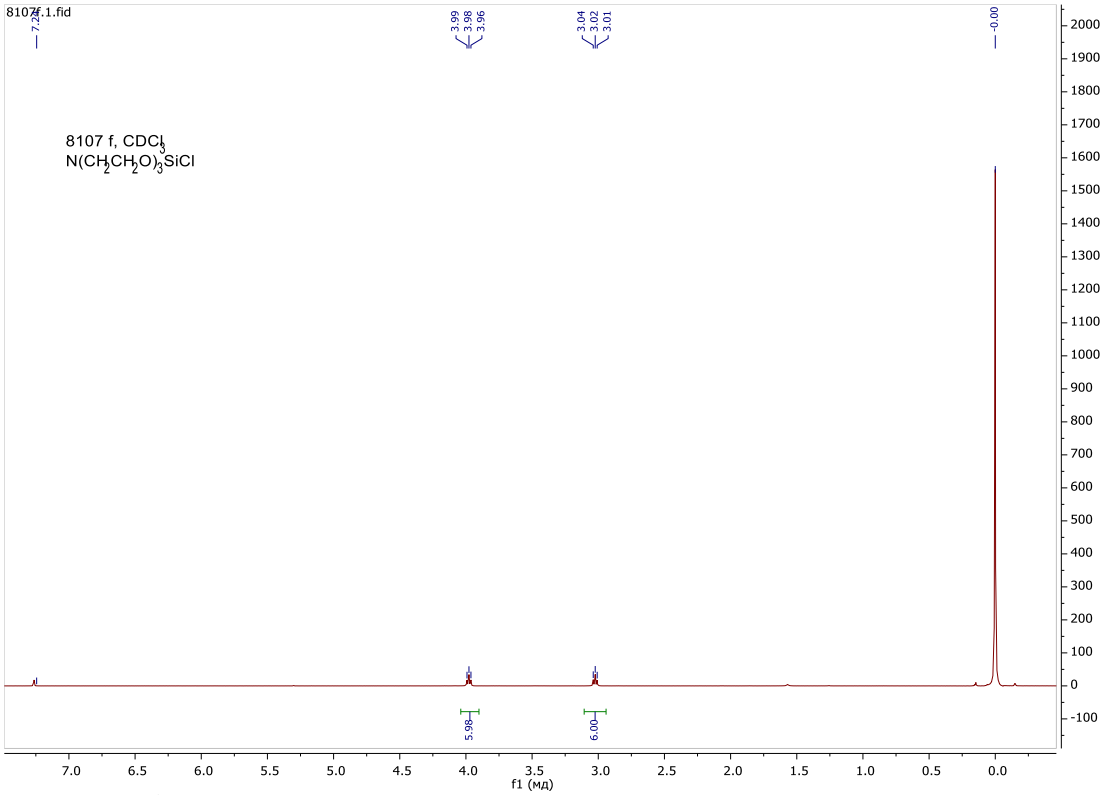


Figure S1. ^{1}H NMR spectrum of $ClSi(OCH_2CH_2)_3N$

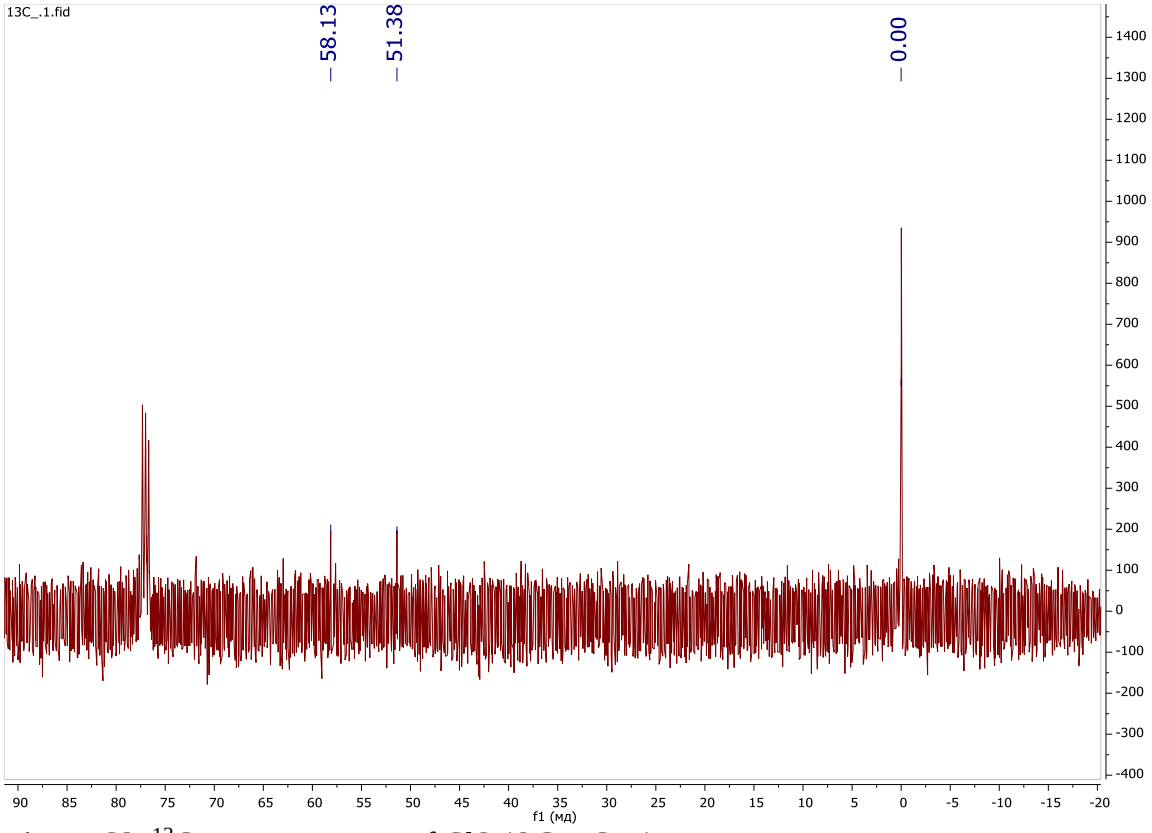


Figure S2. ^{13}C NMR spectrum of $ClSi(OCH_2CH_2)_3N$

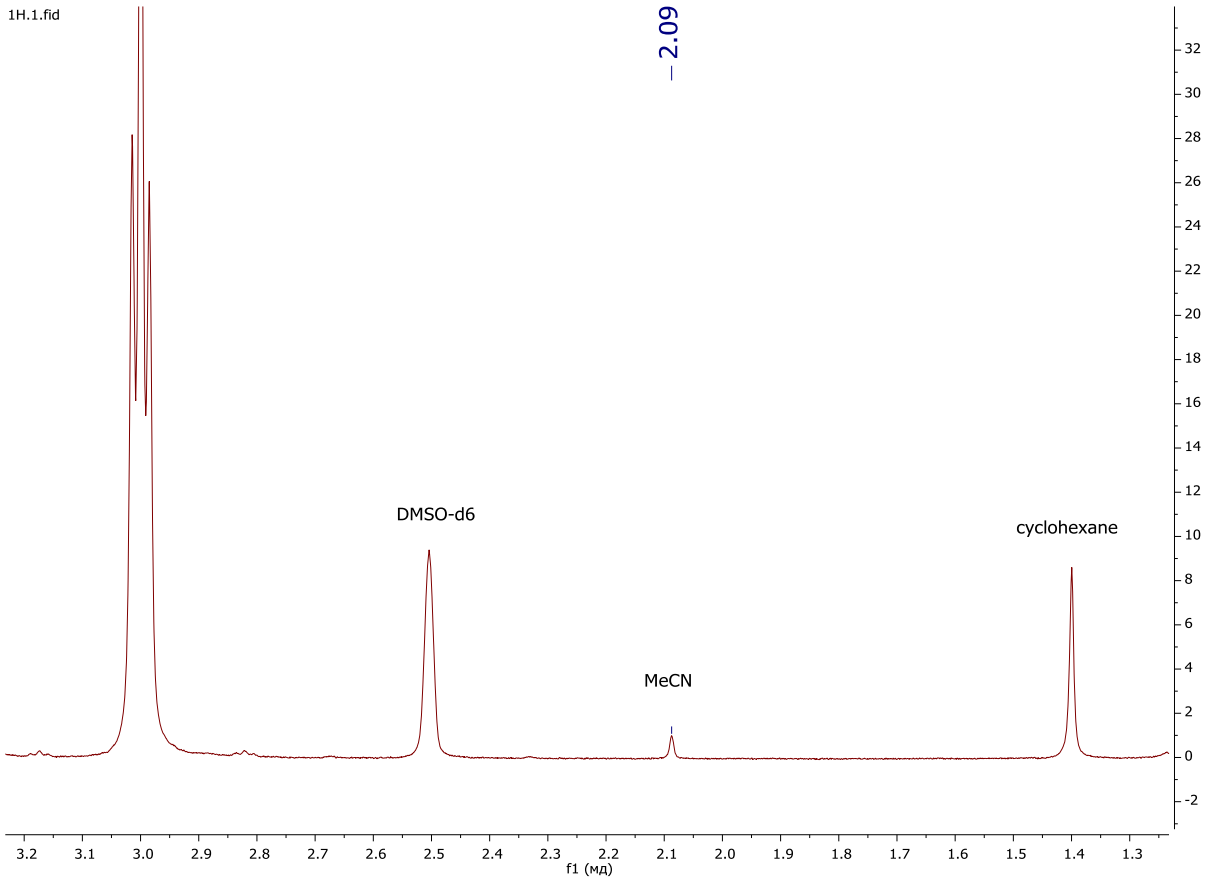


Figure S3 ^{1}H NMR spectrum of $ClSi(OCH_2CH_2)_3N$ in DMSO-d$_6$, cyclohexane as internal standard

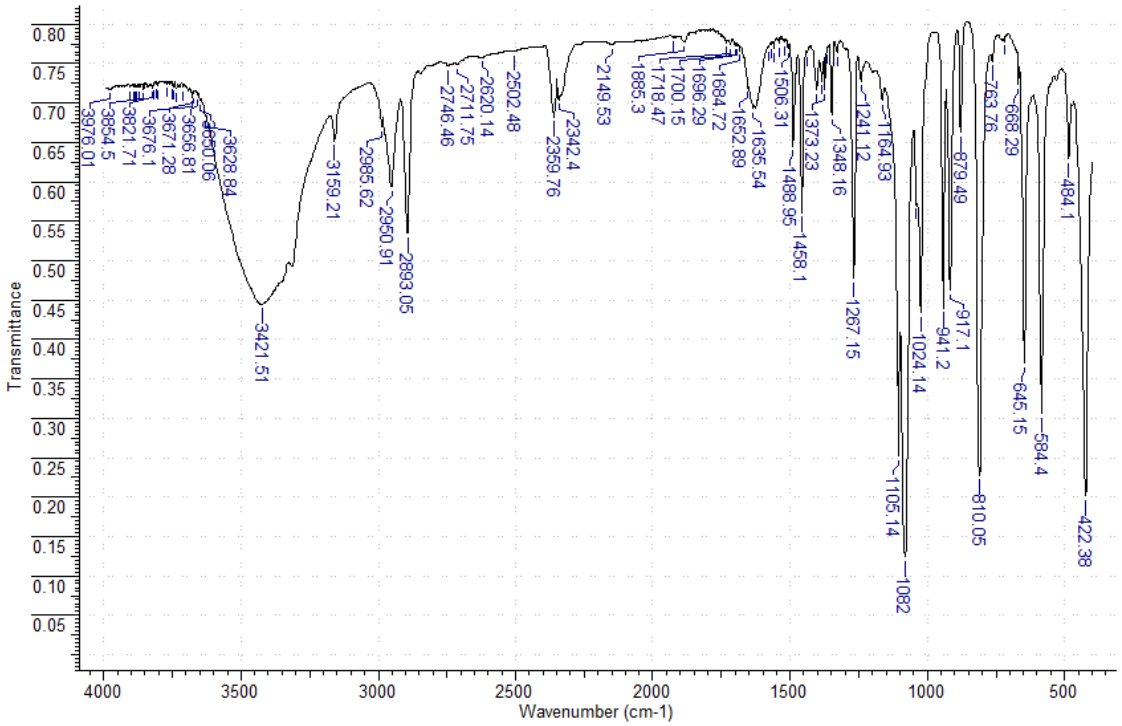


Figure S4 IR spectrum of $ClSi(OCH_2CH_2)_3N$

The X-ray experiment

The X-ray diffraction experiment was carried out on a Bruker D8 Venture single-crystal diffractometer with a Photon 100 detector The orientation matrix and cell parameters were determined and refined using 798 reflections. The cell corresponds to the hexagonal syngony. Details of crystallographic data and experimental conditions are presented in Table 1.

Table S1. Crystallographic data and experimental conditions

Empirical formula	$C_{20}H_{39}Cl_3N_4O_9Si_3$
Formula weight/g mol^{-1}	670.17
Space group, Z	$P6_3$, 2
a, *b*, *c*, Å;	14.6357(10), 14.6357(10), 8.037(15)
α, β, γ /^{o}	90, 90, 120
V, Å^3	1491(4)
μ, mm^{-1}	0.48
Radiation λ, Å	MoK$_\alpha$, 0.71073
T, K	293
Collected/Independent reflections N_1	11325/2240
Reflections with $I > 2\sigma(I)$, N_2	1442
Absorption accounting	multi-scan
R_{int}	0.067
R_1	0.039
Goof	0.963
D_X, Mg m^{-3}	1.493
Weight scheme	$w = 1/[\sigma^2(F_o^2) + (0.0371P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
F000	705.0

Reflex intensity was integrated using Bruker SAINT software. The space group $P6_3$ was determined from the extinction analysis and intensity statistics for all reflections. Accounting for the absorption of x-rays by a crystal was introduced from an analysis of the intensities of equivalent reflections. After averaging the intensities of equivalent reflections, only independent reflections were used.

The search for the model was carried out using the SHELXS program (G.M. Sheldrick, *Acta Cryst*, 2015, **C71**, 3) by direct methods. As a result, the coordinates of all non-hydrogen atoms are found. The resulting structure was refined by the least squares method using the SHELXL program. Complete information on the crystal structure is available from the Cambridge Crystallographic Data Center.

Crystallographic data for the structure reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication (CCDC **2182235**). These data can be obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

The ORTEP view of silatrane is shown in Figure 1 (main text), the selected bond lengths and bond angles are listed in Table S2. The independent part of the unit cell of contains six molecules of 1-chlorosilatrane and 2 molecules of MeCN (Figures S5 and S6).

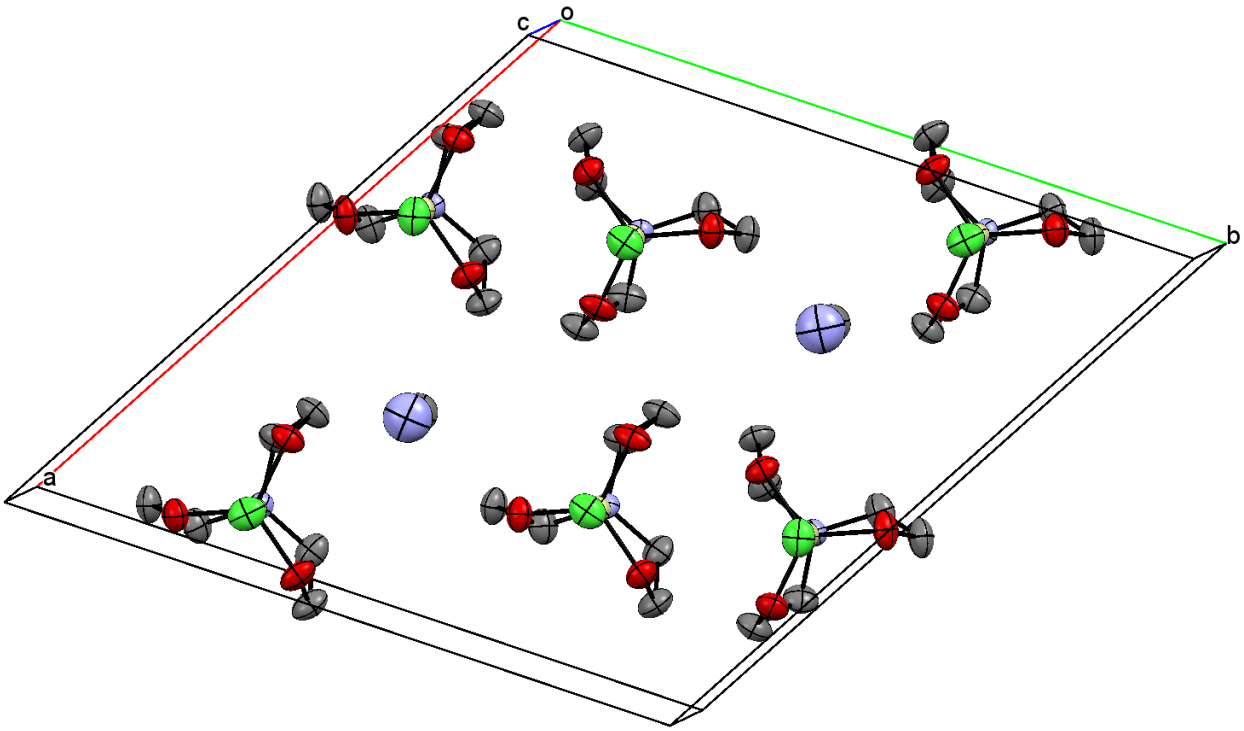


Figure S5. The independent part of the unit cell of contains 6 molecules of 1-chlorosilatrane and 2 molecules of MeCN

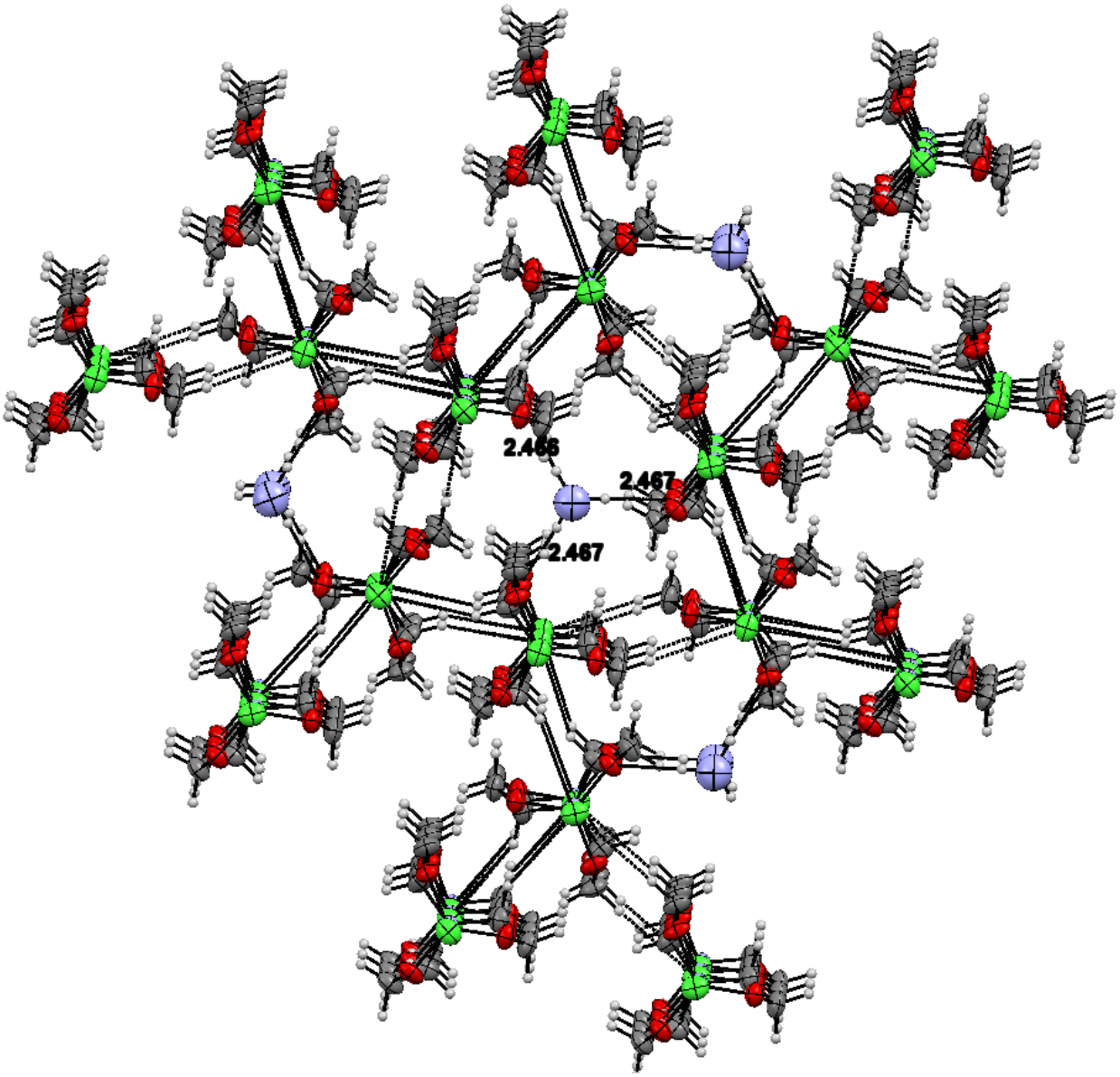


Figure S6. Fragment of a crystal package

Table S2. The main geometrical parameters of solvate 1-chlorosilatrane with MeCN

Atom1	Atom2	Length
N2	C7	1.128
C7	C8	1.459
C8	H	0.96
Cl1	Si1	2.160(4)
Si1	O	1.646(4)
Si1	N1	2.039(5)
O1	C1	1.435(5)
O2	C2	1.426(5)
O3	C3	1.425(4)
N1	C4	1.477(5)
N1	C5	1.475(7)
N1	C6	1.473(4)
C	H	0.971(7)
C1	C4	1.509(6)
C2	C5	1.523(5)
C3	C6	1.494(5)

Atom1	Atom2	Atom3	Angle
Cl1	Si1	O	93
Cl1	Si1	N1	179
O	Si1	O1	119
O	Si1	N1	87
Si1	O	C	118 - 119
Si1	N1	C	105
C	N1	C	112 - 113
O	C	C	109
N1	C	C	105 - 106