

## THE MOLECULAR STRUCTURE OF 1-FLUOROSILATRANE

LÁSZLÓ PÁRKÁNYI\*

*Central Research Institute of Chemistry of the Hungarian Academy of Sciences, H-1525 Budapest,  
P.O. Box 17 (Hungary)*

PÁL HENCSEI, LÁSZLÓ BIHÁTSI,

*Institute for Inorganic Chemistry, Technical University of Budapest, H-1521 Budapest (Hungary)*

and TIBOR MÜLLER

*Institute for General and Analytical Chemistry, Technical University of Budapest, H-1521 Budapest  
(Hungary)*

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### Summary

The crystal structure of 1-fluorosilatrane has been determined by X-ray diffraction. The N → Si dative bond length is 2.042(1) Å, somewhat longer than that in the 1-chloro derivative (2.023 Å). The carbon atoms linked to nitrogen are disordered. The atoms in the close-packed crystal structure are well fixed and have low thermal motion. Deformation electron density calculations show considerable density in the region between the silicon and nitrogen atoms. A mass spectrometric study indicates a strong Si-F bond.

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### Introduction

A wealth of structural information on silatranes has been gathered in recent years. Several attempts have been made to use geometric data in correlations with some physical properties.

One of these attempts was to correlate the inductive Taft constant of the substituent on the silicon atom with the N → Si dative bond length [1]:

$$d = 2.20 - 0.063\sigma_R^*$$

where  $d$  is the bond distance in Å and  $\sigma_R^*$  the inductive Taft constant. Applying the  $\sigma_F^* = 3.06$  value for fluorine, we obtain a dative bond length of 2.007 Å.

Another approach was to correlate the (mean) Sanderson electronegativity of the

substituent with the dative bond length [2]:

$$d = 3.021 X_R^{-0.257}$$

where  $X_R$  is the (mean) Sanderson electronegativity. The value of  $d$  calculated with  $X_F = 5.75$  is 1.927 Å.

The shortest N → Si approach ever found in silatranes was observed in 1-chlorosilatrane ( $d = 2.023$  Å [1]), thus both correlations predict an even shorter N → Si bond distance in agreement with the strong electron-withdrawing property of fluorine.

The  $^1\text{H}$  NMR signals of the hydrogen atoms in the  $\text{CH}_2$  groups attached to the nitrogen atom were found to be sensitive to the interaction between nitrogen and silicon. The  $\delta$ -values seem to be linearly correlated with the N → Si bond distance [3]:

$$\delta = -1.21 d + 5.46$$

The  $\delta$ -value of 2.95 measured in the title compound is smaller than that in the chloro derivative (3.01 [3]), therefore a longer N → Si bond may be expected ( $d = 2.074$  Å).

The primary intention of this paper is to supply the necessary structural data of the title compound.

## Experimental

### *Synthesis*

1-Fluorosilatrane was synthesized according to Frye et al. [4] from the reaction of 1-ethoxysilatrane with hydrogen fluoride in isopropanol solution. The crude product was extracted with ethanol. The melting point could not be determined due to the sublimable character of the compound ( $> 200^\circ\text{C}$ ). IR ( $\text{cm}^{-1}$ ): 325 vs, 380 vs, 475 w, 490 m, 588 m, 630 s, 650 vs, 750 vs, 800 vs, 884 s, 923 vs, 947 vs, 1027 vs, 1050 vs, 1095 vs, 1172 s, 1242 m, 1273 vs, 1350 s, 1390 m, 1460 vs, 1500 vs, 2900 vs, 2960 vs, 3005 s.

### *X-Ray structure determination*

The determination of the unit cell parameters and the collection of intensity data were performed on a computer-controlled Enraf–Nonius CAD-4 diffractometer at room temperature. Crystal data, data collection and least-squares parameters are summarized in Table 1.

The structure was solved by direct methods (MULTAN program, [5]). An ordered model could not be refined below the conventional  $R$  of 0.20 using isotropic thermal parameters. A difference map clearly showed that all the carbon atoms attached to nitrogen were disordered. The disordered atoms were then included with equal (0.5) multiplicities. Further two cycles of isotropic least-squares were calculated in which the multiplicities of the disordered atoms were also refined. At this stage ( $R = 0.156$ ), an empirical spherical absorption correction was applied using the DIFABS program [6]. The minimum and maximum absorption corrections were 0.465 and 1.567. The absorption correction caused  $R$  to drop to 0.086. The multiplicities of the disordered atoms were then fixed and all the hydrogen atomic positions were generated from assumed geometries. The multiplicities of the disordered hydrogen atoms were assigned according to those of the carbon atoms to which they were

TABLE 1  
CRYSTAL DATA, DATA COLLECTION AND LEAST-SQUARES PARAMETERS

Empirical formula	C <sub>16</sub> H <sub>12</sub> NO <sub>3</sub> SiF
<i>M</i> (a.m.u.)	193.3
<i>a</i> (Å)	7.153(1)
<i>b</i> (Å)	7.177(1)
<i>c</i> (Å)	10.775(1)
$\beta$ (°)	131.61(1)
<i>V</i> (Å <sup>3</sup> )	413.6(2)
Space group	<i>Pc</i>
<i>Z</i>	2
<i>D<sub>c</sub></i> (g cm <sup>-3</sup> )	1.552
$\lambda$ (Mo- <i>K<math>\alpha</math></i> ) (Å)	0.71073
$\mu$ (Mo- <i>K<math>\alpha</math></i> ) (cm <sup>-1</sup> )	2.59
2 $\theta$ limits, deg.	3–70
Scan technique	$\theta$ –2 $\theta$
Reflexions with non-zero intensity	1608
Reflexions used in least-squares, NO	1323 ( $F_o^2 \geq 2.0\sigma F_o^2$ )
Numbers of variables, NV	209 <sup>a</sup>
<i>R<sub>o</sub></i>	0.043
<i>R<sub>w</sub></i>	0.054
<i>R<sub>tot</sub></i>	0.06
$(\sum w( F_o  -  F_c )^2 / \text{NO} - \text{NV})^{1/2}$	2.72
Crystal size (mm)	0.02 × 0.33 × 0.55

<sup>a</sup> In two final cycles.

bonded. Non-hydrogen atoms were further refined by anisotropic least-squares. In two final least-squares cycles, the hydrogen atoms were also refined. The disordered atoms (both carbon and hydrogen) behaved well in refinement. The atomic scatter-

TABLE 2  
FINAL ATOMIC COORDINATES ( $\times 10^4$ ) AND *B*(eq)<sup>a</sup> VALUES (Å<sup>2</sup>) FOR THE NON-HYDROGEN ATOMS

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> (eq) Å <sup>2</sup>
Si(1)	0	3009.8(9)	0	1.417(7)
F	2262(3)	2898(4)	–3(2)	2.34(2)
O(2)	–1986(3)	1896(4)	–1772(2)	2.09(5)
O(8)	–46(5)	5291(3)	12(3)	1.77(5)
O(9)	1559(3)	1872(4)	1775(2)	2.90(5)
N(5)	–2859(2)	3142(3)	–8(2)	1.58(3)
C(3)	–4447(6)	1541(6)	–2455(4)	3.5(1)
C(4)	–5125(9)	2780(10)	–1731(6)	3.5(1) <sup>b</sup>
C(4)′	–4579(9)	1650(10)	–1097(6)	1.7(1) <sup>c</sup>
C(6)	–2788(9)	5010(10)	539(6)	2.1(1) <sup>b</sup>
C(6)′	–3900(10)	5010(10)	–563(8)	2.4(1) <sup>c</sup>
C(7)	–1794(8)	6246(5)	–7(5)	2.1(1)
C(10)	470(6)	1545(6)	2464(3)	3.0(1)
C(11)	–1628(8)	2780(10)	1752(5)	1.8(1) <sup>b</sup>
C(11)′	–2378(9)	1630(10)	1093(6)	1.3(1) <sup>c</sup>

<sup>a</sup> *B*(eq) is defined as 4/3 trace (*B.G*) where *B* is the thermal motion tensor and *G* is the real metric tensor. <sup>b,c</sup> Multiplicities: 0.56 and 0.44.

TABLE 3

FINAL ATOMIC COORDINATES ( $\times 10^3$ ), ISOTROPIC TEMPERATURE FACTORS ( $\text{\AA}^2$ ) AND C-H DISTANCES ( $\times 10^2$ ,  $\text{\AA}$ ) FOR THE HYDROGEN ATOMS

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i>	C-H
H(3a)	-446(6)	23(6)	-218(4)	6.0(9)	99(4)
H(3b)	-574(7)	143(5)	-362(4)	7(1)	95(3)
H(4a)	-570(10)	396(10)	-240(7)	5(2)	101(6) <sup>a</sup>
H(4b)	-664(10)	259(10)	-185(8)	6(2)	101(5) <sup>a</sup>
H(4a)'	-405(10)	48(10)	-46(7)	4(2)	99(7) <sup>b</sup>
H(4b)'	-625(9)	190(9)	-141(6)	2(1)	102(5) <sup>b</sup>
H(6a)	-191(8)	512(9)	158(6)	5(1)	85(3) <sup>a</sup>
H(6b)	-445(7)	521(8)	24(5)	3(1)	101(4) <sup>a</sup>
H(6a)'	-479(10)	549(10)	-37(7)	5(2)	86(5) <sup>b</sup>
H(6b)'	-498(10)	490(10)	-166(8)	4(2)	89(5) <sup>b</sup>
H(7a)	-98(6)	670(5)	104(4)	5.4(8)	92(2)
H(7b)	-252(5)	737(6)	-61(3)	4.7(7)	95(4)
H(10a)	158(6)	185(5)	377(4)	6(1)	108(2)
H(10b)	-22(6)	20(6)	216(4)	6.7(9)	103(4)
H(11a)	-287(8)	246(9)	185(5)	3(1)	99(4) <sup>a</sup>
H(11b)	-106(10)	410(10)	237(6)	4(1)	107(7) <sup>a</sup>
H(11a)'	-311(9)	59(10)	47(7)	3(1)	90(6) <sup>b</sup>
H(11b)'	-318(9)	150(10)	168(6)	2(1)	110(3) <sup>b</sup>

<sup>a,b</sup> Multiplicities: 0.56 and 0.44.

ing factors applied were taken from ref. 7. The final atomic parameters are given in Tables 2 and 3 \*.

### Mass spectrometric measurements

Mass spectrometric measurements were performed on a JEOL JMS-O1SG-2 mass spectrometer-data system. The electron-impact source was operated with 75 eV electrons with a trap current of 200  $\mu\text{A}$ . An ion accelerating voltage of 10 kV was employed. The pressure in the ion source was  $< 1 \times 10^{-6}$  mbar, the ion source temperature was  $\sim 400$  K. A direct sample inlet was applied. The resolution was  $R \sim 1200$  (low) and  $R \sim 8000$  and 10000 (high resolution). High-resolution spectra were recorded on-line with the data system and Ilford Q2 photoplates as well. Metastable ions were observed with a defocusing technique.

## Discussion

### The description of the crystal structure

A diagram of the molecule computed from the final atomic coordinates is shown in Fig. 1; bond lengths and angles are given in Table 4. The N  $\rightarrow$  Si approach (2.042(1)  $\text{\AA}$ ) is somewhat longer than that in the chloro derivative (2.023  $\text{\AA}$  [1]).

The partial disorder detected is of the usual type observed in other silatrane crystal structures ( $\alpha$ -1-phenyl- [8], *p*-tolyl- [9], 1-(*m*-chlorophenoxy)- [10] derivatives).

If we regard the silatrane skeleton as a system of three five-membered rings of

\* Lists of structure factors and anisotropic thermal parameters can be obtained from the authors.

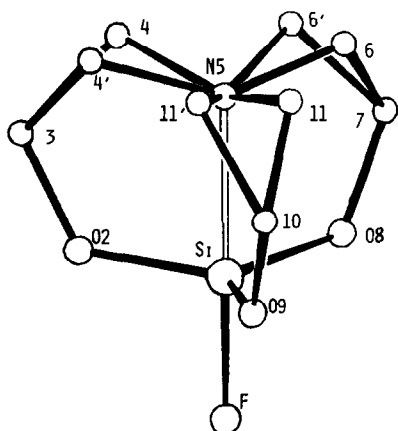


Fig. 1. Molecular diagram of the title compound showing the numbering of atoms. Numbers without a prefix are for carbon atoms. Hydrogen atoms are omitted for clarity.

TABLE 4  
BOND LENGTHS (Å) AND ANGLES (°) WITH THEIR E.S.D.'S

<i>Bond lengths</i>					
Si(1)–F	1.622(1)	O(9)–C(10)	1.408(2)	C(3)–C(4)	1.461(6)
Si(1)–O(2)	1.643(2)	N(5)–C(4)	1.465(5)	C(3)–C(4)′	1.530(3)
Si(1)–O(8)	1.638(2)	N(5)–C(4)′	1.454(6)	C(6)–C(7)	1.480(6)
Si(1)–O(9)	1.654(2)	N(5)–C(6)	1.454(7)	C(6)′–C(7)	1.487(7)
Si(1)–N(5)	2.042(1)	N(5)–C(6)′	1.455(8)	C(10)–C(11)	1.450(7)
O(2)–C(3)	1.409(4)	N(5)–C(11)	1.490(3)	C(10)–C(11)′	1.529(6)
O(8)–C(7)	1.414(4)	N(5)–C(11)′	1.471(6)		
<i>Bond angles</i>					
F–Si(1)–O(2)	93.2(2)	C(4)–N(5)–C(11)′	111.1(5)		
F–Si(1)–O(8)	94.3(2)	C(4)′–N(5)–C(6)	142.1(5)		
F–Si(1)–O(9)	94.0(2)	C(4)′–N(5)–C(6)′	114.7(6)		
F–Si(1)–N(5)	179.7(1)	C(4)′–N(5)–C(11)	111.6(5)		
O(2)–Si(1)–O(8)	119.3(2)	C(4)′–N(5)–C(11)′	74.2(5)		
O(2)–Si(1)–O(9)	120.1(2)	C(6)–N(5)–C(11)	79.1(5)		
O(2)–Si(1)–N(5)	85.8(1)	C(6)–N(5)–C(11)′	115.4(5)		
O(8)–Si(1)–O(9)	119.1(2)	C(6)′–N(5)–C(11)	113.0(6)		
O(8)–Si(1)–N(5)	85.9(1)	C(6)′–N(5)–C(11)′	142.6(6)		
O(9)–Si(1)–N(5)	86.1(1)	O(2)–C(3)–C(4)	110.7(5)		
Si(1)–O(2)–C(3)	120.7(3)	O(2)–C(3)–C(4)′	109.9(5)		
Si(1)–O(8)–C(7)	120.4(3)	N(5)–C(4)–C(3)	107.8(7)		
Si(1)–O(9)–C(10)	120.5(3)	N(5)–C(4)′–C(3)	104.7(6)		
Si(1)–N(5)–C(4)	105.0(4)	N(5)–C(6)–C(7)	106.7(6)		
Si(1)–N(5)–C(4)′	106.1(4)	N(5)–C(6)′–C(7)	106.3(7)		
Si(1)–N(5)–C(6)	106.0(4)	O(8)–C(7)–C(6)	111.0(5)		
Si(1)–N(5)–C(6)′	106.3(4)	O(8)–C(7)–C(6)′	111.4(6)		
Si(1)–N(5)–C(11)	104.1(4)	O(9)–C(10)–C(11)	111.1(5)		
Si(1)–N(5)–C(11)′	105.4(4)	O(9)–C(10)–C(11)′	109.4(5)		
C(4)–N(5)–C(6)	113.0(6)	N(5)–C(11)–C(10)	108.0(6)		
C(4)–N(5)–C(6)′	79.1(6)	N(5)–C(11)′–C(10)	104.9(6)		
C(4)–N(5)–C(11)	143.5(6)				

TABLE 5

OBSERVED AND CALCULATED VALUES OF THE GEOMETRICAL PARAMETERS  $\Delta\text{Si}$  (Å),  $\Delta\text{N}$  (Å) AND  $\alpha(\text{N}-\text{Si}-\text{O})$  (°)

Parameter	Experimental	Calculated	Calculated
$\Delta\text{Si}$	0.117(3)	0.133 <sup>b</sup>	0.126 <sup>c</sup>
$\Delta\text{N}$	0.390(3) <sup>d</sup>	0.416 <sup>e</sup>	
$\alpha$	85.9	85.7 <sup>d</sup>	85.4 <sup>f</sup>

<sup>a</sup> Plane formed by the C(4),C(4)',C(6),C(6)',C(11),C(11)' atoms. <sup>b</sup>  $\Delta\text{Si} = 0.488d - 0.863$  [2]. <sup>c</sup>  $\Delta\text{N} = -0.391d + 1.214$  [2]. <sup>d</sup>  $\alpha = -20.0d + 126.5$  [2]. <sup>e</sup>  $\Delta\text{Si} = 0.62d - 1.14$  [1]. <sup>f</sup>  $\alpha = 120.9 - 17.4d$  [11].

envelope conformation linked to each other by two annellation atoms (Si(1) and N(5)), then the carbon atoms attached to nitrogen are on the flaps of the envelopes. In partially disordered silatrane moieties, the images of the atoms sitting on the flaps appear as if reflected on the other side of the planes formed by the remaining atoms (Si, O, C, N) of the five-membered rings. The average distance of disordered atoms from this plane is 0.47(3) Å in the title compound.

The deviation of the silicon atom ( $\Delta\text{Si}$ ) from the plane of the three (equatorial) oxygen atoms, the deviation of the nitrogen atom ( $\Delta\text{N}$ ) from the plane formed by its substituent carbon atoms, and the mean N–Si–O bond angle ( $\alpha$ ) have been shown [1,2,11] to correlate with the N → Si distance. The observed and calculated values of these parameters are listed in Table 5.

Published Si–F bond distances range from 1.56 to 1.69 Å and the majority of these were observed in SiF<sub>3</sub> groups. Only a few examples could be found in the literature where fluorine is linked to a five-coordinated silicon atom. A 1.668 Å long Si–F (axial) bond was reported [12] for [(CH<sub>3</sub>)<sub>3</sub>PNSiF<sub>3</sub>]<sub>2</sub> and a Si–F (ax) bond length of 1.620 Å was observed in *o*-ClC<sub>6</sub>H<sub>4</sub>COOCH<sub>2</sub>SiF<sub>3</sub> [13]. Equatorial Si–F bonds are shorter by 0.04–0.06 Å. The 1.622(1) Å Si–F bond distance in the title compound falls in the medium range.

### Charge-density maps

The nature of the interaction between nitrogen and silicon in silatranes is still an unsolved problem. Beyond the simple, formal *sp*<sup>3</sup>*d* hybridization model, the formation of a “hypervalent” bond was also suggested [14] and its MO diagram constructed [15]. Although a final and exact answer to the question of the nature of this peculiar interaction is beyond the scope of this paper, we investigated whether there is any significant electron density in the region between nitrogen and silicon.

The rather high density of the crystals ( $D = 1.55 \text{ g cm}^{-3}$ ) compared to other silatranes indicates a close-packed crystal structure. The low thermal parameters (cf.  $B(\text{eq})$  values in Table 2) obtained from least-squares refinement show that in this close-packed structure the atoms are well fixed. The considerable scattering power at relatively high  $\theta$ -values even at room temperature also corroborates the low degree of thermal motion. Four cycles of least-squares refinement were calculated for the non-hydrogen atoms using 620 reflexions ( $F_o^2 \geq 1\sigma(F_o^2)$ ,  $\sin \theta/\lambda = 0.60\text{--}0.85$ ). The starting conventional  $R$  of 0.082 for this set of data dropped to 0.067 during refinement. The atomic parameters obtained in this way were used in structure factor calculation for all reflexions ( $R = 0.058$ ) and difference Fourier synthesis in order to obtain deformation type electron densities. Two electron density maps

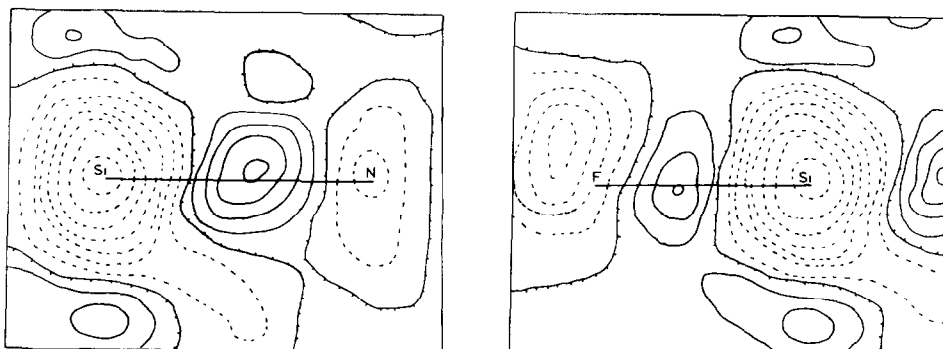


Fig. 2. Deformation electron density maps in the Si(1), O(2), N(5) and the Si(1), O(2), F plane. Contours are drawn at  $0.1 \text{ e}/\text{\AA}^3$  interval and broken lines represent negative values (estimated error is ca.  $0.1 \text{ e}/\text{\AA}^3$ ).

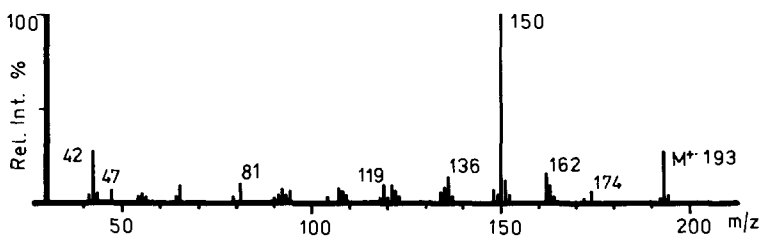


Fig. 3. Low-resolution mass spectrum of the title compound.

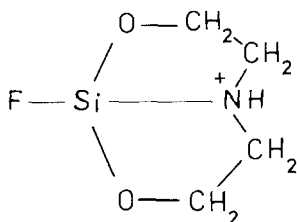


Fig. 4. Assumed structure of the ion at  $m/z$  150.

containing the Si–N and the Si–F bond regions are depicted in Fig. 2. The maps are rather clean and contain negative areas at atomic sites. The region between nitrogen and silicon clearly shows electron density which looks like a “bond” (cf. the Si–F bond region for reference). The maximum peak height of  $0.4 \text{ e}/\text{\AA}^3$  is at the midpoint between the zero contour lines encircling the atoms.

#### Mass spectrometric studies

Earlier MS studies [16–19] on 1-substituted silatranes established the following characteristic features:

- the mass spectra contain a few peaks of low intensity;
- the  $M^+$  ion has low or medium intensity;
- the base-peak is the  $[M - R]^+$  ( $m/z$  174) “silatranyl” ion.

Two different fragmentation patterns are observed with and without the cleavage

TABLE 6  
HIGH RESOLUTION MASS SPECTRAL DATA OF 1-FLUOROSILATRANE

$m/z$	Elemental composition	Exact masses	
		measured	calculated
193	$[\text{C}_6\text{H}_{12}\text{FNO}_3\text{Si}]^+; \text{M}^+$	193.0572	193.0570
192	$[\text{C}_6\text{H}_{11}\text{FNO}_3\text{Si}]^+$	192.0483	192.0492
174	$[\text{C}_6\text{H}_{12}\text{NO}_3\text{Si}]^+$	174.0594	174.0586
172	$[\text{C}_6\text{H}_{10}\text{NO}_3\text{Si}]^+$	172.0435	172.0430
163	$[\text{C}_5\text{H}_{10}\text{FNO}_2\text{Si}]^+$	163.0470	163.0465
162	$[\text{C}_5\text{H}_9\text{FNO}_2\text{Si}]^+$	162.0421	162.0387
150	$[\text{C}_4\text{H}_9\text{FNO}_2\text{Si}]^+$	150.0385	150.0387
148	$[\text{C}_4\text{H}_7\text{FNO}_2\text{Si}]^+$	148.0228	148.0230
136	$[\text{C}_3\text{H}_7\text{FNO}_2\text{Si}]^+$	136.0269	136.0230
134	$[\text{C}_3\text{H}_5\text{FNO}_2\text{Si}]^+$	134.0135	134.0074
121	$[\text{C}_3\text{H}_6\text{FO}_2\text{Si}]^+$	121.0060	121.0121
119	$[\text{C}_3\text{H}_6\text{FNOSi}]^+$	119.0202	119.0203
108	$[\text{C}_2\text{H}_5\text{FO}_2\text{Si}]^+$	108.0023	108.0043
95	$[\text{CH}_4\text{O}_2\text{Si}]^+$	94.9944	99.9965
47	$[\text{SiF}]^+$	46.9790	46.9753
43	$[\text{C}_2\text{H}_3\text{O}]^+$	43.0218	43.0184
	$[\text{C}_2\text{H}_3\text{N}]^+$	43.0431	43.0422
42	$[\text{C}_2\text{H}_4\text{N}]^+$	42.0389	42.0344

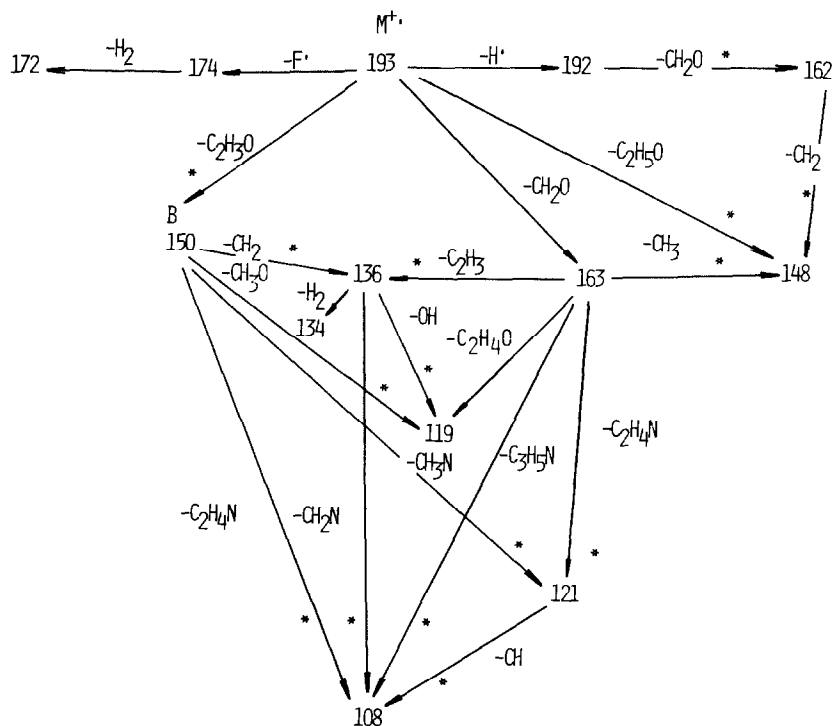


Fig. 5. Fragmentation mechanism of the title compound.



of the R–Si bond. The two fragmentation mechanisms obviously depend strongly on the stability of the R–Si bond.

Because of the great electronegativity difference between F and Si, the formation of a stable F–Si bond is expected which may affect the mass spectroscopic behaviour of the title compound. The mass spectrum is shown in Fig. 3. The molecular ion  $M^{++}$  ( $m/z$  193) is of ca. 27% intensity. Because of the stable F–Si bond the base peak is the  $[M - C_2H_3O]^+$  ion ( $m/z$  150) (Fig. 4), instead of the silatranyl ion ( $m/z$  174). The cleavage of the F–Si bond is hardly observable (< 5%). The stability of the F–Si bond is further substantiated by a peak of ca. 5% intensity at  $m/z$  47 which corresponds, according to the high-resolution measurements, to the  $SiF^+$  ion (Table 6). Metastable transitions obtained by the defocusing technique are shown in Fig. 5. Besides the characteristic  $CH_2O$ ,  $C_2H_3O$  and  $C_2H_4O$  fragments,  $CH_2N$ ,  $CH_3N$ ,  $C_2H_4N$ , etc. are also observed.

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