Journal of Fluorine Chemistry 131 (2010) 719–723

Contents lists available at ScienceDirect

Journal of Fluorine Chemistry

journal homepage: www.elsevier.com/locate/fluor

Interaction of hexafluorosilicic acid with sulfa drugs. Bis(sulfathiazolium) hexafluorosilicate: Spectral data and crystal structure

Vladimir O. Gelmboldt [a], Eduard V. Ganin [b], Larisa V. Koroeva [c], Mark M. Botoshansky [d], Marina S. Fonari [e,*]

[a] Odessa State Medical University of Ministry of Health of Ukraine, Valikhovskiy lane, 2, 65026, Odessa, Ukraine
[b] Odessa State Environmental University of Ministry of Education and Science of Ukraine, Lvovskaya str., 15, 65016, Odessa, Ukraine
[c] Physico-Chemical Institute of Environment and Human Protection of Ministry of Education and Science of Ukraine and National Academy of Sciences of Ukraine, Preobrazhenskaya str., 3, 65082, Odessa, Ukraine
[d] Schulich Faculty of Chemistry, Technion-Israel Institute of Technology, Technion City, 32000 Haifa, Israel
[e] Institute of Applied Physics, Academy of Sciences of Moldova, Academy str., 5, MD2028 Chisinau, Republic of Moldova

ARTICLE INFO

Article history:
Received 27 December 2009
Received in revised form 10 March 2010
Accepted 12 March 2010
Available online 23 March 2010

Keywords:
Hexafluorosilicate
Sulfa drugs
Solubility
Hydrogen bonding
Crystal structure

ABSTRACT

Interaction of hexafluorosilicic acid with sulfa drugs sulfathiazole (**stz**) and sulfalen (2-sulfanilamido-3-methoxypyrazine, **sl**) results in the crystalline salts of the compositions [**stz**H]$_2$[SiF_6] (**I**) and [4-$H_2NO_2SPhNH_3$]$_2$[SiF_6] (**II**). Complex **I** is characterized by IR, mass spectrometry data and single crystal X-ray diffraction. The crystal structure of **I** is stabilized by a network of charge-assisted hydrogen bonding. The relationship between the solubility and H-bonding system in **I**, **II** and related "onium" hexafluorosilicates is discussed. The formation of complex **II**, previously reported as an interaction product of hexafluorosilicic acid with 4-aminobenzenesulfonamide (sulfanilamide), is the result of cleavage of the C–N bond in sulfalen in acidic medium.

1. Introduction

The products of interaction of hexafluorosilicic acid with N-, O-containing organic bases, "onium" fluorosilicates, mainly hexafluorosilicates are of inextinguishable interest as chemical reagents and functional materials for various purposes [1]. As it has been previously noted [1], the system of interionic XH···F (X = N, O) H-bonds in the solid "onium" fluorosilicates has a significant impact on the structural characteristics and properties of the final salts. In particular, the observed inverse-proportional relationship [2,3] between the pyridinium hexafluorosilicates' water solubility and a number of short interionic H-bonds in the crystalline salts has an exponential character. The functionalization of the "onium" cation by the H-donor substituents, differing in the number and nature of the H-donor centers, might serve as a convenient method to detect the directional changes in physico-chemical characteristics of the "onium" hexafluorosilicates.

This work continues the previous research on the correlations between the structure and properties of "onium" hexafluorosilicates [2,3] and is devoted to the interaction products formed by the sulfa drugs sulfathiazole (**stz**) and sulfalen (**sl**) with hexafluorosilicic acid. This choice is explained by our interest to the co-crystals of sulfa drugs, products of their transformations or other active pharmaceutical ingredients (APIs) [4] possessing H-donor/H-acceptor functions with co-crystal partners of different nature [5,6]. In recent years, this topic has received increased attention [7,8]. Sulfa drugs of sulfonamides are a group of antibiotics used as agricultural herbicides and in the treatment of infections of respiratory and urinary tracts in humans. These antibiotics were repeatedly found in aquatic media at concentrations ranging from 0.13 to 1.9 μg/L [9,10]. Members of this class of compounds differ in the N-bound substituent of the sulfonamide linkage. Numerous methods of free-radical-induced oxidative and reductive degradation of sulfa drugs containing heterocyclic five- and six-membered rings have been studied and the authors concluded that the degradation occurs through cleavage at various positions with the formation of sulfanilic acid 4-H_2NPhSO_3H, sulfanilamide 4-$H_2NPhSO_2NH_2$, and aniline $PhNH_2$ as the possible degradation products. On the other hand, it is known that, in acidic media, the sulfa drugs are easily protonated with the formation of cationic forms [9].

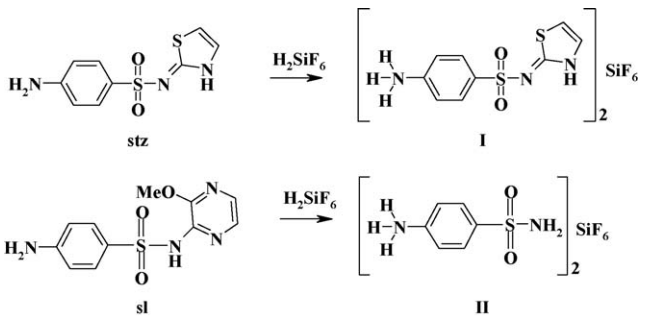
* Corresponding author. Tel.: +373 22 73 81 54; fax: +373 22 72 58 87.
E-mail addresses: vgelmboldt@te.net.ua (V.O. Gelmboldt), edganin@gmail.com (E.V. Ganin), botoshan@tx.technion.ac.il (M.M. Botoshansky), fonari.xray@phys.asm.md, fonari.xray@gmail.com (M.S. Fonari).

0022-1139/$ – see front matter © 2010 Elsevier B.V. All rights reserved.
doi:10.1016/j.jfluchem.2010.03.007

The interaction of hexafluorosilicic acid with sulfa drugs sulfathiazole **stz** and sulfalen **sl** [11] results in the crystalline salts of the compositions [**stz**H]$_2$[SiF$_6$] (**I**) and [4-H$_2$NO$_2$SPhNH$_3$]$_2$[SiF$_6$] (**II**) [12]. The preparation, spectral and solubility data alongside with the crystal structure for **I** are reported herein.

2. Results and discussion

We have previously shown that interaction of hexafluorosilicic acid with *p*-aminobenzoic acid, sulfanilamide and 5-amino-1-benzyl-1,2,3-triazol-4-carbonic acid yielded the corresponding hexafluorosilicates with the aromatic cations formed due to protonation of the terminal amino group [6,12,13]. In a similar way, we expected the formation of the corresponding hexafluorosilicates starting from the sulfa drugs sulfathiazole and sulfalen. However, according to the data of elemental analysis and XRD, the crystalline compound isolated from the reaction solution of hexafluorosilicic acid – **sl** – H$_2$O appeared to be a hexafluorosilicate with the composition [4-H$_2$NO$_2$SC$_6$H$_4$NH$_3$]$_2$[SiF$_6$] (**II**). The formation of **II**, previously reported in [12], can be considered as the result of hydrolytic transformation of sulfalen molecule in the acidic medium due to the cleavage of C–N covalent bond [14] (Scheme 1). The possible reaction by-product with the pyrazine cycle or the product of its degradation does not form a crystalline compound in the reaction conditions and has not been identified.



Scheme 1. Schematic pathway to the formation of crystalline salts **I** and **II**.

Sulfathiazolium hexafluorosilicate (**I**) was obtained by interaction of **stz** with an aqueous solution of hexafluorosilicic acid. Table 1 summarizes the IR spectroscopic characteristics for **I**. Vibrations ν(NH) of the secondary amino group are registered near 3525 and 3445 cm^{-1}, whereas the vibrations of –N$^+$H$_3$ group and –NH fragment of the thiazole ring are identified as a series of bands of average intensity in the range 3260–2630 cm^{-1}. The bands registered at 1320 and 1150 cm^{-1} are assigned to the $\nu_{as,s}$(SO$_2$) vibrations, respectively, and their positions are close to the corresponding absorption bands in the IR spectrum of **II** (1320 and 1175 cm^{-1}) [12]. The deformation vibrations of the cation are also visible in the range of the stretching vibrations of the phenyl and thiazole rings ν_{ring} (1650–1505 cm^{-1}) and in the range of 750–680 cm^{-1} of the ν(SiF) vibrations of the anion (Table 1). At the same time, the region of the δ(SiF$_2$) vibrations is free of the other types of vibrations: a triplet structure of the δ(SiF$_2$) band indicates in favor of the decrease of symmetry of [SiF$_6$]$^{2-}$ anion with regard to O_h that is explained by the resulting interionic H-interactions.

Being one of the most important physical and chemical properties of the pharmaceuticals, the water solubility is crucial for solving the problems of chemical technology, ecology, and pharmacology [15]. As we have recently shown [3] the water solubility (**C**) of a number of pyridinium hexafluorosilicates correlates with the parameter $\mathbf{h = n/d(D\cdots A)_{av}}$, where **n** is a number of short interionic contacts (H-bonds) and $\mathbf{d(D\cdots A)_{av}}$ is an average donor(D)–acceptor(A) distance in the salt crystal structure (taking into account contacts with D$\cdots$A $\leq$ 3.2 Å, (D)–H$\cdots$A $\leq$ 2.6 Å that correspond to strong and moderate H-bonds following the classification suggested by Steiner [16]). The functional dependence of **C** against *h* has an exponential character. Table 2 demonstrates the water solubility data for **I**, **II**, and some other heterocyclic "onium" hexafluorosilicates. As it follows from Table 2, the solubility of **I**, **II**, and [C$_2$H$_6$N$_5$]$_2$[SiF$_6$] (where C$_2$H$_6$N$_5$ is a 3,5-diamino-1,2,4-triazolium cation) fits well within the specified dependency, reflecting the decrease in solubility of compounds with the increasing number of short interionic H-bonds. We do not exclude that the observed relationship between **C** and **h** may be of a common character for the "onium" hexafluorosilicates with the "onium" aromatic cations.

Sulfathiazole has a remarkable solvate-forming ability. Many solvent-containing sulfathiazoles are known and their structures

Table 1
IR spectral data (cm^{-1}) for hexafluorosilicate **I**.

ν(NH)	ν(N$^+$H$_n$)	ν_{ring}*	$\nu_{as,s}$(SO$_2$)	$\nu_{puls.}$	ν(SN)	ν(SiF)**	δ(SiF$_2$)
3525 m.	3260 m.	1650 sh.	1320 m.	1085 s.	915 m.	750 s.	480 sh.
3445 m.	3050 m.	1630 sh.	1150 m.	1015 m.		720 v.s.	455 m.
	2710 m.	1595 m.				680 s.	445 m.
	2630 m.	1570 s.					
		1525 sh.					
		1505 sh.					

m. = medium, s. = strong, v.s. = very strong, sh. = shoulder.
* Also include deformation vibrations δ(HNC), δ(N$^+$H$_3$).
** Also may include deformation vibrations r(N$^+$H$_3$), r(CNH), δ(CCH).

Table 2
Solubility of **I**, **II** and related onium hexafluorosilicates in water.

Compound	Solubility/mol.%, 25 °C	Standard deviation, *S*	*h*/Å^{-1}	References
I	0.10 ± 0.0007	0.0008	2.03	Present work
II	0.06 ± 0.004	0.004	2.10	[12]
[2,6-(H$_2$N)$_2$C$_5$H$_3$NH]$_2$[SiF$_6$]	0.06 ± 0.003	0.004	2.35	[3]
[C$_2$H$_6$N$_5$]$_2$[SiF$_6$]*	0.28 ± 0.016	0.019	1.78	[17]
[3-HO(O)CC$_5$H$_4$NH]$_2$[SiF$_6$]	3.33 ± 0.121	0.138	1.09	[3,18]
[2-HO(O)CC$_5$H$_4$NH]$_2$[SiF$_6$]	5.33 ± 0.251	0.286	0.76	[3]
[2-CH$_3$C$_5$H$_4$NH]$_2$ [SiF$_6$]	11.60 ± 0.82	0.93	0.71	[3,19]

* C$_2$H$_5$N$_5$ = 3,5-diamino-1,2,4-triazole.

have been previously reported [20,21]. Besides, Caira et al. [22] reported the crystal structure of the 1:1 complex of sulfathiazole and cyclodextrin, in which the molecules are hydrogen bonded with each other. In the sulfathiazolium nitrate monohydrate [23] the drug molecule is protonated on its terminal amino group. The planes of the benzene and thiazole rings are inclined in a *gauche* conformation about the S–N bond with a dihedral angle of 87.63(6)°. The crystal structure is stabilized by a network of hydrogen bonding.

Bis(sulfathiazolium) hexafluorosilicate **I** crystallizes in a monoclinic crystal system (space group *C*2/*c* with $Z = 4$). The crystal structure and refinement data for **I** is given in Table 3. Selected bond distances and angles are given in Table 4, while hydrogen-bonding geometry is summarized in Table 5. The formula unit comprises two [**stz**H]$^+$ cations that occupy general positions and one hexafluorosilicate anion that resides on the twofold axis (Fig. 1).

The positions of the N-bound hydrogen atoms were found in the difference Fourier map. The cation is characterized by the common geometry with the S1–O1, S1–O2 and N2–C7 bond distances being in agreement with the double-bond character for sulfoxide and imine groups, respectively. In the sulfathiazolium cation the planes of the benzene and thiazole rings are inclined in a *gauche* conformation about the S–N bond with a C7–N2–S1–C4 dihedral angle of 95.8(2)°. Only major position for the disordered $[SiF_6]^{2-}$ anion is further discussed. The $[SiF_6]^{2-}$ anion has a geometry of a slightly distorted octahedron with the Si–F bond distances range of 1.654(2)–1.679(2) Å and maximal deviation of the *cis*-F–Si–F angle from the right one equal to 1.3°. The crystal structure is stabilized by the network of hydrogen bonding. All fluorine atoms are involved in the hydrogen-bonding system (Table 5). Each $[SiF_6]^{2-}$ anion has NH···F contacts with eight closest [**stz**H]$^+$ cations, while each cation bridges two $[SiF_6]^{2-}$ anions *via* terminal ammonia group and binds one more $[SiF_6]^{2-}$ anion *via* the imine NH– site (Fig. 2(a)). The self-assembling of the [**stz**H]$^+$ cations occurs *via* a couple of the inversion-related N1···N2 hydrogen bonds; N···N separation being 2.925(3) Å (Fig. 2(b)).

The crystal packing represents the alternation of the H-bonded rows of $[SiF_6]^{2-}$ anions and [**stz**H]$^+$ cations. Thus, the interaction of hexafluorosilicic acid with sulfathiazole results in the formation of bis(sulfathiazolium) hexafluorosilicate, whereas the reaction involving sulfalen leads to its hydrolysis with the cleavage of the C–N bond and formation of bis[(4-aminosulfonyl)benzeneammonia] hexafluorosilicate. The solubility of the studied salts, as for the previously studied hexafluorosilicates with functionalized pyridinium cations [2,3], correlates with the number of short interionic H-bonds in their structures.

3. Experimental

Commercially available reagents were used as received. Compounds **I**–**II** were analysed for C, H, N and S in a Perkin Elmer 240C. The IR-absorption spectra were recorded on a spectrophotometer Specord 75IR (range 4000–400 cm^{-1}, samples as

Table 3
Crystal data and structure refinement parameters for **I**.

	I
Empirical formula	$(C_9H_{10}N_3O_2S_2)_2[SiF_6]$
Formula weight	654.73
Crystal system	Monoclinic
Space group	*C*2/*c*
Unit cell dimensions	
a (Å)	17.723(3)
b (Å)	11.684(2)
c (Å)	11.938(2)
β (°)	91.78(3)
Cell volume (Å^3)	2470.9(7)
Z	4
D_{calc} (g/cm^3)	1.760
μ (mm^{-1})	0.520
F(000)	1336
θ range for data collection (°)	2.30–25.02
Limiting indices	$-21 \le h \le 20$ $-13 \le k \le 13$ $-14 \le l \le 12$
Reflections collected	10,129
Reflections with $I > 2\sigma(I)$	1908
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	2175/87/218
Goodness-of-fit on *F*2	1.021
Final *R* indices [$I > 2\sigma(I)$]	$R_1 = 0.0389$ $wR_2 = 0.1054$
R indices (all data)	$R_1 = 0.0448$ $wR_2 = 0.1086$
Largest diff. peak and hole (e Å^{-3})	0.431/−0.269

Table 4
Selected intermolecular distances (Å) and angles (°) for **I**.

	I		**I**
Si(1)–F(1)	1.675(4)	Si(1)–F(3)	1.679(2)
Si(1)–F(2)	1.664(4)	Si(1)–F(4)	1.654(2)
F(1)–Si(1)–F(2)	180.00(1)	F(2)–Si(1)–F(3)	89.77(12)
F(1)–Si(1)–F(3)	90.23(12)	F(2)–Si(1)–F(4)	89.09(14)
F(1)–Si(1)–F(4)	90.91(14)	F(3)–Si(1)–F(4)	91.31(12)
S(1)–O(1)	1.435(2)	S(1)–O(2)	1.430(2)
S(1)–N(2)	1.598(2)	S(1)–C(4)	1.781(2)
S(2)–C(8)	1.725(4)	S(2)–C(7)	1.726(3)
N(1)–C(1)	1.469(3)	N(2)–C(7)	1.333(3)
N(3)–C(7)	1.341(3)	N(3)–C(9)	1.375(4)
C(8)–C(9)	1.313(5)		
O(2)–S(1)–O(1)	119.18(14)	C(2)–C(1)–N(1)	120.28(19)
O(2)–S(1)–N(2)	111.82(12)	C(6)–C(1)–N(1)	118.1(2)
O(1)–S(1)–N(2)	106.16(12)	C(5)–C(4)–S(1)	118.69(16)
O(2)–S(1)–C(4)	107.10(11)	C(3)–C(4)–S(1)	120.57(18)
O(1)–S(1)–C(4)	105.30(11)	N(2)–C(7)–N(3)	119.7(2)
N(2)–S(1)–C(4)	106.46(10)	N(2)–C(7)–S(2)	130.7(2)
C(8)–S(2)–C(7)	90.54(17)	N(3)–C(7)–S(2)	109.53(19)
C(7)–N(2)–S(1)	121.53(18)	C(9)–C(8)–S(2)	112.1(2)
C(7)–N(3)–C(9)	115.1(3)	C(8)–C(9)–N(3)	112.8(3)
C(2)–C(1)–C(6)	121.6(2)		

Note: The data are given only for the major position of $[SiF_6]^{2-}$ anion.

Table 5
Geometry of hydrogen bonds for **I**.

D–H···A	*d*(D–H)/Å	*d*(H···A)/Å	*d*(D···A)/Å	∠(DHA)/°	Symmetry transformation for acceptor
N(1)–H(1C)···F(3)	0.85(3)	2.04(2)	2.884(3)	170(2)	$x, 1-y, 1/2+z$
N(1)–H(1A)···F(1)	0.88(2)	2.09(2)	2.936(2)	159(2)	x, y, z
N(1)–H(1A)···F(3)	0.88(2)	2.29(3)	2.869(3)	123(2)	$-x, y, 1/2-z$
N(1)–H(1B)···N(2)	0.89(2)	2.07(2)	2.925(3)	159(3)	$-x, -y, 1-z$
N(3)–H(3A)···F(4)	0.90(4)	2.06(4)	2.931(4)	163(3)	$x, y-1, z$
N(3)–H(3A)···F(2)	0.90(3)	2.47(4)	3.177(3)	135(3)	$x, y-1, z$

Note: The data are given only for the major position of $[SiF_6]^{2-}$ anion.

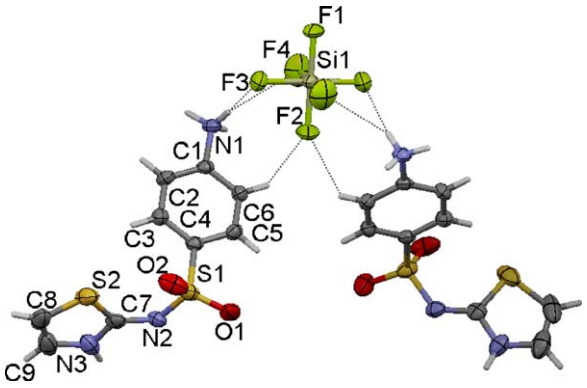


Fig. 1. ORTEP drawing for **I** showing hydrogen bonding. Only the asymmetric unit is numbered and only one position for the disordered $[SiF_6]^{2-}$ anion is shown. Thermal ellipsoids are drawn for 50% probabilities.

suspension in Nujol mulls between KRS-5 windows). The mass spectra were registered on a spectrometer MX-1321 (direct input of a sample in a source, energy of ionizing electrons 70 eV). The isothermal conditions of experiments for detection of the solubility of hexafluorosilicates ($t = 25 \pm 0.2$ °C) were provided with the help of an ultra thermostat U15.

3.1. Synthesis of 4-amino-N-(1,3-thiazol-2-ium)benzenesulfonamide hexafluorosilicate (I)

4-Amino-*N*-(1,3-thiazol-2-yl)benzenesulfonamide (0.255 g, 1 mmol) was dissolved in methanol (15 mL) and hexafluorosilicic acid (45%, 2 mL) was then added to the solution. The reaction mixture was stored under ambient conditions prior to the beginning of crystallization of the reaction product, which was obtained with a quantitative yield. The reaction product was a colorless transparent crystals of the composition (**stz**H)$_2$[SiF$_6$] (**I**) with m.p. 232–234 °C (with decomposition). Anal. found, %: Si 4.11, N 12.93, F 19.18. Calcd. for $C_{18}H_{20}F_6N_6O_4S_4Si$, Si 4.29, N 12.84, F 17.41. Mass spectrum: [M**stz**]$^+$ (m/z = 255, I = 14%), [M**stz**–SO_2]$^+$ (m/z = 191, I = 66%), [C_6H_5NH]$^+$ (m/z = 90, I = 100%).

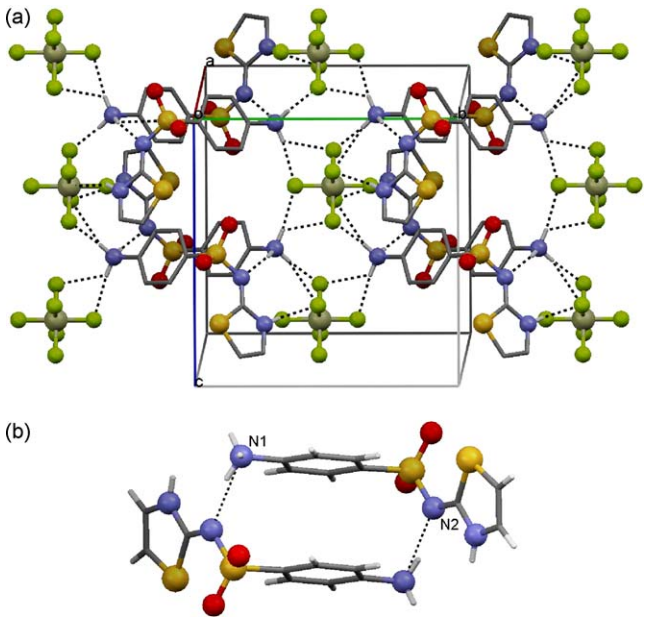


Fig. 2. Fragment of crystal packing in **I**, view along *a*-axis. C-bound H atoms are omitted for clarity (a); self-association pattern of [**stz**H]$^+$ cations in **I** (b).

3.2. Synthesis of the product of interaction of hexafluorosilicic acid with the 4-amino-N-(3-methoxypyrazin-2-yl)benzenesulfonamide, bis(4-ammoniumbenzenesulfonamide) hexafluorosilicate (II)

4-Amino-N-(3-methoxypyrazin-2-yl)benzenesulfonamide (0.280 g, 1 mmol) was dissolved in methanol (7 mL) and hexafluorosilicic acid (45%, 2 mL) was then added to the solution. The reaction mixture was stored under ambient conditions prior to the beginning of crystallization of the reaction product which was obtained with a quantitative yield. The reaction product was a colorless transparent crystals of the composition $(C_6H_9N_2O_2S)_2[SiF_6]$ (**II**) with m.p. 230–232 °C (with decomposition). Anal. found, %: Si 5.49, N 11.61, F 24.01. Calcd. for $C_{12}H_{18}F_6N_4O_4S_2Si$, Si 5.75, N 11.47, F 23.33.

3.3. Structure determination

The X-ray intensity data were collected at room temperature on a Nonius Kappa CCD diffractometer equipped with graphite monochromated Mo Kα radiation using ϕ–ω rotation. Unit cell parameters were obtained and refined using the whole data set. The structure solution and refinement were processed using SHELX-97 program package [24]. Direct methods yielded all non-hydrogen atoms of the asymmetric unit that were treated anisotropically. The $[SiF_6]^{2-}$ anion was disordered over two positions with the occupancies of 0.829(6) and 0.171(6), respectively, and only the major position was refined in anisotropic approximation. The C-bound hydrogen atoms were placed in calculated positions with their isotropic displacement parameters riding on those of the parent atoms, while the N-bound H-atoms were found from differential Fourier maps at an intermediate stage of the refinement and were treated isotropically. Crystallographic data (cif file) for **I** have been deposited with the Cambridge Crystallographic Data Center, CCDC No. 713403. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (fax: +44 1233 336 033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

Acknowledgements

The diffraction data were collected at the Schulich Faculty of Chemistry, Technion, The Haifa (Israel) through the cooperation of Professor Menahem Kaftory whom we would like to acknowledge.

This work was supported by the Russian Foundation for Basic Research and the Academy of Sciences of the Republic of Moldova (project number 08.820.05.037RF).

References

[1] V.O. Gelmboldt, Russ. J. Inorg. Chem. 54 (2009) 916–921.
[2] V.O. Gelmboldt, L.V. Koroeva, Ed.V. Ganin, M.S. Fonari, M.M. Botoshansky, A.A. Ennan, J. Fluorine Chem. 129 (2008) 632–636.
[3] V.O. Gelmboldt, Ed.V. Ganin, M.S. Fonari, L.V. Koroeva, Yu.Ed. Ivanov, M.M. Botoshansky, J. Fluorine Chem. 130 (2009) 428–433.
[4] (a) N. Blagden, M. de Matas, P.T. Gavan, P. York, Adv. Drug Del. Rev. 59 (2007) 617–630;
(b) K.R. Morris, U.J. Griesser, C.J. Eckhardt, J.G. Stowell, Adv. Drug Del. Rev. 48 (2001) 91–114;
(c) S. Byrn, R. Pfeiffer, M. Ganey, C. Hoiberg, G. Poochikian, Pharm. Res. 12 (1995) 945–954;
(d) P. Vishweshwar, J.A. McMahon, M.L. Peterson, M.B. Hickey, T.R. Shattock, M.J. Zaworotko, Chem. Commun. (2005) 4601–4603;
(e) N. Shan, M.J. Zaworotko, Drug Discov. Today 13 (2008) 440–446;
(f) P. Vishweshwar, J.A. McMahon, J.A. Bis, M.J. Zaworotko, J. Pharmaceut. Sci. 95 (2006) 499–516.
[5] (a) M.S. Fonari, Ed.V. Ganin, Yu.A. Simonov, W.-J. Wang, Acta Cryst. E62 (2006) o911–o913;
(b) Yu.A. Simonov, M.S. Fonari, G.G. Duca, M.V. Gonta, Ed.V. Ganin, A.A. Yavolovskii, M. Gdaniec, J. Lipkowski, Tetrahedron 61 (2005) 6596–6601.

[6] B. Moulton, B.S. Luisi, M.S. Fonari, S.S. Basok, Ed.V. Ganin, V.Ch. Kravtsov, New J. Chem. 31 (2007) 561–568.
[7] M.R. Caira, Mol. Pharmaceut. 4 (2007) 310–316.
[8] (a) C.B. Aakeroy, M.E. Fasulo, J. Desper, Mol. Pharmaceut. 4 (2007) 317–322; (b) J.A. McMahon, J.A. Bis, P. Vishweshwar, T.R. Shattock, O.L. McLaughlin, M.J. Zaworotko, Z. Kristallogr. 220 (2005) 340–350.
[9] A.L. Boreen, W.A. Arnold, K. Mcneill, Environ. Sci. Technol. 38 (2004) 3933–3940.
[10] S.P. Mezyk, T.J. Neubauer, W.J. Cooper, J.R. Peller, J. Phys. Chem. A 111 (2007) 9019–9024.
[11] E.N. Padeiskaya, L.M. Polukhina, Pharmaceut. Chem. J. 11 (1977) 139–143.
[12] V.O. Gelmboldt, A.A. Ennan, Ed.V. Ganin, Yu.A. Simonov, M.S. Fonari, M.M. Botoshansky, J. Fluorine Chem. 125 (2004) 1951–1957.
[13] M.S. Fonari, Yu.A. Simonov, J. Lipkowski, V.O. Gelmboldt, Ed.V. Ganin, A.A. Yavolovski, Crystallogr. Rep. 48 (2003) 630–637.
[14] D.N. Jones (Ed.), Comprehensive Organic Chemistry, Vol. 3. Sulfur Compounds, Pergamon Press, Oxford, 1979.
[15] (a) S.H. Yalkowsky, Y. He, Handbook of Aqueous Solubility Data, CRC, Boca Raton, FL, USA, 2003; (b) R. O'Shea, H.E. Moser, J. Med. Chem. 51 (2008) 2871–2878; (c) G.P. Stahly, Cryst. Growth Des. 9 (2009) 4212–4229.
[16] T. Steiner, Angew. Chem. Int. Ed. 41 (2002) 48–76.
[17] E. Goreshnik, V.O. Gelmboldt, L.V. Koroeva, Ed.V. Ganin, Abstr. Int. Conf. ded. to the 50th anniversary from the Foundation of the Institute of Chemistry of the Academy of Sciences of Moldova, Chisinau, Moldova, 2009, p. 32.
[18] V.O. Gelmboldt, L.Kh. Minacheva, Ed.V. Ganin, V.S. Sergienko, Russ. J. Inorg. Chem. 53 (2008) 875–878.
[19] A. Pevec, A. Demšar, J. Fluorine Chem. 129 (2008) 707–712.
[20] A.L. Bingham, D.S. Hughes, M.B. Hursthouse, R.W. Lancaster, S. Tavener, T.L. Threlfall, Chem. Commun. (2001) 603–604.
[21] N. Blagden, R.J. Davey, H.F. Lieberman, L. Williams, R. Payne, R. Roberts, R. Rowe, R. Docherty, J. Chem. Soc., Faraday Trans. 94 (1998) 1035–1044.
[22] M.R. Caira, V.J. Griffith, L.R. Nassimbeni, R. Luigi, B.V. Oudtshoom, J. Incl. Phenom. 17 (1994) 187–201.
[23] A. Banu, G.M.G. Hossain, Acta Crystallogr. E62 (2006) o1573–o1574.
[24] G.M. Sheldrick, Acta Crystallogr. A64 (2008) 112–122.