Monatshefte für Chemie 134, 1–9 (2003)
DOI 10.1007/s00706-002-0529-0

Monatshefte für Chemie
Chemical Monthly
Printed in Austria

Synthesis, Crystal Structure and Raman Spectrum of Hydrazinium(+2) Fluoroarsenate(III) Fluoride, $N_2H_6AsF_4F$

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Received April 18, 2002; accepted July 15, 2002

Summary. Hydrazinium(+2) fluoroarsenate(III) fluoride was prepared by the reaction of hydrazinium(+2) fluoride and liquid arsenic trifluoride. $N_2H_6AsF_4F$ is stable at 273 K, but decomposes slowly at room temperature. $N_2H_6AsF_4F$ crystallizes in the orthorhombic space group Pnn2 with $a = 774.0(2)$ pm, $b = 1629.2(4)$ pm and $c = 436.6(1)$ pm; $V = 0.5506(3)\,nm^3$, $Z = 4$ and $d_c = 2.461\,g\,cm^{-3}$. The structure consists of $N_2H_6^{2+}$ cations, AsF_4^- anions, and F^- anions and is interconnected by a hydrogen bonding network. Distorted trigonal-bipyramidal AsF_4^- units are very weakly interconnected and form chains along the *b* axis. Bands in the *Raman* spectrum are assigned to the vibrations of $N_2H_6^{+2}$ cations and AsF_4^- anions.

Keywords. Fluoroarsenates(III); Crystal structure; Raman spectroscopy; Hydrazinium(+2).

Introduction

The first attempt to prepare hydrazinium(+2) fluoroarsenates(III) dates back to 1953, when *Pugh* [1] dissolved As_2O_3 in hydrofluoric acid and reacted it with hydrazine hydrate. The reaction product presumably reacted with moist air and only $N_2H_6F_2$ and As_2O_3 were found after the isolation of the solid product. However, similar reactions of SbF_3 and BiF_3 with hydrazinium(+2) fluoride in water solution yielded $N_2H_5HSbF_5$ and $N_2H_5HBiF_5$, respectively. In 1980 the syntheses of $(N_2H_6)_2F_2SbF_5$ and $N_2H_6SbF_5$ from 40% HF water solution of $N_2H_6F_2$ and SbF_3 have been reported [2–4]. During a similar procedure *Helmolt* failed to prepare ammonium fluoroarsenate(III) [5]. By measuring the specific conductivities [6] it has been found that the addition of either BF_3, SbF_5, or KF to the AsF_3 liquid increases its specific conductivity considerably and the ionic compounds $K^+AsF_4^-$ and $AsF_2^+SbF_6^-$ were isolated from liquid AsF_3. It has been shown

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that besides KF, alkali metal fluorides RbF, CsF as well as TlF react in liquid AsF_3 and a whole set of alkali metal fluoroarsenates(III) was isolated [7] in 1957. Utilizing the method of preparation in liquid AsF_3, by dissolving hydrazinium(+2) fluoride in AsF_3 at 353 K, the new compound $N_2H_6AsF_4F$ has been prepared.

Results and Discussion

Syntheses

$N_2H_6F_2$ dissolves in liquid AsF_3 and reacts with it to produce the new compound $N_2H_6AsF_4F$, which crystallizes from the AsF_3 solution after cooling to room temperature. Because of the lower density than liquid AsF_3 ($d = 2.6659\,\mathrm{g\,cm^{-3}}$ at 273 K) [8], $N_2H_6AsF_4F$ ($d_c = 2.461\,\mathrm{g\,cm^{-3}}$ at 200 K) collects at the top of the liquid AsF_3 as transparent crystals. At room temperature $N_2H_6AsF_4F$ decomposes slowly into $N_2H_6F_2$ by releasing AsF_3 gas: $N_2H_6AsF_4F \rightarrow N_2H_6F_2 + AsF_3$.

Description of the Crystal Structure

$N_2H_6AsF_4F$ crystallizes in the space group Pnn2, with $a = 774.0(2)$ pm, $b = 1629.2(4)$ pm, and $c = 436.6(1)$ pm, with $V = 0.5506(3)\,\mathrm{nm^3}$ and with four formula units in the unit cell. The structure of $N_2H_6AsF_4F$ consists of $N_2H_6^{2+}$ cations, AsF_4^- anions and F^- anions (Fig. 1) and is interconnected by a hydrogen bonding network. The $N_2H_6^{2+}$ cations adopt the usual staggered configuration and the N–N bond lengths (143.2(7) pm), (143.8(7) pm) (Table 1) are close to the weighted mean of the nine N–N bond lengths (142.6 pm) in other $N_2H_6^{2+}$ salts [9]. As1 in $N_2H_6AsF_4F$ has three fluorine atoms at distances 173.2(3) pm, 173.9(3) pm, and 174.4(3) pm, in average 173.8 pm, which is 3.2 pm longer than the average bond in the AsF_3 molecule in the gas phase (170.6(2) pm), determined from electron diffraction [10]. The angles F11–As1–F13 (91.1(2)°), F11–As1–F12

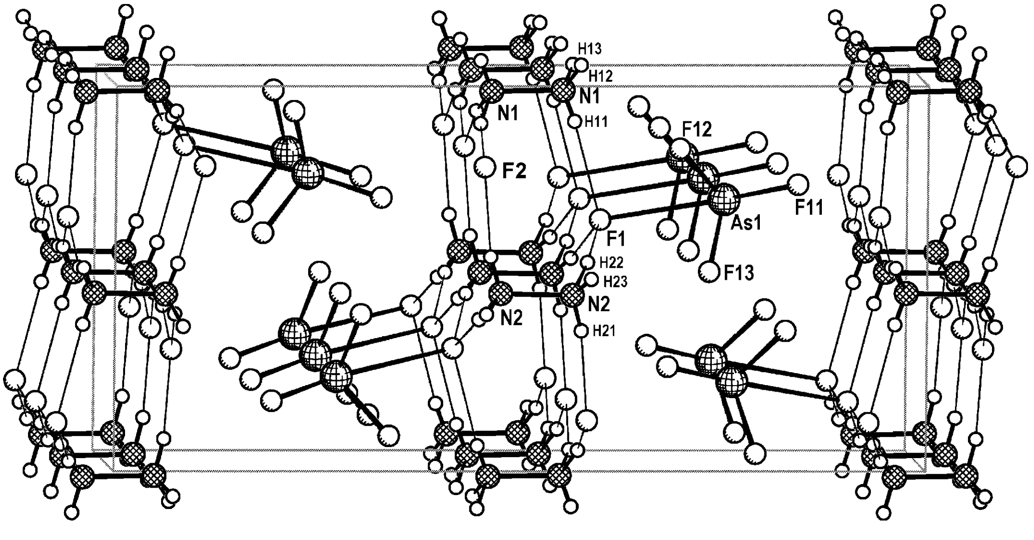

Fig. 1. Packing diagram of $N_2H_6AsF_4F$

Table 1. Selected distances (Å) and angles (°) in $N_2H_6AsF_4F$

As1–F11	1.739(3)	F12–As1–F11	90.9(1)
As1–F12	1.732(3)	F12–As1–F13	93.5(1)
As1–F13	1.744(3)	F11–As1–F13	91.1(2)
N1–N1	1.432(7)		
N2–N2	1.438(7)		

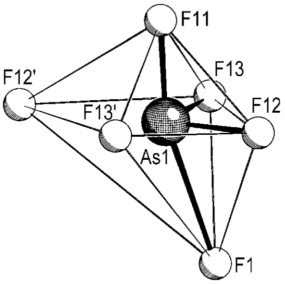

Fig. 2. The primary and secondary coordination of As1 in $N_2H_6AsF_4F$

(90.9(1)°), and F12–As1–F13 (93.5(1)°), in average 91.8°, are close to the corresponding angles in the AsF_3 molecule (96.2(2)°) [10]. The fourth fluorine atom F1 connects (Fig. 2) to As1 at 259.2(4) pm, which is considered as an unusual long bond, by 84.2 pm longer than the sum of covalent radii [11, 12] (175 pm), however, appreciably shorter than the sum of the *van der Waals* radii (335 pm) [11]. There is another interaction to F atom F13′ at a distance of 287.0(4) pm to As1 (Fig. 2). F13′ may be hardly regarded as a bridging F atom and the arsenic entities are very weakly connected by asymmetric bridges As1–F13′···As1′ into chains running parallel to the *b* axes. The next shortest distance from As1 to the fluorine atom is As1–F12′ at 334.8(5) pm, which is just longer than the *van der Waals* distance. F11, F12 and F13 interacts with positive centers on the cation $N_2H_6{}^{2+}$, while F1 and F2 form acceptor hydrogen bonds to nitrogen atoms [13–15] (Table 2). The F^- ions F2 are not coordinated, but they are linked by three very strong hydrogen bonds [16] (N1–H12···F2 (256 pm), N1–H13···F2 (258 pm), N2–H21···F2

Table 2. Intermolecular hydrogen bonds in $N_2H_6AsF_4F$

A	H	B	A···B	A–H	H···B	A–H···B
N1	H11	F1	2.69	0.69	2.01	173.3
N1	H12	F2	2.56	0.94	1.66	159.5
N1	H13	F2	2.58	0.80	1.80	163.3
N2	H21	F2	2.56	0.76	1.81	166.8
N2	H22	F1	2.64	0.97	1.68	168.2
N2	H23	F1	2.67	0.74	1.96	161.2
N1	H11	F12	3.05	0.69	2.68	116.1
N1	H11	F11	3.07	0.69	3.42	103.6
N2	H21	F13	2.82	0.76	2.82	130.8

(256 pm)) to hydrogen atoms in three different $N_2H_6^{2+}$ ions. Angles between bonds connecting F2 and hydrogen atoms vary from 119° to 121° with an average of 120° and the arrangement is almost planar. Besides to F2, the $N_2H_6^{2+}$ cations are linked similarly by hydrogen bonds to F1: F1–H23 $\cdots$ N2 (267 pm), F1–H22 $\cdots$ N2 (264 pm), and F1–H11 $\cdots$ N1 (269 pm). Here the bonds are longer and the angles between hydrogen atoms and F1 range from 109° to 128°, in average 116°, which reflects an interaction with As1.

Raman Spectrum

The *Raman* spectrum of $N_2H_6AsF_4F$ from 250 cm^{-1} to 1060 cm^{-1} is shown in Fig. 3. The bands are tentatively assigned to the vibrations of the $N_2H_6^{2+}$ cation and the AsF_4^- anion (Table 3) by comparison to the *Raman* spectra of known $N_2H_6^{2+}$ salts [17], fluoroarsenates(III) [18] and fluoroantimonates(III) [19–22], taking into account the mass differences. The band at 1033 cm^{-1} is assigned to

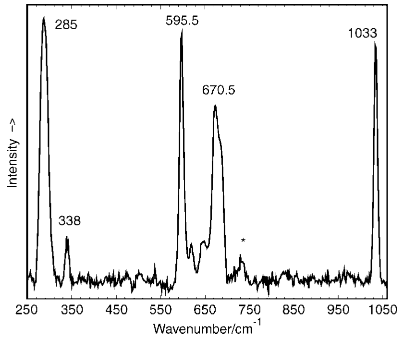

Fig. 3. *Raman* spectrum of $N_2H_6AsF_4F$ (The band marked with asterisk is due to the reactor wall – FEP)

Table 3. *Raman* spectrum of $N_2H_6AsF_4F$ (*Raman* intensities are given in parentheses, sh = shoulder)

$N_2H_6AsF_4F$	Assignment
2879 (3.9)	$(NH_3^+)_s$
2740 (1.2)	
2398 (0.5)	
2189 (5.7)	
1993 (1.2)	$(NH_3^+)_d$
1649 (1.6)	
1033 (9.0)	$(NN)_s$
1016 (0.3)	?
682 (5.4) sh	$(AsF)_s$ eq
670.5 (6.7)	
646 (1.4)	
619 (1.4)	$(AsF)_s$ ax
595.5 (9.4)	
338 (1.7)	$(AsF)_d$
285 (10.0)	

the N–N stretching vibration of the cation and it appears in the *Raman* spectra of all hydrazinium(+2) salts in the narrow region around 1030 cm^{-1}; it is usually a very intense band and it is absent from the infrared spectra [17] of hydrazinium(+1) compounds. The bands at higher frequencies are assigned to the stretching and deformation vibrations of the NH_3^+ units of the $N_2H_6^{2+}$ cation. The arsenic species in $N_2H_6AsF_4F$ is distorted considerably and its symmetry is C_s in contrast to a regular AsF_4^- unit in $CsAsF_4$ with C_{2v} symmetry [18]. Strong bands at 682 cm^{-1}, 670.5 cm^{-1}, and 595.5 cm^{-1} as well as moderate bands at 646 cm^{-1} and 619 cm^{-1} are assigned to the stretching vibrations of the AsF_4^- unit, while bands at 338 cm^{-1} and 285 cm^{-1} belong to deformation vibrations.

Arsenic Atom Coordination

The local environment of As1 in $N_2H_6AsF_4F$ is interesting from the point of primary and secondary coordination, the term developed by *Alcock* [23] (Fig. 2). The three fluorine atoms located at 173.2(3) pm, 173.9(3) pm, and 174.4(3) pm from As1 may be considered as the primary coordination, while two fluorine atoms at 259.2(4) pm and 287.0(4) pm complete the secondary coordination. Based on two types of coordination the As entity may be viewed in different ways. Taking into account the primary coordinated fluorine atoms only, the AsF_3 molecule with slightly elongated As1–F bonds results. The fourth fluorine atom F1 connects at 259.2(4) pm in the proper direction for AsF_4^- ion geometry. Going further from As1 there is the fifth fluorine atom F13′ at 287.0(4) pm in a proper direction to define a distorted tetragonal pyramid, with F1, F11, F13, and F13′ in a basal plane and F12 at the apices. Taking into account also fluorine atom F12′ at 334.8(5) pm from As1, distorted octahedra caped with lone pair may be described. The ratios of the distances As1–F1 and As1–F13′ to the average bond length As–F in the primary As1 coordination are 1.49:1 and 1.66:1 and especially the interaction between As1 and F13′ may be regarded as extremely weak. In a similar compound, $N_2H_6SbF_5$ [3], the product of the reaction of the less basic (compared to AsF_3) SbF_3 with $N_2H_6F_2$, the corresponding ratios of the distances Sb–F1 and Sb–F5 to the average bond length Sb–F in the primary Sb coordination are 1.15:1 and 1.20:1, which permits the formulation SbF_5^{2-} for the antimony species. In studies of the stereochemistry of O and F complexes of Sn(II), Sb(III), Te(IV), I(V), and Xe(VI), the interatomic distances longer than 310 pm were generally ignored [24] and the bonds Sb–F in fluoroantimonates(III) longer than 300 pm were found not to have the directional character [25]. For the calculated covalent bond As–F, 175 pm, being 20 pm shorter than the covalent Sb–F bond [11], the interaction As1-F13′ with the interatomic distance of 287.0(4) pm was considered too weak to have any influence. *Fourcade* observed three types of coordination in alkaline fluoroantimonates(III) [25], AX_6E (monocaped octahedron) ($E =$ lone pair), AX_5E (octahedron), and AX_4E (trigonal bipyramid). In addition, intermediate cases between these three types exist. The arsenic species in $N_2H_6AsF_4F$ may also be viewed as an intermediate case between the defined coordination types, as As1 forms three short bonds, two weaker interactions, and in addition there is one interaction at the *van der Waals* distance. For the discussion the definition "AsF_4^-" for the arsenic species in $N_2H_6AsF_4F$ is used, because this anion, although distorted in $N_2H_6AsF_4F$, exists also in other fluoroarsenates(III) [6, 7, 18, 26].

According to the VSEPR theory [27, 28] the four fluorine atoms and the lone pair in $AsF_4{}^-$ occupy positions to define a trigonal-bipyramid, in $N_2H_6AsF_4F$ the elongation of one apex deforms the trigonal-bipyramid and the lone pair is located in the equatorial position. The environment of As1, regarding the bond distances and the geometry, is close to the environment of As1 in the $As_2F_7{}^{-2}$ anion in the structure of KAs_2F_7, where besides the primary coordinated fluorine atom at 175(1) pm, and two at 171(1) pm, the two secondary coordinated fluorine atoms are located at 265(1) pm and 283(1) pm from As1.

A further support for the formulation of the arsenic species as $AsF_4{}^-$ is provided by the *Raman* spectrum of $N_2H_6AsF_4F$, in which the stretching bands appear at 682 cm^{-1}, 670.5 cm^{-1}, 646 cm^{-1}, 619 cm^{-1}, and 595.5 cm^{-1}, compared to 660 cm^{-1} and 592 cm^{-1} in $CsAsF_4$ [18], while the stretching bands in the *Raman* spectrum of AsF_3 [29] appear much higher, at 738.5 cm^{-1} and 698.8 cm^{-1}.

Interestingly enough, the noncoordinated fluorine atom F2 is held in position by strong hydrogen bonds only. Noncoordinated fluorine atoms are not usual in structures of hydrazinium(+2) compounds, but they were already observed in the compounds $(N_2H_6)_2F_2MF_6$ (M = Ge, Ti, Sn) [30–32] and $(N_2H_6)_3Zr_2F_{13}F$ [33].

Experimental

Reagents

AsF_3 has been prepared by a modified procedure in a batch process from As_2O_3 and anhydrous HF at 470 K in a 300 cm^3 stainless steel reactor [34]. The product has been purified by bubbling gaseous AsF_3 through the FEP trap filled with concentrated H_2SO_4 and collecting the product in a FEP trap held at 215 K. $N_2H_6F_2$, Fluka has been dried at 303 K in a PFA tube on a high vacuum line for 20 h prior to use.

Synthesis of Hydrazinium(+2) Fluoroarsenate(III) Fluoride (**1**, $N_2H_6AsF_4F$)

In a typical synthesis 100 g AsF_3 was condensed onto 0.1424 g of $N_2H_6F_2$ in a PFA reaction vessel at 77 K. The reaction mixture was heated to 353 K on a water bath while stirring and kept at this temperature until all $N_2H_6F_2$ which floated on the surface of the AsF_3, dissolved. Upon cooling to room temperature white crystals precipitated out of the AsF_3 liquid and collected on the surface. The reaction vessel was cooled to 273 K and the volatiles (AsF_3) pumped off on a high vacuum line and condensed in a cold trap held at 77 K. The composition of the volatiles was checked by infrared spectroscopy and only bands attributable to gaseous AsF_3 were found. Mass loss *vs.* time curve obtained at 273 K (Fig. 4) clearly shows a change of the slope at a composition $N_2H_6F_2{:}AsF_3 = 1{:}1$. $N_2H_6AsF_4F$ gradually decomposes at room temperature by releasing AsF_3, which condenses back to the sample of $N_2H_6AsF_4F$, and causes drops to appear and the sample becomes sticky. However, it is stable at 273 K and was stored at this temperature for two days without decomposition. The new compound $N_2H_6AsF_4F$ was decomposed on a vacuum line ($p = 10^{-2}$ Pa) at room temperature and the composition of the white powder left in the reactor was $N_2H_6F_2$ only, as checked by x-ray powder diffraction.

Chemical Analyses

The content of total fluoride ion was determined with a fluoride ion selective electrode after total decomposition of the sample using alkaline carbonate fusion with $NaKCO_3$ [35, 36]. $N_2H_6AsF_4F$: observed F 45.1%; calculated F 46.57%.

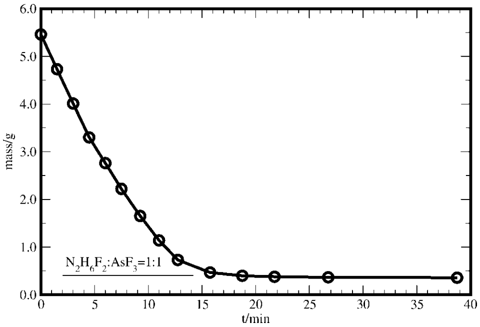

Fig. 4. Dependence of the total mass of the sample on the time of pumping off volatiles in the course of the $N_2H_6AsF_4F$ preparation at 273 K

X-Ray Powder Diffraction

X-ray diffraction patterns of the powdered solid samples were obtained on a *Debye-Scherrer* camera using CuK_α radiation.

X-Ray Structure Determination

Crystals immersed in perfluorodecalin (ABCR) were selected under microscope and mounted onto the free end of thin-walled quartz capillaries by flash freezing in a low temperature dry nitrogen flow. The data collection procedure, crystal data, and structure determination methods are summarized in Table 4

Table 4. Data for the crystal structure determination of $N_2H_6AsF_4F$

Empirical Formula	$N_2H_6AsF_4F$
Formula Weight	203.97
Crystal System	orthorhombic
Space Group	Pnn2 (#34)
Lattice Parameters	$a = 774.0(2)$ pm $b = 1629.2(4)$ pm $c = 436.6(1)$ pm $V = 0.5506(3)$ nm^3
Z value	4
D_{calc}	2.461 Mg/m^3
F_{000}	392.00
$\mu(MoK\alpha)$	6.192 mm^{-1}
Mosaicity:	0.366(11)
No. of Reflections Measured	Total: 4176 Unique: 1309 ($R_{int} = 0.037$)
Absorption	multi-scan (trans. factors: 0.3343–0.540)
Reflections ($I > 2\sigma(I)$)	1157
Reflection/Parameter Ratio	11.93
R1	0.030 ($I > 2\sigma(I)$); 0.035 (all data)
wR2	0.072 ($I > 2\sigma(I)$); 0.073 (all data)
Goodness of Fit on F^2 (All data)	1.09
Max. and min. peak residual	1087 e_0/nm^3 and -718 e_0/nm^3
Temperature	200(2) K

Table 5. Fractional atomic coordinates and displacement parameters

Atom	x	y	z	U_{eq} (pm^2)[a]
As1	0.27002(5)	0.73771(2)	0.4284(2)	186(1)
F1	0.3328(3)	0.5900(2)	0.2401(5)	187(5)
F2	0.3289(3)	0.9357(2)	0.2012(5)	217(6)
F11	0.2192(4)	0.8229(2)	0.6507(7)	354(7)
F12	0.1269(3)	0.6776(2)	0.6351(6)	269(6)
F13	0.4444(3)	0.7142(2)	0.6656(6)	317(6)
N1	0.4990(5)	0.9561(2)	0.7038(8)	149(8)
N2	0.5020(5)	0.5441(2)	0.7357(8)	148(7)
H11	0.582(7)	0.941(3)	0.72(1)	170
H12	0.425(6)	0.939(3)	0.86(1)	170
H13	0.441(7)	0.941(3)	0.56(1)	170
H21	0.596(7)	0.557(3)	0.72(1)	170
H22	0.429(7)	0.565(3)	0.57(1)	170
H23	0.450(6)	0.566(3)	0.85(1)	170

[a] One third of the trace of orthogonalized U_{ij} tensor

and the final positional parameters with their standard deviations in Table 5. All measurements were made on a Mercury CCD area detector coupled with a Rigaku AFC7 diffractometer with graphite monochromated Mo-K_α radiation. The data were collected at a temperature of 200 ± 2 K to a maximum 2θ value of 60.3° and processed using CrystalClear (Rigaku) [37]. The structure was solved by direct methods (SIR92) [38] and expanded using *Fourier* techniques. The non-hydrogen atoms were refined anisotropically [39]. Positions of hydrogen atoms were refined with fixed isotropic parameters.

Additional details on the single crystal structure determination can be obtained from the Fachinformationszentrum Karlsruhe, (FIZ): D-76344 Eggenstein-Leopoldshafen (Germany), on quoting the depository number CSD-412507, the names of the authors and the journal citation.

Vibrational Spectroscopy

Raman spectra were recorded on a Renishaw *Raman* microscope 1000 with He-Ne (632.8 nm) laser excitation source. Using a 10 microscope objective the laser beam was focused onto a spot of approximately 1 μm in diameter, and the collected scattered light was passed through a spectrophotometer onto a CCD detector. Attempts to record infrared spectra as Nujol mulls were not successful as the compound $N_2H_6AsF_4F$ decomposes readily at room temperature into $N_2H_6F_2$ and AsF_3, and spectra recorded show bands of decomposition products. The infrared spectra of gaseous products released during the decomposition of $N_2H_6AsF_4F$ were recorded using an all nickel two gauge infrared cell with AgCl windows and a 10 cm beam path. A Perkin Elmer FTIR 1710 infrared spectrometer was utilized to record infrared spectra of solid and gaseous samples.

Acknowledgement

The financial support from the Ministry of Education, Science and Sport of Slovenia is gratefully acknowledged.

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