

Crystal Structures of Hydrazinium(II) Salts of [SbF₆]⁻ and [Sb₂F₁₁]⁻

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Dedicated to Dr. Karel Lutar (the Ph.D. Thesis Tutor of Z.M) who did Preliminary Experiments in 1992 and Deceased in 2000

Keywords: Hydrazinium ions; Antimony; Fluorides; Crystal structure

Abstract. N₂H₆(Sb₂F₁₁)₂ was synthesized by the reaction of N₂H₆F₂ with excess of SbF₅ in anhydrous hydrogen fluoride (aHF). It crystallizes in the triclinic space group $P\overline{1}$ (No. 2) with a = 6.6467(3) Å, b = 8.3039(4) Å, c = 8.3600(5) Å, a = 76.394(5) °, $\beta = 70.161(5)$ °, $\gamma = 70.797(5)$ °, V = 405.90(4) Å³ at 150 K, Z = 2. When it is redissolved in aHF, it solvolysis with the release of SbF₅ yielding N₂H₆(SbF₆)₂

1 Introduction

The synthesis of hydrazinium(II) hexafluoridoantimonate, $N_2H_6(SbF_6)_2$ was first time reported in 1990.^[1] It was characterized by chemical analysis and vibrational spectroscopy. Its thermal decomposition was also studied. In the same paper it was given that its X-ray powder diffraction pattern (measured at ambient temperature) can be indexed on the basis of a monoclinic cell with a = 8.22(2) Å, b = 10.04 Å, c = 9.51 Å, $\beta = 97.2(2)^{\circ}$ and V = 780 Å³. Since no other structure information have been reported, we decided to reinvestigate its synthesis and try to prepare single crystals of $N_2H_6(SbF_6)_2$.

In this paper, syntheses and crystallizations in the system $N_2H_6F_2$ / SbF₅ were carried out in anhydrous hydrogen fluoride (aHF) and have yielded two hydrazinium(II) salts containing the [SbF₆]⁻ and [Sb₂F₁₁]⁻ anions. $N_2H_6(SbF_6)_2$ and $N_2H_6(Sb_2F_{11})_2$ were isolated and their crystal structures were determined and are described in this paper.

2 Results and Discussion

2.1 Syntheses

 $N_2H_6(Sb_2F_{11})_2$ can be prepared by reaction of $N_2H_2F_2$ with SbF_5 in anhydrous hydrogen fluoride (aHF) [Equation (1)]:

$$N_2H_2F_2 + 4SbF_5 \xrightarrow{aHF} N_2H_6(Sb_2F_{11})_2$$
(1)

Although the starting molar ratio $n(\text{SbF}_5)/n(N_2\text{H}_6\text{F}_2)$ was higher than four, the final product was still contaminated with

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which crystallizes in the monoclinic *C*2/*c* space group (No. 15) with a = 7.3805(3) Å, b = 12.3248(5) Å, c = 10.4992(4) Å, $\beta = 92.218(4)$ °, V = 954.33(7) Å³ at 150 K, and Z = 8. No other phases were observed in crystallization products when different molar ratios of N₂H₆F₂/SbF₅ (1:1,2:3,1:3) in aHF were used as starting materials.

small amount of $N_2H_6(SbF_6)_2$. In previous report only $N_2H_6(SbF_6)_2$ has been detected as the final product.^[1] The reason is most likely in different concentrations of SbF_5 in aHF. When redissolved, $N_2H_6(Sb_2F_{11})_2$ solvolysis in aHF [Equation (2)]:

$$N_2H_6(Sb_2F_{11})_2 \xrightarrow[-298\ K]{aHF} N_2H_6(SbF_6)_2 + 2SbF_5$$
(2)

Redissolving of dry solid Sb_2F_{11} salts with the use of aHF is an effective way to eliminate SbF_5 from Sb_2F_{11} .,^[2,3] Both colorless compounds decompose when they are exposed to air due to the reaction with moisture.

2.2 Crystal Structures of $N_2H_6(SbF_6)_2$ and $N_2H_6(Sb_2F_{11})_2$

The crystal structures of hydrazinium $[N_2H_6]^{2+}$ salts with fluoridometallate anions are limited to metals (*M*) in the oxidation states $M^2 = Be$,^[4] Sn;^[5] $M^{+3} = B$,^[6] Ga,^[7] In,^[8] As,^[9] Sb;^[10] and $M^{+4} = Ti$,^[11,12] Zr,^[13,14] Si,^[15] Ge,^[16] Sn.^[17] Although N₂H₆($M^{V}F_6$)₂ (M = P, As, Sb) are known,^[1] no data about their or crystal structures of other N₂H₆($M^{V}F_6$)₂ ($M = M^{5+}$] compounds have been reported so far.

The crystal data and refinement results of $N_2H_6(SbF_6)_2$ and $N_2H_6(Sb_2F_{11})_2$ are summarized in Table 1. Selected bond length and angles are given in Table 2.

The crystal structure of $N_2H_6(SbF_6)_2$ consists of $[N_2H_6]^{2+}$ cations and $[SbF_6]^-$ anions held together by hydrogen bonds (Figure 1). The N–N distance determined at 150 K is 1.457(8) Å and it is longer than usually observed.^[4,6,13,15,16] It is comparable to N–N bond lengths found in $(N_2H_6)_2F_2(SnF_6)$ (at 123 K: 1.46(3) Å – eclipsed conformation of $N_2H_6)^{[17]}$ and $N_2H_6TiF_6$ (at room temperature: 1.474(7) Å – staggered conformation of N_2H_6).^[12]

The symmetry constraints of the space group C_2/c require that the hydrogen atoms of the N₂H₆ group are in eclipsed (*cis*)

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Table 1. Summary of crystal data and refinement results for $N_2H_6(SbF_6)_2$ and $N_2H_6(Sb_2F_{11})_2$.

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	$N_2H_6(SbF_6)_2$	$N_2H_6(Sb_2F_{11})_2$
T /K	150	150
Crystal system	monoclinic	triclinic
a /Å	7.3805(3)	6.6467(3)
b /Å	12.3248(5)	8.3039(4)
c /Å	10.4992(4)	8.3600(5)
$a /^{\circ}$	/	76.394
β /°	92.218(4)	70.161(5)
γ /°	/	70.797(5)
V/Å ³	954.33(7)	405.90(4)
Space group	C_2/c	$P\bar{1}$
Z	8	2
$D_{\rm calcd}$ /g·cm ⁻³	3.5184	3.8413
λ/Å	0.71073	0.71073
μ /mm ⁻¹	5.826	6.813
GOF indicator ^{a)}	1.048	1.029
$R_1^{(b)}$	0.028	0.028
$wR_2^{c)}$	0.070	0.082

a) GOF = $[\Sigma w (F_o^2 - F_c^2)^2 / (N_o - N_p)]^{1/2}$, where $N_o = \text{no. of refins and} N_p = \text{no. of refined parameters. b)} R_1 = \Sigma ||F_o| - |F_c|| / \Sigma ||F_o|. c) wR_2 = <math>[\Sigma w (F_o^2 - F_c^2)^2 / \Sigma (w (F_o^2)^2)]^{1/2}$.

Table 2. Selected distances /Å and angles /° in the crystal structures of $N_2H_6(SbF_6)_2$ and $N_2H_6(Sb_2F_{11})_2$.

	$N_2H_6(SbF_6)_2$	$N_2H_6(Sb_2F_{11})_2$
N–N	1.457(8)	1.439(8)
Sb1-F1	$2 \times 1.869(3)$	1.827(2)
Sb1-F2	$2 \times 1.885(3)$	1.851(2)
Sb1–F3	$2 \times 1.887(3)$	1.873(3)
Sb1-F4	/	1.876(2)
Sb1-F5	/	1.881(2)
Sb1-F11	/	2.011(2)
Sb2-F4	$2 \times 1.873(3)$	/
Sb2-F5	$2 \times 1.875(3)$	/
Sb2-F6	$2 \times 1.879(3)$	1.843(3)
Sb2–F7	/	1.857(3)
Sb2–F8	/	1.861(3)
Sb2–F9	/	1.867(2)
Sb2-F10	/	1.878(2)
Sb2-F11	/	2.036(2)
Sb ₂ F ₁₁ angles		
Sb1-F _b -Sb2 ^{a)}	/	170.74(14)
Torsion angle ^{b)}	/	≈ 7

a) Bending of two SbF5 groups about Fb (bridging fluorine), which is expressed in terms of the bridge angle *a*. b) Torsion of two planar SbF4,eq groups from eclipsed to staggered conformation expressed in the torsion angle (ψ) .^[2,18]

position (Figure 2). There is a twofold axis going through the center of N–N bond. Such eclipsed conformation is rare and it has been observed previously in $(N_2H_6)_2F_2(SnF_6)^{[17]}$ and $(N_2H_6)_2F_2(TiF_6).^{[11]}$

Although hydrogen bonds exists with a continuum of strengths, it is useful for practical reasons to use classification such as strong, moderate and weak hydrogen bonds.^[19] There are no strong (quasi-covalent nature) hydrogen bonds with short F···N = 2.2–2.5 Å contacts^[19] present in the crystal structure. On the basis of proposed model for hydrogen atoms, considering the packing of the $[N_2H_6]^{2+}$ and $[SbF_6]^-$ ions and using the proposed parameters H···F = 1.5–2.2 Å, N···F = 2.5–



Figure 1. Packing of $[N_2H_6]^{2+}$ cations and $[SbF_6]^-$ anions in the crystal structure of $N_2H_6(SbF_6)_2$.



Figure 2. Part of the crystal structure of $N_2H_6(SbF_6)_2$ showing only moderate strong hydrogen bonds (dashed lines) between $[N_2H_6]^+$ and $[SbF_6]^-$ ions; thermal ellipsoids are drawn at the 50% probability level. Symmetry codes: (i) –*x*, 1–*y*, 1–*z*; (ii) 1–*x*, 1–*y*, 1–*z*; (iii) 0.5+*x*, 0.5+*y*, *z*; (iv) 1+*x*, *y*, *z*; (v) 0.5+*x*, 0.5+*y*, *z*; (vi) 0.5–*x*, 0.5+*y*, 0.5–*z*; (vii) 1–*x*, *y*, 0.5–*z*; (viii) –*x*, *y*, 0.5–*z*

3.2 Å and N–H···F > 130° for moderate (mainly electrostatic) hydrogen bonds,^[19] then each $[N_2H_6]^{2+}$ cation is involved in moderate hydrogen bonding with six SbF₆ units, i.e. each hydrogen atom of the NH₃ group forms one single contact with different SbF₆ unit (Figure 2).

There are two crystallographic unique SbF₆ units. All six fluorine atoms of Sb(1)F₆ participate in moderate strong hydrogen. Comparison of Sb–F distances (Table 2) shows that both SbF₆ octahedra are quite regular, indicating that in addition to previously mentioned moderate hydrogen bonds there are a lot of weak (electrostatic/dispersion)^[19] hydrogen bonding interactions.

The crystal structure of N₂H₆(Sb₂F₁₁)₂ is composed of $[N_2H_6]^{2+}$ cations and $[Sb_2F_{11}]^-$ groups (Figure 3). The examples of compounds where the charge of double charged cation is compensated by two single charged $[Sb_2F_{11}]^-$ anions are not very common. No $M(Sb_2F_{11})_2$ compounds with simple M^{2+} cations ($M^{2+} = Sn$, Pb, transition or alkaline earth metal) have been reported so far.^[20,21] The reason must be in unfavorable packing of small metal M^{2+} cations and large $[Sb_2F_{11}]^-$ anions. Only compounds with complex double charged cations, as for example $[AuXeF_4]^{2+,[22]}$ $[Se_2I_4]^{2+,[23]}$ and $[M(CO)_n]^{2+}$ (M = Hg, Pd, Pt, Fe, Ru, Os; n = 2-6),^[24] are known.

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Figure 3. Packing of $[N_2H_6]^{2+}$ cations and $[Sb_2F_{11}]^-$ anions in the crystal structure of $N_2H_6(Sb_2F_{11})_2$.

The N–N bond length in N₂H₆ unit equals 1.439(8) Å and is slightly shorter than in SbF₆ salt. In N₂H₆(Sb₂F₁₁)₂ the hydrogen atoms of N₂H₆ group are in usual staggered (*trans*) position. If the same approach is used as for N₂H₆(SbF₆)₂ compound, then each $[N_2H_6]^{2+}$ cation is involved in six moderate strong hydrogen bonds with two Sb₂F₁₁ units (Figure 4). Similar as before, a large number of weak hydrogen bonding contacts are present.



Figure 4. Part of the crystal structure of $N_2H_6(Sb_2F_{11})_2$ showing only moderate strong hydrogen bonds (dashed lines) between $[N_2H_6]^{2+}$ and $[Sb_2F_{11}]^-$ ions; thermal ellipsoids are drawn at the 50% probability level. Symmetry codes: (i) 1+*x*, *y*, *z*; (ii) 1–*x*, 2–*y*, 1–*z*; (iii) 2–*x*, 2–*y*, 1–*z*.

With almost linear dihedral angle (approx. 171°) and very small torsion angle (7°, Table 2) the $[Sb_2F_{11}]^-$ anion (Figure 4) is very closed to ideal D_{4h} symmetry.^[25]

3 Conclusions

The single crystals of only two phases, i.e. $N_2H_6(SbF_6)_2$ and $N_2H_6(Sb_2F_{11})_2$, were grown in the $N_2H_6F_2/SbF_5/aHF$ system and their crystal structures were determined. The unit cell of the crystal structure of $N_2H_6(SbF_6)_2$, determined on a single crystal at 150 K, is in disagreement with previously reported one indexed on the basis of X-ray powder diffraction (XPD) photograph obtained at ambient temperature. There could be two reasons. First, that the indexation gave wrong result due to the poor quality of obtained XPD photograph or that there is phase transition resulting in high- and low-temperature modifications of $N_2H_6(SbF_6)_2$. Unfortunately, single crystals of $N_2H_6(SbF_6)_2$ were too small to be filled in quartz capillaries and measured at ambient temperature.

4 Experimental Section

4.1 Apparatus, Techniques and Reagents

Volatile materials (anhydrous HF, F_2 , Sb F_5) were handled in a nickel vacuum line and an all PTFE vacuum system equipped with PTFE valves as described previously.^[26] The nonvolatile materials were manipulated in a dry box (M. Braun, Germany). The residual water in the atmosphere within the dry-box never exceeded 1 ppm. The reactions were carried out in FEP (tetrafluoroethylene-hexafluoropropylene; Polytetra GmbH, Germany) reaction vessels (height 250–300 mm with inner diameter 15.5 mm and outer diameter 18.75 mm) equipped with PTFE valves ^[27] and PTFE coated stirring bars. Prior to their use all reaction vessels were passivated with elemental fluorine (Solvay Fluor and Derivate GmbH, Germany).

 $N_2H_6F_2$ and SbF₃ (Alfa Aesar, 99%) were used as supplied. SbF₅ was prepared in a similar way as reported for AsF₅,^[28] i.e. by static fluorination of SbF₃ with elemental fluorine in a nickel autoclave at 110 °C. Higher temperature should be avoided since otherwise SbF₅ further reacts with reaction vessel's walls forming Ni(SbF₆)₂. Anhydrous HF (Linde, Fluorwasserstoff 3.5) was treated with K₂NiF₆ (Ozark-Mahoning, 99%) for several hours prior to use.

X-ray powder diffraction photographs were obtained using the Debye-Scherrer technique with Ni-filtered Cu- K_{α} radiation. Samples were loaded into quartz capillaries (0.3 mm) in a dry-box. Intensities were estimated visually. Hydrazine was determined potentiometrically.^[29]

4.2 Synthesis of $N_2H_6(Sb_2F_{11})_2$ and $N_2H_6(SbF_6)_2$

 $N_2H_6F_2$ (0.502 g, 6.98 mmol) was loaded in a reaction vessel in a glove-box. SbF_5 (7.10 g, 32.8 mmol) and anhydrous hydrogen fluoride (4 mL, aHF) were condensed onto the $N_2H_6F_2$ and the reaction vessel was brought to room temperature. Immediately, a clear colorless solution was obtained. The volatiles were slowly pumped off at room temperature, leaving behind colorless solid. The final mass of the isolated solid was 6.335 g [calcd. for $N_2H_6(Sb_2F_{11})_2$: 6.553 g] corresponding to the molar ration $N_2H_6F_2$:SbF₅ = 1:3.85 on the basis of starting amount of $N_2H_6F_2$. Chemical analysis of N_2H_4 in $N_2H_6F_2$ ·3.85SbF₅: calcd. 3.52 wt %; found 3.5 wt %. X-ray powder diffractions data show only the presence of $N_2H_6(Sb_2F_{11})_2$.

A large amount of aHF (8 mL) was condensed onto $N_2H_6(Sb_2F_{11})_2$ (200 mg). Volatiles were pumped away and fresh aHF was added. This was repeated three times. The measured X-ray powder diffraction data of the bulk material and of the simulated one from single crystal determination of $N_2H_6(SbF_6)_2$ were essentially identical.

4.3 Crystal Growth of $N_2H_6(Sb_2F_{11})_2$ and $N_2H_6(SbF_6)_2$

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A T-shaped apparatus consisting of two FEP tubes (6 mm and 19 mm outer diameter) was used for single crystal growth. The product of reaction between N2H6F2 and SbF5 corresponding to molar ration $N_2H_6F_2$:SbF₅ = 1:3.85 was loaded (approximately 150 mg) into the wider arm of the crystallization vessel in a dry-box. Afterwards aHF (4 mL) was condensed onto the starting material at 77 K. The crystallization mixture was brought up to ambient temperature and the clear solution, which had developed, was decanted into the narrower arm. The evaporation of the solvent from this solution was carried out by maintaining a temperature gradient corresponding to about 10 K between both tubes for 2 months. The effect of this treatment was to enable aHF to be slowly evaporated from the narrower into the wider tube leaving the crystals. Two different types of crystals were obtained, i.e. N₂H₆(Sb₂F₁₁)₂ and N₂H₆(SbF₆)₂. No other phases were observed when different molar ratios of N2H6F2 / SbF5 (1:1, 2:3,1:3) in aHF were used as starting materials.

Crystallization products were immersed in perfluorinated oil (ABCR, FO5960, melting point 263 K) in a dry-box. Single crystals were selected from the crystallization products under the microscope outside the dry-box and transferred into the cold nitrogen stream of the diffractometer.

4.4. Crystal Structure Determination of $N_2H_6(Sb_2F_{11})_2$ and $N_2H_6(SbF_6)_2$

Single-crystal data for both compounds were collected on a Gemini A diffractometer equipped with an Atlas CCD detector, using graphite monochromatized Mo- K_{α} radiation. Data were treated using the Crysalis software suite program package.^[30] Structures were solved by charge flipping method using Superflip^[31] program (Olex crystallographic software^[32]) and refined with SHELXL-2013^[33] software, implemented in program package WinGX.^[34] Hydrogen atoms were localized from peaks of electron density in different Fourier maps. Because of varied N–H distances the final refinement was performed with geometrical restrictions for hydrogen atoms positions. The figures were prepared using DIAMOND 3.1 software.^[35]

Further details of the crystal structure investigations may be obtained from the Fachinformationszentrum Karlsruhe, 76344 Eggenstein-Leopoldshafen, Germany (Fax: +49-7247-808-666; E-Mail: crysdata@fiz-karlsruhe.de, http://www.fiz-karlsruhe.de/request for deposited data.html) on quoting the depository numbers CSD-429156 $(N_2H_6(Sb_2F_{11})_2)$ and -429157 $(N_2H_6(SbF_6)_2)$.

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