Pages: 5





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[XeF₅]⁺/Metal and [XeF₅]⁺/Non-Metal Mixed-Cation Salts of Hexafluoridoantimonate(V)

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New types of $[XeF_5]^+$ salts, i.e., $NO_2XeF_5(SbF_6)_2$ and $XeF_5-[Cu(SbF_6)_3]$, are derived from reactions between XeF_5SbF_6 and NO_2SbF_6 or $Cu(SbF_6)_2$, respectively. The crystal structure of the former consists of $[NO_2]^+$ and $[XeF_5]^+$ cations and $[SbF_6]^-$ anions. The main feature of the crystal structure of $XeF_5[Cu(SbF_6)_3]$ are rings of CuF_6 octahedra that share ap-

Introduction

The synthesis of XeF₆ was reported for the first time in 1962,^[1] followed by three later reports in 1963.^[2-4] It was observed fairly quickly that XeF₆ is a very good fluoride ion donor forming $[XeF_5]^+$ or $[Xe_2F_{11}]^+$ salts. None of the compounds reported so far contain any additional cations and studies on crystal structure that we are aware of have stated that they are simple $[Xe_2F_{11}]^+$ or $[Xe_5]^+$ salts of various fluorido anions, with square planar $[MF_4]^-$ (M = Ag, Au),^[5,6] octahedral $[MF_6]^-$ (M = As, V, Nb, Ru, Au, Pt),^[5,6] polymeric $[MF_5]^-$ (M = Cr, Ge),^[5,6] octahedral $[MF_6]^{2-}$ (M = Ni, Pd),^[5,6] or oligometric $[Ti_4F_{19}]^{3-}$ geometries.^[7] Exceptions are XeF₂·n(XeF₅AsF₆) (n = 2, 1, 0.5),^[8] XeF_{2} ·($XeF_{5}RuF_{6}$),^[9] and ($XeF_{5}CrF_{5}$)· XeF_{4} ^[10] where additional ligands (XeF₂, XeF₄) are present and the recently reported $[XeF_5]^+$ and $[Xe_2F_{11}]^+$ salts with $[OsO_3F_3]$ and $[\mu$ -F(OsO₃F₂)₂] anions.^[11]

Results and Discussion

Synthesis of NO₂XeF₅(SbF₆)₂ and XeF₅[Cu(SbF₆)₃]

The reaction between XeF_5SbF_6 and NO_2SbF_6 at ambient temperature in anhydrous hydrogen fluoride (aHF) as a solvent yields the first mixed-cation $[NO_2]^+/[XeF_5]^+$ compound $NO_2XeF_5(SbF_6)_2$. In a similar way, but using $Cu(SbF_6)_2$ instead of NO_2SbF_6 , $XeF_5[Cu(SbF_6)_3]$ has been synthesized (Scheme 1). Both compounds represent the first exes with SbF₆ octahedra connected into an infinite tridimensional framework. This arrangement leads to the formation of cavities within which $[XeF_5]^+$ cations are located. Raman spectra of both $[NO_2]^+/[XeF_5]^+$ and $[XeF_5]^+/Cu^{2+}$ mixed-cation hexafluorido-antimonates(V) are reported herein.

examples where in addition to $[XeF_5]^+$ other cations are present. The compounds are well soluble in aHF allowing growth of colorless single crystals of very good quality.

$$NO_{2}SbF_{6} + XeF_{5}SbF_{6} \xrightarrow{aHF} NO_{2}XeF_{5}(SbF_{6})_{2}$$

$$298 K \xrightarrow{aHF} Cu(SbF_{6})_{2} + XeF_{5}SbF_{6} \xrightarrow{aHF} XeF_{5}[Cu(SbF_{6})_{3}]$$

Scheme 1. Syntheses of NO₂XeF₅(SbF₆)₂ and XeF₅[Cu(SbF₆)₃].

Crystal Structure and Raman Spectra of NO₂XeF₅(SbF₆)₂ and XeF₅[Cu(SbF₆)₃]

The crystal structure of NO₂XeF₅(SbF₆)₂ consists of $[NO_2]^+$ and $[XeF_5]^+$ cations and $[SbF_6]^-$ anions.^[12] The Xe1 and F4 atoms of the $[XeF_5]^+$ cations are situated at the 2c crystallographic site (a special position) on a fourfold axis; the F3 atom is located in a general position, resulting in three additional symmetry-generated F3' atoms. All three atoms of the $[NO_2]^+$ cations are located on a twofold axis. Additionally, the nitrogen atom is placed at the intersection of three twofold axes (special position 2a) resulting in equal N-O distances. The Sb atoms occupy the 4f special position on a twofold axis resulting in three pairs of equivalent Sb–F bonds of the $[SbF_6]$ anion. In the latter, the F4 and F5 atoms are disordered between two positions around the F3-Sb1-F3' axis, with almost a 3:2 occupancy ratio between F4a and F5a atoms on the one side and F4b and F5b on the other.

The $[XeF_5]^+$ cation is based on pseudo-octahedral AX₅E VSEPR arrangements of bond pairs (X) and lone pairs (E), which give rise to a square-pyramidal geometry. The Xe–F_{ax} bond [1.811(2) Å] is shorter than the remaining four

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Date: 04-02-15 12:31:21

Pages: 5

Xe–Fe_{eq} distances [1.832(1) Å]. Each [XeF₅]⁺ forms four secondary contacts (2.786 Å) with F3 atoms coordinated to four different SbF₆ groups. The Sb–F3 distances involved in secondary bonding with [XeF₅]⁺ cations are 1.894(1) Å. In the linear [NO₂]⁺ cations the N–O bond lengths are equal to 1.101(2) Å (Figure 1).



Figure 1. Packing of $[NO_2]^+$ and $[XeF_3]^+$ cations, and $[SbF_6]^-$ anions in the crystal structure of $NO_2XeF_5(SbF_6)_2$. Thermal ellipsoids are drawn at the 50% probability level. Only one of the two orientations of the disordered $[SbF_6]^-$ anions is depicted.

The main feature of the XeF₅[Cu(SbF₆)₃] structure are rings of CuF₆ octahedra sharing apexes with SbF₆ octahedra connected into an infinite tridimensional framework (Figure 2).^[13] In this arrangement cavities are formed where [XeF₅]⁺ cations are located (Figure 3). Because of the secondary Xe···F contacts they are slightly shifted from the center of the cavities.



Figure 2. The crystal structure of $XeF_5[Cu(SbF_6)_3]$ showing rings of CuF_6 units sharing apexes with SbF_6 units, connected into an infinite tridimensional framework. The $[XeF_5]^+$ cations are located in the cavities (dark spheres: Sb; bright spheres: Cu; XeF_5 units have thicker Xe–F bonds).



Figure 3. Part of the crystal structure of $XeF_5[Cu(SbF_6)_3]$ showing the $[XeF_5]^+$ cation located in the cavity (bright octahedra: SbF_6 units, dark octahedra CuF_6 units). The secondary contacts between the $[XeF_5]^+$ cation and four $[SbF_6]^-$ anions are also shown; thermal ellipsoids are drawn at the 50% probability level.

A similar motif has been observed in AM(AsF₆)₃ (A = [H₃O]⁺, [NH₄]⁺, O₂⁺, NO⁺, K⁺, M = Mn; Fe, Co, Ni, Zn) compounds where single charged cations (A) are also found in a tridimensional framework consisting of MF₆ and AsF₆ octahedra (Figures S1 and S2).^[14–16] There are two crystallographically independent Cu atoms. The CuF₆ octahedra are distorted and their coordination can be described as 4+2 elongated octahedra [2×1.918(2)/2×1.968(2) Å and 2×2.128(2) Å] for Cu1 and as 2+4 compressed octahedra [2×1.926(2) Å and 2×2.038(2)/2×2.073(2) Å] in the case of Cu2.

Such unusual compressed coordination has been observed before in CuFAsF₆, H₃OCuF(AsF₆)₂, KCuAlF₆, and CsCuAlF₆.^[17,18] The shape of [XeF₅]⁺ is similar to that in NO₂XeF₅(SbF₆)₂ with an Xe–F_{ax} bond length [1.798(2) Å] shorter than in the case of Xe–F_{eq} [1.827(2)–1.835(2) Å]. Each XeF₅ group forms four secondary bond contacts (2.739, 2.872, 2.88, and 3.068 Å) with F atoms belonging to four [SbF₆]⁻ anions. The Sb–F distances of three crystallographically unique SbF₆ groups can be divided into three groups. The shortest are in the range 1.835(2)–1.847(2) Å for terminal F atoms (F_t). The Sb–F_t bond lengths where F_t are involved in secondary contacts with Xe atoms are slightly longer [1.870(2)–1.877(2) Å] but shorter than Sb–F_b bonds where fluorine atoms (F_b) bridge Sb and Cu atoms [1.894(2)–1.952(2) Å].

Raman Spectra of NO₂XeF₅(SbF₆)₂ and XeF₅[Cu(SbF₆)₃]

Raman spectra of $NO_2XeF_5(SbF_6)_2$ (1) and $XeF_5[Cu-(SbF_6)_3]$ (2) are shown in Figure 4 and given in Table 1.

The Raman spectra recorded for the powdered samples obtained by synthesis and the Raman spectra recorded for the single crystals of (1) and (2) were essentially identical. The strong Raman band at 654 cm⁻¹ in the Raman spectrum of (1) could be assigned to a symmetric stretching (v_1) mode and the weak ones at 547/552 cm⁻¹ and 288 cm⁻¹ to

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Date: 04-02-15 12:31:21

Pages: 5



Figure 4. Raman spectra of $NO_2XeF_5(SbF_6)_2$ (1) and $XeF_5[Cu-(SbF_6)_3]$ (2).

Table 1. Experimental Raman frequencies and intensities for $NO_2XeF_5(SbF_6)_2$ (1) and $XeF_5[Cu(SbF_6)_3]$ (2).

1	2	Assignments		
		$[XeF_5]^+$	$[SbF_6]^-$	$[NO_2]^+$
1405(50)				$v_1(\Sigma^+)$
	723(5)		$v_3(F_{1u})$	
	715(5)		$v_3(F_{1u})$	
	705(10)		$v_3(F_{1u})$	
687(5)				
678(90)	681(sh)	$v_1(A_1)$		
654(100)	675(100)		$v_1(A_{1g})$	
	654(10)	$v_7(E)$. 2.	
	635(sh)			
632(50)	631(95)	$v_2(A_1)$		
626(40)	610(20)	$v_2(A_1)$		
581(10)		$v_4(B_1)$		
571(15)	574(5)	$v_4(B_1)$		
552(10)			$v_2(E_g)$	
547(10)			$v_2(E_g)$	
	494(1)		0	
418(10)	406(15)	$v_8(E)$		
362(1)		$v_3(A_1)$		
312(3)	308(sh)	$v_3(A_1)$		
	297(5)	$v_3(A_1)$		
	290(sh)			
288(10)	283(4)		$v_5(F_{1g})$	
281(sh)			0	
274(sh)	272(2)	$v_5(B_1)$		
263(1)		$v_5(B_1)$		

 $v_2(\text{SbF}_6)$ and to bending (v_5) modes, respectively. The corresponding bands of the [SbF₆]⁻ anion in the Raman spectrum of NO₂SbF₆ are at 661(vs), 576(w)/568(ms) and 283(m)/294(w) cm⁻¹.^[19] The Raman band at 1405 cm⁻¹ is assigned to the [NO₂]⁺ cation.^[19] As already mentioned, the crystal structure of (**2**) resembles the structure type of AM-(AsF₆)₃ compounds^[14–16] where strong interactions between M²⁺ cations and [AsF₆]⁻ anions are observed via shared bridging fluorine atoms resulting in a similar tridimensional framework (Figure S1) as shown in Figure 2. Further investigations show that (H₃O)Cu(SbF₆)₃ appears to be isostructural and its Raman spectra show that the v_1 mode of the SbF₆ group is shifted to 672 cm⁻¹, meanwhile $v_2(\text{SbF}_6)$

and $v_5(\text{SbF}_6)$ were observed at 596 and 271/290 cm⁻¹, respectively (Figure S2). For this reason, the strongest band in the Raman spectrum of (2) (Figure 4) at 675 cm⁻¹ was assigned to $v_1(\text{SbF}_6)$ and the weak band at 283 cm⁻¹ to $v_5(\text{SbF}_6)$. The bands between 705–723 cm⁻¹, which are otherwise Raman inactive in O_h symmetry, are assigned to $v_3(\text{SbF}_6)$ because of the lowering of the symmetry in the solid state. The remainder of the vibrational bands are assigned to $[\text{XeF}_5]^+$ cations where $(\text{Xe}-\text{F})_{ax}$ stretching vibrations (v_1), the in-phase (Xe–F)_{eq} stretching vibrations in the XeF₄ moiety (v_2), and the out-of-plane in-phase bending vibration of XeF₄ (v_3) are found in the ranges 650–680, 580–630, and 330–360 cm⁻¹, respectively.^[20]

The weak bands at 620–660 belong to the doubly degenerate asymmetric stretching vibration of the XeF₄ moiety (v_7), medium weak bands at 410/420 cm⁻¹ to the F_{ax}–Xe–F_{eq} bending vibration (v_8), very weak bands at 260/270 cm⁻¹ to the out-of-plane bending (out of phase) vibration of XeF₄ (v_5), and the medium weak bands at 570/580 cm⁻¹ to the out-of-phase stretching vibration of XeF₄ (v_4). Since the ranges of different vibration modes observed in various [XeF₅]⁺ salts overlap^[20] the given assignment of vibration modes of the [XeF₅]⁺ cation is only tentative. The possibility that some of the cation vibrational bands overlap with the anionic ones could also not be completely excluded.

Conclusions

 $NO_2XeF_5(SbF_6)_2$ and $XeF_5[Cu(SbF_6)_3]$ are the first examples of $[XeF_5]^+$ /metal and $[XeF_5]^+$ /non-metal mixed-cation compounds. Their syntheses shows that the preparation of various new $[XeF_5]^+$ salts should be explored, which may lead to many new mixed-cation $[XeF_5]^+$ phases in the future.

Experimental Section

General: Volatile materials (anhydrous HF, F_2 , and SbF₅) were handled in a nickel vacuum line and an all-PTFE vacuum system equipped with PTFE valves as described previously.^[21] The nonvolatile materials were manipulated in a dry box (M. Braun, Germany). The residual water in the atmosphere within the dry box never exceeded 2 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 and PTFE-coated stirring bars. Prior to their use all reaction vessels were passivated with elemental fluorine (Solvay Fluor and Derivate GmbH, Germany).

 CoF_3 ,^[22] XeF₂,^[23] and Cu(SbF₆)₂^[24] were prepared as described previously. NO₂F was prepared by a modified method^[25] by reaction between NO₂ and CoF₃ in a nickel autoclave at 270 °C. NO₂SbF₆ and XeFSbF₆ were prepared by reaction between NO₂F or XeF₂ and SbF₅ in anhydrous HF. XeF₅SbF₆ was synthesized by reaction between XeFSbF₆ and elemental F₂ in aHF as solvent in the presence of a UV light source [400 W medium pressure mercury lamp (Baird and Tatlock, London, Type 400 LQ)].

Raman spectra with a resolution 0.5 cm⁻¹ were recorded at room temperature with a HORIBA JOBIN YVON LabRam-HR spec-

Pages: 5

www.eurjic.org

trometer equipped with an Olympus BXFM-ILHS microscope. Samples were excited by the 632.8 nm emission line of a He–Ne laser with regulated power in the range 20–0.0020 mW, which equals a power of 17–0.0017 mW focused to a 1 μ m spot on the top surface of the sample using a 50x microscope objective. The Raman spectra of both the bulk material and the single crystal were essentially identical. X-ray powder diffraction patterns 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. In a dry box corresponding amounts of XeF₅SbF₆ and NO₂SbF₆ [or Cu(SbF₆)₂] were loaded into a FEP reaction vessel. aHF (3 mL) was condensed onto the solid at 77 K and the reaction mixture was brought to ambient temperature. The reaction mixtures were stirred for Ld of ambient temperature resulting in along colorloss equations.

for 1 d at ambient temperature resulting in clear colorless solutions. Volatiles were slowly pumped off at ambient temperature for 2 h leaving behind a colorless solid. The final masses of the isolated solids were 151 mg (calcd. for $NO_2XeF_5[SbF_6]_2$: 158 mg) and 190 mg (calcd. for $[XeF_5]Cu[SbF_6]_3$: 187 mg). The Raman spectra were recorded, and the X-ray powder diffraction diagrams were obtained.

For both NO₂XeF₅[SbF₆]₂ and XeF₅[Cu(SbF₆)₃] a T-shaped apparatus consisting of two FEP tubes (19 and 6 mm outer diameter) was used for single crystal growth. NO₂XeF₅[SbF₆]₂ and XeF₅-[Cu(SbF₆)₃] (approximately 90 mg) were loaded into the wider arm of the crystallization vessel in the dry box. aHF (\approx 3 mL) was then condensed onto the starting material at 77 K. The crystallization mixture was brought up to ambient temperature and the clear colorless solution 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 5 wk. The result of this treatment was to slowly condense the aHF from the narrower into the wider tube, leaving behind the crystals.^[26] Selected single crystals were then placed inside 0.5 mm quartz capillaries in a dry box and their Raman spectra recorded.

Supporting Information (see footnote on the first page of this article): Details of crystal structure determination, Figures S1 and S2 showing the crystal structures of $AM(AsF_6)_3$ ($A = A^+$, $M = M^{2+}$) compounds and the Raman spectrum of $H_3OCu(SbF_6)_3$ (Figure S3).

Acknowledgments

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FULL PAPER

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- [13] Crystal data for XeF₅[Cu(SbF₆)₃]: colorless block, $M_r = 997.12$, monoclinic space group $P2_1/n$, a = 10.8166(4), b = 10.2511(4), c = 15.4289(4) Å, $\beta = 90.073(3)^\circ$, V = 1710.79(10) Å⁻³, Z = 4, $\rho_{calcd.} = 3.871$ gcm⁻³, F(000) = 1772, T = 150 K; 29493 reflections up to $\theta = 29.9^\circ$ collected, thereof 4293 with $I > 2\sigma(I)$, 4457 independent reflections, 257 parameters. Final *R* indices: R1 = 0.019 (all), wR2 = 0.038 (all). The final difference Fourier synthesis gave a min./max. residual electron density of 0.82/-2.31 eÅ⁻³.
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* Eurje uropean Journal of Inorganic Chemistry Pages: 5

Noble Gas Chemistry

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[XeF₅]⁺/Metal and [XeF₅]⁺/Non-Metal Mixed-Cation Salts of Hexafluoridoantimonate(V)

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 $NO_2XeF_5(SbF_6)_2$ and $XeF_5[Cu(SbF_6)_3]$ are the first examples of $[XeF_5]^+$ /metal and $[XeF_5]^+$ /non-metal mixed-cation compounds. The crystal structure of NO_2 - $XeF_5(SbF_6)_2$ is composed of $[NO_2]^+$ and $[XeF_5]^+$ cations and $[SbF_6]^-$ anions. In the crystal structure of $XeF_5[Cu(SbF_6)_3]$ the $[XeF_5]^+$ cations are located in cavities present in the tridimensional framework made up of CuF_6 and SbF_6 octahedra sharing joint apexes.