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Undecafluorodiarsenate Anion: Synthesis and Crystal Structure of $(MeS)_2CSH^+As_2F_{11}^-$

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Dimethyltrithiocarbonate reacts with the superacidic systems HF/MF_5 and DF/MF_5 (M = As, Sb) to form $(MeS)_2CSX^+MF_6^-$ (X = H, D). With an excess of AsF_5 the reaction leads to $(MeS)_2CSX^+As_2F_{11}^-$ (X = H, D). The latter salt crystallizes in the monoclinic space group $P2_1/c$ (No. 14 with $a = 7.687(1)$ Å, $b = 21.327(6)$ Å, $c = 8.704(1)$ Å, and $\beta = 91.99(1)°$) with four formula units per cell. The undecafluorodiarsenate anion contains an As−F−As bridge with two As−F bonds significantly longer than the nonbridging bonds and an As−F−As angle of 159.1(6)°. The Raman, IR, and ^1H, ^{75}As, ^{19}F, and ^{13}C NMR spectra of $(MeS)_2CSX^+SbF_6^-$ and $(MeS)_2CSX^+As_2F_{11}^-$ (X = H, D) are reported.

Introduction

A considerable amount of information has been accumulated on the behavior of pentafluorides as Lewis acids in anhydrous hydrogen fluoride. Gillespie and Moss used conductivity data and ^{19}F NMR to show that at about 20 mol % SbF_5 in HF the dominant anionic species is $Sb_2F_{11}^-$. Up to 10 mol % SbF_5 in HF the principal anion is SbF_6^-, and beyond 30 mol % SbF_5 the $Sb_3F_{16}^-$ anion and higher polymeric species are formed.[1] In 1969, Brownstein reported the formation of an $AsSbF_{11}^-$ (eq 1) and an $As_2F_{11}^-$ anion, which were characterized by NMR and IR spectroscopy.[2]

$$X^+SbF_6^- + AsF_5 \rightarrow X^+AsSbF_{11}^- \qquad (1)$$

Christe and Maya observed a band at 491 cm^{-1} in the low-temperature spectrum of $NOF_3/2AsF_5$ which was attributed to polymeric arsenic fluoro anions because on pumping on the solid this band disappeared and the composition changed to NOF_3/AsF_5.[3] Subsequent investigations of solutions of the Lewis acids SbF_5, AsF_5, and PF_5 in HF by Gillespie and co-workers provided evidence for an $As_2F_{11}^-$ anion. They compared the conductivity of an AsF_5 solution to that of a SbF_5 solution. On the basis of their measurements at 0 and −83.6 °C, they concluded that the $As_2F_{11}^-$ anion is formed essentially quantitatively according to eq 2.[4] Furthermore they were able to measure the ^{19}F NMR

$$2HF + 2AsF_5 \rightarrow H_2F^+ + As_2F_{11}^- \qquad (2)$$

$$R_4N^+AsF_6^- + AsF_5 \rightarrow R_4N^+As_2F_{11}^- \quad (R = Bu, Et) \qquad (3)$$

spectra of $Bu_4N^+As_2F_{11}^-$ and $Et_4N^+As_2F_{11}^-$ (eq 3) in SO_2 solution.[4,5] At higher temperature the spectra consist of only one sharp single signal, in contrast to the quartet of an AsF_6^- anion, due to As−F spin−spin coupling, and on cooling, the line was replaced by a more complex spectrum. O'Donnel et al. and Holloway, Schrobilgen, and co-workers reported Raman spectra of dimeric arsenic fluoro anions.[6,7] In 1979, Holloway et al. carried out X-ray powder diffraction studies on $M^{2+}(As_2F_{11}^-)_2$ species, but until now no crystal structure with an $As_2F_{11}^-$ anion has been published.[8] It should be noted that the structure of the $(MeS)_2CSH^+$ cation is unprecedented in the literature, too.

Recently we were informed by K. O. Christe that he and co-workers also were able to determine the crystal structure of an $As_2F_{11}^-$ anion and that this result was submitted for publication.[9] Nevertheless, except Raman and IR spectra, all previous attempts to characterize the undecafluorodiarsenate anion were made in solutions, but to compare the conformations of polymeric arsenic and antimony fluoro anions it is necessary to characterize them by X-ray studies. Polymeric fluoro anions of antimony are known, the $As_3F_{16}^-$ anion was mentioned once by Gillespie, and no polymeric phosphorus fluoro anions are known in the literature. In 1997, we successfully isolated an $As_2F_{11}^-$ salt. The first attempt to solve its crystal structure failed, but a later attempt succeeded. The existence of the $As_2F_{11}^-$ anion was also confirmed by a second additional crystal structure of $Me_3AsH^+As_2F_{11}^-$ which was solved by our work group.[10]

Experimental Section

Apparatus and Materials. All synthetic work and sample handling were performed by employing standard Schlenk techniques and a standard vacuum line (stainless steel or glass). SbF_5 (Merck) was fractionally distilled. The syntheses of $(MeS)_2CS$ and DF, using D_2SO_4 and CaF_2, and the drying of HF with F_2 and SO_2 with CaH_2 were carried out by known literature methods.[11,12] Reactions in HF/DF were carried out in a KEL-F reactor with a KEL-F valve. Infrared

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spectra were recorded with a Bruker IFS 113v spectrophotometer. Spectra of dry powders were obtained on a CsI plate in a cooled IR cell.[13] The Raman spectra were recorded with a T64000 (ISA) using a CCD detector (EEV CCD15-11) and an Ar^+ ion laser (514.5 nm) from Spectra Physics. The spectra were recorded in a glass cell cooled with liquid nitrogen. The NMR spectra were recorded with a Bruker DPX 300 spectrometer with TMS, $CFCl_3$, and $KAsF_6$ as external standards. Single crystals were placed in Lindemann capillaries in a cooled stream of dry nitrogen, and an X-ray diffraction study was carried out at −100 °C using an Enraf Nonius Mach3 automated diffractometer.

Synthesis of $(MeS)_2CS$. Na_2S (256 mmol, 20 g) was dissolved in 50 mL of H_2O, and 260 mmol (20.3 g) of CS_2 was added. After 1 h the solvent and unreacted CS_2 were removed under a dynamic vacuum at room temperature. Afterward 300 mmol (15.12 g) of CH_3Cl was added to the remaining salt. The mixture was kept at −10 °C for 1 h, and then the excess of CH_3Cl was removed under a dynamic vacuum at 0 °C. The remaining yellow liquid was dried with $MgSO_4$ and distilled at 25 °C with a yield of about 70% (24.7 g) based on the used Na_2S.

Synthesis of $(MeS)_2CSX^+SbF_6^-$ (X = H, D). In a 50 mL KEL-F reactor, 4 mmol (0.87 g) of SbF_5 was dissolved in 3 g of HF/DF. The solution was frozen at −196 °C, and 4 mmol (0.55 g) of $(MeS)_2CS$ was added under an inert atmosphere (N_2). The mixture was warmed to −78 °C, and the excess of HF/DF was slowly removed under a dynamic vacuum at −78 °C. The remaining colorless solid was identified as $(MeS)_2CSX^+SbF_6^-$ (X = H, D) by vibrational and NMR spectroscopy. The colorless salt is unstable at room temperature but can be held under inert conditions at −70 °C without decomposition.

Synthesis of $(MeS)_2CSX^+As_2F_{11}^-$ (X = H, D). In a 50 mL KEL-F reactor, 4 mmol (0.68 g) of AsF_5 was dissolved in 3 g of HF/DF. The solution was frozen at −196 °C, and 1 mmol (0.14 g) of $(MeS)_2CS$ was added under an inert atmosphere (N_2). The mixture was warmed to −78 °C, and the excess of HF/DF and AsF_5 was removed slowly under a dynamic vacuum at −78 °C. The remaining colorless solid was identified as $(MeS)_2CSX^+As_2F_{11}^-$ (X = H, D) by vibrational and NMR spectroscopy. The colorless salt can be stored under inert conditions at −70 °C without decomposition. The remaining $(MeS)_2CSH^+As_2F_{11}^-$ contained crystals suitable for X-ray diffraction studies.

Results and Discussion

Synthesis and Properties. The $(CH_3S)_2CS$ starting material is prepared by using Na_2CS_3 and an alkyl halogenide, such as CH_3Cl or CH_3I. By distillation at 0 °C under reduced pressure, the excess of alkyl halogenide can be removed, and the $(CH_3S)_2CS$ residue is distilled at room temperature under reduced pressure (eq 4). Dimethylthiomercaptocarbenium hexafluoroantimonate can be isolated at −78 °C (eq 5) by protonation of dimethyltrithiocarbonate in the superacidic systems XF/SbF_5 (X = H, D) with stoichiometric amounts of pentafluorometalates. The resulting slightly yellow salt is sensitive toward moisture and soluble in SO_2 and decomposes above −20 °C. Under inert conditions the salt can be stored for several weeks without decomposition at −70 °C. With an excess of AsF_5 in XF (X = H, D), the corresponding $As_2F_{11}^-$ salt is formed (eq 6). The resulting slightly yellow crystals are well soluble in HF and SO_2 and decompose at −38 °C to products which were not further characterized.

$$Na_2CS_3 + 2CH_3X \rightarrow (CH_3S)_2CS + 2NaX \quad (X = Cl, I) \quad (4)$$

$$(CH_3S)_2CS + XF + SbF_5 \rightarrow (CH_3S)_2CSX^+SbF_6^- \quad (X = H, D) \quad (5)$$

$$(CH_3S)_2CS + XF + 2AsF_5 \rightarrow (CH_3S)_2CSX^+As_2F_{11}^- \quad (X = H, D) \quad (6)$$

^1H and ^{13}C NMR Spectra of $(MeS)_2CSH^+SbF_6^-$ and $(MeS)_2CSH^+As_2F_{11}^-$. The ^1H NMR spectra of freshly prepared $(MeS)_2CSH^+As_2F_{11}^-$ and $(MeS)_2CSH^+SbF_6^-$ dissolved in SO_2 with TMS as external standard at −60 °C show singlets at 3.31 and 3.33 ppm, respectively, for the methyl groups and singlets at 7.25 and 7.54 ppm, respectively, for the mercapto group. In the ^{13}C NMR, the singlets at 22.1 and 22.2 ppm are assigned to the CH_3 groups and the singlets in the region of 231 ppm are assigned to the carbon of the CS_3 group. These results are comparable to the spectra of the $(HS)_3C^+$ cation observed by Olah.[14]

^{19}F and ^{75}As NMR Spectra of $(MeS)_2CSH^+As_2F_{11}^-$. The ^{19}F NMR spectrum of freshly prepared $(MeS)_2CSH^+As_2F_{11}^-$ dissolved in SO_2 with $CFCl_3$ and $KAsF_6$ as external standard at −60 °C shows a singlet at 51.3 ppm. As reported, on further cooling the signal will collapse and be replaced by signals comparable to those of $Bu_4N^+As_2F_{11}^-$ (21.1, 48.6, and 85 ppm at −140 °C).[4] Due to As−F spin−spin coupling, the ^{75}As spectrum shows no signal in the measured range whereas $KAsF_6$ shows a septet.

Crystal Structure of $(MeS)_2CSH^+As_2F_{11}^-$. Dimethylthiomercaptocarbenium undecafluorodiarsenate crystallizes in the monoclinic space group $P2_1/c$ (No. 14) with four molecules per unit cell (Table 1).[15] In Table 2, the bond lengths and angles are listed. Figure 1 shows the salt with atom labels, and the arrangement of the $(MeS)_2CSH^+As_2F_{11}^-$ in the unit cell is depicted in Figure 2. For the data reduction, a semiempirical correction determined from ψ-scan data and structure solution and refinement programs in the SHELXTL package and PARST were used.[16−19] The arsenic atoms were found by the Patterson method. All other atoms except the mathematically added methyl protons were determined by difference Fourier synthesis. A final refinement with anisotropic (except H atoms) thermal parameters gave a value of $R = 0.088$.

The anion consists of two strongly distorted octahedrons which are linked at one corner. The bridging As(1)−F(1) and As(2)−F(1) bonds with lengths of about 186.7(4) and 192.0(6) pm are significantly longer than the other As−F bonds, which are in the range of 163.8(7)−169.6(8) pm. By comparison with known crystal structures of $Sb_2F_{11}^-$ salts, the As(1)−F(1)−As(2) angle of 159.1(6)° is larger than the Sb−F−Sb angle of 146.2(4)° in $(CF_3)_2C(F)OH_2^+Sb_2F_{11}^-$ or 147.6(3)° in $[Hg(CO)_2][Sb_2F_{11}]_2$.[20,21] Due to the strong cationic and anionic interactions, a maximum deviation of 7.1(3)° from an ideal octahedron is observed for the AsF_6 units. The two octahedral AsF_6 units are

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(15) More details about the crystal structure are available at the Cambridge Crystallographic Data Centre (CCDC), 12 Union Road, Cambridge CB2 1EZ, U.K., under the number CCDC 134476 for $(MeS)_2CSH^+$-$As_2F_{11}^-$.

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Table 1. Crystal Data and Structure Refinement for $(MeS)_2CSH^+As_2F_{11}^-$

empirical formula	$C_3H_7As_2F_{11}S_3$
fw	498.11
temp (K)	173(2)
wavelength (Å)	0.710 69
cryst syst	monoclinic
space group	$P2_1/c$
a (Å)	7.687(1)
b (Å)	21.327(6)
c (Å)	8.7040(10)
β (deg)	91.99(1)
vol (Å^3)	1426.1(5)
Z	4
density (calcd) (mg/m^3)	2.320
abs coeff (mm^{-1})	5.229
F(000)	952
cryst size (mm)	0.3 × 0.25 × 0.2
θ range for data collection (deg)	3.02−27.97
index ranges	$-10 \le h \le 10$ $-28 \le k \le 28$ $-11 \le l \le 11$
reflns collected	4801
indept reflns	2327 ($R_{int} = 0.0413$)
abs correction	ψ-scans
max and min transm	0.9974 and 0.6327
refinement meth	full-matrix least squares on F^2
data/restraints/params	2327/0/176
goodness-of-fit on F^2	0.991
final R^a indices [$I > 4\sigma(I)$]	R1 = 0.0482, wR2 = 0.1097
R^a indices (all data)	R1 = 0.0882, wR2 = 0.1227
extinction coeff	0.0040(6)
largest diff peak and hole (e Å^{-3})	0.752 and −0.625

[a] $R = \sum||F_o| - |F_c||/\sum|F_o|$.

Table 2. Bond Lengths (pm), Selected Angles (deg), and Interionic Distances (pm) for $(MeS)_2CSH^+As_2F_{11}^-$

As(1)−F(1)	186.7(4)	F(1)−As(1)−F(2)	87.8(4)
As(1)−F(2)	165.2(7)	F(1)−As(1)−F(3)	83.6(4)
As(1)−F(3)	169.6(8)	F(1)−As(1)−F(4)	177.9(4)
As(1)−F(4)	167.3(4)	F(1)−As(1)−F(5)	86.0(3)
As(1)−F(5)	169.4(5)	F(1)−As(1)−F(6)	86.1(3)
As(1)−F(6)	168.7(5)	F(1)−As(2)−F(7)	175.9(3)
As(2)−F(1)	192.0(6)	F(1)−As(2)−F(8)	82.9(3)
As(2)−F(7)	167.6(4)	F(1)−As(2)−F(9)	84.5(3)
As(2)−F(8)	168.8(8)	F(1)−As(2)−F(10)	87.7(4)
As(2)−F(9)	165.4(5)	F(1)−As(2)−F(11)	87.7(4)
As(2)−F(10)	166.4(5)		
As(2)−F(11)	163.8(7)	As(1)−F(1)−As(2)	159.1(6)
C(1)−S(1)	172.2(6)	S(2)−C(1)−S(3)	121.2(4)
C(1)−S(2)	168.1(8)	S(2)−C(1)−S(1)	120.6(5)
C(1)−S(3)	168.2(8)	S(3)−C(1)−S(1)	118.2(5)
S(2)−C(2)	180.4(9)	C(1)−S(2)−C(2)	103.7(4)
S(3)−C(3)	177.4(8)	C(1)−S(3)−C(3)	104.2(4)
S(1)−H(1)	133(12)	C(1)−S(1)−H(1)	96(4)

positioned in a gauche conformation with a torsion angle F(11)−As(2)···As(1)−F(6) of 47.24° to assume the maximum F···F range.

Looking at the central carbon atom, the S−C(1)−S angles correspond within the 3σ range to an ideal 120° angle as expected for an sp^2 hybridization. The geometry of the CS_3 skeleton with a longer C(1)−S(1) bond of 172.2(6) pm and two shorter C(1)−S(CH_3) bonds of about 168.1(8) pm and 168.2(8) pm indicates that the positive charge is not equally distributed over the CS_3 fragment, and obviously the shorter methylthio carbon bonds show a higher degree of double-bond character. The proton H(1) and the two methyl carbon atoms C(2) and C(3) are positioned slightly out of the CS_3 plane.

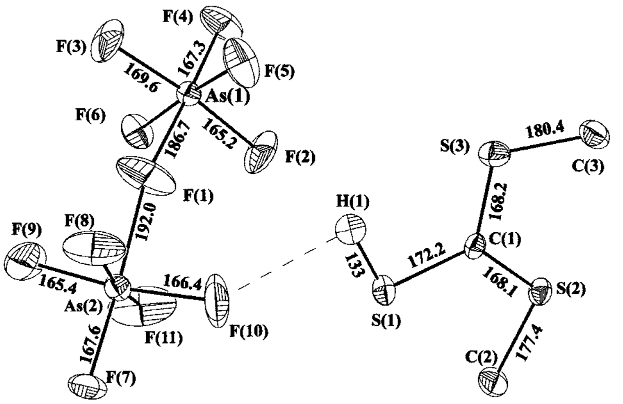

Figure 1. Projection of $(MeS)_2CSH^+As_2F_{11}^-$. Thermal ellipsoids are drawn at the 25% probability level.

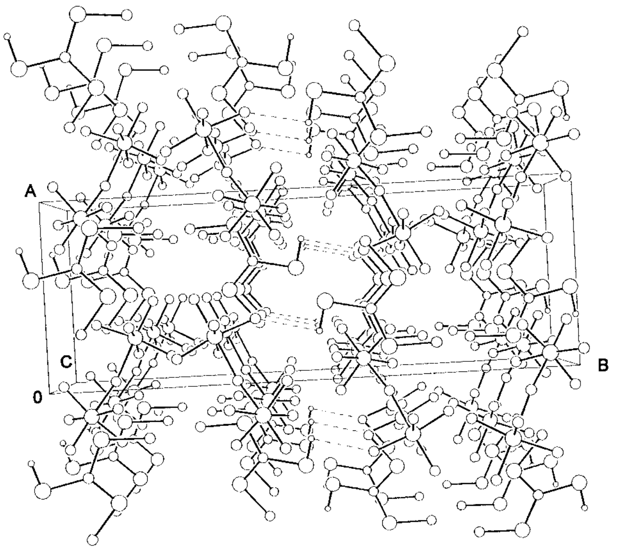

Figure 2. Projection of the $(MeS)_2CSH^+As_2F_{11}^-$ salt with contacts.

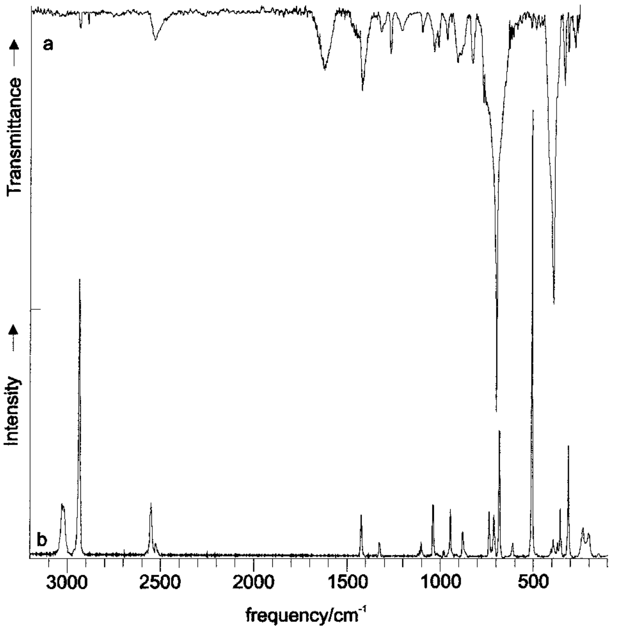

Figure 3. Infrared spectrum (trace a) and Raman spectrum (trace b) of $(MeS)_2CSH^+As_2F_{11}^-$.

Overall the crystal consists of AC planes with alternating ion pairs. Along the *B* axis the layers are piled up with hydrogen bridges between two planes. The distance between the donor atom S(1) and acceptor atom F(10) is about 300.3(7) pm and thus 10% below the sum of the van der Waals radii.[22] There

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Table 3. Raman and IR Frequencies and Possible Assignment of $(MeS)_2CSX^+SbF_6^-$ and $(MeS)_2CSX^+As_2F_{11}^-$ (X = H, D)

$(MeS)_2CSX^+SbF_6^-$				$(MeS)_2CSX^+As_2F_{11}^-$				
X = H		X = D		X = H		X = D		
IR	Raman	IR	Raman	IR	Raman	IR	Raman	possible assignment
3035w	3028(13)		3029(20)		3028(12)	3028w	3028(16)	
							3019(14)	νCH_3
	2936(43)	2937w	2935(47)	2933vw	2936(62)	2934vw	2936(84)	
2498w	2506(9)	1819m	1824(13)	2536w	2552(12)	1852w	1856(16)	νSX
1419w	1424(10)	1423m	1425(10)	1422m	1424(10)	1419m	1423(15)	
1314w	1317(4)	1317w	1318(4)	1320w	1325(4)	1320w	1326(3)	δCH_3
				1269w				
				1215vw		1224w		
						1197w		
1096m	1101(3)	1098vw		1099w	1101(4)	1097w		
1078w		1063w						$\nu C{=}S$
1031w	1034(8)	1034s	1033(8)	1032w	1038(12)	1036s	1039(13)	
		972w	968(5)	964w		968m		
		964m	957(3)					ρCH_3
935m	940(4)	936s	939(7)		946(11)	939s	943(8)	
				908m				
861m	875(3)	876vw			878(6)	875vw		νCS
818m		822vw		824w		819w		
	708(7)	708vs	711(10)		710(9)	714vs	711(9)	
703m		700vs						$\nu S{-}CH_3$
		677s	683(4)					
	645(29)	650vs	646(28)					τCSX
					608(3)		608(4)	
		560m	568(3)					
		552m		544w				$\nu C{-}S$
				531w				
496w	505(100)	497m	504(100)		508(100)		507(100)	
469s		467w		461w		465m		γCS_3
434m								
403s								
349m	353(8)	353m	354(8)		353(11)		353(11)	δCS_2
		327w						
	313(13)		309(12)		310(25)	311w	307(26)	
			238(4)		231(7)		229(7)	
			208(5)		200(5)		198(6)	τCH_3
				767m		757vs		
					736(10)		737(18)	νMF
655vs	657(5)	659vs		701vs		703vs		
572s	575		576(3)		683(28)		683(38)	
				484vw		482w		$\nu As{-}F{-}As$
289vs	280(12)	287vs	280(10)		392(4)	396vs	392(6)	δMF

are additional hydrogen bonds, but because of their longer interatomic distances they are not considered.

Raman and IR Spectra of $(MeS)_2CSX^+SbF_6^-$ and $(MeS)_2CSX^+As_2F_{11}^-$ (X = H, D). The infrared and Raman spectra of $(MeS)_2CSH^+As_2F_{11}^-$ are shown in Figure 3. The observed frequencies of the $(MeS)_2CSX^+$ salts of $As_2F_{11}^-$ and SbF_6^- (X = H, D) are summarized in Table 3. The assignments for the $(CH_3S)_2CSX^+$ cation were made by comparison to the calculated frequencies of $(CH_3S)_2CS$ and several alkylxanthogenates.[23−25]

On assumption that the $(CH_3S)_2CSX^+$ cation (X = H, D) has C_1 point symmetry, 33 Raman and IR active fundamental vibrations are expected. The antisymmetric and symmetric stretching modes of the methyl groups are observed in the region of 2933 cm^{-1}−3035 cm^{-1}. The νSH mode is observed at 2500 cm^{-1}, and the H/D shift to 1819 cm^{-1} for the SbF_6^- compound is nearly in accordance with the expected shift.[26] The νSH vibrational modes in the $As_2F_{11}^-$ salt are shifted ca. 40 cm^{-1} to higher frequencies because the interaction between anions and cations is weaker than in the SbF_6^- compound. The deformation and rocking modes of the methyl groups are observed in their normal ranges of 1425−1269 and 972−900 cm^{-1}. A sharp Raman line and IR band is detected at 465 cm^{-1} and is assigned to the CS_3 out of plane deformation mode.

A comparison of the spectra of the $As_2F_{11}^-$ and the SbF_6^- salts shows that the frequencies of 767 and 736 cm^{-1} occur only in the arsenic species. The symmetric degradation is responsible for the two additional As−F stretching modes. The band at 484 cm^{-1} is assigned to the stretching modes of the bridging As−F−As.[3,7] A complete assignment of the vibrational frequencies would require force field calculations.

Supporting Information Available: Tables listing detailed crystallographic data, atomic positional parameters, and bond lengths and angles and figures depicting the projection of $(MeS)_2CSH^+As_2F_{11}^-$ and IR and Raman spectra. This material is available free of charge via the Internet at http://pubs.acs.org.

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