

Vibrational and ^{119}Sn Mössbauer spectra and X-ray crystal structure of ammonium tetrafluorodimethylstannate(IV)

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Abstract

Ammonium tetrafluorodimethylstannate(IV) has been prepared and characterized by IR, Raman and ^{119}Sn Mössbauer spectroscopies. The Mössbauer parameters place this compound at the frontier between the dimethyltin(IV) compounds that gave rise to the quadrupole splitting (QS) *vs.* C–Sn–C angle correlation, and the dimethyltin(IV) salts of strong protic acids and superacids. The Mössbauer QS, as well as the IR and Raman spectra, indicate that the C–Sn–C angle is 180° . The IR-active $\nu(\text{Sn–F})$ E_u mode appears at 345 cm^{-1} , and this mode is related to the Mössbauer QS for *trans*- $[\text{SnF}_4\text{L}_2]$ because strong donors have a negative partial quadrupole splitting (pqs) and weaken the Sn–F bond. The IR spectrum also suggests the existence of normal (not polyfurcated) N–H $\cdots$ F hydrogen bonds. The X-ray crystal structure contains two crystallographically independent centrosymmetric $[\text{Me}_2\text{SnF}_4]^{2-}$ anions, but only one kind of NH_4^+ cation. The cations and the anions are linked by nearly linear N–H $\cdots$ F hydrogen bonds giving rise to a three-dimensional network. The average Sn–F distance is $2.127\text{ \AA}$ and the usefulness of covalent radii to predict accurate bond distances is challenged. An equation to calculate the average Sn–F distance in complexes $[\text{SnF}_4\text{L}_2]$ from the Mössbauer QS is proposed.

Key words: Tin; Mössbauer spectroscopy; Infrared spectroscopy; Raman spectroscopy; Hydrogen bonding; Crystal structure

1. Introduction

The Mössbauer quadrupole splitting (QS) of octahedral tin(IV) chloride [1] and bromide [2,3] complexes is related to the Sn–X (X = Cl or Br) distances. Furthermore, QS is also related to the $\nu(\text{Sn–X})$ frequencies [2,4]. These relationships indicate that ligands with negative partial quadrupole splitting (pqs) values are strong donors that weaken the Sn–X bond, thus leading to long Sn–X distances and low $\nu(\text{Sn–X})$ frequencies. In the case of octahedral complexes $[\text{SnF}_4\text{L}_2]$, only one crystal structure, of $[\text{SnF}_4(\text{bipy})]$, has been reported [5], and a few Mössbauer data of inorganic [6] and organotin complexes [7–9] are available in the literature. Bearing in mind that alkyl groups have the most negative pqs values [10], complexes $[\text{R}_2\text{SnF}_4]^{2-}$ should display the longest Sn–F distances and the lowest $\nu(\text{Sn–F})$ frequencies of $[\text{SnF}_4\text{L}_2]$ compounds. Therefore, a knowledge of the crystal structures of compounds containing the $[\text{R}_2\text{SnF}_4]^{2-}$ anion would be very desirable, but the lack of structural data for this

type of compound has been noted recently [11,12]. Consequently, we have tried to grow single crystals of compounds containing the $[\text{Me}_2\text{SnF}_4]^{2-}$ anion, and we have been successful in the case of $(\text{NH}_4)_2[\text{Me}_2\text{SnF}_4]$ which was first prepared by Wilkins and Haendler [13]. Further interest in the structure of this compound is provided by the possibility of N–H $\cdots$ F hydrogen bonds and their influence on the crystal structure [14]. This paper describes the IR, Raman and Mössbauer spectra, and the X-ray crystal structure of $(\text{NH}_4)_2[\text{Me}_2\text{SnF}_4]$.

2. Experimental details

2.1. Preparation

Crystallization of an aqueous solution of Me_2SnF_2 and NH_4F in a 1:2 molar ratio [13] yielded crystals of both $(\text{NH}_4)[\text{Me}_2\text{SnF}_3]$ and $(\text{NH}_4)_2[\text{Me}_2\text{SnF}_4]$. Nevertheless, slow evaporation of an aqueous solution of the same reagents in a 1:3 molar ratio yielded large colourless crystals of $(\text{NH}_4)_2[\text{Me}_2\text{SnF}_4]$ only. Anal.

Found C, 9.1; H, 5.2; N, 10.6. $C_2H_{14}F_4N_2Sn$ calc: C, 9.2; H, 5.4; N, 10.7%. IR: 3200s, 3045vs, broad, 2996sh, 2919vs, 2885sh, 2856vs, 2187m, 1927m, 1720w, 1492s, 1456s, 1424m, 1412m, 1203w, 790m, 588m, 583m, 345s, 292sh, 230sh cm^{-1} ; Raman: 540vvs, 527vvs, 353vw, 261sh, 246m, 221w, 190w, 132w cm^{-1} ; Mössbauer: IS = 1.12, QS = 4.18, $\Gamma_1 = 1.05$, $\Gamma_2 = 1.07$ mm s^{-1} .

2.2. Physical measurements

Infrared spectra (4000–220 cm^{-1}) were recorded on a Perkin Elmer 1600 FT-IR instrument, using Nujol and Fluorolube mulls between CsI windows. Raman spectra (600–100 cm^{-1}) were recorded on a Jarrell-Ash spectrophotometer, model 25-300, using Ar (5145 Å) and Kr (6471 Å) lasers with the powdered sample sealed in a capillary tube. The Mössbauer spectrum at liquid N_2 temperature was obtained as described previously [15], but velocity calibration was carried out with the six lines of an α -Fe foil. The isomer shift is referred to $BaSnO_3$ at room temperature, and the reproducibility of the Mössbauer parameters was ± 0.02 mm s^{-1} .

A single crystal of $(NH_4)_2[Me_2SnF_4]$ suitable for X-ray diffraction experiments was cut from a bigger fragment.

2.3. Crystal data

$C_2H_{14}F_4N_2Sn$, $M = 260.8$, monoclinic, $A2/m$ (No. 12), $a = 11.023(2)$, $b = 10.514(2)$, $c = 7.564(1)$ Å, $\beta = 103.64(1)^\circ$, $V = 851.9(3)$ Å³ (by least-squares refinement on diffractometer angles for 25 reflections), $Z = 4$, $D_c = 2.034$ g cm^{-3} , $D_m = 2.02$ g cm^{-3} (by flotation in $CCl_4/CHBr_3$), $F(000) = 504$, $\mu(Mo K\alpha) = 30.1$ cm^{-1} , $T = 295$ K.

2.4. Data collection and processing

Reflection data were collected on an Enraf-Nonius CAD4 diffractometer. The intensity data were measured in the range $1 \leq \theta \leq 24^\circ$ ($-13 \leq h \leq 13$, $0 \leq k \leq 12$, $0 \leq l \leq 9$), using the ω - 2θ scan mode with ω scan width = $0.80 + 0.35 \tan \theta$ and a variable scan speed with maximum recording time 60 s. A total of 794 unique reflections were measured, using graphite monochromated Mo $K\alpha$ radiation ($\lambda = 0.71069$ Å); of these, 677 with $I \geq 2\sigma(I)$ were considered as observed. No significant crystal decay was detected.

2.5. Structure analysis and refinement

After corrections for Lorentz and polarization factors, the structure was solved by Patterson and Fourier methods. An empirical absorption correction [16] applied at the end of the isotropic refinement by using unit weights led to a conventional R value of 0.049. Anisotropic refinement for the non-hydrogen atoms

TABLE 1. Fractional atomic coordinates for $(NH_4)_2[Me_2SnF_4]$

Atom	x	y	z
Sn(1)	0	0	0
Sn(2)	0.5	0	0.5
C(1)	0.0809(8)	0	0.2824(12)
C(2)	0.4715(9)	0	0.2149(12)
F(1)	0	0.2030(4)	0
F(2)	0.1767(4)	0	-0.0658(6)
F(3)	0.3594(3)	0.1412(3)	0.4812(4)
N	0.2230(5)	0.2986(6)	0.2237(7)
H(1)	0.147(8)	0.263(8)	0.154(11)
H(2)	0.209(8)	0.377(8)	0.306(11)
H(3)	0.270(7)	0.231(9)	0.320(13)
H(4)	0.269(8)	0.319(9)	0.157(12)

led to $R = 0.036$. At this stage, a difference Fourier map revealed the positions of the ammonium ion H atoms, but the H atoms corresponding to the methyl groups could not be located. Final refinements were carried out with anisotropic temperature factors for the non-hydrogen atoms and fixed isotropic thermal parameters for the H atoms. Final R and R_w values were 0.033 and 0.049 for 677 observed reflections (number of variables = 62); maximum shift/c.s.d. = 0.01. In order to prevent bias on $\langle F \rangle$ vs. $\langle F_o \rangle$ or $\langle (\sin \theta)/\lambda \rangle$, the last steps of the refinement were carried out with weights calculated by means of PESOS [17]. Scattering factors for neutral atoms, and anomalous dispersion correction for Sn, were taken from the International Tables [18], and most of the calculations were carried out with XRAY80 [19]. The final atomic coordinates are given in Table 1. Full lists of structure factors and anisotropic thermal parameters are available from the author, and the data have also been deposited at the Cambridge Crystallographic Data Centre.

3. Results and discussion

3.1. Mössbauer and vibrational spectra

Aubke and co-workers have reported a linear correlation between the IS and QS values for dimethyltin(IV) derivatives of strong protic acids and superacids [9]. The compounds included in the correlation have very high QS values and are clearly distinct from the dimethyltin(IV) complexes that had formed the experimental bases for the application of the point-charge model to dimethyltin(IV) compounds and the calculation of C–Sn–C angles from the Mössbauer QS [20]. Whereas the Mössbauer parameters of $K_2[Me_2SnF_4]$ (IS = 1.38, QS = 4.12 mm s^{-1}) [7,8] deviate significantly from the correlation, $Li_2[Me_2SnF_4]$ (IS = 1.31, QS = 4.60 mm s^{-1}) fits [9]. Furthermore, the parameters of the title compound (IS = 1.12, QS = 4.18 mm

s^{-1}) also fit the correlation. A possible explanation would be that the polarizing power of the small Li^+ ions, and $N-H \cdots F$ hydrogen-bond formation in the ammonium salt, reduce the nucleophilicity of the F^- anions around the Me_2Sn group, therefore behaving like the strong protic acid and superacid derivatives that are the basis of the correlation [9]. Moreover, the QS of $(NH_4)_2[Me_2SnF_4]$ indicates that the C–Sn–C group is essentially linear [20], and the title compound is at the frontier between the dimethyltin(IV) compounds that gave rise to the QS *vs.* C–Sn–C angle correlation [20] and the dimethyltin(IV) salts of strong protic acids and superacids reported by Aubke and co-workers [9].

The IR spectrum of $(NH_4)_2[Me_2SnF_4]$ (see Experimental section) shows $\nu_{as}(Sn-C)$ at 588 and 583 cm^{-1} , but no band assignable to $\nu_s(Sn-C)$. The Raman spectrum, in contrast, shows $\nu_s(Sn-C)$ at 540 and 527 cm^{-1} , but no $\nu_{as}(Sn-C)$. Therefore, the C–Sn–C fragment must be linear, as indicated by the Mössbauer QS. On the basis of a normal-coordinate analysis of $K_2[Me_2SnF_4]$ [21], a very weak band at 353 cm^{-1} in the Raman spectrum of $(NH_4)_2[Me_2SnF_4]$ can be assigned tentatively to $\nu(Sn-F)$ (A_{1g}). Nevertheless, the IR-active $\nu(Sn-F)$ E_u mode is more unambiguously assigned at 345 cm^{-1} . We have reported linear relationships between the Mössbauer QS and the IR active $\nu(Sn-X)$ (E_u) frequency for *trans*- $[SnCl_4L_2]$ [4] and *trans*- $[SnBr_4L_2]$ [2]. In the case of *trans*- $[SnF_4L_2]$ very few data are available (Table 2), but they seem to indicate a similar behaviour. The trend shown in Table 2 can be rationalized by considering that strong donors have a negative pqs and weaken the Sn–F bond, thus lowering $\nu(Sn-F)$.

A linear correlation of the four points ($r = 0.997$) gives the relation E_u (cm^{-1}) = $[(-46 \pm 3) QS + (551 \pm 8)]$. The intercept (i.e. the E_u frequency corresponding to QS = 0) is close to the $\nu(Sn-F)$ (F_{1u}) frequency of the octahedral $[SnF_6]^{2-}$ (559 cm^{-1}) [22]. Similarly, the intercepts of the correlations $\nu(Sn-X)$ (E_u) *vs.* QS for *trans*- $[SnX_4L_2]$, 317 cm^{-1} for X = Cl [4] and 221 cm^{-1}

for X = Br [2], are also close to the $\nu(Sn-X)$ (F_{1u}) for octahedral $[SnX_6]^{2-}$ (311 and 224 cm^{-1} for X = Cl and Br, respectively) [22]. This is due to the fact that the symmetry species E_u derives from the species F_{1u} in the correlation from the O_h to the D_{4h} point groups [23]. Therefore, the $[SnX_6]^{2-}$ anions behave as a particular case of $[SnX_4L_2]$ complexes with $L = X^-$.

The bands at 2996 [$\nu_{as}(CH_3)$], 2919 [$\nu_s(CH_3)$], 1424–1412 [$\delta_{as}(CH_3)$], 1203 [$\delta_s(CH_3)$] and 790 cm^{-1} [$\rho(Sn-CH_3)$] in the IR spectrum of $(NH_4)_2[Me_2SnF_4]$ (see Experimental section) can be assigned to vibrations involving the Me_2Sn^{IV} group [21]. The rest of the bands above 700 cm^{-1} must correspond to vibrations involving the ammonium ion. The internal modes of vibration of the NH_4^+ ion under T_d symmetry, are ν_1 [$\nu_s(N-H)$], ν_2 [$\delta_s(NH_2)$], ν_3 [$\nu_{as}(N-H)$] and ν_4 [$\delta_{as}(NH_2)$]; while the external modes are usually denoted by ν_5 (translation) and ν_6 (libration) [24]. The N–H stretching region in the vibrational spectra of ammonium salts usually consists of a broad, complex absorption (3400–2800 cm^{-1}) in which extensive Fermi resonance occurs between the N–H stretching fundamentals and several overtone and combination modes, such as $2\nu_4$, $2\nu_2$, $\nu_2 + \nu_4$, $\nu_1 + \nu_5$, *etc.* [24]. This complexity is also evident in the IR spectrum of $(NH_4)_2[Me_2SnF_4]$, although the bands at 2187, 1927, 1720 and 1492–1456 cm^{-1} can be assigned to $\nu_2 + \nu_6$, $\nu_4 + \nu_6$, ν_2 and ν_4 , respectively [25].

Dunsmuir and Lane proposed three criteria for hydrogen bonding of the ammonium ion in crystals: (i) observation of both $\nu_2 + \nu_6$ and $\nu_4 + \nu_6$ combination modes in the IR spectrum, (ii) the appearance of ν_4 above 1400 cm^{-1} , and (iii) a broadening of the lattice bands in the far IR region [25]. The first criterion was later shown to relate more to the type than to the strength of the hydrogen-bonding present, because the combination modes are usually observed in the room-temperature spectra of ammonium salts in which normal hydrogen bonds are present; but not in those where the hydrogen bonds are polyfurcated [26]. In our compound the lattice bands are expected in the region where skeletal vibrations of the $[Me_2SnF_4]^{2-}$ anion should appear, and they are therefore not assigned. However, ν_4 appears above 1400 cm^{-1} and combination modes involving the libration ν_6 are clearly present in the IR spectrum, so that $(NH_4)_2[Me_2SnF_4]$ should have normal (not polyfurcated) $N-H \cdots F$ hydrogen bonds.

3.2. X-Ray crystal structure

The unit cell of $(NH_4)_2[Me_2SnF_4]$ contains two crystallographically independent centrosymmetric $[Me_2SnF_4]^{2-}$ anions, but only one kind of NH_4^+ cation. Every cation is linked to four different octahedral

TABLE 2. Mössbauer (mm s^{-1}) and IR (cm^{-1}) data for *trans*- $[SnF_4L_2]$

L	QS ^a	Ref.	$\nu(Sn-F)(E_u)$	Ref.
Me ^b	+4.18	This work	345	This work
Me ^c	+4.12	7, 8	372	21
Py	−0.45	6	569	6
THF	−1.03	6	601	6

^a The sign of the QS has been determined experimentally for $K_2[Me_2SnF_4]$ [8]. For the other compounds, the sign has been assigned on the basis of chemical considerations [2,4] and better correlation with the $\nu(Sn-F)$ mode. ^b NH_4^+ salt. ^c K^+ salt.

anions by N–H···F hydrogen bonds, and every anion is connected to eight cations, giving rise to a three-dimensional network. Bond and contact distances and angles are collected in Table 3. The NH_4^+ ion is close to tetrahedral with average N–H distances and H–N–H angles of 0.98 Å and 109°, respectively. Every ammonium H atom is involved in a nearly linear hydrogen bond to a F atom with H···F distances ranging from 1.67(8) Å to 1.89(10) Å. In order to maximize the number of hydrogen bonds, every F atom is connected to two H atoms, because the compound contains two ammonium H atoms for every F atom. Therefore, the three-dimensional structure is governed by the hydrogen bonding. Interestingly, while $\text{K}_2[\text{Me}_2\text{SnF}_4]$ crystallizes as a dihydrate from aqueous solutions [13], the title compound crystallizes anhydrous, probably because all the F atoms are already involved in hydrogen bonds. The fact that the hydrogen bonds are normal (not polyfurcated) agrees with the predictions from IR spectroscopy (see above).

The tin atoms in $(\text{NH}_4)_2[\text{Me}_2\text{SnF}_4]$ are sites of $2/m$ symmetry, so that the two crystallographically independent octahedral anions (see Fig. 1) are centrosymmetric. Therefore, both C–Sn–C angles are exactly of 180° consistent with the IR, Raman and Mössbauer spectroscopic evidence. The geometry around both Sn atoms is very similar (see Table 3), with bond angles close to the regular octahedral ones, and it is not surprising that the Mössbauer spectrum shows only one doublet. On the other hand, the presence of the two independent anions in the unit cell can account for the splitting of $\nu_{\text{as}}(\text{Sn}-\text{C})$ and $\nu_{\text{s}}(\text{Sn}-\text{C})$ in the IR and Raman spectra, respectively. The average Sn–F bond distance,

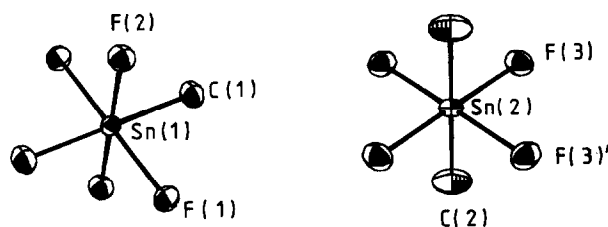


Fig. 1. ORTEP view of the two crystallographically independent centrosymmetric anions in $(\text{NH}_4)_2[\text{Me}_2\text{SnF}_4]$.

2.127 Å, is significantly longer than for the octahedral $[\text{SnF}_6]^{2-}$ (1.955 Å) [27–29], consistent with the idea that the strong donor methyl groups weaken the Sn–F bonds. Recently, a new value of 0.54 Å for the covalent radius of fluorine has been proposed [30]. Taking into account the accepted covalent radius of tin (1.40 Å) [31], the calculated Sn–F distance is 1.94 Å in good agreement with the experimental distance in $[\text{SnF}_6]^{2-}$ (1.955 Å) [27–29], but too short when compared to the Sn–F distance in the title compound (2.127 Å). We have noted previously that the range of Sn–Cl distances in octahedral complexes $[\text{SnCl}_4\text{L}_2]$ spans a range of 0.29 Å [1]. Therefore, the application of the concept of covalent radii is questionable for calculating accurate bond distances.

In the case of $[\text{SnX}_4\text{L}_2]$ (X = Cl or Br), we have quantified the relationship between the average Sn–X bond distances and the donor power of the ligands by means of the correlation with the Mössbauer pqs of the ligands, where $4\text{pqs} = -\text{QS}$ for *trans* compounds or 2 QS for *cis* compounds [1–3]. In these correlations, the intercept is close to the Sn–X distance in $[\text{SnX}_6]^{2-}$ and the slope is close to that calculated from $[\text{R}_2\text{SnX}_4]^{2-}$ and $[\text{SnX}_6]^{2-}$. In fact, a straight line calculated from only the two points can be used to calculate Sn–X distances, which are consistent with the experimental ones within ± 0.02 Å. In the case of $[\text{SnF}_4\text{L}_2]$, only the points corresponding to $(\text{NH}_4)_2[\text{Me}_2\text{SnF}_4]$ ($4\text{pqs} = -4.18 \text{ mm s}^{-1}$; $d(\text{Sn}-\text{F}) = 2.127 \text{ Å}$) and $[\text{SnF}_6]^{2-}$ ($4\text{pqs} = 0$; $d(\text{Sn}-\text{F}) = 1.955 \text{ Å}$) [27–29] are available, but by comparison with $[\text{SnX}_4\text{L}_2]$ (X = Cl or Br) [1–3], a linear relationship is also expected. In that case, eqn. (1) may be used to predict approximate QS or $d(\text{Sn}-\text{F})$ data.

$$d(\text{Sn}-\text{F}) = -0.041(4\text{pqs}) + 1.955 \text{ Å} \quad (1)$$

According to eqn. (1), *cis*- $[\text{SnF}_4(\text{bipy})]$ ($d(\text{Sn}-\text{F}) = 1.934 \text{ Å}$) [5] should have a very small and unresolvable QS (*ca.* 0.26 mm s^{-1}), and an average Sn–F distance as short as 1.88 Å is expected for *cis*- $[\text{SnF}_4(\text{MeCN})_2]$ (QS = 0.97 mm s^{-1}) [6]. The wide range of Sn–F

TABLE 3. Bond and contact distances (Å) and angles (°) in $(\text{NH}_4)_2[\text{Me}_2\text{SnF}_4]$

Sn(1)–C(1)	2.112(8)	C(1)–Sn(1)–F(1)	90
Sn(2)–C(2)	2.105(9)	C(1)–Sn(1)–F(2)	92.6(3)
Sn(1)–F(1)	2.135(4)	C(2)–Sn(2)–F(3)	90.1(2)
Sn(1)–F(2)	2.121(5)	F(1)–Sn(1)–F(2)	90
Sn(2)–F(3)	2.126(3)	F(3)–Sn(2)–F(3)'	91.5(1)
N–H(1)	0.95(8)	H(1)–N–H(2)	113(7)
N–H(2)	1.07(9)	H(1)–N–H(3)	110(7)
N–H(3)	1.06(9)	H(1)–N–H(4)	110(8)
N–H(4)	0.82(10)	H(2)–N–H(3)	104(7)
N...F(1)	2.820(6)	H(2)–N–H(4)	111(8)
N...F(2)''	2.768(7)	H(3)–N–H(4)	109(8)
N...F(3)	2.722(6)	N–H(1)...F(1)	175(8)
N...F(3)'''	2.707(7)	N–H(2)...F(2)''	177(7)
H(1)...F(1)	1.87(8)	N–H(3)...F(3)	171(8)
H(2)...F(2)''	1.70(9)	N–H(4)...F(3)'''	173(8)
H(3)...F(3)	1.67(9)		
H(4)...F(3)'''	1.89(10)		

Symmetry codes: (') $1-x, y, 1-z$; (") $x, \frac{1}{2}+y, \frac{1}{2}+z$; (""') $x, \frac{1}{2}-y, z-\frac{1}{2}$.

distances in $[\text{SnF}_4\text{L}_2]$ complexes further challenges the concept of covalent radii in these compounds.

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