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Organotin complexes with 1-methyl-2(3H)-imidazolinethione. The crystal structure of dichloro[1-methyl-2(3H)-imidazolinethione]dimethyltin(IV)

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Abstract

The compounds $[SnMe_2X_2(Hmimt)]$, $[SnMe_2X_2(Hmimt)_2]$ and $(Et_4N)[SnMe_2X_3(Hmimt)]$ (X = Cl or Br; Hmimt = 1-methyl-2(3*H*)-imidazolinethione) have been prepared and characterized by conductivity measurements and by IR, Raman, Mössbauer and ^1H, ^{13}C and ^{119}Sn NMR spectroscopy. The structure of $[SnMe_2Cl_2(Hmimt)]$ was determined by X-ray diffraction. Its crystals are triclinic, space group $P\bar{1}$, with a 9.702(2), b 9.375(2), c 7.119(1) Å, α 68.1(2), β 86.6(2), γ 85.6(2)°, U 598.7 Å^3, $Z = 2$, $R = 0.062$, $R_w = 0.058$ and consist of chlorine-bridged $[SnMe_2Cl_2(Hmimt)]$ dimers in which the hexacoordinate tin atoms have distorted octahedral environments and the thiones are S-bonded. Neighbouring dimers are linked by NH ··· Cl bonds. Mössbauer and vibrational data suggest octahedral coordination for the tin atom in $[SnMe_2X_2(Hmimt)_2]$ and $(Et_4N)[SnMe_2X_3(Hmimt)]$.

1. Introduction

In previous work [1] we prepared the compound $[SnMe_2Br_2(Hmimt)]$ (Hmimt = 1-methyl-2(3*H*)-imidazolinethione). X-ray diffraction analysis showed that in this compound the tin atom has a 5 coordinate bipyramidal trigonal environment bound to two methyl carbon atoms and a sulphur atom in equatorial positions and two bromine atoms in axial positions. This interesting geometry prompted us to initiate a detailed structural study of the chlorine analogue and of the possibility of increasing the coordination number of the tin atom. This paper reports the crystal and molecular structure of $[SnMe_2Cl_2(Hmimt)]$ and the preparation of the compounds $[SnMe_2X_2(Hmimt)_2]$ and $(Et_4N)[SnMe_2Cl_3(Hmimt)]$ (X = Cl or Br). X-ray diffraction analysis of $[SnMe_2Cl_2(Hmimt)]$ shows a hexacoordinate tin environment achieved by chlorine bridging. The vibrational and Mössbauer spectra of the other compounds are in keeping with the assumption that in these cases hexacoordination is achieved by coordination of a second Hmimt or a halide ion.

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2. Experimental details

2.1. Materials

Dimethyltin dichloride and dimethyltin dibromide (Ventron) and Hmimt (Aldrich) were used as supplied. Solvents were purified by standard methods.

2.2. Preparation of compounds

$[SnMe_2Cl_2(Hmimt)]$. A solution of Hmimt (5.66 mmol) in CH_2Cl_2 (20 ml) was added dropwise to a solution of $SnMe_2Cl_2$ (5.56 mmol) in CH_2Cl_2 (15 ml). After stirring, the solvent was evaporated off and the beige solid obtained was dried *in vacuo*. Anal. Found: C, 22.3; H, 3.5; N, 9.4. $C_6H_{12}Cl_2N_2SSn$ calc.: C, 21.6;

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H, 3.6; N, 8.4%. M.p. 121°C. Λ_M (MeCN, 10^{-3} M) 1.9 S cm^2 mol^{-1}. Crystals suitable for the X-ray analysis were obtained from $CHCl_3$ solutions.

[$SnMe_2Cl_2(Hmimt)_2$]. A solution of $SnMe_2Cl_2$ (2.83 mmol) in CH_2Cl_2 (15 ml) was added dropwise to a solution of Hmimt (5.66 mmol) in $CHCl_3$ (20 ml). After stirring for several days, the solvent was evaporated off and a beige solid obtained. Anal. Found: C, 27.5; H, 4.1; N, 13.2. $C_{10}H_{18}Cl_2N_4S_2Sn$ calc.: C, 26.8; H, 4.1; N, 12.5%. M.p. 116°C. Λ_M (MeCN, 10^{-3} M) 2.6 S cm^2 mol^{-1}.

[$SnMe_2Br_2(Hmimt)$]. A solution of Hmimt (3.36 mmol) in CH_2Cl_2 (20 ml) was added dropwise to a solution of $SnMe_2Br_2$ (3.36 mmol) in CH_2Cl_2 (15 ml). Upon stirring, the white solid which formed was filtered off and dried *in vacuo*. Anal. Found: C, 17.2; H, 2.9; N, 6.5. $C_6H_{12}Br_2N_2SSn$ calc.: C, 17.1; H, 2.9; N, 6.6%. M.p. 142°C. Λ_M (MeCN, 10^{-3} M) 3.0 S cm^2 mol^{-1}.

[$SnMe_2Br_2(Hmimt)_2$]. A solution of $SnMe_2Br_2$ (3.10 mmol) in CH_2Cl_2 (20 ml) was added dropwise to a solution of Hmimt (6.20 mmol) in CH_2Cl_2 (20 ml). After stirring, the solvent was evaporated off and the white solid obtained was dried *in vacuo*. Anal. Found: C, 22.4; H, 3.2; N, 10.4. $C_{10}H_{18}Br_2N_4S_2Sn$ calc.: C, 22.4; H, 3.4; N, 10.4%. M.p. 141°C. Λ_M (MeCN, 10^{-3}M) 3.6 S cm^2 mol^{-1}.

(Et_4N)[$SnMe_2Cl_3(Hmimt)$]. A solution of Hmimt (3.23 mmol) in acetone (15 ml) was added dropwise to a solution of $SnMe_2Cl_2$ (3.23 mmol) in acetone (15 ml). The mixture was stirred for 3 d, a solution of $Et_4NCl \cdot H_2O$ (3.23 mmol) in MeOH (15 ml) was added dropwise, and after stirring 1 d the solvent was evaporated off and the white solid obtained was dried *in vacuo*. Anal. Found: C, 33.2; H, 6.1; N, 7.9. $C_{14}H_{32}Cl_3N_3SSn$ calc.: C, 33.7; H, 6.5; N, 8.4%. M.p. 94°C. Λ_M (MeCN, 10^{-3} M) 156.0 S cm^2 mol^{-1}.

(Et_4N)[$SnMe_2Br_3(Hmimt)$]. A solution of Hmimt (5.76 mmol) in acetone (20 ml) was added dropwise to a solution of $SnMe_2Br_2$ (5.76 mmol) in acetone (20 ml). The mixture was stirried for 3 d, a solution of Et_4NBr (5.76 mmol) in MeOH (20 ml) was added dropwise, and after stirring 1 d the solvent was evaporated off and the beige solid obtained was dried *in vacuo*. Anal. Found: C, 26.6; H, 4.9; N, 6.3. $C_{14}H_{32}Br_3N_3SSn$ calc.: C, 26.6; H, 5.1; N, 6.6%. M.p. 122°C. Λ_M (MeCN, 10^{-3} M): 185.6 S cm^2 mol^{-1}.

2.3. Physical measurements

Analytical data were obtained with a Carlo-Erba 1108 apparatus. Melting points were determined on a Büchi apparatus. IR spectra were recorded in Nujol mulls or KBr discs with a Perkin–Elmer 180 apparatus. Raman spectra were recorded with a Dilor Omars 90 apparatus. Molar conductivities of 10^{-3} M in acetonitrile solutions were measured with a WTW LF-3 conductivity meter. NMR spectra of "fresh" solutions in $CDCl_3$ with a tin concentration of 0.1 M were recorded with a Bruker WM-250 spectrometer; chemical shifts are relative to external $SiMe_4$ for ^1H and ^{13}C, and to $SnMe_4$ for ^{119}Sn.

2.4. Determination of the structure

A well formed crystal of approximate dimensions 0.3 × 0.4 × 0.4 mm was mounted on a Phillips PW 1100 diffractometer to determine the cell dimensions and to measure intensity data.

Crystal data: $C_6H_{12}Cl_2N_2SSn$, $M = 333.83$ triclinic, space group $P\bar{1}$, a 9.702(2), b 9.375(2), c 7.119(1) Å, α 68.1(2), β 86.6(2), γ 85.6(2)°, U 598.7 Å^3, $Z = 2$, D_c 1.852 g cm^{-3}, $F(000) = 324$, μ(Mo Kα) 27.2 cm^{-1}.

Data collection: 2891 independent reflections in the range $4.2 < 2\theta < 56°$ were collected by the θ–2θ step scan method using monochromated Mo Kα radiation (λ 0.7107 Å). Reflections with $F \geq 7\sigma(F)$ were considered as observed (2623). The intensities were corrected for Lorentz and polarization effects, but not for absorption.

Determination and refinement of the structure: The positions of the Sn and Cl atoms were obtained from a three-dimensional Patterson-Fourier synthesis. The remaining non hydrogen atoms were located in a subsequent electron density map. The hydrogen atoms were located in a Fourier difference map and isotropically refined. Final refinements were carried out with anisotropic thermal parameters for all non hydrogen atoms. Scattering factors for all atoms were those incorporated in the program SHELX 76 [2]. The final R and R_w ($w = [\sigma^2(F) + 0.015F^2]^{-1}$) values were 0.062 and 0.058 respectively. Positional parameters for the non hydrogen atoms are listed in Table 1. Full lists of

TABLE 1. Fractional coordinates with equivalent isotropic thermal parameters (Å^2)

Atom	x	y	z	$U_{iso/eq}$ [a]
Sn(1)	0.27723(2)	0.52241(3)	0.14042(4)	0.0343(2)
Cl(1)	0.4573(1)	0.2978(2)	0.2261(2)	0.0548(4)
Cl(2)	0.0858(1)	0.7546(1)	0.0642(2)	0.0464(4)
S(1)	0.1140(1)	0.3752(1)	0.4168(2)	0.0453(4)
N(1)	0.2343(4)	0.0807(4)	0.5653(6)	0.041(1)
N(2)	0.1154(4)	0.1275(4)	0.3014(6)	0.043(1)
C(1)	0.2291(5)	0.4946(6)	−0.1301(7)	0.047(2)
C(2)	0.3925(5)	0.6499(7)	0.2616(9)	0.050(2)
C(3)	0.1579(4)	0.1901(5)	0.4263(6)	0.037(1)
C(4)	0.3039(9)	0.0985(8)	0.729(1)	0.066(2)
C(5)	0.2431(7)	−0.0511(5)	0.5223(9)	0.052(2)
C(6)	0.1694(6)	−0.0222(6)	0.3574(9)	0.053(2)

[a] $U_{iso/eq}$ is defined as one third of the trace of the orthogonalized U_{ij} tensor.

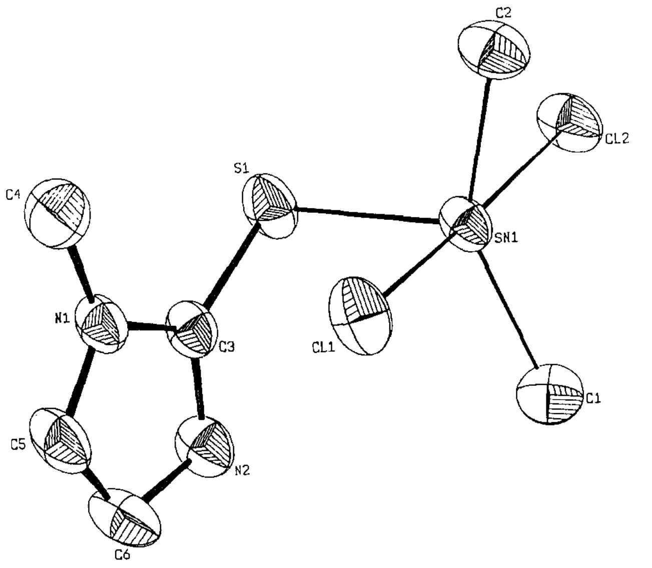

Fig. 1. The molecular structure of $[SnMe_2Cl_2(Hmimt)]$ with the atom numbering scheme.

TABLE 2. Selected bond distances (Å) and angles (°) for $[SnMe_2Cl_2(Hmimt)]$

Sn(1)–Cl(1)	2.542(6)	Sn(1)–Cl(2)	2.665(6)
Sn(1)–S(1)	2.495(6)	Sn(1)–C(1)	2.120(6)
Sn(1)–C(2)	2.124(8)	S(1)–C(3)	1.732(5)
N(1)–C(3)	1.339(6)	N(1)–C(4)	1.45(1)
N(1)–C(5)	1.375(8)	N(2)–C(3)	1.332(7)
N(2)–C(6)	1.378(7)	C(5)–C(6)	1.34(1)
N(2)–Cl(2) [a]	3.151(8)	Sn(1)–Cl(1) [b]	3.587(9)
C(1)–Sn(1)–C(2)	144.2(3)	S(1)–Sn(1)–C(2)	106.5(2)
S(1)–Sn(1)–C(1)	108.5(3)	Cl(2)–Sn(1)–C(2)	85.9(3)
Cl(2)–Sn(1)–C(1)	89.8(3)	Cl(2)–Sn(1)–S(1)	85.6(2)
Cl(1)–Sn(1)–C(2)	93.5(3)	Cl(1)–Sn(1)–C(1)	91.8(3)
Cl(1)–Sn(1)–S(1)	92.7(2)	Cl(1)–Sn(1)–Cl(2)	178.0(1)
Sn(1)–S(1)–C(3)	100.1(3)	C(4)–N(1)–C(5)	124.6(6)
C(3)–N(1)–C(5)	109.1(5)	C(3)–N(1)–C(4)	126.2(7)
C(3)–N(2)–C(6)	109.6(6)	N(1)–C(3)–N(2)	107.1(6)
S(1)–C(3)–N(2)	126.0(5)	S(1)–C(3)–N(1)	126.8(5)
N(1)–C(5)–C(6)	107.3(7)	N(2)–C(6)–C(5)	106.8(7)
N(2)–H–Cl(2) [a]	167.22(5)		

[a] Symmetry code: $-x, -y+1, -z$.
[b] Symmetry code: $-x+1, -y+1, -z$.

atomic coordinates, thermal parameters and structure factors are available from the authors.

3. Results and discussion

3.1. Description of the structure

The structure of $[SnMe_2Cl_2(Hmimt)]$ is shown in Fig. 1 with the atom-numbering scheme. Selected bond distances and angles are listed in Table 2. The compound exists as dimers in which the two monomers are bound together by two chlorine bridges between the Sn atoms. The dimers are interconnected through NH ··· Cl hydrogen bonds.

Although the Sn ··· Cl distance in the bridges is slightly greater than those found in other dimeric complexes [3,4], it is less than the sum of the Van der

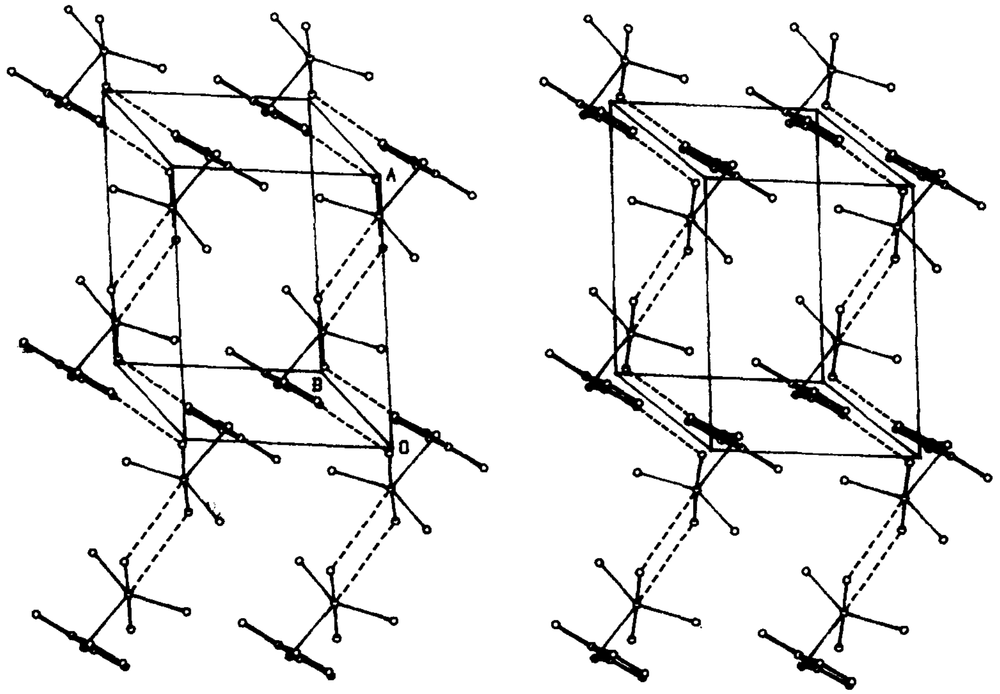

Fig. 2. Stereoscopic view of the molecular packing of $[SnMe_2Cl_2(Hmimt)]$ showing the intermolecular interactions.

Waals radii [5]. The tin atom must therefore be considered as having a distorted octahedral environment with its "own" two chlorine atoms in the axial plane and the two methyl groups, the thione donor S atom and the bridging chlorine of the other monomer in the equatorial plane. This octahedral configuration, may be considered to be the result of the distortion of the bipyramidal trigonal environment in the monomer by the long range interaction with the chlorine atom of the second monomer; and indeed the C–Sn–C angle is widened from the 120° expected for a bpt environment to 144.2°. Like the Sn–Cl bridge distances [3,4], this compares well with the values found in other dinuclear complexes. Note that in these monomers the donor S atom is equatorial and the bridge is formed through a Cl atom *trans* to another Cl, both axial, whereas in the case of O-donors [3,4,6] the bridge is formed by a Cl *trans* to an O atom.

The H atom on N(2) is involved in intermolecular hydrogen bonding with chloride ligands (see Fig. 2). The N(2)–Cl(2) distance (3.151 Å) is shorter than those found in other neutral [7,8] or ionic [9,10] complexes. The H(N(2))–Cl(2) distance, (2.086 Å) and the N(2)–H–Cl(2) angle (167.2°) show the hydrogen bond to be strong. These hydrogen bonds connect the dimer units throughout the lattice, giving rise to a polymeric structure.

The Hmimt binds to the tin through the S atom and retains the thione form of free Hmimt [11], the labile hydrogen being attached to N(2). The S(1)–C(3) bond distance (1.732(5) Å) shows that, as in other complexes of this ligand the π character of the C=S bond is reduced upon coordination. Other distances and angles in the coordinated Hmimt molecule are in keeping with the values discussed previously (ref. 12 and references therein). The Sn–S–C angle is less than angles M–S–C in other complexes of this ligand [13] and Sn–S–C in pyridinethione complexes [8], possibly because of the hydrogen bonding.

Comparison of the above structure with that of [$SnMe_2Br_2$(Hmimt)] [1] shows several analogies and differences. In both complexes the axial positions are occupied by halogen atoms, and also in both complexes the two Sn–X bond distances are different; in the bromide the reason is probably the participation of the bromine in a hydrogen bond, and in the chloride the participation of Cl(1) in a hydrogen bridge and Cl(2) in a chlorine bridge. The Sn–C bond distances are practically equal in this case and the Sn–S bond distances greater than that found in [$SnMe_2Br_2$(Hmimt)] despite the small acceptor capacity of the bromide, this difference probably being due to the strong hydrogen bond in this case. The greatest difference between the two structures is the C–Sn–C angle, an average 132.9° in the bromide as against 144.2° in the chloride, probably due to the formation of the bridge and the resulting transition from bpt coordination in the bromide to a distorted octahedron in the chloride.

TABLE 3. Mössbauer parameters at 80.0 K (mm/s)

		δ [a]	ΔE_Q	Γ	ΔE_Q^{calc}
[$SnMe_2Cl_2$(Hmimt)]	(1)	1.55	3.63	0.81	3.4 [b] 3.6 [c]
[$SnMe_2Br_2$(Hmimt)]	(2)	1.45	3.13	0.82	3.2 [d] 3.1 [e]
[$SnMe_2Cl_2(Hmimt)_2$]	(3)	1.52	3.90	0.84	3.8
[$SnMe_2Br_2(Hmimt)_2$]	(4)	1.56	3.88	0.81	–
(Et_4N)[$SnMe_2Cl_3$(Hmimt)]	(5)	1.50	3.96	0.94	–
(Et_4N)[$SnMe_2Br_3$(Hmimt)]	(6)	1.58	3.79	0.89	–

[a] Relative to room temperature $CaSnO_3$. [b] Calculated for a hexacoordinate tin environment. [c] Calculated for a pentacoordinate tin environment. [d] Calculated for the A tin site [1]. [e] Calculated for the B tin site [1].

3.2. Mössbauer spectra

The Mössbauer spectra of the compounds are typical of diorganotin(IV) compounds (see Table 3). The range of the isomer shifts is rather small, 0.13 mm/s, but a trend is nevertheless evident if [$SnMe_2Cl_2$ (Hmimt)] is ignored: replacement of chlorine atoms by bromine results in very small increases in δ (0.04 and 0.08 mm/s for compounds **3**, **4** and **5**, **6**, respectively), while coordination of a sixth ligand to compound **2** to produce the hexacoordinate derivatives **4** and **6** increases δ by 0.11 and 0.13 mm/s, respectively. The anomalous behaviour of [$SnMe_2Cl_2$(Hmimt)] may be a consequence of the large distortion of the crystal structure and the short Sn ··· Cl distance. The replacement of the thioimidazole sulphur atoms by the halogens (compounds **3**, **5** and **4**, **6**) does not significantly influence the isomer shift.

The small values of the line width for all the compounds, 0.81–0.94 mm/s, is indicative of a single tin site. Though the X-ray analysis for [$SnMe_2Br_2$(Hmimt)] shows two slightly different molecules [1], point charge calculations using the experimental bond angles for the two molecules give theoretical quadrupole splitting values of 3.2 and 3.1 mm/s, which are too close to each other and to the experimental value to differentiate between the two sites. Similarly the calculated ΔE_Q values do not unambiguously show the coordination number of [$SnMe_2Cl_2$(Hmimt)], being 3.4 mm/s for the hexacoordinate form and 3.6 mm/s for the pentacoordinate form, while the experimental value is 3.63 mm/s. For the other four compounds, the crystal structures of which are not known, point charge calculations suggest distorted octahedral geometries with C–Sn–C angles ranging from about 150 to 160°.

TABLE 4. Significant bands in the range 600–180 cm^{-1} for the compounds prepared

		ν_{asym}(Sn–C)	ν_{sym}(Sn–C)	ν(Sn–S)	ν_{asym}(Sn–Cl)
$[SnMe_2Cl_2(Hmimt)]$	IR	570m	515ms [a]	340m	245s,b
	R	564w	515vs	337m	–
$[SnMe_2Br_2(Hmimt)]$	IR	560m	515m [a]	350m	–
	R	567m	517vs	350m	–
$[SnMe_2Cl_2(Hmimt)_2]$	IR	560m	–	–	230s,b
	R	–	489s	–	–
$[SnMe_2Br_2(Hmimt)_2]$	IR	560m	–	–	–
	R	–	492s	–	–
$(Et_4N)[SnMe_2Cl_3(Hmimt)]$	IR	565m	–	–	260s
	R	–	499vs	–	240s
$(Et_4N)[SnMe_2Br_3(Hmimt)]$	IR	565m	–	–	–
	R	–	492vs	–	–

[a] +Hmimt vibration.

3.3. Vibrational spectra

The shifts that ligand bands in the range 3200–600 cm^{-1} undergo upon coordination are similar to those found in other complexes [12]. The significant bands in the range 600–180 cm^{-1} are listed in Table 4.

The IR and Raman spectra of $[SnMe_2X_2(Hmimt)]$ show the two Sn–C stretching vibrations, the Raman ν_{sym}(Sn–C) band being very strong. The presence of both vibrations in both the IR and Raman spectra is consistent [14,15] with the non-linearity shown by the X-ray and Mössbauer studies. A ligand IR band at 530 cm^{-1} hinders analysis of the relationship between the intensities of the asymmetric and symmetric bands, which was useful in other systems [16]. According to the X-ray study, the Cl–Sn–Cl fragment of $[SnMe_2Cl_2(Hmimt)]$ is almost linear. However, the Raman spectrum of neither $[SnMe_2X_2(Hmimt)]$ shows a strong ν_{sym}(Sn–X) band. In the IR spectrum of the chloride, ν_{asym}(Sn–Cl) is identified as the band at 245 cm^{-1}, in keeping with the tin coordination number of 6; neither the IR nor the Raman spectra of the chloride show bands at wavenumbers > 300 cm^{-1} (the region typical of pentacoordinate monomers [16–18] or incipient dimers [6]) that are not present in the spectra of the bromide. Thus ν_{asym}(Sn–X) appears in the chloride at a position similar to that observed in most hexacoordinate bridged complexes [4,16], though a position suggesting pentacoordination has been reported [3b] for dimeric sulphine complexes in spite of their having shorter bridges than $[SnMe_2Cl_2(Hmimt)]$.

When a second Hmimt molecule is coordinated, to form $[SnMe_2X_2(Hmimt)_2]$, the Mössbauer spectra (*vide supra*) suggest a larger C–Sn–C angle than in $[SnMe_2X_2(Hmimt)]$. This widening of the angle does not greatly affect the position of ν_{asym}(Sn–C). Though the position of ν_{asym}(Sn–Cl) in the IR spectrum of the chloride is typical of coordination number six [16,19], we found no Raman band assignable to ν_{sym}(Sn–X). This is not necessarily indicative of an angular X–Sn–X fragment, because (for example) no such band was detected for the all-*trans* anion $[SnMe_2Cl_4]^{2-}$ either (ref. 20). Probably $[SnMe_2X_2(Hmimt)_2]$, like related systems [8,17], have an all-*trans* structure.

For $(Et_4N)[SnMe_2X_3(Hmimt)]$ the ν(Sn–C) data are

TABLE 5. NMR parameters for the $SnMe_2$ fragment (δ in ppm and J in Hz)

Compound	$\delta(^1H)$	$^2J(^{119}Sn-^1H)$	$\delta(^{13}C)$	$^1J(^{119}Sn-^{13}C)$	$\delta(^{119}Sn)$ [a]
$SnMe_2Cl_2$	1.21	68.5	6.3	469.2 [b]	141.9
$[SnMe_2Cl_2(Hmimt)]$	1.29	74.7	9.8	535.5 [b]	65.5
$[SnMe_2Cl_2(Hmimt)_2]$ [c]	1.33	79.3	10.1	–	22.0
$(Et_4N)[SnMe_2Cl_3(Hmimt)]$	1.34	88.2	18.2	708.7	−101.4
$SnMe_2Br_2$	1.38	66.1	7.2	442.5	67.8
$[SnMe_2Br_2(Hmimt)]$	1.45	71.0	10.0	491.2	19.2
$[SnMe_2Br_2(Hmimt)_2]$ [c]	1.47	72.8	10.8	513.4 [b]	−13.3 [d]
$(Et_4N)[SnMe_2Br_3(Hmimt)]$	1.64	82.6	20.0	–	−109.2

[a] δ values were obtained from solutions of recently prepared donor–acceptor mixtures $\{[SnR_2X_2] = 0.1$ M$\}$. [b] ^{119}Sn and ^{117}Sn satellites not resolvable, $^1J(^{119}Sn-^{13}C) = J_{obs.} \times 1.023$ [23]. [c] ^1H and ^{13}C measurents were made on saturate solutions (concentration < 0.1 M). [d] When the spectrum was recorded, crystals were visible within the tube.

also in keeping with the Mössbauer data for the C–Sn–C fragment. If these compounds are considered as derived from the pentacoordinate anions $[SnMe_2X_3]^-$, the position of ν(Sn–Cl) is in keeping with the increase in the coordination number of tin from five to six [18,20].

3.4. NMR spectra

Table 5 lists ^{1}H, ^{13}C and ^{119}Sn chemical shifts and coupling constants for the $Sn^{IV}Me_2$ fragment of the acceptors and complexes. Chemical shifts for the ligand moieties have been omitted because they differ very little from those of the free donor.

The ^{1}H and ^{13}C NMR signals of the complexes are shifted downfield with respect to the free acceptors, in keeping with the donor–acceptor interaction reflected by the shielding of $\delta(^{119}Sn)$. The coupling constants $^2J(^{119}Sn-^1H)$ and $^1J(^{119}Sn-^{13}C)$ are smaller than expected for pentacoordination in $[SnMe_2X_2(Hmimt)]$ and hexacoordination in $[SnMe_2X_2(Hmimt)_2]$, showing dissociation in $CDCl_3$. $[SnMe_2Cl_2(Hmimt)_2]$ has a value of ^2J close to 80 Hz, in keeping with a coordination number of five [21], whereas for $[SnMe_2Br_2(Hmimt)_2]$ the value of ^2J is indicative of greater dissociation. This, and the values of $\delta(^{119}Sn)$ [22], are in keeping with $SnMe_2Cl_2$ being a better acceptor than $SnMe_2Br_2$.

In the case of the tetraethylammonium salts, the deshielding of the ^{1}H and ^{13}C signals and the upfield shift of $\delta(^{119}Sn)$ with respect to $[SnMe_2X_2(Hmimt)]$ show that the additional halide is coordinated in $CDCl_3$. For $(Et_4N)[SnMe_2Cl_3(Hmimt)]$ $\delta(^{119}Sn)$, ^1J and ^2J nevertheless indicate coordination number five (the value of ^2J is very similar to the value for $(Et_4N)[SnMe_2Cl_3]$ [18]). This shows that the Hmimt is totally dissociated in $CDCl_3$.

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