Introduction

The separation of metals by gas chromatography of their volatile metal chelates of quadridentate Schiff bases such as *N,N'*-ethylenebis(acetylidenemine) (H_2EDAA) is a technique which has met with some success [2,3]. Whereas diorganotin(IV) derivatives of *N,N'*-ethylenebis(salicylideneimine) (H_2salen) have been isolated and studied in some detail [4,5], attempts to prepare covalent diorganotin(IV) derivatives of H_2EDAA have resulted in a singular lack of success [6], although complexes of H_2EDAA with organotin(IV) chlorides are readily obtained [7—10]. In this paper we report the synthesis and properties of $Me_2Sn^{IV}EDAA$, $Me_3Sn^{IV}HEDAA$, and $Sn^{II}EDAA$.

Experimental

All manipulations were performed under an atmosphere of dry nitrogen or argon. Solvents were dried and freed from dissolved oxygen by standard methods, and redistilled immediately before use. Infrared spectra were obtained using a Perkin—Elmer 457 spectrometer, ^1H NMR spectra using a Varian HA-100 spectrometer, and tin-119*m* Mössbauer spectra on a Harwell spectrometer at 77 K against a $Ba^{119m}SnO_3$ source. Data reduction to Lorentzian line shapes was effected by usual least-squares methods. Mass spectra were obtained at 70 eV using an AEI MS-902 instrument.

Synthesis of the compounds

(a) $Me_2SnEDAA$. H_2EDAA (1.78 g, 0.008 mol) in benzene (20 ml) was added to $Me_2Sn(NEt_2)_2$ (2.35 g, 0.008 mol) also in benzene (20 ml) and the mixture refluxed. After 3 h, the mixture was allowed to cool, the solvent removed in vacuo, and the product recrystallised from heptane. M.p. (sealed tube) ca. 170°C (decomp.). Found: C, 45.60; H, 6.37; N, 7.21; mol. wt., 356 (osmometry in benzene). $C_{14}H_{24}N_2O_2Sn$ calcd.: C, 45.32; H, 6.52; N, 7.55%, mol. wt. 371.

(b) $Me_3SnHEDAA$. H_2EDAA (2.15 g, 0.001 mol) and Me_3SnNEt_2 (2.40 g, 0.001 mol) were dissolved in benzene (40 ml) and refluxed for 12 h. Removal

TABLE 1

SPECTROSCOPIC DATA FOR $Me_3SnHEDAA$, $Me_2SnEDAA$ AND SnEDAA

Compound	Tin-119*m* Mössbauer data (mm s^{-1})						Infrared data (cm^{-1})
	I.S.	Q.S.	Γ_1	Γ_2	I_1	I_2	$\nu(C=O)/\nu(C=N)$
$Me_3SnHEDAA$	1.40	3.49	0.84	0.84	0.87	1.00	1590vs, v(br); 1515s
$Me_2SnEDAA$	1.12	3.07	0.90	0.94	0.91	1.00	1585s(sh); 1560vs; 1515vs
SnEDAA	2.76	1.65	1.36	1.42	0.93	1.00	1605s(sh); 1580s; 1510s
Oxidised SnEDAA	2.83	1.80	0.98	1.15	0.94	1.00	
	0.12	0	1.59		1.23		
$Me_2SnSalen$ [d]	1.13	3.46					

[a] Satisfactory integration values were observed. [b] $\nu(N—H)$ 3150vw, (br). [c] $\tau(N—H)$ 3.48 cm^{-1}. [d] Ref. 5.

of the solvent in vacuo yielded $Me_3SnHEDAA$ as a white solid. M.p. (sealed tube) 94—96°C. Found: C, 47.41; H, 7.42; N, 7.92. $C_{15}H_{28}N_2O_2Sn$ calcd.: C, 46.41; H, 7.19; N, 7.22%. The compound decomposed on attempted recrystallisation from benzene.

(c) Sn^{II}EDAA. H_2EDAA (2.15 g, 0.001 mol) and $(C_5H_5)_2Sn$ (2.49 g, 0.001 mol) were stirred in benzene (40 ml) for 4 h, after which time the reaction mixture was filtered, the volume reduced by half and left to crystallise. Golden crystals of Sn^{II}EDAA appeared on standing. M.p. (sealed tube) ca. 150°C (decomp.). Found: C, 42.89; H, 5.66; N, 8.5. $C_{12}H_{18}N_2O_2Sn$ calcd.: C, 42.27; H, 5.32; N, 8.22%.

Infrared, ^{1}H NMR, and tin-119*m* Mössbauer spectral data are listed in Table 1.

Structure determination of Sn^{II}EDAA

Pale yellow needles of Sn^{II}EDAA suitable for intensity measurements were obtained by recrystallisation from hot benzene. The crystals are air sensitive, but stable to X-rays, and a suitable specimen of approximate dimensions 0.3 × 0.7 × 0.4 mm³ was mounted in a Lindemann capillary and used for both the initial photography and the subsequent intensity data collection.

Crystal data. $C_{12}H_{18}N_2O_2Sn$, mol. wt. 340.98, a 8.2815(5), b 15.3707(5), c 24.7351(5) Å, β 120.753(9)°, $V = 1538.33(2)$ Å^3, $Z = 8$, $F(000) = 340$, μ(Mo-K_α) 33.30 cm^{-1}. Space group $P2_1/c$ from systematic absences $h0l$ ($l = 2n + 1$) and $0k0$ ($k = 2n + 1$).

The space group and initial cell dimensions were determined from oscillation and zero and first layer Weissenberg photographs obtained on an equi-inclination Weissenberg camera. Relative intensities were collected with Mo-K_α radiation (λ 0.71069 Å) using a Hilger and Watts Y290 four-circle diffractometer. Accurate cell dimensions were obtained by least squares refinement using ca. 20 reflections. Each reflection was counted for 30 sec and the two associated background counts for 10 sec each. The reflections were brought to the same relative intensities by reference reflections taken for every 100 reflections recorded; the orientation of the diffractometer being checked for every 200 reflections. The background counts were low and were included in the usual manner. All reflec-

	^{1}H NMR [a]				
ν(Sn—C)	τ(Sn—Me) (ppm)	$^2J(^{117,119}Sn—H)$ (Hz)	τ(Me) (ppm)	τ(CH) (ppm)	$\tau(CH_2—CH_2)$ (ppm)
555m [b]	9.33 [c]	58, 61	7.88; 8.43	5.00	7.28, 7.31, 7.39
561m; 533vw	9.21	95, 101	8.09; 8.14	5.18	6.62
			7.87; 8.38	5.03	7.01

TABLE 2

FRACTIONAL ATOMIC COORDINATES WITH ESTIMATED STANDARD DEVIATIONS IN PARENTHESES

Atom	Population parameter	x/a	y/b	z/c
Sn(1)	1.0	0.1515(3)	0.1375(1)	0.2503(1)
Sn(2)	0.5	0.148(1)	0.3615(6)	0.0908(3)
Sn(3)	0.5	0.130(1)	0.3634(6)	0.4107(3)
O(1)	1.0	0.902(3)	0.184(1)	0.251(1)
O(2)	0.5	0.288(9)	0.205(4)	−0.001(3)
O(3)	0.5	0.037(9)	0.209(3)	0.499(2)
O(4)	0.5	0.043(5)	0.207(2)	0.166(1)
O(4')	0.5	0.294(4)	0.206(2)	0.335(1)
O(6)	0.5	0.895(9)	0.305(4)	0.067(2)
O(7)	0.5	0.933(7)	0.319(3)	0.438(2)
N(1)	1.0	0.975(4)	0.564(2)	0.250(1)
N(2)	0.5	1.00(1)	0.000(5)	0.429(3)
N(3)	0.5	0.354(9)	0.472(5)	0.463(3)
N(4)	0.5	0.22(1)	0.455(4)	0.019(3)
N(5)	0.5	0.04(1)	0.511(6)	0.434(3)
N(6)	0.5	0.220(6)	0.043(3)	0.196(1)
N(6')	0.5	0.405(5)	0.048(2)	0.308(2)
C(1)	1.0	0.633(5)	0.205(2)	0.252(2)
C(2)	1.0	0.774(3)	0.147(2)	0.249(1)
C(3)	1.0	0.753(4)	0.059(2)	0.249(1)
C(4)	1.0	0.135(6)	0.493(2)	0.249(2)
C(5)	1.0	0.198(5)	0.396(2)	0.249(2)
C(14)	0.5	0.063(8)	0.205(3)	0.121(2)
C(15)	0.5	0.156(7)	0.136(3)	0.103(2)
C(16)	0.5	0.244(7)	0.059(3)	0.145(2)
C(17)	0.5	0.76(1)	0.495(4)	0.403(3)
C(20')	0.5	0.534(7)	0.277(3)	0.427(2)
C(14')	0.5	0.470(7)	0.202(3)	0.382(2)
C(15')	0.5	0.606(7)	0.143(4)	0.397(2)
C(16')	0.5	0.560(7)	0.056(3)	0.357(2)
C(17')	0.5	0.725(5)	−0.001(3)	0.391(2)
C(18)	0.5	0.920(8)	0.429(4)	0.276(3)
C(19)	0.5	0.74(1)	0.459(4)	0.278(3)
C(18')	0.5	0.84(1)	0.421(4)	0.229(3)
C(19')	0.5	0.66(1)	0.461(4)	0.230(3)
C(20)	0.5	−0.006(9)	0.272(5)	0.070(4)

tions with a corrected count of less than three times the estimated standard deviation of the total counts were considered as non-observed and were discarded, reducing the total number of reflections from 5050 to 3364. This was further reduced to 2154 by discarding the 1210 reflections of lowest intensity to enable adequate computer storage for refinement. The intensities were corrected for Lorentz and polarisation effects, but no correction was applied for absorption because of the low μ value.

Structure determination and refinement. The positional parameters of the tin atoms could not be satisfactorily obtained from a three-dimensional Patterson synthesis, only one tin atom being located with certainty. However, direct methods showed that the two tin atoms were in fact a unit weight atom at the posi-

TABLE 3

ANISOTROPIC THERMAL PARAMETERS WITH ESTIMATED STANDARD DEVIATIONS IN PARENTHESES [a,b]

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Sn(1)	2.6(1)	3.4(1)	2.8(1)	−0.4(1)	1.38(8)	0.0(1)
Sn(2)	6.9(5)	9.7(5)	7.6(5)	−0.6(5)	2.8(4)	−1.5(5)
Sn(3)	9.0(5)	9.8(5)	8.5(5)	1.1(5)	4.4(4)	1.2(5)
O(1)	2(1)	2(1)	7(2)	0(1)	3(1)	0(1)
O(2)	5(5)	2(4)	3(5)	0(4)	0(4)	−2(4)
O(3)	6(4)	2(3)	3(4)	2(3)	4(3)	2(3)
O(4)	9(2)	11(3)	9(2)	−1(2)	5(2)	−2(2)
O(4′)	7(2)	9(3)	10(3)	0(2)	4(2)	0(2)
O(6)	7(5)	9(4)	3(3)	−4(4)	4(4)	−2(3)
O(7)	6(3)	7(3)	7(3)	0(2)	1(2)	2(2)
N(1)	3(1)	1(2)	5(2)	0(1)	3(1)	0(2)
N(2)	8(5)	10(6)	5(4)	1(4)	5(4)	5(4)
N(3)	4(4)	8(5)	10(6)	−5(3)	4(4)	6(4)
N(4)	4(5)	3(5)	6(4)	0(4)	3(4)	0(3)
N(5)	10(6)	11(6)	3(4)	2(4)	2(4)	6(5)
N(6)	11(3)	11(3)	5(2)	−1(2)	3(2)	−2(2)
N(6′)	8(2)	9(3)	7(3)	−1(2)	3(2)	−1(2)
C(1)	2(2)	3(2)	6(2)	1(2)	3(2)	1(2)
C(2)	1(1)	4(2)	0(1)	0(1)	0(1)	0(2)
C(3)	2(2)	1(1)	3(2)	−1(1)	1(2)	−1(1)
C(4)	2(3)	7(2)	0(3)	0(2)	0(2)	0(3)
C(5)	6(2)	1(1)	2(3)	1(1)	2(2)	2(2)
C(14)	12(4)	9(4)	11(3)	0(3)	9(3)	0(3)
C(15)	12(3)	8(3)	10(4)	−3(3)	10(3)	5(4)
C(16)	11(4)	8(4)	7(3)	−2(3)	5(3)	0(3)
C(17)	10(6)	11(4)	11(6)	4(4)	1(5)	9(4)
C(20′)	11(4)	8(4)	9(4)	0(3)	1(3)	−4(3)
C(14′)	7(4)	9(4)	9(3)	1(3)	3(3)	1(3)
C(15′)	11(3)	7(4)	10(3)	0(4)	6(3)	2(3)
C(16′)	5(3)	10(4)	11(4)	0(3)	1(3)	2(3)
C(17′)	3(2)	9(4)	12(4)	3(2)	1(2)	4(3)
C(18)	3(3)	2(4)	4(3)	−1(3)	3(2)	0(3)
C(19)	6(6)	2(4)	1(4)	−2(4)	1(4)	−1(3)
C(18′)	1(5)	2(4)	7(4)	0(4)	2(4)	2(3)
C(19′)	4(4)	4(4)	4(4)	−3(3)	0(3)	0(3)
C(20)	0(3)	4(5)	11(8)	0(3)	1(4)	2(5)

[a] Thermal parameters as listed have been multiplied by 10^2. [b] U_{ij} are of the form: $\exp - 2\pi^2(h^2U_{11}a^{*2} + k^2U_{22}b^{*2} + l^2U_{33}c^{*2} + hkU_{12}a^*b^* + hlU_{13}a^*c^* + klU_{23}b^*c^*)$

tion indicated from the Patterson synthesis and two half-weight atoms located at two other general positions. The positional parameters of these tin atoms (with the two half-weight tin atoms given population parameters of 0.5) were used to phase the initial structure factor calculation. After successive Fourier syntheses followed by block-diagonal least-squares anisotropic refinement, the positions of 33 light atoms were located. However, at this stage it was noted that the EDAA ligand attached to the unit weight tin atom was disordered such that one half of the ligand occupies equally two equivalent positions. Hence, light atoms in the disordered positions were assigned population parameters of 0.5, as were the located atoms attached to the two half-weight tin atoms. Only the two oxygen and two nitrogen atoms of the two half-weight molecules could be located with any certainty; other possible positions located from the Fourier synthesis deteriorated

TABLE 4a

BOND LENGTHS (A) WITH ESTIMATED STANDARD DEVIATIONS IN PARENTHESES

Bond	Length (A)	Bond	Length (A)
Sn(1)—O(1)	2.20(3)	N(6')—C(19')	1.58(8)
Sn(1)—O(4)	2.08(4)	C(1)—C(2)	1.50(6)
Sn(1)—O(4')	2.10(3)	C(2)—C(3)	1.36(5)
Sn(1)—N(1)	2.28(3)	C(3)—C(4)	1.35(6)
Sn(1)—N(6)	2.23(5)	C(4)—C(5)	1.58(6)
Sn(1)—N(6')	2.29(4)	C(14)—C(20)	1.49(11)
O(1)—C(2)	1.18(5)	C(14)—C(15)	1.50(10)
O(4)—C(14)	1.22(9)	C(15)—C(16)	1.49(7)
O(4')—C(14')	1.32(5)	C(16)—C(17)	1.54(10)
N(1)—C(4)	1.35(8)	C(20')—C(14')	1.50(8)
N(1)—C(18)	1.50(8)	C(14')—C(15')	1.34(8)
N(1)—C(18')	1.57(8)	C(15')—C(16')	1.58(8)
N(6)—C(16)	1.37(9)	C(16')—C(17')	1.48(7)
N(6)—C(19)	1.41(8)	C(18)—C(19)	1.58(13)
N(6')—C(16')	1.24(5)	C(18')—C(19')	1.65(13)

into non-chemical positions on refinement, and were not included. Two cycles of block-diagonal least-squares refinement on the 36 weighted atoms located resulted in convergence at an *R* value of 0.141. The scattering factors used were those for neutral atoms [11]. Final fractional atomic coordinates and anisotropic

TABLE 4b

BOND ANGLES WITH ESTIMATED STANDARD DEVIATION IN PARENTHESES

Bond Angle	Angle (°)	Bond Angle	Angle (°)
O(1)—Sn(1)—O(4)	85.1(15)	C(5)—C(4)—N(1)	116(4)
O(1)—Sn(1)—O(4')	82.7(13)	Sn(1)—N(1)—C(18)	117(4)
O(1)—Sn(1)—N(1)	81.9(12)	C(4)—N(1)—C(18)	115(4)
O(1)—Sn(1)—N(6)	138.5(12)	C(4)—N(1)—C(18')	112(5)
O(1)—Sn(1)—N(6')	138.6(17)	N(1)—C(18)—C(19)	106(5)
O(4)—Sn(1)—O(4')	118.2(15)	C(18)—C(19)—N(6)	107(6)
O(4)—Sn(1)—N(1)	119.0(13)	C(19)—N(6)—C(16)	118(6)
O(4)—Sn(1)—N(6)	81.4(16)	C(19)—N(6)—Sn(1)	113(5)
O(4)—Sn(1)—N(6')	136.2(20)	N(6)—C(16)—C(17)	120(5)
O(4')—Sn(1)—N(1)	118.7(15)	N(6)—C(16)—C(15)	122(6)
O(4')—Sn(1)—N(6)	137.7(17)	C(17)—C(16)—C(15)	98(5)
O(4')—Sn(1)—N(6')	78.8(15)	C(16)—C(15)—C(14)	120(6)
N(1)—Sn(1)—N(6)	71.0(18)	C(15)—C(14)—C(20)	107(7)
N(1)—Sn(1)—N(6')	75.1(14)	C(15)—C(14)—O(4)	127(5)
N(6)—Sn(1)—N(6')	63.6(15)	C(20)—C(14)—O(4)	126(7)
Sn(1)—O(1)—C(2)	132(2)	Sn(1)—N(1)—C(18')	120(4)
Sn(1)—O(4)—C(14')	132(4)	N(1)—C(18')—C(19')	99(5)
Sn(1)—N(1)—C(4)	124(3)	C(18')—C(19')—N(6')	111(7)
Sn(1)—N(6)—C(16)	129(4)	C(19')—N(6')—Sn(1)	101(3)
Sn(1)—N(6')—C(16')	135(4)	C(19')—N(6')—C(16')	124(5)
O(1)—C(2)—C(1)	114(4)	N(6')—C(16')—C(17')	133(5)
O(1)—C(2)—C(3)	126(4)	C(17')—C(16')—C(15')	107(4)
C(1)—C(2)—C(3)	119(4)	N(6')—C(16')—C(15')	119(5)
C(2)—C(3)—C(4)	131(4)	C(16')—C(15')—C(14')	120(4)
C(3)—C(4)—C(5)	119(5)	C(15')—C(14')—O(4')	131(5)
C(3)—C(4)—N(1)	125(4)	C(15')—C(14')—C(20')	113(4)
		O(4')—C(14')—C(20')	116(5)

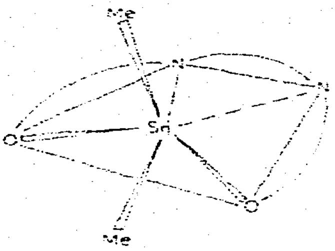

Fig. 1. Proposed structure for $Me_2SnEDAA$.

thermal parameters for the located atoms are listed in Tables 2 and 3, respectively. Intramolecular bond distances and angles are collected in Table 4.

Discussion

The synthesis of the compounds was accomplished by the reaction of H_2EDAA with Me_3SnNEt_2, $Me_2Sn(NEt_2)_2$ and $Sn(C_5H_5)_2$ according to equations 1—3. All the complexes were readily hydrolysed releasing H_2EDAA. SnEDAA underwent

$$Me_3SnNEt_2 + H_2EDAA \rightarrow Me_3SnHEDAA + Et_2NH \qquad (1)$$

$$Me_2Sn(NEt_2)_2 + H_2EDAA \rightarrow Me_2SnEDAA + 2\ Et_2NH \qquad (2)$$

$$Sn(C_5H_5)_2 + H_2EDAA \rightarrow SnEDAA + 2\ C_5H_6 \qquad (3)$$

oxidative-hydrolysis in air as evidenced by the appearance of a resonance at ca. 0 mm s^{-1} in the Mössbauer spectrum. The highest peaks observed in the mass

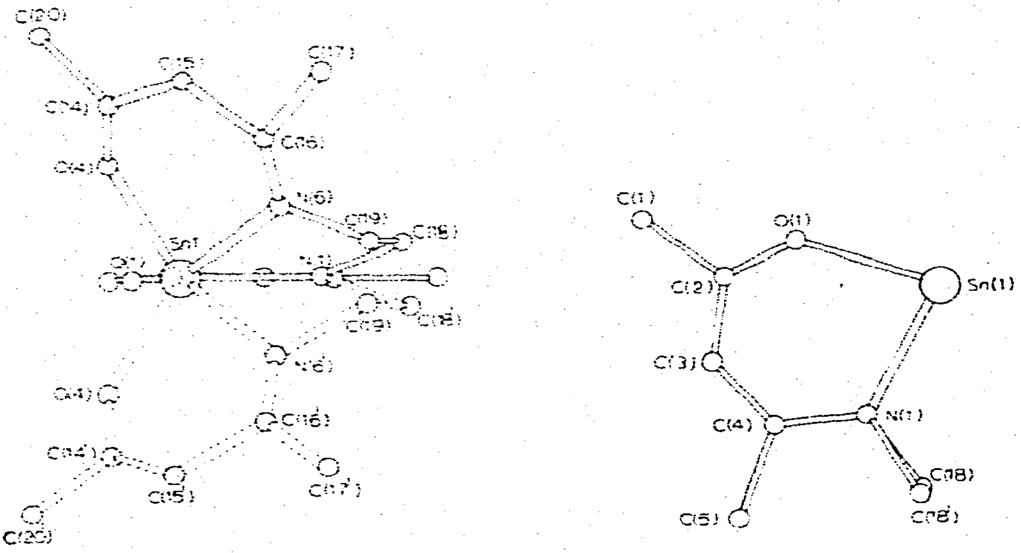

Fig. 2. (a) Diagram of SnEDAA showing atom labelling of the two equivalent disordered portions of the EDAA ligand. (b) Diagram of a portion of SnEDAA showing the atom labelling of the unit weight elements of the EDAA ligand.

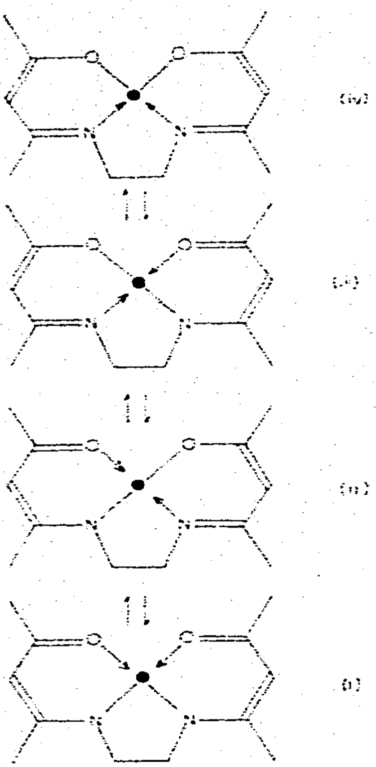

Fig. 3. Possible canonical forms of SnEDAA.

spectra of all three compounds corresponded to the respective polyisotopic parent ions, indicating monomeric character for all three, as was also apparent in benzene solution for $Me_2SnEDAA$ by osmometry.

The structure of $Me_2SnEDAA$ most probably closely resembles that of Me_2Sn-Salen [4], in which the central $[SnO_2N_2]$ unit is essentially planar (Fig. 1). Some measure of corroboration of this hypothesis comes from the almost identical values of the Mössbauer isomer shifts of the two compounds. However, the value of the quadrupole splitting of $Me_2SnEDAA$ is significantly smaller (3.07 mm s^{-1}) than that of $Me_2SnSalen$ (3.46 mm s^{-1}), and corresponds to a much smaller C—Sn—C bond angle of ca. 130° [12].

The stereochemistry about the tin in SnEDAA is that of a distorted square pyramid, the tin atom lying significantly above the basal plane (ca. 0.94 Å). The tin lone pair of electrons presumably occupies the vacant apical position. The molecule is disordered such that half of the ligand occupies equally two equivalent orientations (Fig. 2). The tin—oxygen bond distances (2.08(4), 2.10(3) and 2.20(3) Å) are very short considering the range of previously reported Sn—O covalent bonds (2.11—2.59 Å), indicating highly covalent character for the Sn—O bonds. The tin—nitrogen bond distances (2.28(3), 2.23(5) and 2.29(4) Å) are also shorter than the coordinate bond distances reported for the tin(II) ethylenediaminotetraacetate complexes [13,14] (2.38—2.45 Å), but are similar to the tin—

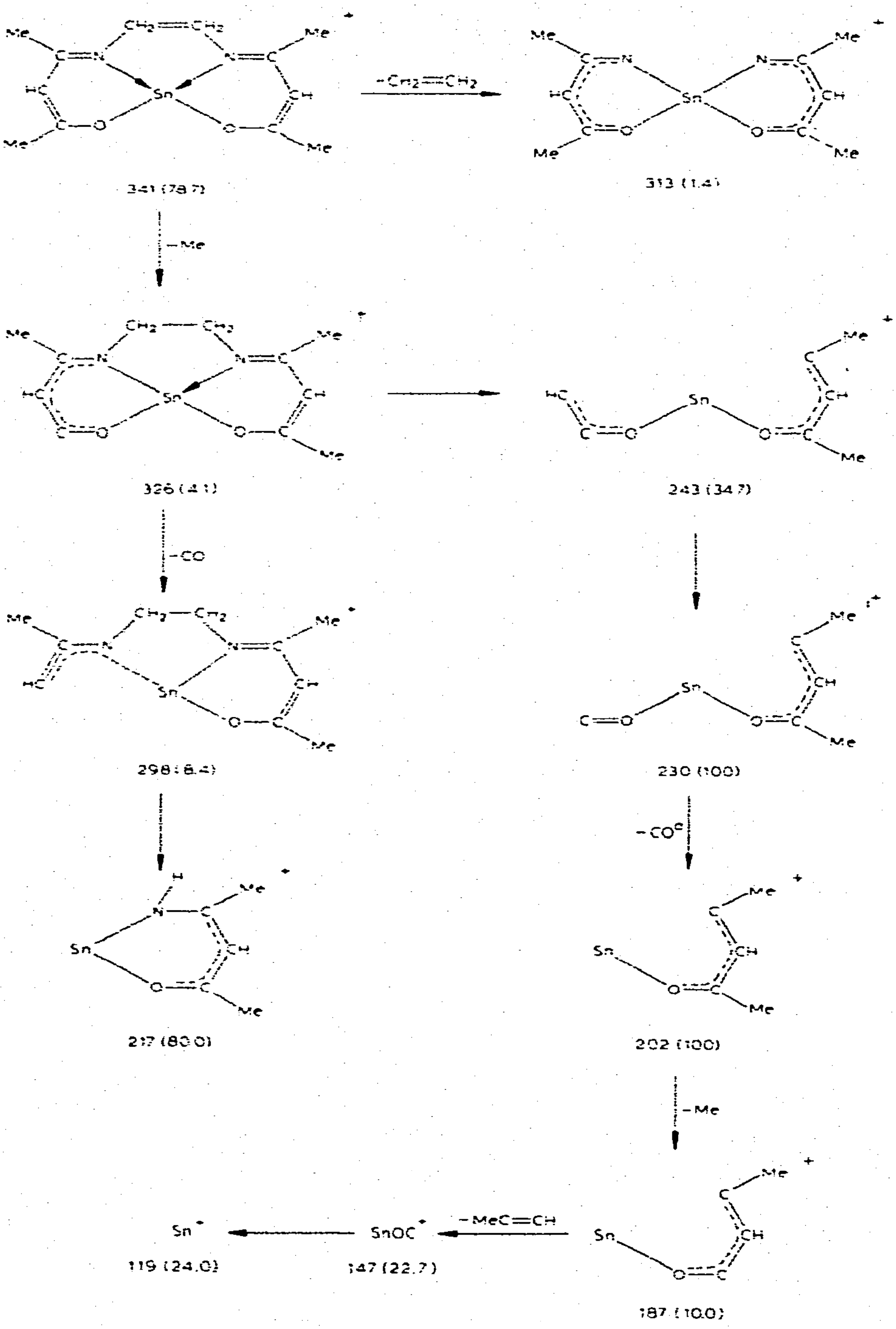

Fig. 4. Fragmentation pattern for SnEDAA. ^{a} $SnO_2C_4HMe_2^+ \rightarrow SnOC_3HMe_2^+ + CO$ m^*_{120}(calc.) 178.3; m^*_{120}(obs.) 178.1.

TABLE 5

MASS SPECTRAL DATA FOR $Me_2SnEDAA$ AND $Me_3SnHEDAA$

$Me_2SnEDAA$		$Me_3SnHEDAA$		Assignment
m/e	Rel. int.	*m/e*	Rel. int.	
		372	12.5	$Me_2SnHEDAA^+$
371	0.75			$Me_2SnEDAA^+$
356	100			$MeSnEDAA^+$
341	2.83	341	11.25	$SnEDAA^+$
328	3.42			$[SnEDAA - (MeCO)]^+$
312	1.00			^{a} $[313 - H]^+$
298	1.83	298	100	^{a}
		268	26.25	$[298 - 2Me]^+$
243	15.0			^{a}
230	30.8	230	16.25	^{a}
217	11.7	217	14.4	^{a}
202	30.8			^{a}
187	8.3			^{a}
		164	76.25	Me_3Sn^+
147	13.3			^{a}
134	36.7			$MeSn^+$
119	12.5			Sn^+

[a] See Fig. 4 for assignment.

nitrogen bond distances reported for tin(II) phthalocyanine (2.24—2.27 Å) [15]. In this case the tin atom lies ca. 1.1 Å above the plane of the four nitrogen atoms (cf. 0.94 Å in the present case). This difference is most probably a result of the acyclic nature of the EDAA quadridentate ligand compared with the cyclic phthalocyanato ligand, in which the Sn^{2+} cation, but not the Sn^{4+} cation, is too large to fit. The Sn—O and Sn—N bond distances suggest that the bands to both oxygen and nitrogen atoms are to some degree covalent in character, and that the overall bonding can be best described as an equilibrium of the four canonical forms Fig. 3, i—iv.

The bond distances between the nitrogen and carbon atoms of the ethylenediamine residue (1.50(8), 1.57(8), 1.41(8) and 1.58(8) Å) are all considerably longer (by ca. 0.2 Å) than the nitrogen—carbon distances of the acetyleneiminato residue (1.35(8), 1.37(9) and 1.24(5) Å). This implies that there is a good deal of double bond character present in this bond and the tin—nitrogen interactions must be considered as being more coordinate in nature (Fig. 3, iv).

The fragmentation pattern for SnEDAA taken at 70 eV is shown in Fig. 4, and reflects the stronger tin—oxygen bonding. The mass spectra for $Me_2SnEDAA$ and $Me_3SnHEDAA$ are listed in Table 5, and are qualitatively similar to that of SnEDAA following the loss of methyl groups from tin.

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