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Diorganotin(IV) Compounds Derived from N-Methyl-2,2′-diphenolamine and 2,2′-Diphenolamine

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ABSTRACT: *The reaction of N-methyl-2,2′-diphenolamine* **1** *and 2,2′-diphenolamine* **2** *with some diorganotin(IV) oxides [R^1_2SnO: R^1 = Me, n-Bu, t-Bu and Ph] led to the syntheses of diorgano[N-methyl-2,2′-diphenolato-O,O′,N]tin (IV)* **3–6** *and diorgano[2,2′-diphenolato-O,O′,N]tin (IV)* **7–9**. *All compounds (except* **7***) studied in this work were characterized by ^{1}H, ^{13}C, ^{119}Sn NMR, infrared, and mass spectroscopy. Their ^{119}Sn NMR data show that the tin atom is tetracoordinated in $CDCl_3$ but penta and hexacoordinated in DMSO-d_6.* © 1999 John Wiley & Sons, Inc. Heteroatom Chem 10: 133–139, 1999

INTRODUCTION

Diorganotin(IV) compounds with Sn–O, Sn–S, and Sn ← N bonds, especially those derived from aminoalcohols and aminoacids, have received remarkable attention in recent years because they have shown cytotoxic activity. Some of them are characterized by a bicyclic structure due to an intramolecular nitrogen–tin coordination.

These compounds have been tested in vitro against various human cancer cell lines [1–9]. Also, they have been the subject of extensive studies because of their structural interest due to the fact that they show nitrogen–tin interaction [10–20].

We have been interested in the synthesis of new diorganotin(IV) compounds derived from aminoalcohols and aminoacetic acids mainly in order to study their structural characteristics, stereochemistry, and, in the future, to investigate their biological properties.

This article describes the syntheses and characterization by spectroscopic methods of diorgano[N-methylamino-2,2′-diphenolato-O,O′,N]tin(IV) **3** to **6** and diorgano[amino-2,2′-diphenolato-O,O′,N]tin(IV) **7** to **9** (Figure 1). Compound **7** was not fully characterized by ^{1}H, ^{13}C, and ^{119}Sn NMR spectroscopy because it is insoluble in common organic solvents; only its infrared and mass spectroscopic data are given in Table 3 and Figure 3, respectively.

DISCUSSION

The compounds diorgano[N-methylamino-2,2′-diphenolato-O,O′,N]tin(IV) **3** to **6** were obtained as gray, air-stable solids by the reaction of N-methyl-2,2′-diphenolamine **1** with diorganotin(IV) oxides [R_2SnO: R = Me, *n*-Bu, *tert*-Bu, and Ph].

The reaction of 2,2′-diphenolamine **2** with some diorganotin(IV) oxides [R_2SnO: R = Me, *n*-Bu, and Ph] gave diorgano[amino-2,2′-diphenolato-O,O′,N]tin(IV) compounds **7** to **9** as black air-stable solids having poor solubility in common organic solvents. Due to the insolubility of the dimethyltin-derived

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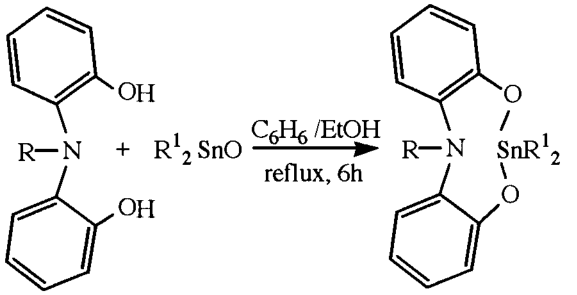


1: R=CH$_3$
2: R=H

3: R = CH$_3$, R^1= CH$_3$
4: R = CH$_3$, R^1= n-Bu
5: R = CH$_3$, R^1= t-Bu
6: R = CH$_3$, R^1= Ph
7: R = H, R^1= CH$_3$
8: R = H, R^1= n-Bu
9: R = H, R^1= Ph

FIGURE 1 Synthesis of diorganotin(IV) compounds **3–9**.

compound **7**, only its data of infrared (Table 3) and mass spectroscopy were obtained (Figure 3).

The ^{1}H and ^{119}Sn NMR data of compounds **3** to **6** are given in Table 1. Their spectra were recorded in CDCl$_3$ and DMSO-d$_6$. The aromatic protons of the compounds **3** and **4** exhibit the coupling pattern ABCD in both solvents; however, compounds **5** and **6** are not very soluble in DMSO-d$_6$, a precipitate being observed in the tube, and their spectra exhibit broad signals and an unresolved pattern, respectively, at δ 6.4–8.0. The SnR$^1_2$ groups of **3** and **5** exhibit two signals for each R^1 group, and **4** shows two triplets for the CH$_3$ groups of the Sn(n-Bu)$_2$ groups in CDCl$_3$; however, the spectra of **3** and **4** in DMSO-d$_6$ exhibit only one triplet. Also, **5** exhibits a singlet at δ 1.05 in DMSO-d$_6$; however, it also shows the two signals for the SnR$^1_2$ groups. It is possible that **5** decomposes in DMSO-d$_6$, and the same can be said for **6**. The N-CH$_3$ group of the compounds **3** to **6** exhibit a singlet in CDCl$_3$ and a coupling with the tin atom, which could indicate the presence of an intramolecular N → Sn coordination bond.

However, this coupling should be through the H-C-N-C-C-O moiety because their ^{119}Sn NMR spectra in deuteriochloroform exhibit values of $\delta(^{119}\mathrm{Sn})$ within the range of tetracoordinated compounds, depending on the nature of the groups bonded to tin [22,25]. Therefore, ^{119}Sn NMR spectra of **3** to **5** and **6** in DMSO-d$_6$ exhibit upfield values of $\delta(^{119}\mathrm{Sn})$ due to the effect of the solvent, within the range of penta and hexacoordinated compounds [21–25]. In general, the $\delta(^{119}\mathrm{Sn})$ values in the five-coordinate compounds appear at ca. 60–150 ppm upfield. On the corresponding four-coordinate analogs, six-coordination gives rise to an additional upfield shift of ca. 130–200 ppm [22]. Compounds **3** and **4** exhibit values of $\delta(^{119}\mathrm{Sn})$ typical of a pentacoordinated tin atom, while **5** exhibits a value of $\delta(^{119}\mathrm{Sn})$ expected for an equilibrium between a tetra- and pentacoordinated tin atom. In fact, its ^{1}H NMR spectrum exhibits broad signals for the aromatic protons.

The ^{119}Sn NMR spectrum of **6** exhibits six signals at $\delta(^{119}\mathrm{Sn})$ -225.9 (49.5%), -236.0 (41%), -367.3 (100%), -418.4 (30.8%), -479.7 (25.8%), and -527 (29.3%). This can be the result of decomposition or an equilibrium between isomers resulting from complex decomposition of the compound in DMSO-d$_6$. In fact, its ^{1}H NMR spectrum also exhibits an unresolved pattern. Compound **8** is poorly soluble in CDCl$_3$ and only exhibits an unresolved pattern. In DMSO-d$_6$ it is somewhat more soluble, but it also exhibits an unresolved pattern. Its ^{119}Sn NMR spectrum exhibits a value at $\delta(^{119}\mathrm{Sn})$ -359, which is within the range of an n-dibutyltin hexacoordinated compound. Compound **9** is slightly soluble in both solvents, but its ^{1}H NMR spectrum exhibits unresolved patterns. Its ^{119}Sn NMR spectrum in CDCl$_3$ exhibits a value at $\delta(^{119}\mathrm{Sn})$ -44.6 corresponding to tetracoordinated tin, while, in DMSO-d$_6$, at -225, it is within the range of pentacoordinated tin.

Table 2 shows the ^{13}C NMR spectra for the compounds **3** to **6**, **8**, and **9**.

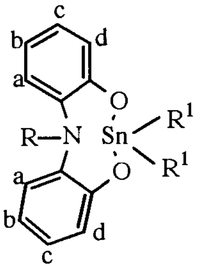
The assignments for C$_1$ to C$_7$ were achieved by comparison with the chemical shifts of some known diorganotin(IV) derivatives of N,N′-bis(o-hydroxyphenylene)ethylene diamine, the structures of which were assigned by 1D and 2D ^{1}H and ^{13}C NMR spectroscopy [18].

The assignments for the R^1-Sn carbons were deduced by comparison with the chemical shifts reported for analogous diorgatin compounds [2,18,20]. The compounds **3**, **4**, and **5** exhibit two signals in deuteriochloroform attributable to the carbons of the SnR$^1_2$ groups.

The assignment of the carbon atoms for compound **9** was achieved by a HETCOR experiment in deuteriochloroform in which the signals of H$_a$, H$_b$, H$_c$, and H$_d$ are correlated with the signals at $\delta(^{13}\mathrm{C})$ 128.3, 118.8, 129.2, and 121.4, respectively, the unresolved pattern of the SnPh$_2$ group at $\delta(^1\mathrm{H}_0)$ 7.58–7.64 is correlated with the signal at $\delta(^{13}\mathrm{C})$ 137.2 (C$_0$), and the unresolved pattern at $\delta(^1\mathrm{H}_{\mathrm{m,p}})$ 7.37–7.42 with the signals at $\delta(^{13}\mathrm{C})$ 127.9 (C$_{\mathrm{m}}$) and 129.4 (C$_{\mathrm{p}}$).

The infrared spectra of all compounds (see Table 3) show the band due to the $\nu_{\text{C-H arom}}$ in the range 3075–3050 cm^{-1}, a band due to the $\nu_{\text{C-H aliph}}$ in the

TABLE 1 ^{1}H and ^{119}Sn NMR Data for Compounds **3** to **6, 8,** and **9**



	R	R^1		R	R^1
3	CH_3	CH_3	**6**	CH_3	Ph
4	CH_3	n-Bu	**8**	H	n-Bu
5	CH_3	tert-Bu	**9**	H	Ph

Compound	N-R^c	CH_{arom}	Sn-R^1	δ(^{119}Sn)
3	3.02 (s: 16.5)	a: 6.93 (dd: 7.5, 1.5) b: 6.76 (ddd: 7.5, 1.5) c: 7.12 (ddd: 7.5, 1.5) d: 7.34 (dd: 7.5, 1.5)	0.79 (s) 0.62 (s) ^{2}(J^{119}Sn-C-^{1}H):64.8	−20.0
3^a	3.05 (a: 13.5)	a: 6.65 (dd: 7.5, 1.5) b: 6.55 (ddd: 7.5, 1.5) c: 7.00 (ddd: 7.5, 1.5) d: 7.40 (dd: 7.5, 1.5)	0.25 (s) ^{2}($J^{119/117}$Sn-C-^{1}H):72/69	−190
4	2.98 (s: 14.0)	a: 6.86 (dd: 8.0, 1.6) b: 6.68 (ddd: 8.0, 1.6) c: 7.03 (ddd: 8.0, 1.6) d: 7.27 (dd: 8.0, 1.6)	0.87 (*t*: 7.3) 0.89 (t: 7.3) 1.22–1.62 (m)	−55.1
4^a	3.0 (s: 12.0)	a: 6.60 (dd: 7.5, 1.5) b: 6.50 (ddd: 7.5, 1.5) c: 6.85 (ddd: 7.5, 1.5) d: 730 (dd: 7.5, 1.5)	0.80 (*t*: 7.5) 1.0–1.6 (m)	−207
5	3.28 (s: 13.8)	a: 6.99 (dd: 7.5, 1.5) b: 6.70 (ddd: 7.5, 1.5) c: 7.15 (ddd: 7.5, 1.5) c: 7.54 (dd: 7.5, 1.5)	0.98 (s) ^{3}($J^{119/117}$Sn-C-^{1}H):110.7/106.2 1.53 (s) ^{3}($J^{119/117}$Sn-C-^{1}H):112.2/106.9	−186 −207^a
6	3.23 (s: 24.4)	a: 7.04 (dd: 7.5, 1.5) b: 6.81 (ddd: 7.8, 1.5) c: 7.14 (ddd: 7.8, 1.5) d: 7.46 (dd: 7.5, 1.5)	$H_{m,p}$: 7.37–7.42 (m) H_0: 7.58–7.64 (m)	−44.9 −367^a
8		6.4–7.2 (m)	0.6–1.9 (m)	
8^a	7.4 (b)	6.4–7.2 (m)	0.6–1.9 (m)	−359
9		6.80–7.80 (m)^c	6.80–7.80 (m)^c	−44.6
9^a		6.50–8.00 (m)^c	6.50–8.00 (m)^c	−225

δ(^{1}H) relative to $Si(CH_3)_4$, δ (^{119}Sn) relative to $Sn(CH_3)_4$, |*J*| Hertz; solvent: $CDCl_3$.

[a] Solvent: DMSO-d_6;

[b] (J^{119}SnOCCN-C-^{1}H); b: broad; m: unresolved pattern; s: singlet; dd: doublet of doublets; ddd: doublet of doublet of doublets.

[c] The signals of aromatic protons are overlapped with those of the $SnPh_2$ groups.

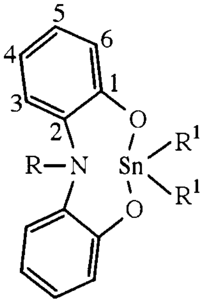
range 2975–2852 cm^{-1} and the strong band due to $\nu_{Sn\text{-}O}$ between 550 and 640 cm^{-1}.

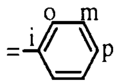
The mass spectra of compounds **3** to **6**, **8**, and **9** exhibit the fragmentation pattern proposed in Figure 2. The mass spectra of compounds **7** and **8** do not exhibit the molecular ion. Compound **7** shows a different fragmentation pattern in comparison with the mass spectra of the other compounds.

All the spectra exhibit other radical ions, and they are given in Figure 3. Compounds **6** and **9** show a fragment ion at *m*/*z* = 309 with the relative abundances 18% and 43%, respectively, assigned to the fragment ion $HO(H_2O)SnPh_2]^+$. Compounds **3** and **7** exhibit the fragment ion at *m*/*z* = 135 assigned to $SnCH_3]^+$ with relative abundances 12% and 11%, respectively.

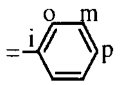
Compounds **5** and **8** exhibit the fragment ion at *m*/*z* = 57 with relative abundances 92% and 34%, respectively, assigned to the fragment ion $C_4H_9]^+$. The spectra of compounds **7** and **8** show fragment ions as the base peak for **7** at *m*/*z* = 45 assigned to the fragment ion $NH(CH_3)_2]^+$, apparently arising from the coupling between NH and the methyl groups attached at the tin atom. The base peak for **8**

TABLE 2 ^{13}C NMR Data for Compounds **3–6**, **8**, and **9**



3 R = CH_3 $R^1 = {}^{\alpha}CH_3$ **6** R = CH_3 R^1 = 

4 R = CH_3 $R^1 = {}^{\alpha}CH_2\text{-}{}^{\beta}CH_2\text{-}{}^{\gamma}CH_2\text{-}{}^{\delta}CH_3$ **8** R = H $R^1 = {}^{\alpha}CH_2\text{-}{}^{\beta}CH_2\text{-}{}^{\gamma}CH_2\text{-}{}^{\delta}CH_3$

5 R = CH_3 $R^1 = {}^{\alpha}C({}^{\beta}CH_3)_3$ **9** R = H R^1 = 

Compound	*C_1*	*C_2*	*C_3*	*C_4*	*C_5*	*C_6*	*C_7*	*Sn-R^1*			
3	154.4	135.8	121.4	118.8	129.1	118.9	47.8	C_α 1.29			
	[22.3]	[26.9]	[28.4]		[7.7]	[24.5]		−1.42			
3^a	156.4	138.4	123.0	115.9	126.8	117.6	44.3	C_α 4.1			
4	156.2	136.5	122.4	117.5	128.1	119.7	46.5	C_α 18.7	C_β 26.3	C_γ 25.8	C_δ 12.6
	[23.0]	[21.5]	[9.7]			[19.5]		[432]	26.4	26.0	12.7
								20.7	[21.5]		
								[415]			
4^a	156.8	139.0	122.8	115.9	126.6	117.7	44.2	C_α 21.9	C_β 27.5	C_γ 26.1	C_δ 13.6
	[22.4]	[24.0]				[24.4]			[22.5]		
5	158.5	135.1	122.7	116.3	128.7	118.8	51.6	C_α 42.2	C_β 29.8 [10.7]		
								41.4	30.6 [10]		
5^a	154.1	136.7	122.7	117.6	127.9	118.1	50.3	C_α 46.8	C_β 31.9		
6	154.4	135.8	128.3	118.8	129.1	121.4	47.6	C_i 137.1	C_o 137.2	C_m 128.6	C_p 130.4
	[15.0]	[28]	[25.9]			[27.0]			[39.0]	[39]	[13.0]
8^a	154.0	138.0	123.0	115.6	127.0	117.8		C_α 23.5	C_β 27.2	C_γ 25.7	C_δ 13.4
9	156	136.9	128.6	118.1	129.6	118.5		C_i 137.3	C_o 136.1	C_m 129.2	C_p 130.5

δ: ppm; |*J*| Hertz *J*(^{119}Sn-^{13}C); solvent: $CDCl_3$.
^{a}Solvent: DMSO-d_6.

TABLE 3 Infrared Data for Compounds **3** to **9**

Compound	*νC-H arom*	*νC-H aliph*	*νSn-O*
3	3050	2950 2925	550
4	3074	2936 2864	558
5	3056	2930 2858	542
6	3050	2970	581
7	3050	2963	581
8	3066	2934 2852	560
9	3075	2975	640

ν(cm^{-1}), KBr.

is at $m/z = 44$, and this can be assigned to the fragment ion $N(CH_3)_3]^+$ or $C_3H_8]^+$. The base peaks for the other compounds are given in Figure 3.

EXPERIMENTAL

NMR spectra were recorded on Varian EM-390, Jeol GLX-270, and Jeol Eclipse-400 spectrometers. All ^{1}H and ^{13}C chemical shifts are reported relative to TMS and ^{119}Sn to $Sn(CH_3)_4$, $CDCl_3$ and DMSO-d_6 being used as solvents. Mass spectra were obtained with a Hewlett-Packard 59940-A instrument, and infrared spectra were determined on a Perkin-Elmer 16F PC FT-IR spectrometer. Melting points were taken in open capillary tubes on a Gallenkamp MFB-595 apparatus and are uncorrected.

The procedure outlined in the following section is general for the preparation of diorganotin(IV) derivatives of N-methyl-2,2-diphenolamine **1** and 2,2-diphenolamine **2**.

Dimethyl [N-methylamino-2,2′-diphenolato-O,O′,N]tin(IV) 3

A solution of 0.28 g (1.37 mmol) of N-methyl-2,2-diphenolamine **1** in 100 mL of an 80/20 benzene/ethanol mixture was placed in a flask equipped with a magnetic stirrer and a Dean-Stark trap, and 0.22 g (1.37 mmol) of dimethyltin(IV) oxide was added. The suspension was refluxed during 6 hours. After the mixture had attained room temperature, the solvent was evaporated under vacuum. The residual

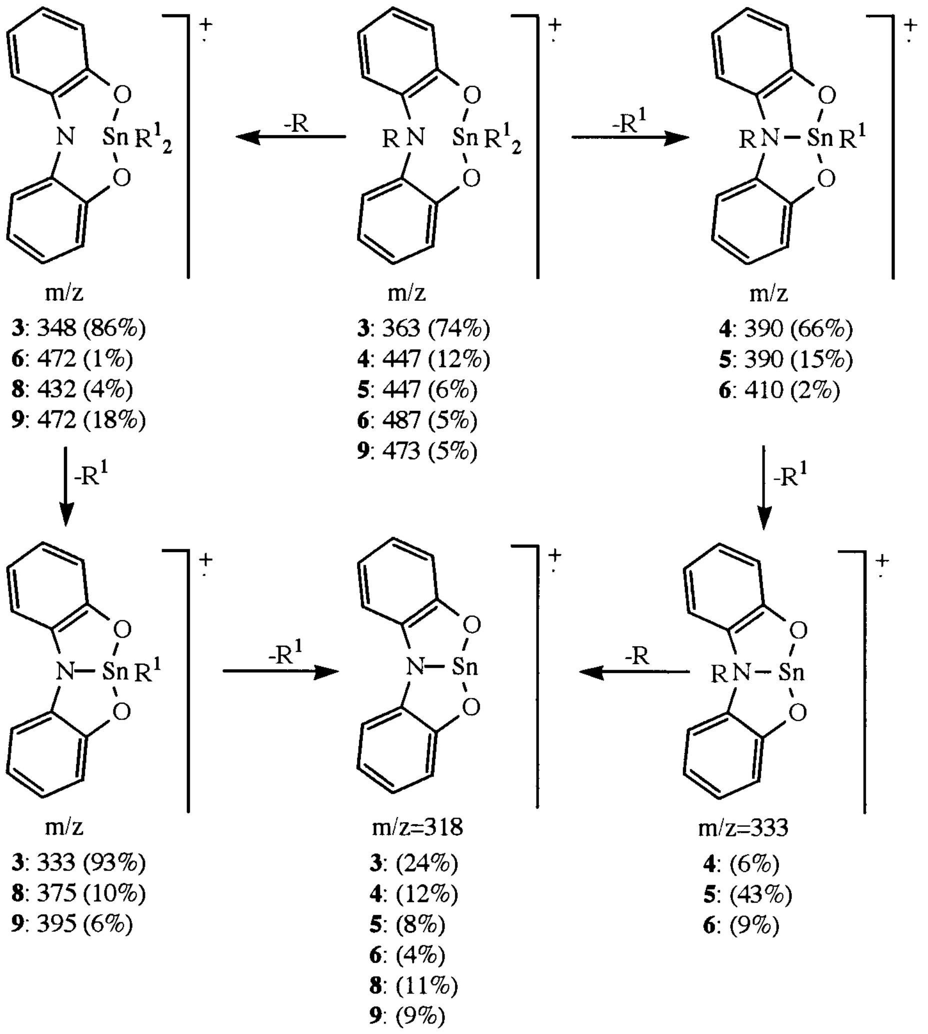


FIGURE 2 Mass spectral data for compounds **3** to **6**, **8**, and **9**.

gray solid was recrystallized from acetone to yield 0.48 g (94.6%) of compound **3** with mp 157–158°C.

di-n-Butyl[N-methylamino-2,2′-diphenolato-O,O′,N]tin(IV) 4

A 0.28 g (1.37 mmol) amount of compound **1** and 0.34 g (1.37 mmol) of di-*n*-butyltin(IV) oxide gave 0.55 g (88.6%) of compound **4** (gray solid), recrystallized from acetone, mp 140°C.

di-Tert-butyl[N-methylamino-2,2′-diphenolato-O,O′,N]tin(IV) 5

A 0.12 g (0.55 mmol) amount of compound **1** and 0.14 g (0.55 mmol) of di-*tert*-butyltin(IV) oxide gave 0.25 g (97%) of compound **5** (gray solid), recrystallized from acetone, mp 104°C.

Diphenyl[N-methylamino-2,2′-diphenolato-O,O′,N]tin(IV) 6

The reaction of 0.19 g (0.93 mmol) of compound **1** and 0.26 g (0.93 mmol) of diphenyltin(IV) oxide gave 0.43 g (97%) of compound **6** (gray solid), recrystallized from acetone, mp 155°C (dec).

Synthesis of Dimethyl[amino-2,2′-diphenolato-O,O′,N]tin(IV) 7

The reaction of 0.18 g (0.88 mmol) of 2,2-diphenolamine **2** and 0.14 g (0.88 mmol) of dimethyltin (IV) oxide gave 0.26 g (84.7%) of compound **7** (black solid) recrystallized from acetone/ether. Its melting point could not be determined. The melting point apparatus reached 340°C, but no melting was observed.
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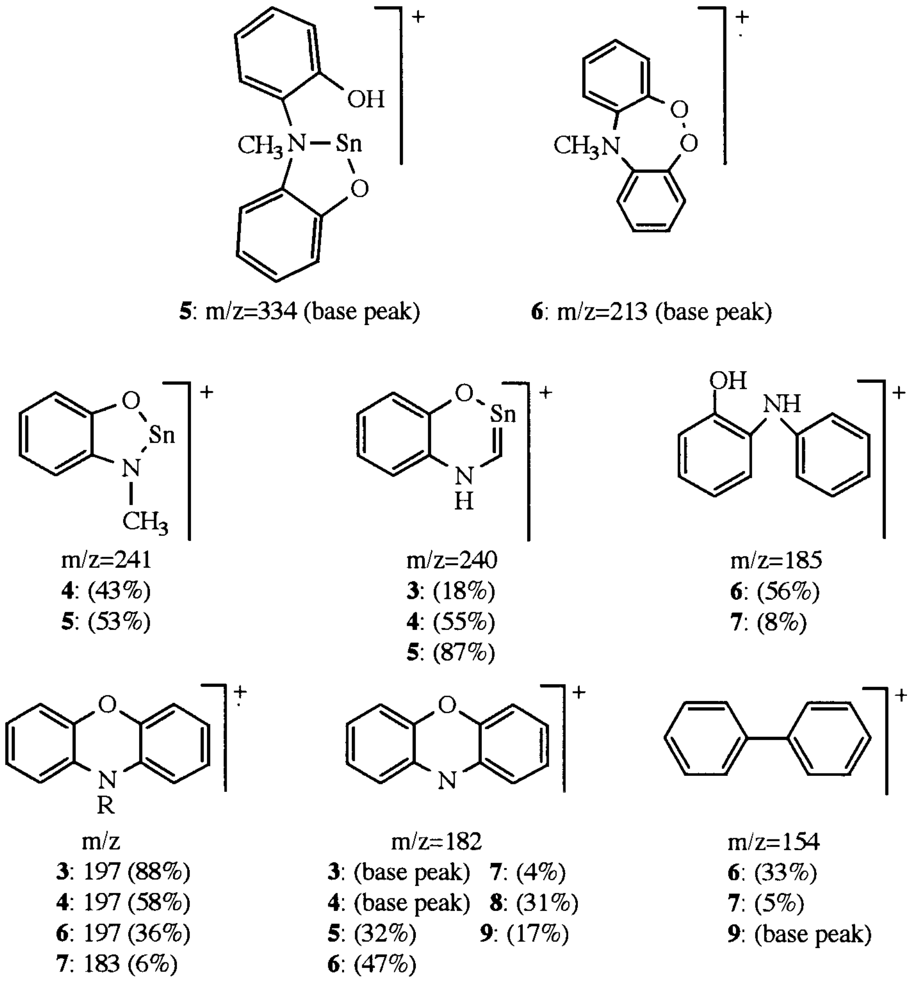

FIGURE 3 Other fragment ions for compounds **3** to **9**.

di-n-Butyl[amino-2,2′-diphenolato-O,O′,N]tin(IV) 8

The reaction of 0.15 g (0.73 mmol) of compound 2 and 0.18 g (0.73 mmol) of di-*n*-butyltin(IV) oxide gave 0.28 g (88.6%) of compound 8 (black solid), recrystallized from acetone/ether. Its melting point could not be determined.

The melting point apparatus reached 340°C, but no melting was observed.

Diphenyl[amino-2,2′-diphenolato-O,O′,N]tin(IV) 9

A 0.34 g (1.67 mmol) amount of compound 2 and 0.48 g (0.48 mmol) of diphenyltin(IV) oxide gave 0.72 g (89.0%) of compound 9 (black solid, recrystallized from acetone/benzene). Its melting point could not be determined.

The melting point apparatus reached 350°C, but no melting was observed.

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