

Mass Spectral Studies of Some Triorganotin Derivatives Containing Carboxylates

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The mass spectra of 16 triorganotin compounds having the general formula R_3SnL where $R=CH_3, C_4H_9, C_6H_5, C_6H_{11}$ and $L=trans\text{-}3\text{-}(2\text{-furanyl})\text{-}2\text{-propenoate, trans}\text{-}m\text{-methyl cinnamate, 2-}(2,3\text{-dimethylphenyl})\text{aminobenzoate}$ and $trans\text{-}3\text{-}(2\text{-thienyl})\text{-}2\text{-propenoate}$, were investigated. The possible fragmentation pattern is discussed in detail.

Key words: Mass spectra, Organotin carboxylates, Triorganotins.

Introduction

Different spectroscopic techniques have been employed for the characterization of organotin carboxylates¹⁻³. Among such techniques, mass spectrometry has been growing in structural elucidation and an interpretation of organotin carboxylates⁴⁻⁸. Bond dissociation energies for these compounds are therefore relatively low in organotin compounds, particularly larger ones, parent ions are not observed and the molecules suffer considerable fragmentation in the mass spectrometer. Smaller organotin molecules often show the molecular ion peak as well as a characteristic series of fragmentation peaks⁹. The first mass spectrometric studies of organotin compounds appeared in 1966^{10,11}, whereas more reports are available on different aspects of mass spectrometry of organotin compounds¹²⁻¹⁸. Previously, we studied different aspects of organotin carboxylates^{6-8,19-22}. In the present paper we are going to report the mass spectrometry and fragmentation pattern of triorganotin derivatives in some detail.

Experimental

Instrumentation

The mass spectral measurements of the compounds were made on MAT 112S, MAT 8500 and Varian MAT 312 Mass Spectrometers. The m/z values are computed according to $H=1, C=12, O=16, S=32$ and $Sn=120$.

Synthesis

The following triorganotin derivatives were prepared by methods reported earlier^{6,7,19,20}.

1. Trimethyltin (trans-3-(2-furanyl)-2-propenoate)

$[(C_4H_3O)CHCHCOOSn(CH_3)_3]^+$ 302 (not observed);
 $[(C_4H_3O)CHCHCOOSn(CH_3)_2]^+$ 287 (91); $[(C_4H_3O)CHCHSn(CH_3)_2]^+$ 243 (100);
 $[(C_4H_3O)CHCHSn]^+$ 213 (35); $[Sn(CH_3)_3]^+$ 165 (30); $[Sn(CH_3)_2]^+$ 150 (18);
 $[(C_4H_3O)CHCHCOOH]^+$ 138 (7); $[SnCH_3]^+$ 135 (42); $[SnH]^+$ 121 (55); $[Sn]^+$ 120 (18);
 $[(C_4H_3O)CHCH]^+$ 93 (14); $[C_5H_5]^+$ 65 (75).

2. Trimethyltin (trans-m-methyl cinnamate)

$[(CH_3)C_6H_4CHCHCOOSn(CH_3)_3]^+$ 326 (not observed);
 $[(CH_3)C_6H_4CHCHCOOSn(CH_3)_2]^+$ 311 (100); $[(CH_3)C_6H_4CHCHSn(CH_3)_2]^+$ 267 (55);
 $[(CH_3)C_6H_4CHCHSn]^+$ 237 (15); $[Sn(CH_3)_3]^+$ 165 (27); $[(CH_3)C_6H_4CHCHCOOH]^+$ 162 (47);
 $[(CH_3)C_6H_4CHCHCOO]^+$ 161 (36); $[Sn(CH_3)_2]^+$ 150 (35); $[(CH_3)C_6H_4CHCH]^+$ 117 (51);
 $[(CH_3)C_6H_4]^+$ 91 (31).

3. Trimethyltin (2-(2,3-dimethylphenyl)aminobenzoate)

$[(CH_3)_2C_6H_3NHC_6H_4COOSn(CH_3)_3]^+$ 405(100);
 $[(CH_3)_2C_6H_3NHC_6H_4COOSn(CH_3)_2]^+$ 390 (60); $[(CH_3)_2C_6H_3NHC_6H_4Sn(CH_3)_2]^+$ 346 (28);
 $[(CH_3)_2C_6H_3NHC_6H_4Sn]^+$ 316 (2); $[(CH_3)_2C_6H_3NHC_6H_4COOH]^+$ 241 (5); $[Sn(CH_3)_3]^+$ 165 (23);
 $[Sn(CH_3)_2]^+$ 150 (5); $[SnCH_3]^+$ 135 (4).

4. Trimethyltin (trans-3-(2-thienyl)-2-propenoate)

$[(C_4H_3S)CHCHCOOSn(CH_3)_3]^+$ 318 (not observed);
 $[(C_4H_3S)CHCHCOOSn(CH_3)_2]^+$ 303 (11); $[(C_4H_3S)CHCHCOOSn(CH_3)Sn(CH_3)_2]^+$ 259 (9);
 $[(C_4H_3S)CHCHSn]^+$ 229 (10); $[(C_4H_3S)CHCHCOOH]^+$ 154 (100); $[(C_4H_3S)CHCH]^+$ 109 (42);
 $[SnH]^+$ 121 (45).

5. Triphenyltin (trans-3-(2-furanyl)-2-propenoate)

$[(C_4H_3O)(CHCHCOOSnC_6H_5)_3]^+$ 488 (20);
 $[(C_4H_3O)CHCHCOOSn(C_6H_5)_2]^+$ 411 (100); $[(C_4H_3O)CHCHCOOSn]^+$ 257 (5);
 $[Sn(C_6H_5)]^+$ 197 (50); $[C_{12}H_{10}]^+$ 154 (35);
 $[(C_4H_3O)CHCHCOOH]^+$ 138 (8); $[SnH/Sn]^+$ 121 (35); $[(C_4H_3O)CHCH]^+$ 93 (6); $[C_6H_5]^+$ 77 (10);
 $[C_5H_5]^+$ 65 (15).

6. Triphenyltin (trans-m-methyl cinnamate)

$[(CH_3)C_6H_4CHCHCOOSn(C_6H_5)_3]^+$ 512 (8); $[(CH_3)C_6H_4CHCHCOOSn(C_6H_5)_2]^+$ 435 (100);
 $[(CH_3)C_6H_4CHCHSn(C_6H_5)_2]^+$ 391 (30); $[(CH_3)C_6H_4CHCHSn(C_6H_5)]^+$ 314 (9);
 $[(CH_3)C_6H_4CHCHSn]^+$ 237 (6); $[(CH_3)C_6H_4Sn]^+$ 211 (15);
 $[(CH_3)C_6H_4CHCHCOO]^+$ 161 (3); $[(CH_3)C_6H_4CHCH]^+$ 117 (11); $[(CH_3)C_6H_4]^+$ 91 (8).

7. Triphenyltin (2-(2,3-dimethylphenyl)aminobenzoate)

$[(CH_3)_2C_6H_3NHC_6H_4COOSn(C_6H_5)_3]^+$ 591 (92);
 $[(CH_3)_2C_6H_3NHC_6H_4COOSn(C_6H_5)_2]^+$ 514 (31);
 $[(CH_3)_2C_6H_3NHC_6H_4Sn(C_6H_5)_2]^+$ 470 (10); $[(CH_3)_2C_6H_3NHC_6H_4Sn]^+$ 316 (7);
 $[(CH_3)_2C_6H_3NHC_6H_4COOH]^+$ 241 (64); $[C_8H_9Sn]^+$ 225 (100);
 $[Sn(C_6H_5)_3]^+$ 351 (88); $[Sn(C_6H_5)_2]^+$ 274 (11); $[Sn]^+$ 120 (31).

8. Triphenyltin (trans-3-(2-thienyl)-2-propenoate)

$[(C_4H_3S)CHCHCOOSn(C_6H_5)_3]^+$ 504 (0.4);

$[(C_4H_3S)CHCHCOOSn(C_6H_5)_2]^+$ 427 (31); $[(C_4H_3S)CHCHSn(C_6H_5)_2]^+$ 384 (2);
 $[(C_4H_3S)CHCHSn]^+$ 229 (0.2); $[(C_4H_3S)CHCHCOOH]^+$ 154 (100); $[SnH]^+$ 121 (46);
 $[(C_4H_3S)CHCH]^+$ 109 (36).

9. Tributyltin (trans-3-(2-furanyl)-2-propenoate)

$[(C_4H_3O)CHCHCOOSn(C_4H_9)_3]^+$ 428 (not observed);
 $[(C_4H_3O)CHCHCOOSn(C_4H_9)_2]^+$ 371 (45); $[(C_4H_3O)CHCHSn(C_4H_9)_2]^+$ 327 (45);
 $[Sn(C_4H_9)_2]^+$ 234 (5); $[(C_4H_3O)CHCHSn]^+$ 213 (10); $[C_4H_3O)CHCHCOOH]^+$ 138 (100);
 $[SnH/Sn]^+$ 121 (12); $[C_8H_{18}]^+$ 114 (22); $[(C_4H_3O)CHCH]^+$ 93 (5); $[C_5H_5]^+$ 65 (4).

10. Tributyltin (trans-*m*-methyl cinnamate)-

$[(CH_3)C_6H_4CHCHCOOSn(C_4H_9)_3]^+$ 452 (not observed);
 $[(CH_3)C_6H_4CHCHCOOSn(C_4H_9)_2]^+$ 395 (100);
 $[(CH_3)C_6H_4CHCHSn(C_4H_9)_2]^+$ 351 (3); $[Sn(C_4H_9)_3]^+$ 291 (7); $[Sn(C_4H_9)_2]^+$ 234 (11); $[SnC_4H_9]^+$ 177
(5); $[(CH_3)C_6H_4CHCHCOO]^+$ 161 (38); $[(CH_3)C_6H_4CHCH]^+$ 117 (40);
 $[C_8H_{18}]^+$ 114 (3) $[(CH_3)C_6H_4]^+$ 91 (37).

11. Tributyltin (2-(2,3-dimethylphenyl)aminobenzoate)

$[(CH_3)_2C_6H_3NHC_6H_4COOSn(C_4H_9)_3]^+$ 531 (not observed);
 $[(CH_3)_2C_6H_3NHC_6H_4COOSn(C_4H_9)_2]^+$ 474 (44);
 $[(CH_3)_2C_6H_3NHC_6H_4Sn(C_4H_9)_2]^+$ 430 (43); $[(CH_3)_2C_6H_3NHC_6H_4SnC_4H_9]^+$ 373 (28);
 $[(CH_3)_2C_6H_3NHC_6H_4Sn]^+$ 316 (100); $[(CH_3)_2C_6H_3NHC_6H_4COOH]^+$ 241 (38);
 $[(CH_3)_2C_6H_3NHC_6H_4]^+$ 196 (48); $[SnH]^+$ 121 (42);
 $[Sn]^+$ 120 (40); $[C_6H_3NH]^+$ 90 (18); $[C_5H_5]^+$ 65 (39).

12. Tributyltin (trans-3-(2-thienyl)-2-propenoate)

$[(C_4H_3S)CHCHCOOSn(C_4H_9)_3]^+$ 444 (0.5);
 $[(C_4H_3S)CHCHCOOSn(C_4H_9)_2]^+$ 387 (100);
 $[(C_4H_3S)CHCHSn(C_4H_9)_2]^+$ 343 (3);
 $[(C_4H_3S)CHCHSn]^+$ 229 (18); $[(C_4H_3S)CHCHCOOH]^+$ 154 (6); $[SnH]^+$ 121 (3);
 $[(C_4H_3S)CHCH]^+$ 109 (9).

13. Tricyclohexyltin (trans-3-(2-furanyl)-2-propenoate)

$[(C_4H_3O)CHCHCOOSn(C_6H_{11})_3]^+$ 506 (not observed);
 $[(C_4H_3O)CHCHCOOSn(C_6H_{11})_2]^+$ 423 (100); $[(C_4H_3O)CHCHSn(C_6H_{11})_2]^+$ 379 (7);
 $[(C_4H_3O)CHCHCOOSnC_6H_{11}]^+$ 340 (11); $[(C_4H_3O)CHCHSnC_6H_{11}]^+$ 296 (6);
 $[Sn(C_6H_{11})_2]^+$ 286 (22); $[(C_4H_3O)CHCHCOOSn]^+$ 257 (35);
 $[(C_4H_3O)CHCHCOO]^+$ 137 (32); $[Sn]^+$ 120(18);
 $[(C_6H_{11})]^+$ 83 (54); $[C_5H_5]^+$ 65 (60).

14. Tricyclohexyltin (trans-*m*-methyl-cinnamate)

$[(CH_3)C_6H_4CHCHCOOSn(C_6H_{11})_3]^+$ 530 (not observed);
 $[(CH_3)C_6H_4CHCHCOOSn(C_6H_{11})_2]^+$ 447 (100); $[(CH_3)C_6H_4CHCHCOOSnC_6H_{11}]^+$ 364 (4);
 $[(CH_3)C_6H_4CHCHSnC_6H_{11}]^+$ 320 (4); $[(CH_3)C_6H_4CHCHCOOSn]^+$ 281 (18); $[(CH_3)C_6H_4CHCHSn]^+$
237 (13); $[(CH_3)C_6H_4CHCHCOO]^+$ 161 (10); $[(CH_3)C_6H_4]^+$ 91 (5); $[C_6H_{11}]^+$ 83 (10).

15. Tricyclohexyltin (2-(2,3-dimethylphenyl)aminobenzoate)

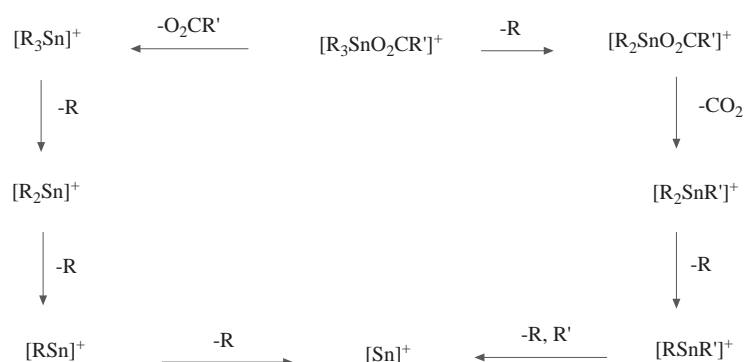
$[(CH_3)_2C_6H_3NHC_6H_4COOSn(C_6H_{11})_3]^+$ 609 (1.5);
 $[(CH_3)_2C_6H_3NHC_6H_4COOSn(C_6H_{11})_2]^+$ 526 (100); $[(CH_3)_2C_6H_3NHC_6H_4Sn]^+$ 316 (37);
 $[(CH_3)_2C_6H_3NHC_6H_4COOH]^+$ 241 (13).

16. Tricyclohexyltin (trans-3-(2-thienyl)-2-propanoate)

$[(C_4H_3S)CHCHCOOSn(C_6H_{11})_3]^+$ 522 (not observed);
 $[(C_4H_3S)CHCHCOOSn(C_6H_{11})_2]^+$ 439 (46); $[(C_4H_3S)CHCHSn(C_6H_{11})_2]^+$ 395 (0.2);
 $[(C_4H_3S)CHCHSn]^+$ 229 (7.5); $[(C_4H_3S)CHCHCOOH]^+$ 154 (100); $[SnH]^+$ 121 (40); $[(C_4H_3S)CHCH]^+$
 109 (41).

Results and Discussion

The 80 eV monoisotopic mass fragmentation of the reported compounds with general fragmentation pattern is given in Scheme 1. Table 1 shows the common fragments with % abundance. The molecular ion peak in most organometallic compounds is not observed²³. We also observed similar pattern for all compounds except 3, 5-8, 12 and 15⁶. Here we discuss all these compounds with regard to the carboxylate groups.



Fragmentation Pattern of Triorganotin Carboxylates.

R = Alkyl or aryl group attached to tin.

R' = Carboxylate motif.

Scheme 1.

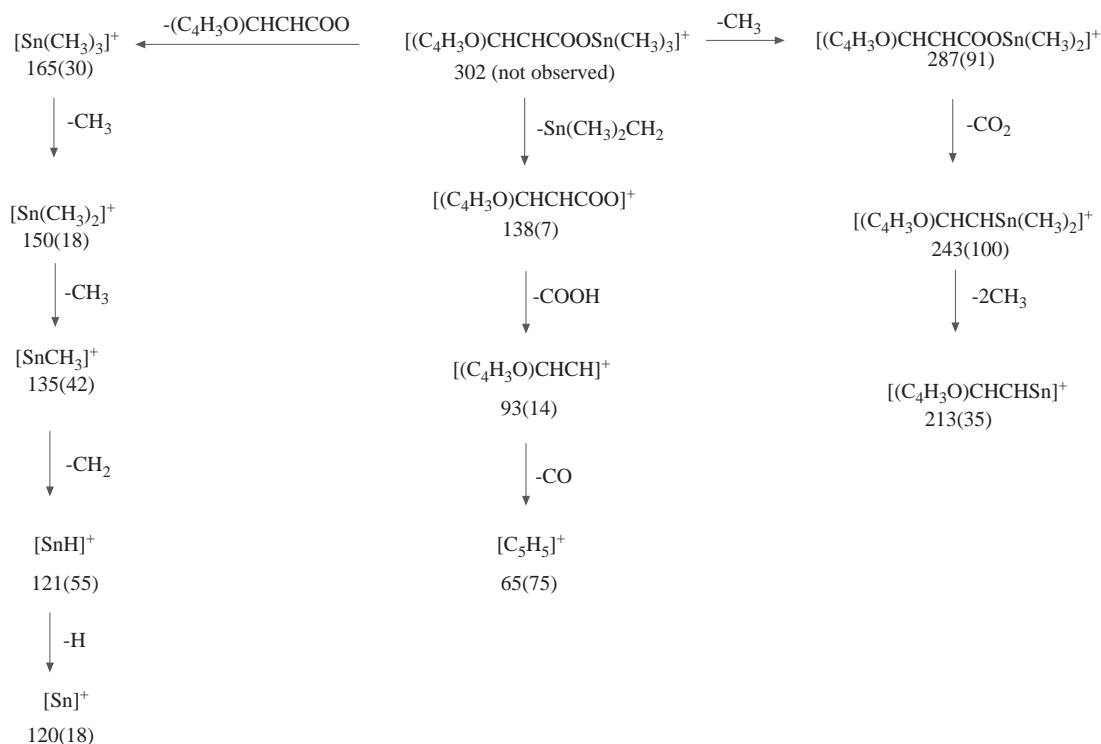
Table 1. Common Fragments of Investigated Compounds.

Fragment ion	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
$R_3SnO_2CR'^+$	-	-	100	-	20	8	92	0.4	-	-	-	0.5	-	-	1.5	-
$R_2SnO_2CR'^+$	91	100	60	11	100	100	31	31	45	100	44	100	100	100	100	46
$R_2SnR'^+$	100	55	-	9	-	30	-	2	30	3	28	3	7	-	-	0.2
SnR'^+	35	15	28	10	5	6	10	-	10	-	100	18	-	13	37	7.5
R_3Sn^+	30	27	23	-	-	-	88	-	-	7	-	-	-	-	-	-
R_2Sn^+	18	35	5	-	-	-	11	-	5	11	-	-	-	-	-	-
RSn^+	42	-	4	-	50	-	-	-	-	5	-	-	-	-	-	-
SnH/Sn^+	55/18	-	4	45	35	-	31	46	12	-	42/40	3	18	-	-	-
HO_2CR'/O_2CR'	7	47/36	5	100	8	3	64	100	100	38	38	6	32	10	13	100

I. Triorganotin compounds of trans-3-(2-furanyl)-2-propenoate.

Compounds 1, 5, 9 and 13 are triorganotin derivatives of trans-3-(2-furanyl)-2-propenoate, and Scheme 2 shows a representative fragmentation pattern of the series. All the triorganotin compounds in this series follow almost the same pattern as that shown for trimethyltin (trans-3-(2-furanyl)-2-propenoate in Scheme 2. The m/z value of each fragment is given along with % relative abundance in parenthesis. The molecular ion peak is not observed at m/z 302 but a very intense peak is observed at 287 due to loss of the CH₃ radical, which follows the base peak at m/z 243 due to elimination of CO₂, and the further loss of two CH₃ radicals gives a peak of m/z 213. The loss of ligand from the molecular ion gives a peak due to $[Sn(CH_3)_3]^+$ at

m/z 165, which with successive loss of CH_3 radicals, gives fragments at m/z 150 and 135 for $[\text{Sn}(\text{CH}_3)_2]^+$ and $[\text{Sn}(\text{CH}_3)]^+$ respectively, and further loss of CH_2 and H gives m/z 121 and 120 for $[\text{SnH}]^+$ and $[\text{Sn}]^+$ respectively. A peak corresponding to the ligand appears at m/z 138 that on elimination of COOH and CO gives peaks at m/z 93 and 65 respectively. The common ions observed are $[\text{R}_2\text{SnO}_2\text{CR}']$, $[\text{R}_2\text{SnR}']$, $[\text{SnH}/\text{Sn}]^+$ and $[\text{R}'\text{CO}_2/\text{R}'\text{CO}_2\text{H}]^+$.



Scheme 2.

II. Triorganotin compounds of trans-m-methyl cinnamate.

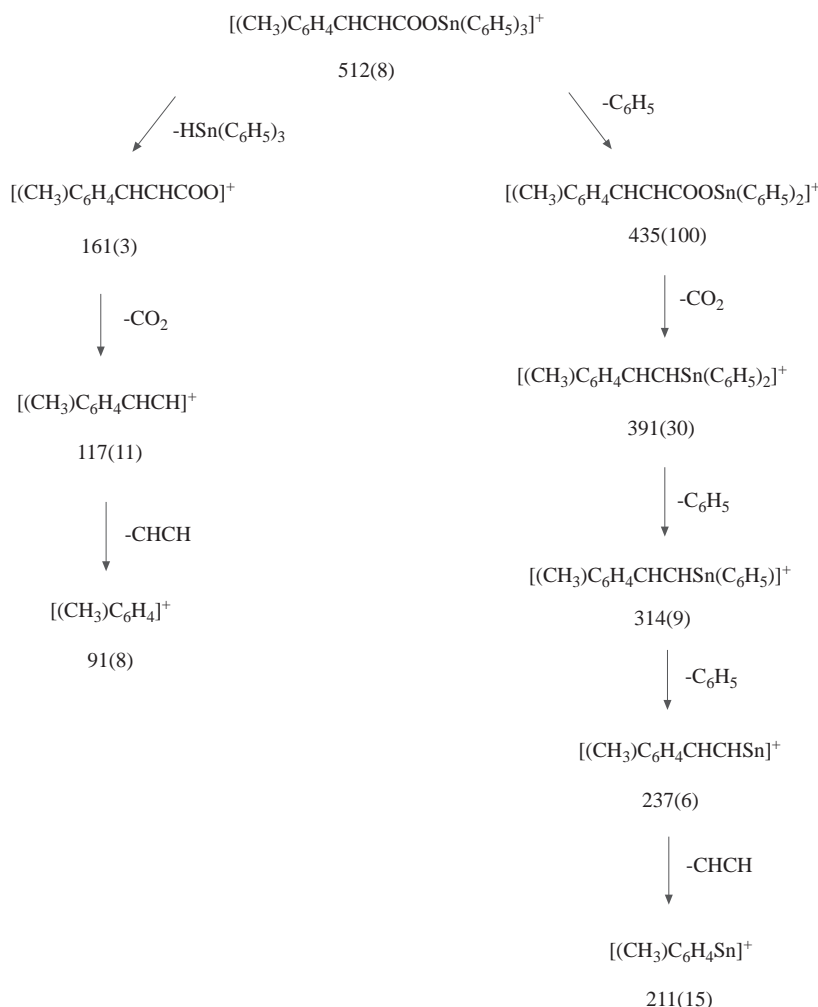
Compounds of this series are 2, 6, 10 and 14, while the representative fragmentation pattern is given in Scheme 3. As shown in this scheme, a low intensity molecular ion peak is present. The loss of C_6H_5 and CO_2 from the parent ion gives at m/z 435 and 391 for $[(\text{CH}_3)\text{C}_6\text{H}_4\text{CHCHCOOSn}(\text{C}_6\text{H}_5)_2]^+$ and $[(\text{CH}_3)\text{C}_6\text{H}_4\text{CHCHSn}(\text{C}_6\text{H}_5)_2]$ respectively. The elimination of two successive C_6H_5 groups results in $[(\text{CH}_3)\text{C}_6\text{H}_4\text{CHCHSn}]^+$ m/z 237, which on elimination of C_2H_2 gives m/z 211 for $[(\text{CH}_3)\text{C}_6\text{H}_4\text{Sn}]^+$. The ligand peak at m/z 161 after elimination of CO_2 and C_2H_2 gives m/z 91.

The base peak in these compounds is observed after elimination of R group from parent molecular ion. The common fragment observed is $\text{R}_2\text{SnR}'$ except in compound 14. The ligand peak at m/z 161 is observed in all compounds.

III. Triorganotin Compounds of 2-(2,3-dimethylphenyl)aminobenzoate.

Compounds 3, 7, 11 and 15 represent the triorganotin derivatives of 2-(2,3-dimethylphenyl)aminobenzoate, and Scheme 4 shows the representative fragmentation pattern of the tributyltin derivative, which is similar, as shown in Schemes 2 and 3. One interesting feature is that trimethyltin 2-(2,3-dimethylphenyl)aminobenzoate yielded molecular ion as base peak. None of the other compounds exhibit this behaviour. The common fragments observed in these compounds are $[\text{R}_2\text{SnO}_2\text{R}']^+$, $[\text{SnR}]^+$ and $[\text{HO}_2\text{CR}'/\text{O}_2\text{CR}']^+$. The m/z 241 for ligand shows loss of $[\text{CO}_2/\text{CO}_2\text{H}]^+$ to give m/z 196, fragment $[(\text{CH}_3)_2\text{C}_6\text{H}_3\text{NHC}_6\text{H}_4]^+$, which yields

$[\text{C}_6\text{H}_3\text{NH}]^+$ m/z 90 after elimination of the $[(\text{CH}_3)_2\text{C}_6\text{H}_4]^+$ radical.

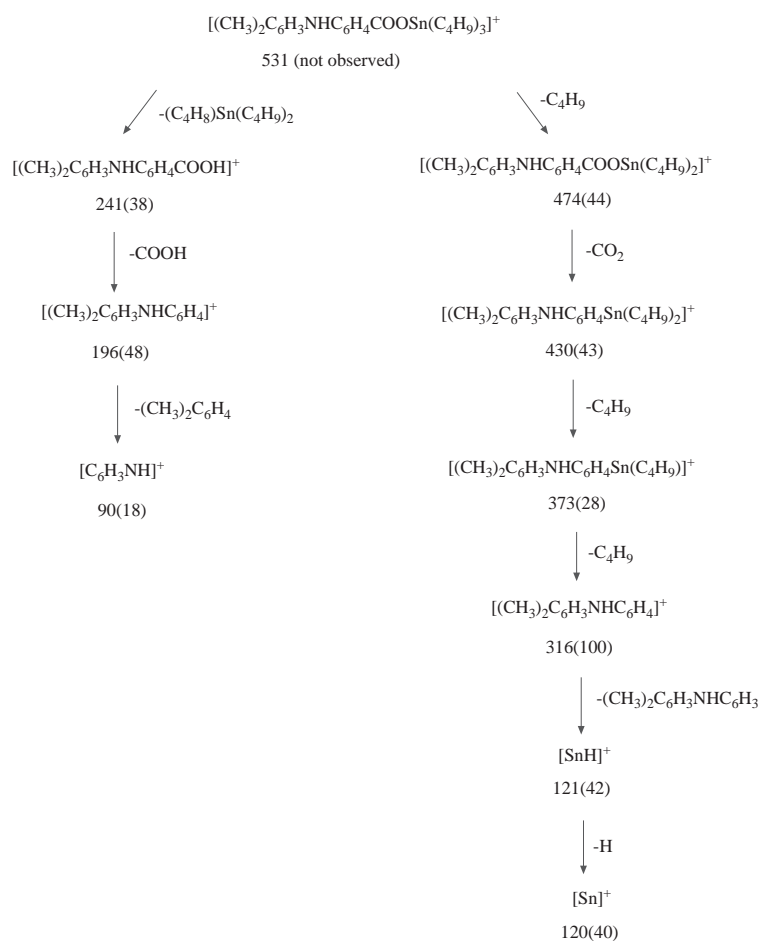


Scheme 3.

IV. Triorganotin compounds of trans-3-(2-thienyl)-2-propenoate

Compounds 4, 8, 12 and 16 are the triorganotin compounds of trans-3-(2-thienyl)-2-propenoate and Scheme 5 shows the representative fragmentation pattern of tricyclohexyltin trans-3-(2-thienyl)-2-propenoate, which has a similar pattern to that discussed for other compounds. Common ions observed include $[\text{R}_2\text{SnO}_2\text{CR}']^+$, $[\text{R}_2\text{SnR}']^+$ and $[\text{HO}_2\text{CR}' / \text{O}_2\text{CR}']^+$.

Thus from the above discussion it is concluded that triorganotin derivatives of carboxylates follow the same general pattern as shown in Scheme 1⁸, which suggests that primary decomposition follows the elimination of R, which on further elimination of R and CO_2 gives $[\text{R}_2\text{SnR}']^+$ or $[\text{SnR}']^+$. The $[\text{R}_3\text{Sn}]^+$, $[\text{R}_2\text{Sn}]^+$ and $[\text{RSn}]^+$ peaks are not present in most cases, suggesting that fragmentation through these species is not favourable⁸. In most cases, the base peak is observed after the elimination of the R group, whereas in some cases, ligands form the base peak (Table 1). The representative spectrum is given in Figure 1.



Scheme 4.

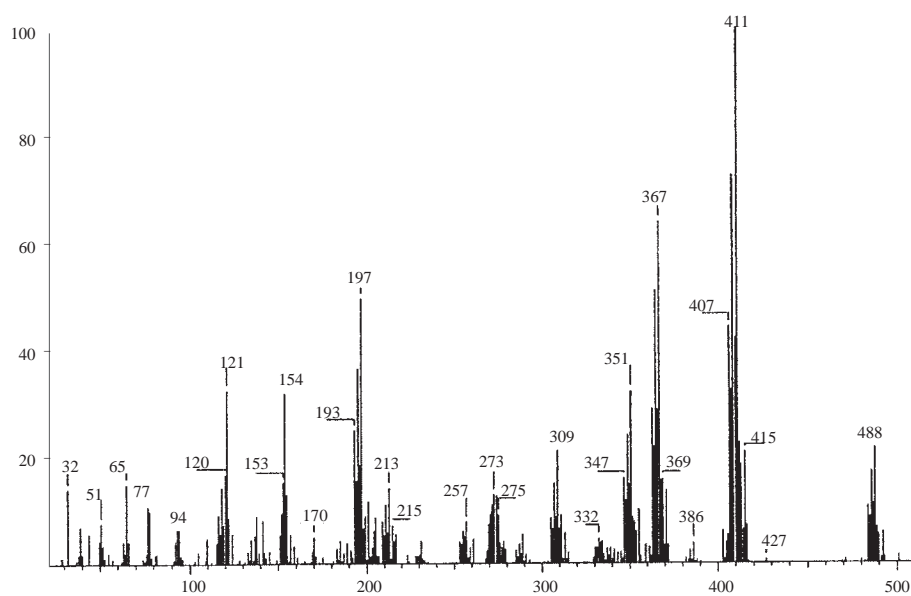
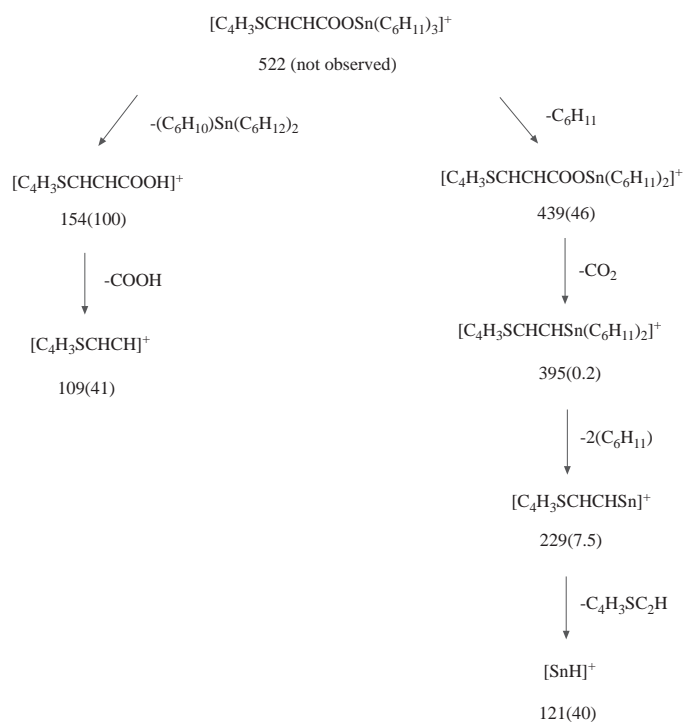


Figure 1. Mass spectrum of triphenyl (trans-3-(2-furanyl)-2-propenoate), x-axis; m/z value, y-axis; relative intensity.



Scheme 5.

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