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Review article

Review of ^{13}carbon nuclear magnetic resonance characterizations of dimethyltin carboxylates

Harpreet Kaur, Archana Thakur *

Chemistry Department, Chandigarh University, Mohali, Punjab, India

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ABSTRACT

Diorganotin carboxylates have received much interest in past decades due to their rich structural chemistry and wide range of applications in various fields. This review study provides an in-depth analysis of carbon NMR data of dimethyltin complexes. The absorptions shown by the carbonyl carbon, methyl groups attached to tin and the other carbons present in the complexes were presented in this study. The effects of nature and the number of substituents attached are also described in this report.

1. Introduction

The significant rise in research activity in the chemistry of organotin complexes in past decades is probably because organotin compounds can be studied using various physical methods. The incredible structural and stereochemical diversity ability is an essential characteristic of organotin compounds. These compounds' synthesis, properties, industrial and biological applications were studied in all respect. The ambient nature of carboxylate ligands is due to the numerous structural motifs identified in this compound family [1, 2, 3]. Many factors, including the flexibility of coordination geometries, the number of coordinations, and the manner of coordination, all played a role in the structural diversity of organotin (IV) carboxylates [4]. In industry, diorganotin dicarboxylates are used as polymerization and transesterification catalysts [5, 6]. Dimethyltin derivatives have a wide range of properties, with anti-cancer, anti-tumour, and anti-bacterial prominent [7, 8]. In the α-glucosidase enzyme assay, some organotin complexes inhibited the enzyme's activity, while the standard drug acarbose inhibited poorly [9]. A surprising finding was that the 4-(diethylamino)benzic acid-derived dimethyltin (IV) complex outperformed the dibutyltin (IV) and triorganotin (IV) derivatives in terms of anti-bacterial activity [10]. Organotin carboxylates have a wide spectrum of applications and possible uses as indicated by the reports in the literature [11]. Organotin carboxylate compounds were used for their biocidal properties and effectiveness as medicinal compounds [12, 13]. Unique structural and diverse properties of organotin carboxylates make these compounds as significant class with applications as catalyst [14], anti-cancer [15], anti-fungal [16, 17], anti-bacterial [18], insecticidal [19], and wood preservatives agents [20].

The spectroscopic and technological developments enabled researchers from various disciplines to use these techniques not only for the complex structural purposes of molecules but also for wide-ranging applications. ^{13}C Nuclear Magnetic Resonance (NMR) study has attained the status of one of the best spectroscopic methods for significant structural characterization, containing organic groups, among the currently useful spectroscopical techniques (Infrared, proton, carbon and tin NMR, Mössbauer spectroscopic studies and Mass spectrometry). In order to get a better insight of the structure chemistry of organotin carboxylates, the ^{13}C NMR spectra of several dimethyltin carboxylates were investigated, and the results were presented in this paper.

2. ^{13}C NMR data of dimethyltin carboxylates

Two dimethyltin carboxylate complexes 1(a) and 1(b) of *m*-methyl trans-cinnamic acid ligand were synthesized, and their ^{13}C spectroscopic characterization was carried out. The carbonyl carbon showed resonance at δ 176, and 173 ppm, the tin atom's methyl carbon, and the ligand's methyl group showed peaks at δ 5.2 and 9.8/6.6 ppm and δ 21.23 and 21.3 ppm respectively in different dimethyltin carboxylates [21]. A new dimethyl organotin complex **(2)** synthesized from ligand 2-(2-hydroxybenzylideneamino)benzoic acid showed carbon resonances at δ 173 ppm (COO); 147 (1, 6, 8, 9 carbons); 136, 130, 125 (2, 3, 4, 5, 10, 11, 12, 13 carbons), 115 (7 carbon), 10 (Sn–CH_3) ppm respectively [22]. The carbonyl carbon in [$Me_2Sn(O_2CC_4H_3E)_2$] complexes **3(a), 3(b)** and **3(c)** where E is oxygen or sulphur of 2-thiophene- or 2-furan-carboxylic acids showed resonance at δ 170 ppm while the ring carbons showed resonances between δ 127–134 ppm. In addition to finding the structure of

* Corresponding author.
E-mail addresses: arch9chem@gmail.com, archana.e9358@cumail.in (A. Thakur).

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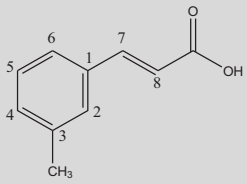
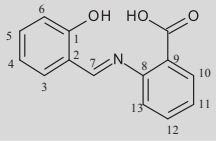
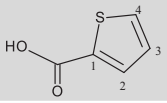
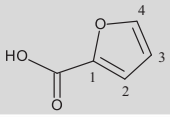
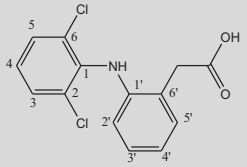
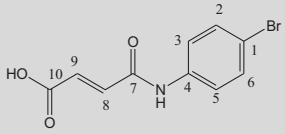
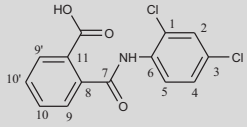
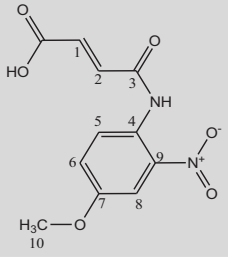
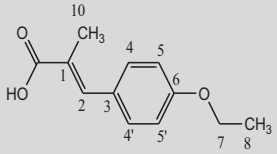
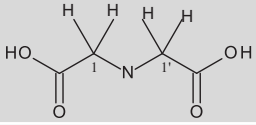
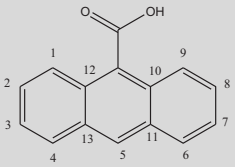
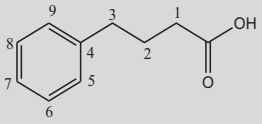
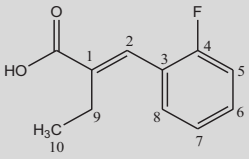
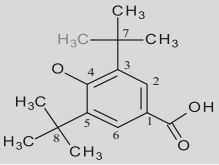
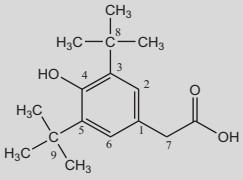
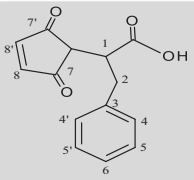
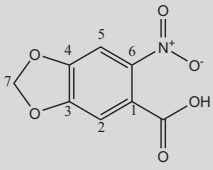
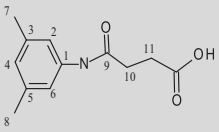
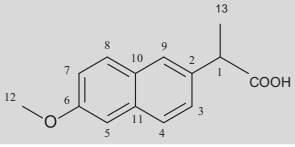
Table 1. List and overview of ligands.

Ligand no.	Ligand name	Structure	Double bond at position	Functional group/ Substituents attached		Reference
				Group	Position	
L1	*m*-Methyl trans-cinnamic acid		1, 3, 5, 7	CH_3	3	[21]
L2	2-(2-Hydroxybenzylideneamino) benzoic acid		2, 4, 6, 7, 9, 11, 13	OH	1	[22]
L3	Thiophene-2-carboxylic acid		1, 3	-	-	[23, 24]
L4	Furan-2-carboxylic acid		1, 3	-	-	
L5	(2-[2,6-Dichlorophenylamino] phenylacetic acid)		2, 4, 6, 1′, 3′, 5′	Cl	2, 6	[25]
L6	4-Bromomaleanilic acid		2, 4, 6, 8	Br; C=O	1; 7	[26]
L7	2 [(2,4-DichloroanilinoCarbonyl)] benzoic acid)		1, 3, 5, 8, 10, 9′	Cl; C=O	1, 3; 7	[27]
L8	4-((4-methoxy-2-Nitrophenyl) amino)-4-oxobut-2-enoic Acid		1, 4, 6, 8	C=O; OCH_3; NO_2	3; 7; 9	[28]
L9	3-(4-Ethoxyphenyl)-2-methylacrylic acid		1, 3, 5′, 5	CH_3; OCH_2CH_3	1; 6	[29]
L10	Iminodiacetic acid		-	-	-	[30]

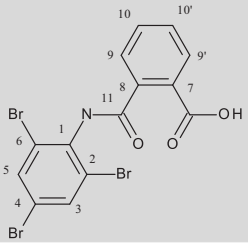
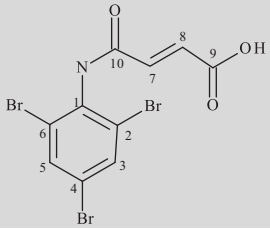
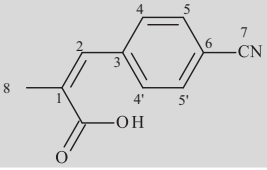
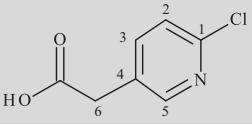
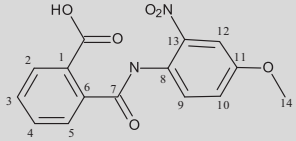
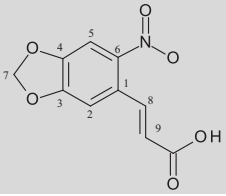
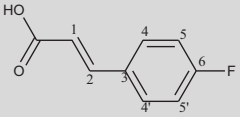
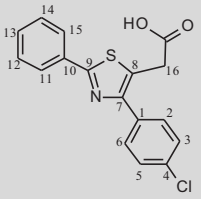
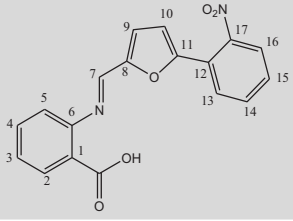
(*continued on next page*)

Table 1 (*continued*)

Ligand no.	Ligand name	Structure	Double bond at position	Functional group/ Substituents attached		Reference
				Group	Position	
L11	Di-(9- anthracene carboxylic acid)		2, 4, 5, 6, 8, 10, 12	-	-	[31]
L12	4-Phenylbutyric acid		5, 7, 9	-	-	[32]
L13	2-(2-Fluorobenzylidene) butanoic acid		1, 4, 6, 8	F; CH_2CH_3	4; 1	[33]
L14	(3,5-Di-tert-butyl-4-hydroxybenzoate)		1, 3, 5	$C(CH_3)_3$; OH	3, 5; 4	[34]
L15	(3-(3,5-Di-tert-butyl-4-hydroxyphenyl) propionate)		1, 3, 5	$C(CH_3)_3$; OH	3, 5; 4	
L16	2-(*N*-Maleoyl)-3-phenylpropanoic acid		3, 5, 5′, 8	C=O	7, 7′	[35]
L17	6-Nitropiperonylic acid		1, 3, 5	NO_2	6	[36]
L18	3-[(3′,5′-Dimethylphenylamido)] propanoic acid		1, 3, 5	C=O; CH_3	9; 3, 5	[37]
L19	(*S*)-(+)-6-Methoxy-alpha-methyl-2-naphthaleneacetic acid		3, 11, 6, 8, 9	CH_3; OCH_3	1; 6	[38]

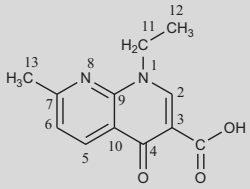
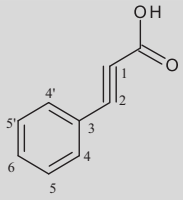
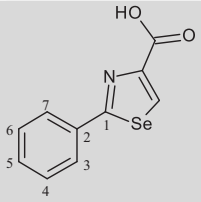
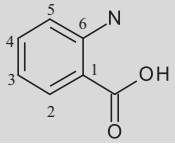
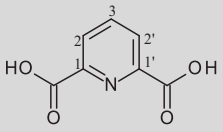
(*continued on next page*)

Table 1 (*continued*)

Ligand no.	Ligand name	Structure	Double bond at position	Functional group/ Substituents attached		Reference
				Group	Position	
L20	2-[(2′,4′,6′-Tribromophenylamido)] benzoic Acid		1, 3, 5, 9, 10′, 11	C=O; Br	7; 2, 4, 6	[39]
L21	3-[(2′,4′,6′-Tribromophenylamido)] propanoic Acid		1, 3, 5, 8	C=O; Br	7; 2, 4, 6	
L22	3-(4-Cyanophenyl)-2-methylacrylic acid		1, 3, 5′, 5	CH_3; CN	1; 7	[40]
L23	6-Chloro-3-pyridineacetic acid		1, 3, 5	Cl	1	[41]
L24	Bis 2-(4-Methoxy-2-nitrophenylcarbamoyl) benzoic acid		2, 4, 6, 8, 10, 12	C=O; NO_2; OCH_3	7; 13; 11	[42]
L25	3,4-Methylenedioxy 6-nitrophenylpropenoic Acid		1, 3, 5, 8	NO_2	6	[43]
L26	3-(4-Fluorophenyl) acrylic acid		1, 3, 5′, 5	F	6	[44]
L27	4-*p* (Chlorophenyl)-2-phenyl-5-thiazoleacetic acid		2, 4, 6, 7, 9, 10, 12, 14	Cl	4	[45]
L28	2-{[5-(2-Nitrophenyl) furan-2-yl] methyleneamino} benzoic acid		1, 3, 5, 7, 8, 10, 12, 14, 16	NO_2	17	[46]

(*continued on next page*)

Table 1 (*continued*)

Ligand no.	Ligand name	Structure	Double bond at position	Functional group/ Substituents attached		Reference
				Group	Position	
L29	1-Ethyl-7-methyl-4-oxo-1,4-dihydro-1,8-naphthyridine-3-carboxylic acid		2, 10, 6, 8	CH_2CH_3; C=O; CH_3	1; 4; 7	[47]
L30	Phenyl acetylene carboxylic acid		4, 6, 4' (C=C) 1 (C≡C)	-	-	[48]
L31	2-Phenyl-4-selenazole carboxylic acid		3, 5, 7	-	-	[49]
L32	2-Aminobenzoic acid		1, 3, 5	NH_2	6	[50]
L33	2,6-Pyridinedicarboxylic acid		1, 2, 2′	-	-	[51]

methyltin (IV) compounds in solution, spectroscopic proof from the coupling constants of the methyl carbons and the tin hydrogen of $[Me_2Sn(O_2CR)]_2O_2$ (where R = Thiophene and furan) revealed that there was no interaction between tin and sulphur or oxygen atoms in the compound [23]. Based on spectroscopic evidence of $\{[Me_2Sn(O_2CR)]_2O\}_2$ (R = Thiophene and furan), the dimeric structure of dicarboxylato tetraorganodistannoxanes with R_2Sn atoms upfield (for exocyclic) and downfield (for endocyclic) resonance. Deshielding of the respective free acids in the heterocyclic ring was shown [24]. A low-intensity peak at δ 178.5 ppm in the ^{13}C NMR of dimethyltin (2-[2, 6-dichlorophenylamino]phenylacetate) complex (4) demonstrated that the carboxylate ion was not in a free state. The shift in the exocyclic methylene carbon and carbon 6 field values towards lower field values revealed the bonding of the metal through the carboxylic group in the methylene carbon [25].

^{13}C NMR results for $Me_2Sn(4\text{-bromomaleanalate})_2$ (5), showed resonances caused by the carboxylate carbon after coordination has been shifted. During the process of complexation, the other carbons in the molecule did not move considerably. With the ^1J value for the molecule, the projected C–Sn–C angle was 109.50°, indicating that the complex was in a tetragonal environment around the tin nucleus [26]. Table 1 gives the list and overview of the ligands. Shahzadi and co-workers reported carbon NMR of dimethyltin-2-[(2,4-dichloroanilino carbonyl)]benzoate complexes **(6).** The position of the carboxylate group in tin bonding was corroborated by the resonance attributed to C, which indicated a shift in the position of the carboxylate group following coordination. The carbon atoms of the methyl groups that are connected to the tin atom are discovered to be in the same location [27]. In dimethyltin complexes **7(a)** and **7(b)** derived from 4-((4-methoxy-2-nitrophenyl)amino)-4--oxobut-2-enoic acid ligand reported in a study. The carboxylate moiety resonances showed a noticeable shift to downfield concerning the Sn due to the electronegative Sn atom withdrawing electron density away from the ligand. In the Lockhart Equation, the C–Sn–C bond angle value for methyltin compounds calculated from the ^2J (^{119}Sn ^1H) coupling was 117° [28]. Figure 1 explains the structures of complexes number 1–10. The ^{13}C NMR of complexes **(8)** of formula $[Me_2SnL_2]$ [where L = 3-(4-Ethoxyphenyl)-2-methylacrylate], showed ^1J [^{119}Sn, ^{13}C], 703 Hz (146°) corresponding to 5-coordinated tin atoms in solutions comparable with literature results. The data was further supported five coordinated structure of the tin atom in solutions by C–Sn–C angles values using Lockhart Equation. The pattern of complexity ranging from six solid-state coordination to five solution coordination attributed to fluxion behaviour [29]. In a methanol solvent, dimethyltin dicarboxylates (9) were made by heating disodium iminodiacetate hydrate using dimethyltin dichloride. The carbon NMR of the complex showed resonances at δ 179 ppm due to carbonyl carbon and CH_2 group attached to carbonyl carbon at δ 51.9 ppm [30]. The dimethyltin complexes **(10)** Me_2Sn $[OC(O)C_{14}H_9]_2$ CH_3OH derived from the ligand anthracenecarboxylic acid, was reported to be characterized by elemental analysis, mass spectrometry, multinuclear nuclear magnetic resonance studies. The ^{13}C NMR resonances due to different Carbons appeared at δ 5.4 ppm (CH_3, ^1J (^{119}Sn–^{13}C), 50.3 (CH_3OH), 124.8, 124.9, 126.1, 126.7, 128.1, 128.4, 129.6, 130.5 ($C_{14}H_9$), and 178.4 (COO) ppm [31]. Table 2 depicts ^{13}C NMR data of complexes number 1–10.

The reactions of Me_2SnCl_2 with $NaO_2C(CH_2)_3Ph$, yielded carboxylates **(11)** of formula $[\{(Me_2SnOC(CH_2CH_2CH_2)_2O\}_2]$. The different carbon NMR peaks observed at δ 179.9, 35.7, 27.4, 34.4, 141.8–126.3, and

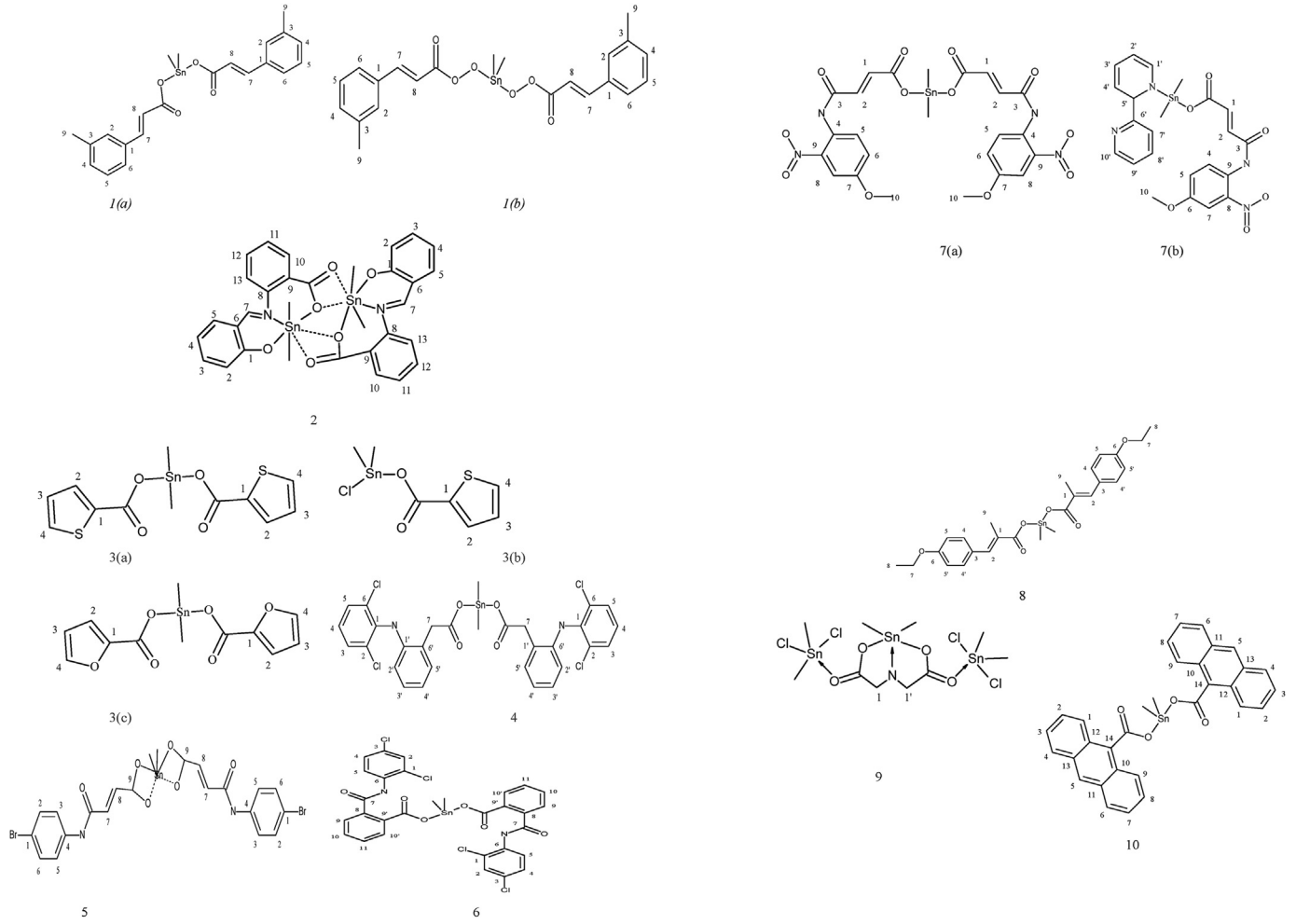


Figure 1. Structure of complex 1–10: (a) Dimethyl complexes 1 (a) and 1(b) with m-methyl trans-cinnamic acid ligand; 2 with 2-(2-hydroxybenzylideneamino)benzoic acid ligand; 3(a), 3(b) with thiophene-2-carboxylic acid and 3(c) with furan-2-carboxylic acid ligand; 4 complex (2-[2,6-dichlorophenylamino]phenylacetic acid) ligand; 5 with 4-bromomaleanilic acid ligand; 6 dimethyltin-2-[(2,4-dichloroanilinocarbonyl)]benzoate complex; 7(a) and 7(b) with 4-((4-methoxy-2-nitrophenyl)amino)-4-oxobut-2-enoic acid ligand; 8 with 3-(4-ethoxyphenyl)-2-methylacrylate ligand; 9 with iminodiacetate hydrate ligand; 10 with anthracene carboxylic acid ligand.

Table 2. ^{13}C NMR data of complexes number 1–10.

Complex	Complex formula	δ (^{13}C) (ppm)				*J* values (Hz)	C–Sn–C angle (°)	Reference
		Carbons attached to Tin (Sn)		Carbons of Ligand				
		Position	Value	Position	Value			
1 (a)	$[Me_2Sn(L^1)_2]$	CH_3	5.20	COO	176.0	^1J [633.00]	132.28	[21]
				1	134.3			
				2	131.5			
				3	138.5			
				4	128.8			
				5	128.5			
				6	125.4			
				7	117.1			
				8	147.0			
				9	21.23			
1 (b)	$\{[(Me_2SnL^1)_2O]_2\}$	CH_3	9.80, 6.60	COO	173.0	^1J [815.00], [756.00]	148.25	
				1	134.0			
				2	130.0			
				3	138.0			
				4	128.69			
				5	128.67			
				6	125.16			
				7	121.19			
				8	144.0			
				9	21.30			

(*continued on next page*)

Table 2 (*continued*)

Complex	Complex formula	δ (^{13}C) (ppm)				*J* values (Hz)	C–Sn–C angle (°)	Reference
		Carbons attached to Tin (Sn)		Carbons of Ligand				
		Position	Value	Position	Value			
2	$[Me_2Sn(L^2)_2]$	CH_3	10	COO	173	^1J [960]	-	[22]
				1, 6, 8, 9	147			
				2, 3, 4, 5, 10, 11, 12, 13	136, 130, 125			
				7	115			
3 (a)	$[Me_2Sn(L^3)_2]$	CH_3	3.5	COO	170.7	^1J [630]	132	[23, 24]
				1	133.7			
				2	133.4			
				3	128.0			
				4	134.6			
3 (b)	$[Me_2SnCl(L^3)]$	CH_3	3.5	COO	169.5	-	-	
				1	132.6			
				2	133.6			
				3	127.8			
				4	134.8			
3 (c)	$[Me_2Sn(L^4)_2]$	CH_3	5.1	COO	165.8	-	-	
				1	144.7			
				2	118.9			
				3	111.8			
				4	146.4			
4	$[Me_2Sn(L^5)_2]$	CH_3	9.5, 6.6	COO	178.5	-	145, 141	[25]
				1	137.9			
				2,6	129.7			
				3,5	128.8			
				4	123.9			
				5′	130.6			
				6′	125.4			
				7′	41.2			
5	$[Me_2Sn(L^6)_2]$	CH_3	4.5	COO	169.0	-	-	[26]
				1	134.2			
				2,6	132.2			
				3,5	132.4			
				4	127.3			
				7	172.8			
				8	121.8			
				9	115.4			
6	$[Me_2Sn(L^7)_2]$	CH_3	14.12	COO	175.7	-	-	[27]
				1	136.7			
				2,4	128.5			
				3,5	134.0			
				6	130.3			
				7	166.3			
				8,11	131.3			
				9,9′	124.0			
				10,10′	114.1			
7	$[Me_2Sn(L^8)_2]$	CH_3	-0.7	COO	164.2	^1J [833]	-	[28]
				1	131.4			
				2	133.8			
				3	170.2			
				4	125.2			
				5	126.8			
				6	121.1			
				7	156.3			
				8	109.5			
				9	142.7			
				10	56.6			

(*continued on next page*)

Table 2 (*continued*)

Complex	Complex formula	δ (^{13}C) (ppm)				J values (Hz)	C–Sn–C angle (°)	Reference
		Carbons attached to Tin (Sn)		Carbons of Ligand				
		Position	Value	Position	Value			
8	$[Me_2Sn(L^9)_2]$	CH_3	4.5	COO	179.1	^2J [703]	146.0	[29]
				1	127.6			
				2	138.3			
				3	131.5			
				4,4′	130.9			
				5,5′	114.3			
				6	159.2			
				7	63.4			
				8	14.8			
				9	14.4			
9	$\{Me_2SnL^{10}\ [Sn(Cl)_2Me_2]_2\}$	CH_3	23.5	COO	171.1	-	152.8, 134.3	[30]
				1,1′	51.9			
10	$[Me_2Sn(L^{11})_2]$	CH_3	5.4	COO	178.4	^1J [642, 625]	133	[31]
				1–13	124.8–130.5			
				14	50.3			

9.1 ppm [32]. The bis [-2-(2-fluorobenzylidene)butanoato] tetramethyldistannoxane complex **(12)** showed eleven peaks at δ 177.5, 130.2, 136.5, 123.7, 160.4, 115.7, 130.3, 130.02, 128.6, 21.5, 13.8 ppm due to carbons numbering from C1 to C-11 and at δ 4.8 ppm due to C–Sn [33]. The organotin (IV) carboxylates **13(a)** and **13(b)** based on 3, 5-di-tert-butyl-4-hydroxyphenyl of formulae $(RCOO)_2SnMe_2$ showed peaks at δ 4.31 $[Sn(CH_3)_2]$, 30.3 $[C(CH_3)_3]$, 31.42 $[ArCH_2CH_2COO]$, 34.31 $[C(CH_3)]$, 36.21 $[ArCH_2\text{-}CH_2CO_2]$, 124.88; 131.02; 135.87; 152.17 ppm due to Ar group and at δ 183.52 ppm due to carbonyl carbon [34]. The ^{13}C NMR spectroscopy data of dimethyltin complexes **(14)**

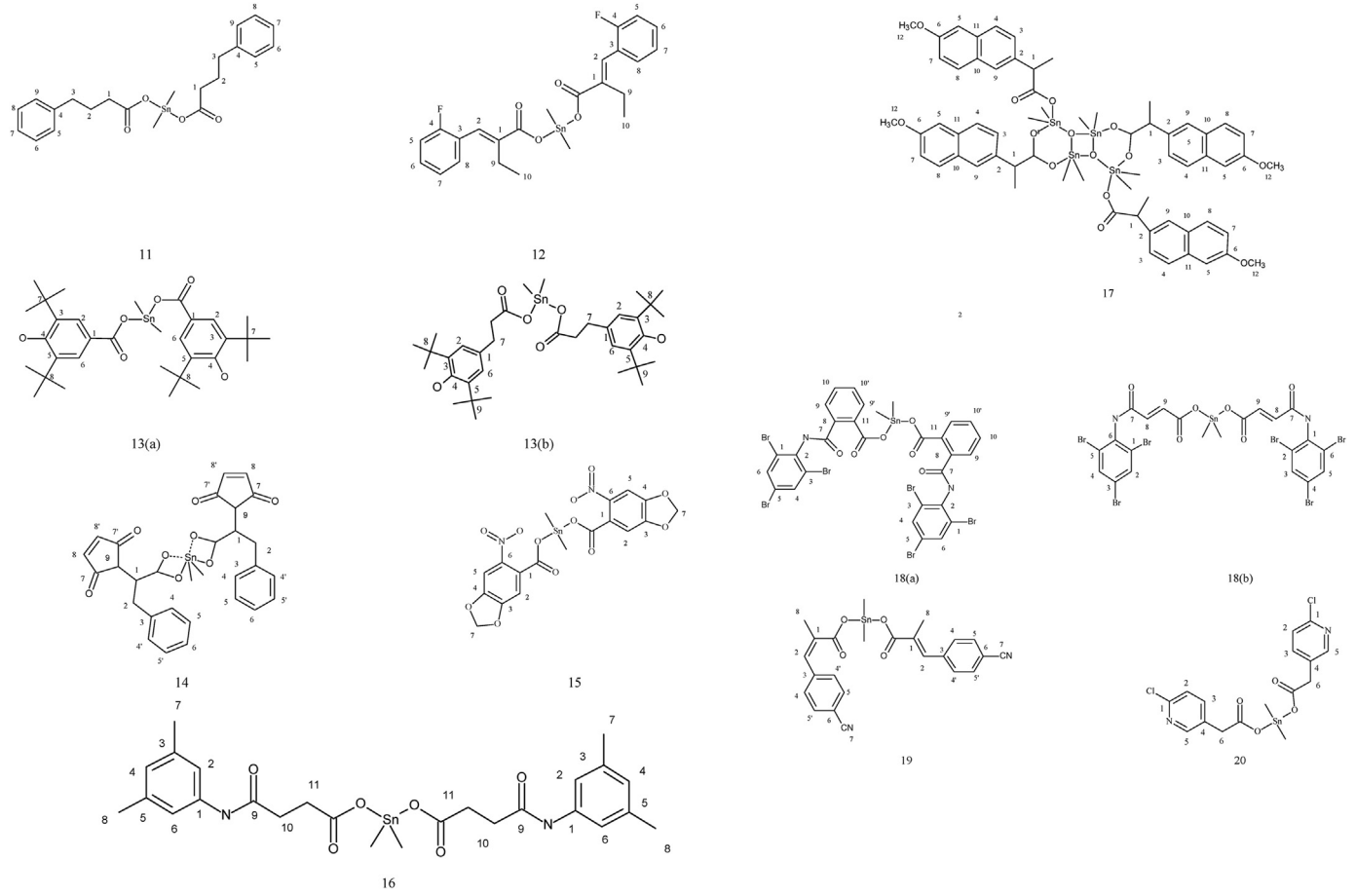


Figure 2. Structure of complex 11–20: dimethyl complexes; **11** with 4-phenylbutyric acid; **12** bis(-2-(2-fluorobenzylidene)butanoato) tetramethyldistannoxane complex; **13(a)** with (3,5-di-tert-butyl-4-hydroxybenzoate) and **13 (b)** with (3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate) ligand; **14** with 2-(*N*-maleoyl)-3-phenylpropanoic acid ligand; **15** with 6-nitropiperonylic acid ligand; **16** with 3-[(3′,5′-dimethylphenylamido)]propanoic acid ligand; **17** with (*S*)-(+)-6-methoxy-alpha-methyl-2-naphthaleneacetic acid ligand; **18(a)** with 2-[(2′,4′,6′-tribromophenylamido)]benzoic acid and **18(b)** with 3-[(2′,4′,6′-tribromophenylamido)]propenoic acid ligand; **19** with 3-(4-cyanophenyl)-2-methylacrylic acid ligand; **20** with 6-chloro-3-pyridineacetic acid ligand.

Table 3. ^{13}C NMR data of complexes number 11–20.

Complex	Compound	δ (^{13}C) (ppm)				*J* values (Hz)	C–Sn–C angle (°)	Reference
		Carbons attached to Tin (Sn)		Carbons of Ligand				
		Position	Value	Position	Value			
11	$[(Me_2Sn(L^{12})_2]$	CH_3	9.1 6.6	COO	179.9	^1J [802/767] ^1J [758/722]	147.1, 143.2	[32]
				1	35.7			
				2	27.4			
				3	34.4			
				4–9	141.8–126.3			
12	$\{[Me_2SnL^{13}]_2O\}_2$	CH_3	4.8	COO	177.5	^1J [14.3] ^1J [248.2] ^1J [21.8] ^1J [8.2] ^1J [3.7] ^1J [3.0]	-	[33]
				1	130.2			
				2	136.5			
				3	123.7			
				4	160.4			
				5	115.7			
				6	130.3			
				7	130.02			
				8	128.6			
				9	21.5			
				10	13.8			
13 (a)	$[Me_2Sn(L^{14})_2]$	CH_3	4.95	COO	176.42	-	-	[34]
				1–6	120.53, 128.10, 135.75, 158.70,			
				8	30.20			
				9	34.37			
13 (b)	$[Me_2Sn(L^{15})_2]$	CH_3	4.31	COO	183.52	-	-	
				1–7	124.88, 131.02, 135.87, 152.17			
				8	31.42			
				9	36.21			
14	$[Me_2Sn(L^{16})_2]$	CH_3	7.4	COO	170.1	-	131.3	[35]
				2	54.3			
				3	34.9			
				4	137.6			
				5,5′	128.7			
				6,6′	133.7			
				7	126.6			
				8	175.2			
				9,9′	128.5			
15	$[Me_2Sn(L^{17})_2]$	CH_3	4.6	COO	174.3	-	-	[36]
				1	129.0			
				2	108.5			
				3	151.3			
				4	149.8			
				5	105.0			
				6	143.2			
				7	103.6			
16	$[Me_2Sn(L^{18})_2]$	CH_3	29.6	COO	177.4	349–396	359.3	[37]
				1	138.03			
				2/6	117.08			
				3/5	139.67			
				4	124.84			
				7/8	21.85			
				9	170.69			
				10	29.26			
				11	31.38			
17	$[Me_2Sn(L^{19})_2]$	CH_3	18.90	COO	180.85	-	-	[38]
				2–11	157.54–105.56			
				6	55.25			
				12	47.43			

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Table 3 (*continued*)

Complex	Compound	δ (^{13}C) (ppm)				*J* values (Hz)	C–Sn–C angle (°)	Reference
		Carbons attached to Tin (Sn)		Carbons of Ligand				
		Position	Value	Position	Value			
18 (a)	$[Me_2Sn(L^{20})_2]$	CH_3	29.6	COO	174.8	-	-	[39]
				1	136.7			
				2/4/6	128.6			
				3/5	134.5			
				7	166.7			
				8	131.0			
				9, 9′	124.1			
				10,10′	115.7			
				11	130.2			
18 (b)	$[Me_2Sn(L^{21})_2]$	CH_3	29.6	COO	175.7	-	-	
				1	135.0			
				2/4/6	129.5			
				3/5	133.7			
				7	164.2			
				8	124.6			
				9	114.4			
19	$[Me_2Sn(L^{22})_2]$	CH_3	4.6	COO	177.6	^1J [704/670]	138.5	[40]
				1	125.3			
				2	140.3			
				3	138.4			
				4	28.2			
				5	132.2			
				6	111.8			
				7	118.5			
				8	14.4			
20	$[Me_2Sn(L^{23})_2]$	CH_3	14.1	COO	173.3	-	-	[41]
				1–5	124.1–150.2			
				6	37.7			

derived from 2-(*N*-maleoyl)-3-phenylpropanoic acid exhibited a number of signals representing magnetically different carbon atoms. The involvement of two C=O entities in the ligand produced two distinct, well-separated, regions ranging from δ 170–175 ppm. At δ 7.4 ppm, the moieties connected to the tin atom produced a signal [35]. The ^{13}C NMR data of dimethytin compounds **(15)** derived from 6-nitropiperonylate, in $CDCl_3$ solvent, showed resonances at δ 129.0, 108.5, 151.3, 149.8, 105.0, 143.2, 103.6 and 174.3 ppm attributed to carbons numbering from C1–C8 carbons. The methyl group attached to tin showed signal at δ 4.6 ppm [36]. Figure 2 depicts the structures of complex number 11–20. Dimethyltin complexes **(16)** developed from a new ligand synthesized by treating succinic anhydride and 3,5-dimethylaniline described in a report. The decrease in the ^{13}C NMR shift upon coordination, in agreement with the results from the carboxylic carbon resonance, demonstrates that the carboxylate group plays a more significant role in the bonding to Sn. In the experimental findings, concerning reports in the literature, the carbon of alkyl groups bonded to tin is found in almost the same position [37]. A complex of formula $\{[Me_2Sn(L)]_2O\}_2$ **(17)** reported in a study where ligand L was (*S*)-(+)-6-methoxy-alpha-methyl-2-naphthaleneaceto anion. A significant downfield shift in the ^{13}C NMR spectrum, including all carbon resonances, was observed in comparison to the ligands, which was attributed to the outflow of electron density from the ligand to the metal atoms. At δ 174.8 ppm, the resonances are attributed to the carbonyl carbon [38]. In dimethyltin complexes 18(a) and 18(b) showed values of δ 163.0–166.8 and 170–176.8 ppm due to the –COO and –C=O group, respectively. Aromatic carbon resonances showed signals in the region reported in the literature [39]. The ^{13}C NMR data of the complex **(19)** showed peaks of δ 177.6, 125.3, 140.3, 138.4, 128.2, 132.2, 111.8, 118.5, 14.4 ppm due to C1–C9 carbons and δ 4.6 ppm due to the methyl carbon attached to the tin [40]. The carbon NMR spectra of organotin complex **(20)** synthesized using 6-chloro-3-pyridineacetic acid ligand and dimethyltin dichloride showed signals at δ 173.3 ppm due to COO group, 124.1–150.2 ppm due to C_6H_5–C group, 37.7 ppm due to C_6H_5–CH_2 group and δ 14.1 ppm due to CH_3 carbon [41]. Table 3 presents ^{13}C NMR data of complexes number 11–20.

The dimethyltin complexes **(21)** derived from 2-(4-methoxy-2-nitrophenylcarbamoyl)benzoic acid ligand exhibited fifteen signals at δ 160.3, 129.6, 127.7, 132.9, 135.7, 124.4, 131.9, 167.0, 121.3, 128.7, 120.9, 156.6, 109.5, 142.5, and 56.0 ppm [C1–C15 carbons]. The Sn–CH_3 carbon showed a signal at δ 0.6 ppm ^1J [^{119}Sn–^{13}C], 800 Hz [42]. The complex of formula $[Me_2SnL_2]$ **(22)**, using 3,4-methylenedioxy-6-nitrophenylpropenoic acid ligand showed resonances due to carboxyl carbon moved to a lower region, indicating the coordination with the carboxyl group [43]. The dimethyltin complexes **(23)** derived from ligand 3-(4-fluorophenyl)acrylic acid ligand showed that C1 of the complexes were shifted downfield due to the drainage of electron density [44]. The NMR spectra of dimethyltin complexes **(24)** of 4-*p*-(chlorophenyl)-2-phenyl-5-thiazoleacetic acid showed signals due to carbonyl Carbon at δ 180 ppm [45]. A new dimethyltin complex **(25)** was synthesized general formula $[Me_2Sn(OBz)_2]$ where OBz represents ligand named 2-{[5-(2-nitrophenyl)furan-2-yl]methyleneamino}benzoic acid and its spectroscopic data were analyzed. Coupling constants ^nJ (^{119}Sn–^{13}C) helps in the structural elucidation of the new complexes. Figure 3 depicts the structures of complex number 21–31.

^{13}C NMR spectra of ligand (NaOBz) showed its shift as (δ ppm): 173.24 (COO); 112.61–178.71 (others) and of the complex as 175.30 (COO); 111.28–178.14 (others) respectively [46]. A new complex **(26)** derived from the ligand (1-ethyl-7-methyl-4-oxo-1,4-dihydro-1,8-naphthyridine-3-carboxylic acid). The spectra showed no appreciable change

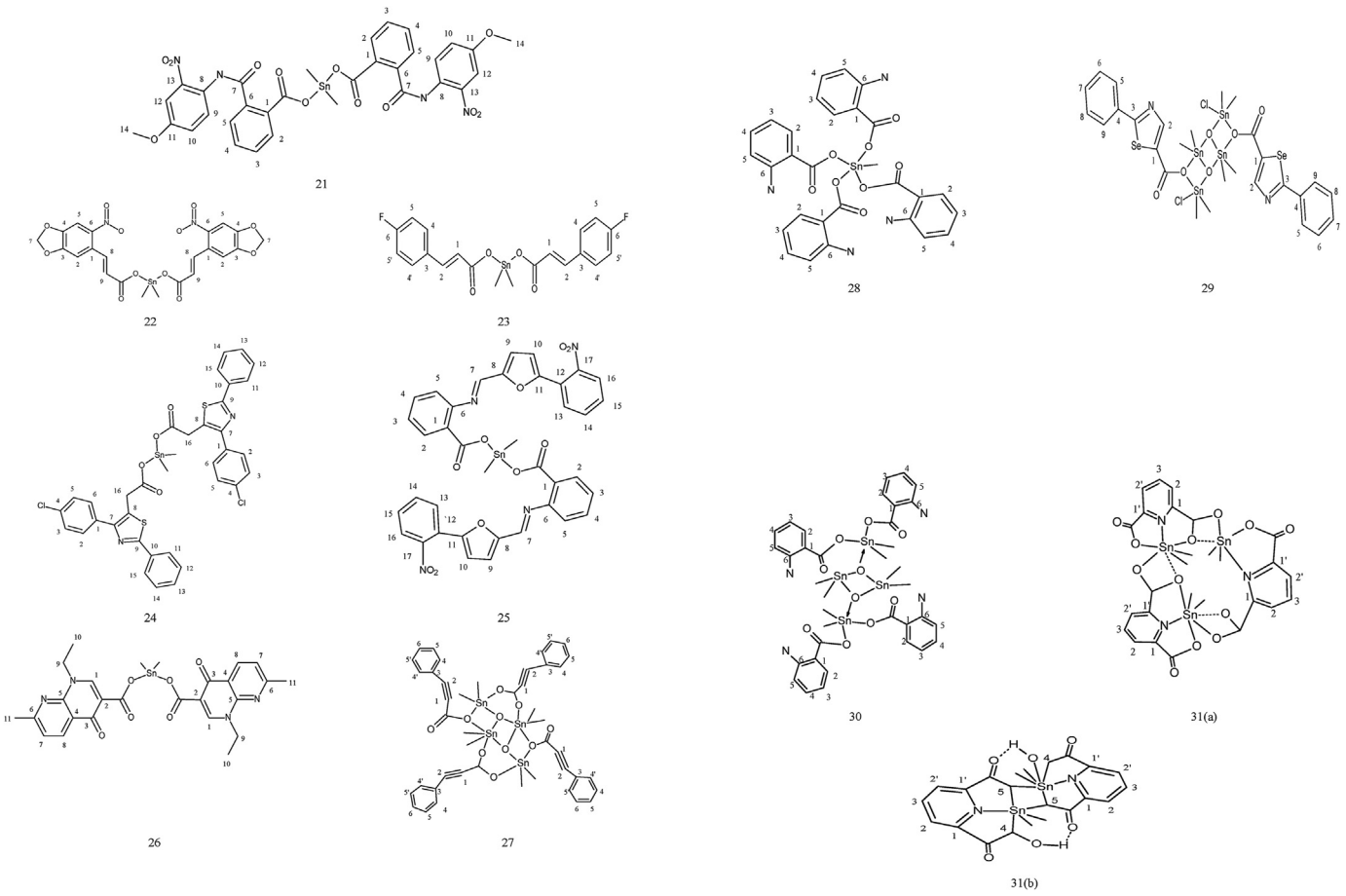

Figure 3. Structure of complex 21–31: dimethyl complexes; **21** with bis 2-(4-methoxy-2-nitrophenylcarbamoyl)benzoic acid; **22** 3,4-methylenedioxy 6-nitrophenylpropenoic acid ligand; **23** with 3-(4-fluorophenyl)acrylic acid and **24** with 4-(*p*-chlorophenyl)-2-phenyl-5-thiazoleacetic acid ligand; **25** with 2-{[5-(2-nitrophenyl)furan-2-yl]methyleneamino}benzoic acid ligand; **26** with (1-ethyl-7-methyl-4-oxo-1,4-dihydro-1,8-naphthyridine-3-carboxylic acid) ligand; **27** with phenyl acetylene carboxylic acid acid; **28** with o-(p-dimethylaminobenzali- dine)benzoic acid ligand; **29** with 2-phenyl-4-selenazole carboxylic acid ligand; 30 with 2-aminobenzoic acid ligand; **31(a)** and **31(b)** with 2,6-pyridinedicarboxylic acid ligand.

in the carbon resonances except the carbonyl carbon [47]. The $[Me_2Sn(pac)]_4$ complex **(27)** derived from the ligand phenyl acetylene carboxylic acid showed resonance values at δ 158.15 (COO); 83.20, 84.23 (C≡C); 120.22–132.95 (aromatic ring); 5.79, 10.14 ppm (methyl carbons attached directly to tin) [48]. The spectroscopic data of dimethyltin complex **(28)** derived from the ligand *o*-(*p*-dimethylaminobenzali- dine)benzoic acid exhibited signals at δ 175.0 (COO); 31.2 (NCH3); 110.7–150.3 (other carbons); 5.5 ppm (Sn–CH_3) [49]. Another

Table 4. ^{13}C NMR data of complex number 21–31.

Complex	Compound	δ (^{13}C) (ppm)				J values (Hz)	C–Sn–C angle (°)	Reference
		Carbons attached to Tin (Sn)		Carbons of Ligand				
		Position	Value	Position	Value			
21	$[Me_2Sn(L^{24})_2]$	CH_3	0.6	COO	160.3	^1J [800]	147	[42]
				1	129.6			
				2	127.7			
				3	132.9			
				4	135.7			
				5	124.4			
				6	131.9			
				7	167.0			
				8	121.3			
				9	128.7			
				10	120.9			
				11	156.6			
				12	109.5			
				13	142.5			
				14	56.0			

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Table 4 (*continued*)

Complex	Compound	δ (^{13}C) (ppm)				J values (Hz)	C–Sn–C angle (°)	Reference
		Carbons attached to Tin (Sn)		Carbons of Ligand				
		Position	Value	Position	Value			
22	$[Me_2Sn(L^{25})_2]$	CH_3	4.34	COO	187.87	-	-	[43]
				1	126.36			
				2	105.93			
				3	153.10			
				4	150.18			
				5	104.86			
				6	141.35			
				7	104.30			
				8	141.25			
				9	123.10			
23	$[Me_2Sn(L^{26})_2]$	CH_3	4.6	COO	171.3	^1J [703]	138.4	[44]
				1	118.3			
				2	145.4			
				3	131.2			
				4,4′	129.5			
				5,5′	116.1, 115.8			
				6	165.8, 162.4			
					129.5			
24	$[Me_2Sn(L^{27})_2]$	CH_3	5.6	COO	170.0	^1J [630]	-	[45]
				1	136.8			
				2,6	112.2			
				3,5	135.8			
				4	131.6			
				7	128.8			
				8	133.5			
				9	148.0			
				10	137.9			
				11,15	134.9			
				12,14	134.6			
				13	128.1			
				16	21.1			
25	$[Me_2Sn(L^{28})_2]$	CH_3	14.95	COO	175.30	-	-	[46]
				1	121.79			
				2	131.77			
				3	112.22			
				4	132.68			
				5	116.05			
				6	150.97			
				7	178.14			
				8	152.61			
				9	111.28			
				10	132.92			
				11	153.18			
				12	114.42			
				13	130.21			
				14	131.05			
				15	133.24			
				16	124.34			
				17	147.34			
26	$[Me_2Sn(L^{29})_2]$	CH_3	1.5	COO1234567891011	178.0	-	-	[47]
				1	148.2			
				2	122.3			
				3	166.6			
				4	109.7			
				5	120.3			
				6	148.6			
				7	164.8			
				8	136.5			
				9	47.4			
				10	15.2			
				11	25.3			

(*continued on next page*)

Table 4 (*continued*)

Complex	Compound	δ (^{13}C) (ppm)				J values (Hz)	C–Sn–C angle (°)	Reference
		Carbons attached to Tin (Sn)		Carbons of Ligand				
		Position	Value	Position	Value			
27	$[Me_2Sn(L^{30})]_4$	CH_3	5.79, 10.14	COO	158.15	-	-	[48]
				1	83.20			
				2	84.23			
				3	132.95			
				4,4′	130.20			
				5,5′	120.22			
				6	128.54			
29	$[(Me_2Sn)_4 (L^{31})_2(Cl)_2]$	CH_3	-	COO	176.21	-	-	[49]
				1	169.13			
				2–7	127.93–151.37			
30	$[Me_2Sn(L^{32})_2]$	CH3	7.3	COO	175.1	-	-	[50]
				1–6	150.4–113.1			
31 (a)	$[Me_2Sn(L^{33})_2]$	CH_3	10.1	COO	168.8	-	159.6–161.6	[51]
				1,1′	141.3			
				2,2′	126.2			
				3	147.2			
31 (b)		CH_3	10.2	COO	166.8	-		
				1,1′	141.3			
				2,2′	126.2			
				3	147.2			

new dimethyltin complex **(29)** of 2-phenyl-4-selenazole carboxylic acid ligand showed two signals due to endocyclic/exocyclic tin atoms at δ 143 and 190 ppm, respectively, indicating dimer in solution [50]. Bis(μ_3-oxo) bis (μ-O-aminobenzoato-O,O′)bis (O-aminobenzoato)tetrakis [di-methyltin (IV)] complex **(30)** showed the resonance signals at δ 175.1 (COO); 150.4–113.1 (aromatic carbons); 7.3 ppm (Sn–CH_3) [51]. The spectroscopic data of two dimethyltin complexes **31(a)** and **31(b)** using ligand 2,6-pyridinedicarboxylic acid showed that the resonances due to carboxylate groups remained at almost the same resonance as that of the ligand. The methyl carbons attached directly to the tin also showed a point of difference between the two complexes [52]. Table 4 represents data related to ^{13}C NMR data of complex number 21–31.

3. Conclusion

The chemical shift of ^{13}C nucleus is influenced by the electronic environment of the complex. The carbonyl carbon in these complexes showed resonances from δ 158–187 ppm. Some of the complexes showing absorption in higher ppm value were having electron donating groups and benzene rings in the structure. The complexes showing absorption in low ppm region were having electron withdrawing groups. As far as methyl groups attached with tin are concerned also showed absorption in different ppm values ranging from δ 0.6–29.6 ppm. The nitro groups containing complexes showed resonances in δ 0.6–1.5 ppm region. While the complexes contacting chloro, bromo and oxygen atoms showed absorption in δ 14–29.6 ppm region. The C–Sn–C bond angle ranges from 132-159° in different dimethyltin complexes. The higher angle was seen in case of nitrogen containing complexes. This spectroscopic technique has contributed a lot in explaining the structural chemistry and in future also will play an important role in this area.

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