J. Chem. Sci. (2019) 131:12
https://doi.org/10.1007/s12039-018-1586-1

REGULAR ARTICLE

New organotin(IV) chlorides derived from *N*-(2-hydroxyphenyl)aryloxy sulfamates. Synthesis, characterization and DSC investigation

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MS received 4 September 2018; revised 17 December 2018; accepted 17 December 2018

Abstract. Three monomeric pentacoordinate organotin complexes were prepared by the reaction of dimethyltin dichloride with three *N*-(2-hydroxy)phenyl substituted aryloxy sulfamates in alkaline medium. The newly synthesized compounds were characterized on the basis of their infrared, CPMAS NMR, powder XRD diffraction and elemental analysis. The XRD powder analyses revealed a tetragonal system for two complexes, whereas the third one was crystallized in the hexagonal system. The thermal decomposition behavior of the synthesized complexes have been investigated and all the organotin compounds have a similar order of thermal stability.

Keywords. Penta-coordinate complex; organotin complexes; sulfamates; powder XRD; thermal stability .

1. Introduction

Sulfamates are found to be suitable intermediates for the design and development of various types of new molecules as therapeutic agents and for biological or pharmaceutical uses. Sulfamate derivatives have been identified as bioactive molecules which occupy a prominent place in medicinal chemistry because of their wide range therapeutic properties, such as anti-cancer,[1–5] antibacterial,[6–10] antioxidant,[11,12] anti-HIV,[13–15] anticonvulsant,[16–19] antibiotic,[13,20,21] anti-obesity,[22] diuretic,[23,24] hypoglycemic,[25] antithyroid,[26] and anti-neuropathic pain[27] activities. In addition, the electronic structure of $-SO_2NH_2$ group in sulfamates and related ligands was considered as moiety par excellence for designing potent carbonic anhydrases inhibitors (CAIs) because of its good chelating to the metal ion of the enzyme.[28–32] Thus, complexation reaction of sulfonyl ($-SO_2NH_2$) and *N*-sulfamoyl ($-SO_2NH-CO$) groups to transition metals has been rarely investigated especially for the purpose of developing new organometallic sulfamate derivatives and exploring their presumed biological activities.[33–35]

On the other hand, organotin complexes have been found to be encouraging agents for stimulating anticancer activity, many of them are as effective or even better than traditional drugs.[36–39] In our previous work, we have reported the synthesis of some new Sn(IV) complexes with 2-alkylaminobenzimidazole ligands.[40] In the current work, we describe synthesis, characterization and thermal studies of dimethyltin dichloride adduct with *N*-(2-hydroxyphenyl) substituted aryloxy sulfamates. The molecular structure of the opted multi-dentate sulfamate might affect the planarity, hydrophobicity, and coordination geometry of tin complexes and perhaps the anticancer activity of tin-based agents.

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Electronic supplementary material: The online version of this article (https://doi.org/10.1007/s12039-018-1586-1) contains supplementary material, which is available to authorized users.

2. Experimental

2.1 *Materials and methods*

The melting point of the complexes was recorded by a differential scanning calorimetric instrument (DSC 131 evo-Setaram, France). The IR spectra were measured using a Nicolet iS5 Infrared Spectrometer in KBr disc (Thermo Scientific, USA). ^{1}H NMR, ^{13}C NMR and ^{119}Sn spectra were recorded by Bruker Avance 300 spectrometer (Bruker, France) at 300 and 75 MHz, respectively. Chemical shifts were reported in ppm from internal TMS for ^{1}H, ^{13}C and ^{119}Sn. Elemental analysis was obtained by an Exeter Analytical CE-440 Elemental instrument (Exeter Analytical, UK). X-ray spectra were recorded by an Oxford X-calibur Gemini diffractometer (Santa Clara, California, USA) equipped with EOS CCD detector using monochromated Mo Ka radiation (k = 0.71073 Å) at room temperature. All reagents and solvents used in this study were commercially available (from Sigma-Aldrich) and were used without further purification. The intermediate compounds were prepared according to the literature methods.

2.2 *Synthesis of substituted phenyloxy (2-hydroxybenzoyl)sulfamates **2a–c***

Freshly prepared and distilled aryloxysulfonyl isocyanate (1 equiv.) and salicylic acid (1 equiv.) were taken in a round bottom flask and anhyd. toluene (10 mL) was added to it followed trimethylamine (0.05 equiv.). Then it was stirred in an argon atmosphere at room temperature. The reaction was monitored by TLC and was found to be completed after 30 min. The crude solid was filtered and recrystallized from carbon tetrachloride.

*2.2.1 Mesityl (2-hydroxybenzoyl)sulfamate **(2a)***: White solid. Yield: 94%. M.p.: 74 °C. IR (cm^{-1}): ν_{OH} 3444, ν_{NH} 3355, ν_{CHarom} 3076, $\nu_{C=O}$ 1658, $\nu_{C=Carom}$ 1612, 1549, 1475, 1448, ν_{SO2} 1350. ^{1}H NMR (δ, ppm in $CDCl_3$) : 7.9–6.6(m; H$_{arom}$, 6H), 5.4 (br, NH, 1H), 4.4 (s, OH; 1H), 2.4 – 2.3 (2 s, CH$_{3arom}$; 9H). ^{13}C NMR (δ, ppm in $CDCl_3$): δ167.2, 160.4, 143.4, 134.1, 129.2, 127.7, 119.9, 117.5, 115.2, 21.8, 16.5. Anal. Calculated(%) for $C_{16}H_{17}NO_5S$: C, 57.30; H, 5.10; N, 4.17; Found(%): C, 57.27; H, 5.09; N, 4.14.

*2.2.2 2,4,6-Trichlorophenyl (2-hydroxybenzoyl)sulfamate **(2b)***: White solid. Yield: 91%. M.p.: 86 °C. IR (cm^{-1}): ν_{OH} 3436, ν_{NH} 3361, ν_{CHarom} 3087, $\nu_{C=O}$ 1682, $\nu_{C=Carom}$ 1614, 1568, 1473, ν_{SO2} 1378. ^{1}H NMR (δ, ppm in $CDCl_3$): 7.7 – 7.0 (m; H$_{arom}$, 6H), 6.2 (br, NH, 1H), 4.1 (s, OH; 1H). ^{13}C NMR (δ, ppm in $CDCl_3$): δ168.2, 161.4, 145.9, 134.3, 130.0, 129.6, 120.1, 117.9, 115.8. Anal. Calculated(%) for $C_{13}H_8Cl_3NO_5S$: C, 39.36; H, 2.03; N, 3.53. Found(%): C, 39.33; H, 2.00; N, 3.52.

*2.2.3 4-Fluorophenyl (2-hydroxybenzoyl)sulfamate **(2c)***: White solid. Yield: 93%. M.p.: 69 °C. IR (cm^{-1}): ν_{OH} 3448, ν_{NH} 3370, ν_{CHarom} 3080, $\nu_{C=O}$ 1690, 1612, 1502, $\nu_{C=Carom}$ 1448, ν_{SO2} 1373. ^{1}H NMR (δ, ppm in $CDCl_3$) : 7.8 – 6.8 (m; H$_{arom}$, 8H), 6.5 (br, NH, 1H), 3.9 (s, OH; 1H). ^{13}C NMR (δ, ppm in $CDCl_3$): δ168.5, 164.1, 161.1, 145.2, 134.1, 129.5, 121.0, 119.9, 118.0, 117.6, 115.4. Anal. Calculated(%) for $C_{13}H_{10}FNO_5S$: C, 50.15; H, 3.23; N, 4.49. Found(%): C, 50.14; H, 3.20; N, 4.47.

2.3 *Synthesis of organotin complexes **3a–c***

The alcoholic solution of 1 mmol of dimethyltin dichloride was added with constant stirring to an alcoholic solution of 1 mmol of Sodium salt of substituted aryloxysulfamate ligand. The brown precipitates were instantly obtained. The precipitates were filtered and washed with water and then with Methanol and dried over calcium chloride in a desiccator.

2.3.1 $[Me_2Sn(L_1)Cl]$***(3a)***: Brown solid. Yield: 54%. M.p.: 373 °C. IR (cm^{-1}): ν_{OH} 3440; $\nu_{C=N}$ 1608 and 1522; ν_{Sn-C} 578; ν_{Sn-N} 507. ^{13}C CPMAS NMR (ppm): 183–164(C=O andC=N); 137–117(C$_{arom}$); 80–40(CH$_{3arom}$), 14–8(CH$_3$ -Sn). Anal. Calculated(%) for $C_{18}H_{22}ClNO_5SSn$: C, 41.69; H, 4.28; N, 2.70. Found(%): C, 41.67; H, 4.25; N, 2.69.

2.3.2 $[Me_2Sn(L_2)Cl]$***(3b)***: Brown solid. Yield: in 41%. M.p.: 338 °C. IR data (cm^{-1}): ν_{OH} 3452; $\nu_{C=N}$ 1610 and 1526; ν_{Sn-C} 571; ν_{Sn-N} 510. ^{13}C CPMAS NMR (ppm): 182 – 164(C=O and C=N); 138 – 116(C$_{arom}$); 14 – 8(CH$_3$ -Sn). Anal. Calculated(%) for $C_{15}H_{13}Cl_4NO_5SSn$: C, 31.07; H, 2.26; N, 2.42. Found(%): C, 31.05; H, 2.25; N, 2.41.

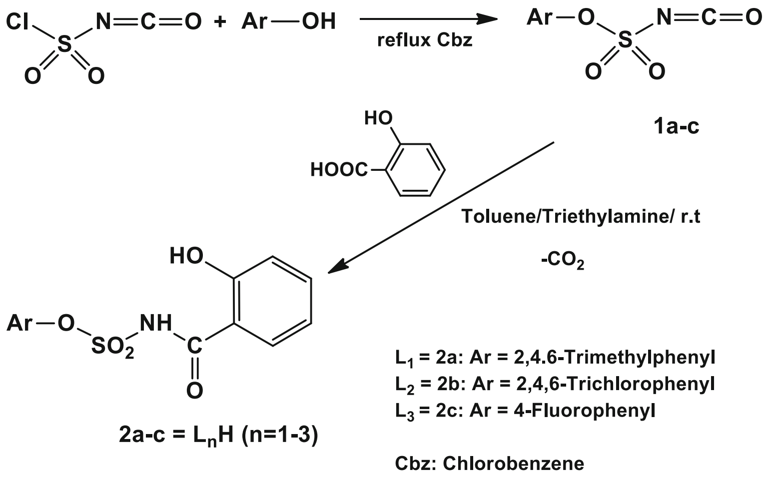
2.3.3 $[Me_2Sn(L_3)Cl]$ ***(3c)***: Brown solid. Yield: 44%, M.p.: 299 °C. IR data (cm^{-1}): ν_{OH} 3444; $\nu_{C=N}$ 1606 and 1526; ν_{Sn-C} 576; ν_{Sn-N} 511. ^{13}C CPMAS NMR (ppm): 185–165 (C=O and C=N); 144–121(C$_{arom}$); 13–8(CH$_3$-Sn). Anal. Calculated (%) for $C_{15}H_{15}ClFNO_5SSn$: C, 36.43; H, 3.06; N, 2.83. Found(%): C, 36.40; H, 3.05; N, 2.80.

3. Results and Discussion

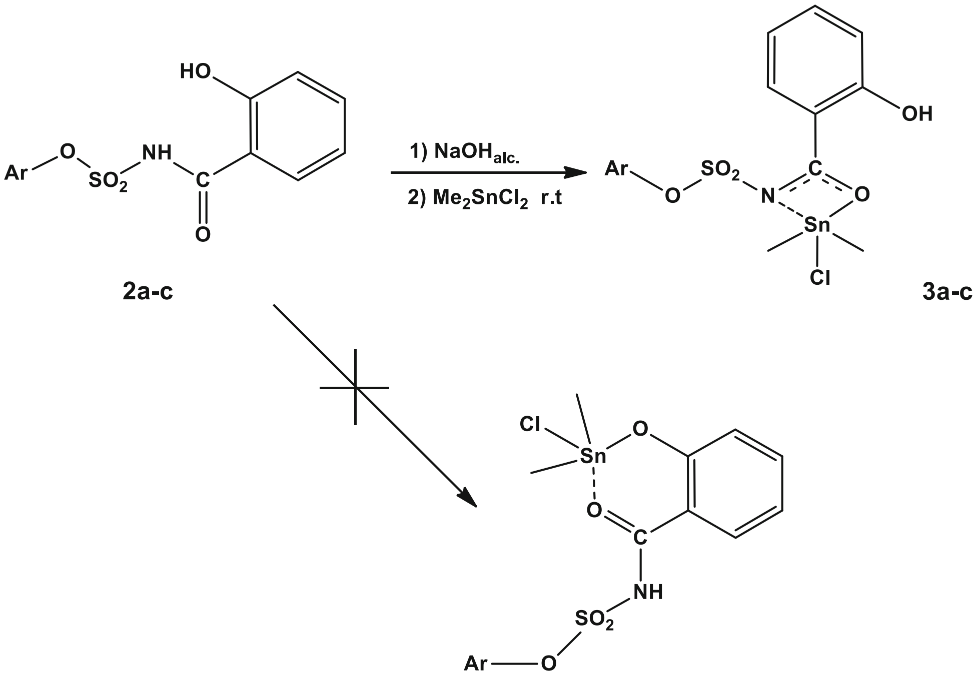
3.1 *Chemistry*

The synthesis of new sulfamate derivative ligands (L$_n$H) **2a–c** has been carried out in a basic medium at room temperature by the addition of 2-hydroxy benzoic acid to a series of substituted aryloxy sulfonylisocyanates **1a–c** which followed by decarboxylation reaction[41] (Scheme 1). The latter isocyanates were prepared according to literature methods.[42,43]

Dimethylthin phenyloxy (2-hydroxybenzoyl)sulfamate complexes **3a–c** [Me$_2$Sn(L$_n$)Cl] were synthesized from *N*-(2-hydroxyphenoyl) substituted aryloxy sulfamates **2a–c** in alkaline medium, and its metal complexes were prepared. As a result of the electro-attractor effect of both sulfonyl and carbonyl groups,



Scheme 1. Synthesis of substituted phenyloxy (2-hydroxybenzoyl)sulfamates **2a–c**.



Scheme 2. Synthesis of sulfamate complexes **3a**–**c**.

the proton sulfamate group NH is more acidic than the proton hydroxyl group OH; therefore, a significant conjugated bidentate ligand would be formed and reacted with the Tin halide, in 1:1 molar ratio, as shown in Scheme 2.

The composition of isolated complexes was confirmed by the findings obtained from elemental analyses which performed by an Exeter Analytical CE-440 Elemental instrument. It has been found that in all ligands (L_nH) **2a**–**c**, one chloride was substituted by monoanionic ligands and furnished with moderate yields their corresponding mononuclear complexes **3a**–**c**.

Upon coordination to tin atom, many absorption bands were markedly shifted. The three complexes **3a**–**c** exhibited broad bands at 3440, 3449, and 3444 cm^{-1} respectively, due to the hydroxyl (OH) chromophore. Thus, the absorption bands observed at 3375 cm^{-1} and 1656 cm^{-1} in IR spectrum of **2c** which assigned to (N-H) and (C=O) groups respectively, were disappeared in the homologous IR spectrum of **3c** in favor of appearance of new bands exhibited at (1609, 1514) cm^{-1} and (565–547) cm^{-1} which might be attributed to (C=N) and (Sn-C) chromophores correspondently (Figure 1). The absorption bands of both carbonyl and imino groups are hardly observed probably because of the high conjugation (Scheme 2).

The ^{13}C NMR spectra of compounds **3a**–**c** displayed one set of resonances which were consistent with the presence of one organotin(IV) moiety and equivalence of the methyl groups attached to tin on the NMR time scale (Figure 2). Indeed, the signals observed at 14–8 ppm were assigned to CH_3-Sn carbon group, whereas, the signals exhibited at 80–40 ppm were attributed to the aromatic methyl (CH_{3arom}) carbon group. The aromatic carbons displayed signals at 137–117 ppm. The C=O and C=N carbon groups presented signals located at 185–165 ppm.

X-ray spectra of synthesized complexes **3a–c** were recorded by an Oxford X-calibur Gemini diffractometer and shown in Figure 3. All spectra revealed sharp peaks with significant diffraction intensity and low background. Indexing of the powder diffraction pattern and searching space group of the crystal specimen were determined using X-pert high score plus software program after peak searching (Figure 3). Particular structural properties and features obtained are presented in Tables 1–3, respectively. The organotin compounds **3a** and **3b** show tetragonal crystal system, while, the crystal structure of complex **3c** belong to the hexagonal system. Crystallite size *Dp* for all complexes **3a–c** have been calculated using Scherrer formula[44] and obtained values are 142 nm, 76 nm, and 70 nm, respectively.

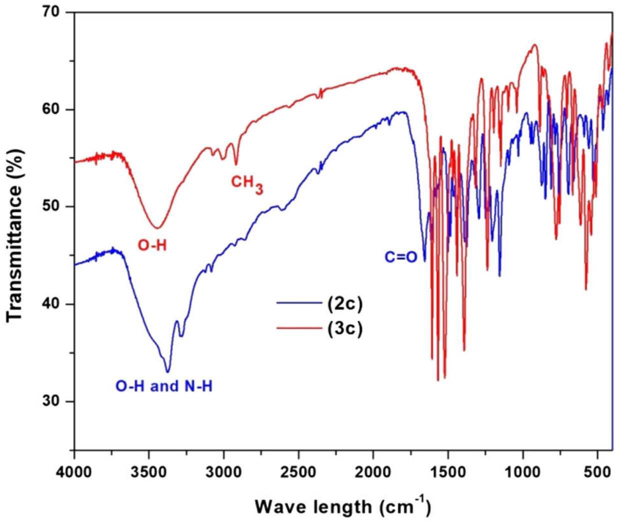


Figure 1. Infrared spectra of ligand **2c** and its corresponding complex **3c**.

$$D_p = \frac{K\lambda}{\beta\cos\theta}$$

D_p is the size of particles of crystals in the form of powder, K is a dimensionless shape factor with a value close to unity, λ is the X-ray wavelength, β is the line broadening at half the maximum intensity (FWHM), after subtracting the instrumental line broadening, in radians and θ is the Bragg angle.

Tables 1–3 show that all the diffraction peaks in the pattern of the synthesized organotin complexes **3a–c** can be readily indexed by a set of lattice parameters. The relative deviations between the calculated and experimental are about 1.13%, 0.37%, and 0.36% for products

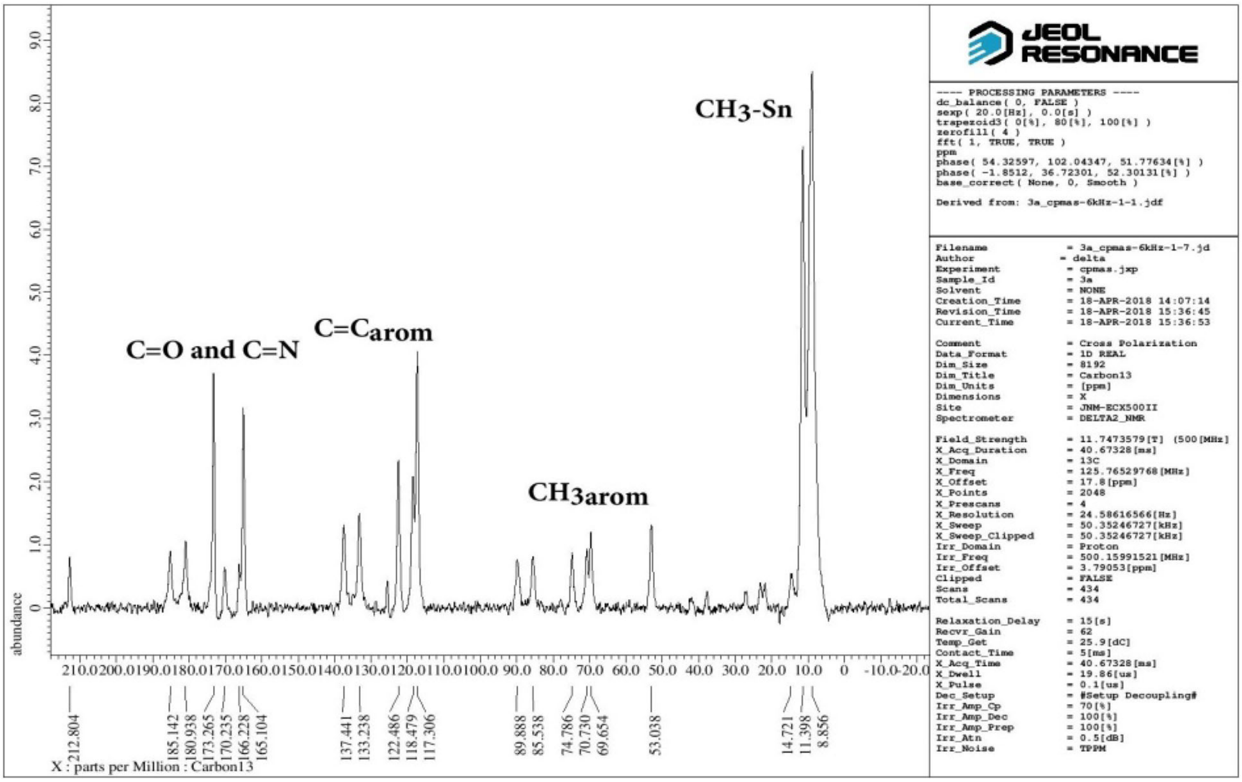


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Figure 2. ^{13}C CPMAS NMR spectrum of organotin complex **3a**.

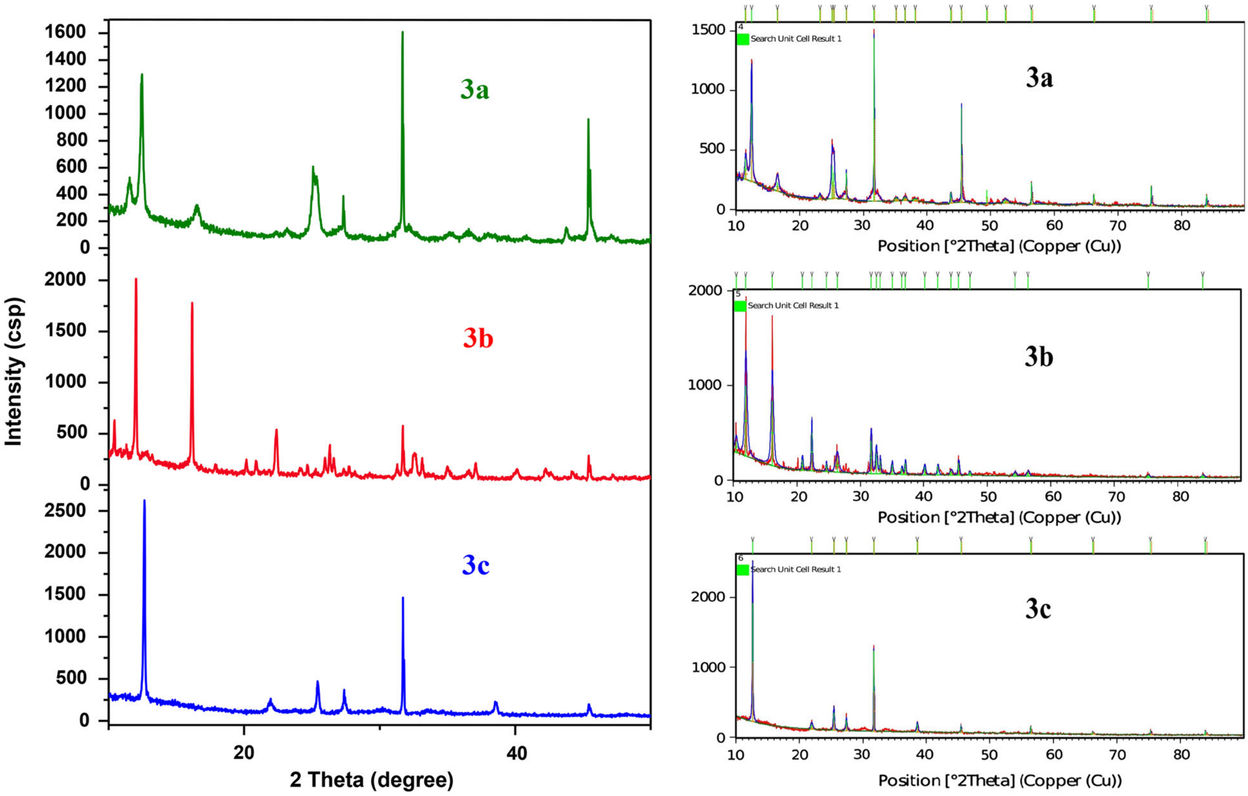

Figure 3. X-ray patterns of complexes **3a–c** and indexation peak using X-pert high score plus software.

Table 1. Experimental data and calculated results for the powder X-ray diffraction pattern of **3a**, tetragonal symmetry.

Peak no.	2θ (c) [°]	2θ (o) [°]	d-sp. (c)[Å]	d-sp. (o) [Å]	Relative intensity (%)	*hkl*
1	11.5707	11.4789	7.641693	7.702639	12.56	100
2	12.4071	12.3876	7.128348	7.139563	47.90	101
3	16.3915	16.4768	5.403492	5.375722	5.26	110
4	23.2616	23.131	3.820846	3.842123	2.31	200
5	24.9628	25.0595	3.564174	3.550645	19.92	202
6	25.3287	25.3407	3.513511	3.511875	15.76	105
7	27.593	27.2856	3.23011	3.2658	16.02	212
8	31.6343	31.6316	2.826082	2.826311	100.00	007
9	35.2044	35.134	2.547231	2.552172	2.24	300
10	36.3925	36.4842	2.466749	2.460762	2.27	302
11	38.2233	38.0732	2.352707	2.361631	1.46	108
12	43.6406	43.7133	2.072384	2.069108	6.21	322
13	45.2256	45.3761	2.003371	1.997076	58.11	218
14	49.3459	49.3517	1.845302	1.845099	8.75	411
15	52.2106	52.2846	1.75059	1.748288	1.93	1011
16	56.5098	56.3931	1.627179	1.63027	13.26	2011
17	66.0679	66.1456	1.413041	1.411568	5.67	0014
18	75.1509	75.2085	1.263187	1.262364	11.88	602
19	83.9897	83.9029	1.151308	1.152279	6.41	5111

Lattice parameters calculated: a = 7.64 Å, b = 7.64 Å, c = 19.78 Å and α = β = γ = 90°
Particle size of complex **3a**: 142 nm, V = 1155.21 Å^3. (2Theta Zero Shift [°], 0.0(1)).

Table 2. Experimental data and calculated results for the powder X-ray diffraction pattern of **3b**, tetragonal symmetry.

Peak no.	2θ (c) [°]	2θ (o) [°]	d-sp. (c) [Å]	d-sp. (o) [Å]	Relative intensity (%)	*hkl*
1	10.4175	10.4552	8.484892	8.454357	14.84	002
2	11.9636	11.9934	7.391633	7.373334	100.00	101
3	16.1204	16.1441	5.493742	5.485749	91.50	111
4	20.9224	20.8978	4.242446	4.247382	14.54	004
5	22.259	22.3631	3.990629	3.972279	53.07	201
6	24.7858	24.6695	3.589231	3.605881	10.22	211
7	26.4248	26.3521	3.370199	3.379341	19.68	212
8	31.6088	31.7311	2.828297	2.817677	45.82	006
9	32.5715	32.5138	2.746871	2.751613	28.86	222
10	33.1246	33.1522	2.702254	2.700069	18.31	301
11	34.9268	35.0121	2.566839	2.560783	13.10	311
12	36.4364	36.5498	2.463877	2.456496	8.53	303
13	37.0534	37.0893	2.424255	2.421989	15.59	007
14	40.2131	40.1444	2.240761	2.244435	10.89	216
15	42.3893	42.2347	2.13062	2.138059	12.02	305
16	44.4152	44.3299	2.038026	2.041746	5.49	401
17	45.4878	45.482	1.99243	1.99267	16.86	118
18	47.2275	47.2446	1.923011	1.922356	4.77	331
19	54.4033	54.3872	1.685099	1.685562	4.36	424
20	56.4471	56.4298	1.62884	1.629297	5.48	1110
21	75.3292	75.3317	1.26064	1.260604	2.95	536
22	83.9728	83.9769	1.151497	1.151451	3.45	635

Lattice parameters calculated: a=8.212 Å, b=8.212 Å, c = 16.97 Å and $\alpha = \beta = \gamma = 90°$,
Particle size of complex **3b**: 76 nm, V = 1144.26 Å^3. (2Theta Zero Shift [°], −0.01(6)).

Table 3. Experimental data and calculated results for the powder X-ray diffraction pattern of **3c**, hexagonal symmetry.

Peak no.	2θ (c) [°]	2θ(o)[°]	d-sp. (c) [Å]	d-sp. (o) [Å]	Relative intensity (%)	*hkl*
1	12.6202	12.6337	7.008465	7.00105	100.00	200
2	21.9487	21.9512	4.046339	4.045873	7.20	220
3	25.3969	25.4268	3.504233	3.500174	21.24	400
4	27.4867	27.392	3.242368	3.253358	12.40	211
5	31.6927	31.7292	2.820999	2.817842	71.49	311
6	38.5047	38.5464	2.336155	2.333723	8.12	600
7	45.6151	45.4493	1.987164	1.994027	7.57	112
8	12.6202	12.6337	7.008465	7.00105	4.02	200
9	21.9487	21.9512	4.046339	4.045873	1.36	220
10	25.3969	25.4268	3.504233	3.500174	2.19	400
11	27.4867	27.392	3.242368	3.253358	1.49	211

Lattice parameters calculated: a = 16.19 Å, b = 16.19 Å, c = 4.10 Å and $\alpha = \beta = 90°$, $\gamma = 120°$
Particle size of complex **3c**: 70 nm, V = 930.13 Å^3. (2 Theta Zero Shift [°], −0.0(1)).

3a, **3b** and **3c** correspondently, which could suggest that all complexes are single-phase compounds.

3.2 *DSC studies for the complexes **3a–c***

The thermal curves exhibited slight endothermic peaks nearly at 373 °C, 338 °C, and 299 °C for the complexes **3a**, **3b**, and **3c,** respectively, which followed by heat flow gradually rise until 649 °C, 652 °C, and 646 °C correspondently (Figure 4). Indeed, the combustion process of complex **3a** was achieved in two different stages. The related DSC curve revealed intense exothermic peak at 664 °C in the temperature range 649–664 °C followed by two successive endothermic peaks at 666 and 669 °C, respectively with an extrapolated combustion enthalpy equal to −699.12 J.g^{-1}. However, the decomposition mechanism of **3b** was carried out in two similar stages. The corresponding DSC curve exhibited

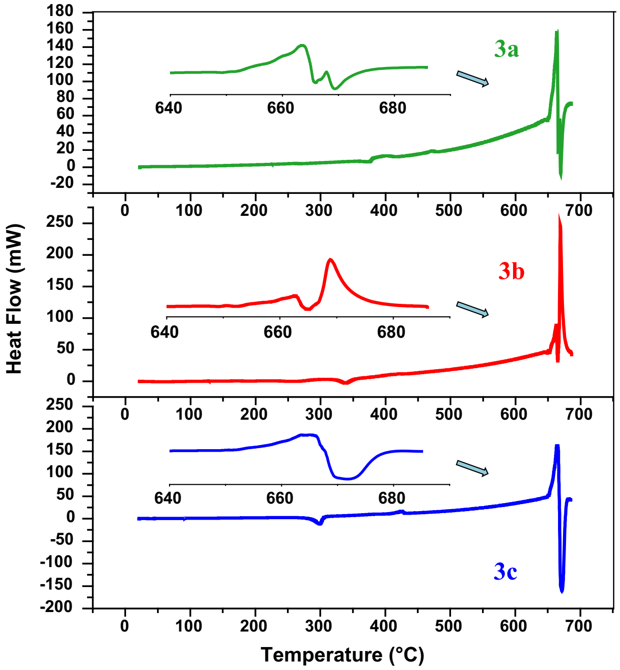

Figure 4. DSC thermograms of complexes **3a–c**.

sharp exothermic peak which presents two maxima at 662 °C and 668 °C, respectively in the temperature range 652–684 °C with total determined enthalpy combustion equal to −343.2 J.g^{-1}. Finally, the combustion proceeding of **3c** was accomplished in two different stages. The associated DSC curve displayed an intense exothermic peak at 665 °C in the temperature range 646–667 °C pursued by an endothermic peak at 671 °C with calculated enthalpy combustion equal to −755.5 J.g^{-1}. It is important to notice that all derivatives **3a**–**c** present higher thermal stability and hardly decompose at low temperature; this may be due to the absence of small molecules which could be attached to the complexes (Figure 4).

4. Conclusions

Three monomeric pentacoordinate organotin(IV) complexes were successfully synthesized and characterized using various analytical approaches. The results of spectroscopic analyses were in agreement with the suggested structures of the compounds. All the complexes **3a**–**c** were obtained *via O*, *N* coordination to tin center by their homologous ligands **2a**–**c**, respectively. The analysis of XRD spectra revealed that products **3a** and **3b** crystalize in the tetragonal system, while, the crystal structure of complex **3c** refers to the hexagonal system. DSC studies showed that all the organotin compounds **3a**–**c** possess higher thermal stability, a similar range of decomposition temperature, and slightly comparable combustion processes.

Supplementary Information (SI)

Supplementary Information for this article is available at www.ias.ac.in/chemsci.

Acknowledgements

The authors wish to acknowledge the approval and the support of this research study by the grant N°. SCI-2017-1-7-F-6928 from the Deanship of Scientific Research in Northern Border University, Arar, KSA.

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