SYNTHESIS AND CHARACTERIZATION BY INFRARED AND MOSSBAUER SPECTROSCOPIES OF SOME NEW SnR₂ (R = Me, Ph, Bu) RESIDUE CONTAINING ARBOXYALKYLPHOSPHONATE ADDUCTS AND DERIVATIVES

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Abstract: Four new carboxyalkylphosphonate SnR₂ residue (R = Me, Ph, Bu) containing derivatives $\{(Cy_2NH_2)_3(O_2CCH_2PO_3)(SnBu_2Cl_2)\}$ adducts and i.e. $\{[(SnMe_2Cl)(O_2CCH_2PO_3H_2)]_4[Cy_2NH_2Cl]\}$ $\{(Me_4N)(SnPh_2Cl)[O_2C(CH_2)_2$ (2), PO_3H]($SnPh_2$)[$O_2C(CH_2)_2PO_3H$] (3) and { $(Cy_2NH_2)_2(O_2CCH_2PO_3H)(SnPh_2Cl_2)_2$ } (4), have been synthesized from one-pot reactions carried out in solution. All compounds have been investigated by spectral techniques (infrared and Mossbauer). The spectral studies have evidenced presence of several characteristic bands, especially v (C=O), v (OH), v (CO₂⁻), v(PO₃²⁻) vibrations coming from carboxyalkylphosphonate ions, with wide absorption due to the NH₂ groups coming from the dicyclohexylammonium counter ion (for 1, 2 and 4) and the intense doublet which show the presence of phenyl groups (for 3 and 4). In the solid state, the proposed structures are discrete or of infinite chain however hydrogen bonding patterns may occur. Event in this investigation is the presence, for compound 3, of two types of arrangement at Sn centers viz. an octahedron and a trigonal bipyramidal whose presence are ascertained by the Mossbauer parameters. The neutral or acidic carboxyalkylphosphonate ions exhibit a diversity of coordination behavior towards the Sn atoms: monochelating (carboxylate and phosphonate O-donor), monochelating through the carboxylate and unidentate coordinating O-donor through the phosphonic acid or acidic phosphonate, bichelating with chelations from carboxylate and acidic phosphonate, and unidentate from both the carboxylate and phosphonic acid.

Keywords: discrete and polymeric structures, octahedral or trigonal bipyramidal environments, supramolecular architectures

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1. INTRODUCTION

Organotin compounds have several applications in various fields such as agriculture, industry and medical sciences [1-5]. The isolation of new organotin (IV) compounds carried considerable efforts because they are of the potential class of biologically active materials. Indeed, they evidenced in the past, antidiabetic [6], antituberculosis [7], antibacterial [8], antimicrobial [9], anti-inflammatory [10], antitumor [11-13] and cardiovascular [14] activities. The carboxyalkylphosphonate complexes are used in organic syntheses as catalysts in esterification reactions [15, 16]. From a structural point of view they have tendency to give rise polymeric structures [17-19].

The presence of two functions *viz* carboxylate and phosphonate, makes carboxyalkylphosphonates polyfunctional ligands able to behave as an anion that can coordinate metal atoms generating polymeric structures in which it adopts polydentate coordination mode. Several investigation studies dealing with carboxyalkylphosphonate complexes have previously been isolated and characterized [20-28]. The Dakar group has already been involved in studies by describing, using FT-IR, FT-Raman and Mossbauer spectroscopies, the structure of the complex {SnMe₂Cl[O₂CCH₂PO₃H]} [29].

In the dynamic of widening our contribution in the field of organotin(IV), and understanding the behavior of carboxyalkylphosphonate, the reactions between $HO_2CCH_2PO_3H_2$ or $HO_2C(CH_2)_2PO_3H_2$ and some diorganotin chloride precursors. The isolation of four new SnR_2 [R = Me, Ph, Bu] residue containing carboxyalkylphosphonate compounds, was promoted. Their composition and structures have been investigated by elemental analyses and spectral studies (FT-IR and Mossbauer).

2. EXPERIMENTAL SETUP

2.1. Salts synthesis

 $(Cy_2NH_2)_2O_2CCH_2PO_3H\cdot 3H_2O$ (L_1) and $(Me_4N)HO_2C(CH_2)_2PO_3H\cdot H_2O$ (L_2), were obtained by partial neutralization of the $HO_2C(CH_2)_nPO_3H_2$ acids (n=1,2) by Cy_2NH (99%) in water or Me_4NOH (25% as aqueous solution), respectively in 1:2 and 1:1 ratio.

2.2. Compounds synthesis

The studied compounds were obtained as white powders after a slow solvent evaporation on mixing:

- L_1 in ethanol with $SnBu_2Cl_2$ in ethanol in ratio 1:3 (1);
- Cy₂NH in ethanol with HO₂CCH₂PO₃H₂ in methanol and SnMe₂Cl₂ in ethanol in ratio 2:1:1 (2);
- L₂ in methanol with SnPh₃Cl in methanol in ratio 2:1 (3);
- L₁ in chloroform with SnPh₂Cl₂ in ethanol in ratio 2:1 (4).

All the mixtures were stirred around two hours before being submitted to a slow solvent evaporation. The analytical data [% calculated and % found], reported below (Table 1) have allowed to suggest the formulae (Table 2).

Table 1. Analytical data.

	Chemical composition [% mass]						
Compound	С		Н		N		
	Calc.	Found	Calc.	Found	Calc.	Found	
1	55.93	55.56	9.39	9.42	4.25	4.17	
2	22.26	21.74	4.27	4.20	0.92	0.89	
3	42.56	42.49	4.41	4.35	1.46	1.43	
4	50.45	50.35	6.01	6.03	2.35	2.30	

Table 2. Compound and chemical formula.

Compound	Chemical formula				
1	$\{(Cy_2NH_2)_3(O_2CCH_2PO_3)(SnBu_2Cl_2)\}$				
2	$\{[(SnMe_2Cl)(O_2CCH_2PO_3H_2)]_4[Cy_2NH_2Cl]\}$				
3	$\{(Me_4N)(SnPh_2Cl)[O_2C(CH_2)_2PO_3H](SnPh_2)[O_2C(CH_2)_2PO_3H]\}$				
4	$\{(Cy_2NH_2)_2(O_2CCH_2PO_3H)(SnPh_2Cl_2)_2\}$				

The results of the Mossbauer analyses are presented in Table 3.

Table 3. Results of the Mossbauer analyses of compounds 1-4.							
Compound Phases		IS (mm/s)	QS (mm/s)	Γ (mm/s)			
		(± 0.05)	(± 0.05)	(±0.04)			
1	1	1.58	3.69	1.00			
2	1	1.27	3.82	1.04			
3	2	1.18	3.32	0.95			
	2	0.45	1.80	0.93			
1	1	0.43	1 77	1.01			

Table 3. Results of the Mossbauer analyses of compounds 1-4.

Elemental analyses have been obtained at the ICMCB-Bordeaux University, France with a CHNS: Flash EA 1112 Thermofisher. Infrared spectra have been recorded at the CRPP-Bordeaux University, France using a Nicolet 6700 FT-IR spectrophotometer on diamond.

Mossbauer spectra were recorded at the ICMCB-Bordeaux, France on a liquid helium cryostat with a HALDER spectrometer. The chemicals were purchased from Aldrich Company-Germany- without any further purification.

3. RESULTS AND DISCUSSION

3.1. FT-IR spectroscopy

Compounds 1-5 have been investigated by FT-IR spectroscopy in ATR mode. In the past, FT-IR carboxylates and phosphonate vibration bands investigation have been reported [29]. The binding modes of the carboxylate moiety were investigated by FT-IR spectroscopy in ATR mode. Indeed, the magnitude of the $\Delta v(\text{COO})$ *i.e.* $v(\text{COO})_{\text{asym}} - v(\text{COO})_{\text{sym}}$ value is usually used to judge the coordination mode of carboxylate groups towards metal atoms [30-32]. Thus, the $\Delta v(\text{COO})$ value of 118 cm⁻¹ in 1, less than 150 cm⁻¹ should be correlated to a carboxylate in chelation mode [33]. Moreover, the presence of carbonyl C=O vibration band at 1723 cm⁻¹ diagnose dissymmetrical in binding, the global species adopting a chelation. In addition, the spectrum of 1 exhibits absorption bands that can be assigned to N–C and C–H bonds of the dicyclohexylammonium ion, and carboxy methyl phosphonate.

The presence of vibration band located in the region $3000-2500~\rm cm^{-1}$ which may be assigned to O–H and/or N–H stretching vibrations is very difficult to be attributed. Vibration bands situated at $1309~\rm cm^{-1}$ and $1289~\rm cm^{-1}$ correspond to C–N stretching and deformation vibrations of the dicyclohexylammonium. The rocking CH₃ and CH₂ vibrations expected about $1000-1100~\rm cm^{-1}$, are partially overlapped by stretching vibrations of the phosphonate which appear about $1129~\rm and~1033~\rm cm^{-1}$, while their elongation vibrations are observed at $746~\rm cm^{-1}$. The carboxylate and phosphonate deformation vibration bands are observed at $783~\rm and~588~\rm cm^{-1}$, respectively. The presence of both $\nu(\rm SnC_2)_{\rm asym}$ at $684~\rm cm^{-1}$ and $\nu(\rm SnC_2)_{\rm sym}$ at $607~\rm cm^{-1}$ is in accordance with a nonlinear $\rm SnC_2$ group [34,35].

The $\Delta\nu(COO)$ value of 189 cm⁻¹ in 2, is correlated to a carboxylate in monodentate mode. The presence of carbonyl C=O vibration band at 1698 cm⁻¹ evidenced a dissymmetrical binding, the global ion adopting chelation from the carboxylate. The spectrum of 2 exhibits absorption bands assignable to N–C and C–H bonds of the dicyclohexylammonium ion, and carboxy methyl phosphonate. Vibration bands situated at 1297 cm⁻¹ and 1206 cm⁻¹ correspond to C–N stretching and deformation vibrations of the dicyclohexylammonium. The shift of ν (P=O) vibration band is also observed enabled by the coordination of the phosphonate moiety.

The rocking CH_3 and CH_2 vibrations expected about $1000\text{-}1100~\text{cm}^{-1}$, are also partially overlapped by stretching vibrations of the phosphonate which appear at 1124, 1085 and $1032~\text{cm}^{-1}$. The elongation vibrations of CH_3 and CH_2 are observed about $722~\text{cm}^{-1}$. The carboxylate and phosphonate deformation vibration bands are observed at 779 and $621~\text{cm}^{-1}$, respectively. The presence of $\nu(SnC_2)_{asym}$ at $706~\text{cm}^{-1}$ and the absence of $\nu(SnC_2)_{sym}$ are in accordance with a linear SnC_2 group [30, 36].

The $\Delta v(COO)$ value of 89 cm⁻¹ in 3, is in accordance with a carboxylate in chelation mode. The presence of carbonyl C=O vibration band at 1715 cm⁻¹ evidences dissymmetry in the binding, the global species adopting a chelation from the carboxylate. The spectrum of compound 3 exhibits absorption bands assignable to N–C and C–H bonds of the dicyclohexylammonium ion, and carboxy ethyl phosphonate. The C–N stretching and deformation vibration bands of the dicyclohexylammonium are attributed to bands appearing at 1243 cm⁻¹ and 1185 cm⁻¹,

respectively. The rocking CH_3 and CH_2 vibrations expected about $1000\text{-}1100~\text{cm}^{-1}$, are overlapped by stretching vibrations of the phosphonate which appear about 1071, 1017 and $997~\text{cm}^{-1}$. The elongation vibrations of CH_3 and CH_2 are also overlapped by vibration bands characteristic of phenyl ligands, observed at 730 and 697 cm⁻¹ corresponding to phenyl groups C-H and C=C elongations, respectively. The carboxylate and phosphonate deformation vibration bands are observed in order at 801 and $660~\text{cm}^{-1}$; the latter is partially overlapped by C-H and C=C elongations of phenyl groups. The presence of $\nu(SnC_2)_{sym}$ observed at 618 cm⁻¹ is in accordance with a nonlinear SnC_2 group [34, 35].

The $\Delta v(\text{COO})$ value of 230 cm⁻¹ in 4 higher than 200 cm⁻¹, is in accordance with a carboxylate in monodentate coordination mode. The presence of carbonyl C=O vibration band at 1720 cm⁻¹ evidenced a pendent oxygen site from the carboxylate. The v (P=O) vibration band is shifted at 1120 cm⁻¹, describing coordination of the phosphonate ligand. Thus, the anion adopts a bridging bidentate coordination fashion through the carboxylate.

The spectrum of 4 ascertained absorption bands assignable to N–C and C–H bonds of the dicyclohexylammonium ion, and carboxy methyl phosphonate. The C–N stretching and deformation vibration bands of the dicyclohexylammonium are attributed to bands appearing at 1347 cm⁻¹ and 1279 cm⁻¹, respectively. The rocking CH₃ and CH₂ vibrations expected about 1000-1100 cm⁻¹, are overlapped by stretching vibrations of the phosphonate which appear in the region 1120-1013 cm⁻¹. The elongation vibrations of CH₃ and CH₂ are also overlapped by vibration bands characteristic of phenyl ligands, observed at 735 and 697 cm⁻¹ corresponding to phenyl groups C–H and C=C elongations, respectively. The carboxylate and phosphonate deformation vibration bands are observed in order at 817 and 588 cm⁻¹. The presence of ν (SnC₂)_{sym} observed at 630 cm⁻¹ is in accordance with a nonlinear SnC₂ group [34, 35].

For compounds wherein the dicyclohexylammonium counter ion is present, absorption bands corresponding to NH_2 bending vibrations are overlapped by the carboxylate vibrations, the scissoring, bending vibrations of CH_2 group too. The same phenomenon is observed for vibrations corresponding to wagging or twisting, the bending vibrations out of plane of CH_2 groups.

Infrared characteristic absorption vibration bands evidence presence of all moieties which are present in the proposed formulae. Thus, the infrared investigations well corroborate the suggested formulae.

3.2. Mossbauer spectroscopy and molecular structures

Compounds 1-4 were also investigated by ¹¹⁹Sn Mossbauer spectroscopy (Table 3). For 1, the Mossbauer parameters (Table 3) evidence one arrangement type at Sn atom. In the past, report for SnBu₂Cl₂ exhibits a quadrupole splitting of 3.38 mm·s⁻¹ describing SnBu₂ residues in a dissymmetrical *trans*-octahedral coordination fashion. Thus, the value of the quadrupole splitting, higher than 3.38 mm·s⁻¹ as well as the isomer shift [37] describes an octahedron at Sn center. Various SnBu₂ containing crystalline structures are known, to date [30, 34-36]. In the solid state, the FT-IR and Mossbauer data suggest a discrete structure with a chelating anion dissymmetrically coordinated to SnBu₂Cl₂ molecule (Figure 1); the counter ions interact with the complex-anion through hydrogen bonding patterns giving rise to a supramolecular topology.

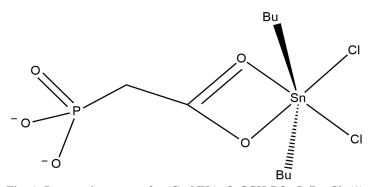


Fig. 1. Proposed structure for $(Cy_2NH_2)_3O_2CCH_2PO_3\cdot SnBu_2Cl_2$ (1).

For 2, the Mossbauer parameters (Table 3) confirm the presence of only *trans*-coordinated octahedral geometry at tin center [30, 36]. Indeed, previous reports for SnMe₂Cl₂ and SnMe₂O₂CCH₂PO₃H complex-cation, which have quadrupole splitting of 3.56 mm·s⁻¹ and 3.83 mm·s⁻¹, describe structures wherein the arrangement at tin centers is

a dissymmetrical *trans*-coordinated octahedron [29, 38]. Therefore, our spectroscopic data suggests an infinite chain structure for the SnMe₂ClO₂CCH₂PO₃H₂ complex, in the solid state. In the structure the anion describes carboxylate chelation and phosphonate monodentate coordination modes (Figure 2).

Fig. 2. Proposed structure for SnMe₂ClO₂CCH₂PO₃H₂·1/4Cy₂NH₂Cl (2).

For 3, the Mossbauer parameters evidence two arrangement types at Sn atoms *i.e.* trigonal bipyramidal and *cis*-octahedral coordination. The FT-IR and Mossbauer data (Table 3) allow to suggest, in the solid state, an infinite chain with alternating SnPh₂ and the SnPh₂Cl moieties bridged by the anion describing carboxylate monochelation *cis*-chelating the SnPh₂moiety, and phosphonate monocoordination *trans*-coordinating the SnPh₂Cl fragment; the phenyl groups are disposed in the basal planes (Figure 3). The adverse cations interact through electrostatic forces with the complex-anion.

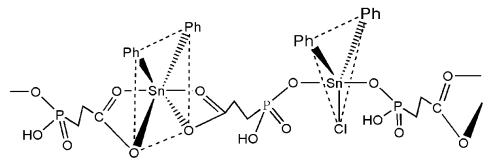


Fig. 3. Proposed structure for (Me₄N)(SnPh₂Cl)O₂C(CH₂)₂PO₃H·SnPh₂O₂C(CH₂)₂PO₃H (3).

For 4, the Mossbauer quadrupole splitting value is consistent with *cis*-octahedral coordination. The spectroscopic data suggests for the complex-anion, in the solid state, a discrete structure describing a monodentate carboxylate and phosphonate bridging tridentate ligand coordinated to two SnPh₂Cl₂ molecules, wherein phenyl groups are in *cis*- positions (Figure 4).

The counter ions interact with the complex-anion through hydrogen bonding patterns affording a supramolecular structure.

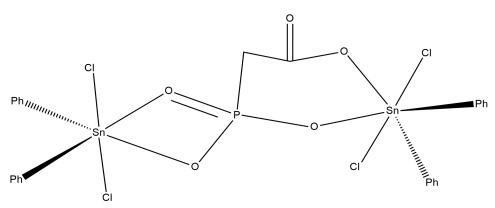


Fig. 4. Proposed structure for $(Cy_2NH_2)_2O_2CCH_2PO_3H\cdot 2SnPh_2Cl_2$ (4).

4. CONCLUSIONS

Carried out in solution, the one-pot reactions between the carboxyalkylphosphonate salts, $(Cy_2NH_2)_2O_2CCH_2PO_3H\cdot 3H_2O$ or $(Me_4N)HO_2C(CH_2)_2PO_3H\cdot H_2O$, and organotin(IV) precursors led to the isolation of four adducts and derivates whose characterizations were merged using FT-IR and Mossbauer techniques. The studied adducts and derivates have a discrete or an infinite chain structure. The carboxyalkylphosphonate acidic or neutral ligands behave as a monochelating, monochelating and monodentate or bichelating. The environments around the tin centers are cis-, trans-octahedral or trigonal bipyramidal. When the counter cations are involved in hydrogen bonding interactions, a supramolecular architecture is obtained.

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