TRIPHENYLTIN (IV) GLUTARATES: SYNTHESIS, FT-IR AND MÖSSBAUER STUDIES

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Abstract: Four new adducts catena-O,O′-glutaratotriphenylstannate, $[(Me_4N)_2O_2C(CH_2)_3CO_2\cdot 4SnPh_3Cl]$ (**1**), $[(Me_4N)_2O_2C(CH_2)_3CO_2\cdot 3SnPh_3Cl]$ (**2**), $[(SnPh_3)_2O_2C(CH_2)_3CO_2\cdot SnPh_3Cl]$ (**2′**) and $[(Cy_2NH_2)_2O_2C(CH_2)_3CO_2\cdot SnPh_3Cl]$ (**3**), have been isolated from the reactions carried out in solution. The compounds have been characterized by FT-IR and Mössbauer spectroscopies except for **2'** whose structure is proposed on the basis of FT-IR data. Overall, the spectra studies sheew existence of several caracteristic bands such as υ (C=O), υ (CO_2^-), υ (Sn–O), υ (Sn–Cl), vibrations of glutarate ions, and the intense doublet showing the presence of phenyl groups. In the solid state, the suggested structures are discrete or of infinite chain. In this work, the geometry at Sn centers, trigonal bipyramidal or tetrahedral, is ascertained by the Mössbauer parameters. The glutarate ions exhibit a monodentate, tridentate or tetradentate coordination behavior towards the Sn atoms.

Keywords: dimeric, discrete and polymeric structures, glutarate, spectroscop, tetrahedral or trigonal bipyramidal environments.

1. INTRODUCTION

Organotin (IV) carboxylates, an important class of compounds, have directed incommensurable efforts because they have merged antibacterial activity [1], antimicrobial activity [2], antidiabetic properties [3], catalytic properties [4] and biological activity [5-7], potential anticancer activity [8-11], fungicidal activity [12], antituberculosis activity [13]. Different modes of coordination of carboxylates toward organotin (IV) fragments describe a diversity of intriguing topologies [14-21]. Moreover, the interactions between a variety of metallic centers and carboxylates have widely been investigated affording countless attracting structural arrangement [22-28]. However, organotin glutarates are quite rare in the literature [29, 30]. Focusing in this area since a while, the Dakar group has undergone wide contributions through several reports [31-38]. As continuation of our involvement, we have isolated new organotin(IV) carboxylates and report herein the synthesis and spectral (FT-IR and Mössbauer) characterization of three based *catena*-O,O′-glutaratotriphenylstannate.

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2. EXPERIMENTAL SETUP

2.1. Salt synthesis

The salt $(Me_4N)_2O_2C(CH_2)_3CO_2{\cdot}5H_2O$ **(L)** was obtained by total neutralization of glutaric acid, $HO_2C(CH_2)_3CO_2H$ (99% purity) by the base Me_4NOH (25% in aqueous solution).

2.2. Compounds synthesis

The studied compounds **1**, **2**, **2′** and **3** were obtained as white powders.

Synthesis of **1**: This compound is obtained by mixing **L (**0.81 mmol) dissolved in ethanol with $SnPh_3Cl$ (95% purity) (1.62 mmol) also dissolved in ethanol in a 1:2 ratio, m.p. (melting point) = 228 °C.

Synthesis of **2**: This compound is obtained by mixing ethanolic solutions of **L** (0.67 mmol) and $SnPh_3Cl$ (95 % purity) (2.68 mmol) in a 1:4 ratio. The cloudy obtained solution was filtrated, and the precipitate characterized as **2**. m.p. >260°C. A secondary product characterized as **2′** has also been collected from slow evaporation of the filtrate, m.p. = 130 °C. The equation of the reaction is as follow:

$$2[(Me_4N)_2O_2C(CH_2)_3CO_2{\cdot}5H_2O] + 6SnPh_3Cl \rightarrow [(Me_4N)_2O_2C(CH_2)_3CO_2{\cdot}3SnPh_3Cl] + [(SnPh_3)_2O_2C(CH_2)_3CO_2{\cdot}SnPh_3Cl] + 2Me_4NCl + 10H_2O \quad (1)$$

Synthesis of **3**: This compound is obtained by mixing ethanolic solutions of Cy_2NH (99 % purity) (2.48 mmol), $HO_2C(CH_2)_3CO_2H$ (99% purity) (2.48 mmol) and $SnPh_3Cl$ (95 % purity) (2.48 mmol) in a 1:1:1 ratio. m.p. = 192 °C. All the mixtures were stirred around two hours before being filtered.

The analytical data reported below (Table 1) have allowed to suggest the formulae.

Table 1. Analytical data of compounds L and 1-3.

Compound	Suggested formulae	Chemical composition [% mass]							
		C		H		N		Sn	
		Calc.	Found	Calc.	Found	Calc.	Found	Calc.	Found
L	$[(Me_4N)_2O_2C(CH_2)_3CO_2{\cdot}5H_2O]$	42.39	42.19	10.87	11.02	7.60	7.46	–	–
1	$[(Me_4N)_2O_2C(CH_2)_3CO_2{\cdot}4SnPh_3Cl]$	56.09	56.18	4.98	5.01	1.54	1.68	26.09	26.15
2	$[(Me_4N)_2O_2C(CH_2)_3CO_2{\cdot}3SnPh_3Cl]$	56,09	56.10	5,27	5.15	1.95	1.85	24.82	24.76
2′	$[(SnPh_3)_2O_2C(CH_2)_3CO_2{\cdot}SnPh_3Cl]$	58,29	58.18	4.23	4.31	–	–	29.30	29.16
3	$[(Cy_2NH_2)_2O_2C(CH_2)_3CO_2{\cdot}SnPh_3Cl]$	64.13	64.20	7.90	7.64	3.18	3.23	13.49	13.51

Elemental analyses have been obtained at the Institute of Condensed Matter Chemistry of Bordeaux (ICMCB) Bordeaux University, France with an elemental analyzer (FlashEA 1112, Thermo Fisher Scientific). The ICP-OES (Inductively Coupled Plasma Optical Emission spectroscopy) was performed at the ICMCB with a Varian 720. Infrared spectra have been recorded at the Paul Pascal Research Center (*CRPP*)-Bordeaux, France using a Nicolet 6700 FT-IR spectrophotometer. Mössbauer spectra were recorded at the ICMCB on a liquid helium cryostat with a HALDER spectrometer. The chemicals were purchased from Aldrich Company, Germany, and used without any further purification.

3. RESULTS AND DISCUSSION

3.1. FT-IR spectroscopy

The binding modes of glutarate in compounds **1**, **2** and **3** were investigated by FT-IR spectroscopy in Attenuated Total Reflectance (ATR) mode. In the IR spectra, the absence of the broad band in the region 3400–2800 cm^{-1}, which appears in the free glutaric acid due to COOH group, indicates deprotonation and thus, the formation of Sn–O bonds through this site. In several investigations, the difference between the asymmetric and symmetric vibration bands, *i.e.* $\Delta\nu(C(O)O)$ $[=\nu(C(O)O)_{asym}-\nu(C(O)O)_{sym}]$ value was used to judge the bonding mode of carboxylate groups to metal centers [18–21, 33, 34]. Indeed, the $\Delta\nu(C(O)O)$ value smaller than 200 cm^{-1} is an indicative for a bidentate or chelation mode, while the value larger than 200 cm^{-1} evidences a carboxylate moiety in an unidentate fashion.

For compound **1**, the glutarate asymmetric and symmetric stretching vibration bands appear at 1538 cm^{-1} and 1427 cm^{-1}, respectively. These bands, in comparison to the free acid, shifted to lower frequencies as a result of the coordination of glutarate ligands to Sn(IV) atoms forming Sn–O bonds. In the IR spectrum, the ν(Sn–O) vibration band which appears at 459 cm^{-1} ascertained the binding of glutarate ligand to tin center. The presence of chlorine atoms is evidenced by the ν(Sn–Cl) vibration band observed at 272 cm^{-1}. The $\Delta\nu$(C(O)O) value of 111 cm^{-1} is smaller than 200 cm^{-1}, indicating a bidentate coordination mode of each carboxylate group [18–21, 33, 34]. The vibration bands characteristic of phenyl ligands, *i.e.* C−H and C=C elongations are observed at 731 and 697 cm^{-1}, respectively.

For compound **2**, the IR spectrum shows two variations between the glutarate asymmetric stretching vibration bands which appear at 1573 cm^{-1} and 1531 cm^{-1}, and symmetric stretching vibration bands appearing at 1428 cm^{-1} and 1326 cm^{-1}. The $\Delta\nu$(C(O)O) value of 145 cm^{-1} smaller than 200 cm^{-1}, indicates a bidentate coordination mode while the $\Delta\nu$(C(O)O) value of 205 cm^{-1} higher than 200 cm^{-1}, describes a monodentate coordination behavior [18–21, 33, 34]. The shift observed for asymmetric and symmetric stretching vibration bands of the ligand are in accordance with the formation of Sn−O bonds, thus deprotonation and coordination. The presence of carbonyl C=O vibration band is observed at 1726 cm^{-1} allows to conclude that the glutarate ligand adopts a tri-O-coordination mode. The vibration bands characteristic of C−H and C=C phenyl ligands elongations are observed at 722 and 696 cm^{-1}, respectively.

For compound **2′**, the IR spectrum exhibit a unique variation between the glutarate asymmetric stretching vibration bands observed at 1527 cm^{-1} and symmetric stretching vibration bands appearing at 1427 cm^{-1}. The $\Delta\nu$(C(O)O) value of 100 cm^{-1} smaller than 200 cm^{-1}, well corroborate polydentate coordination fashion of the glutarate ligand [18–21, 33, 34]. Moreover, the shift noticed for asymmetric and symmetric stretching vibration bands of the ligand are in accordance with the formation of Sn−O bonds, thus deprotonation and coordination too. However, the presence of carbonyl C=O vibration band is observed at 1724 cm^{-1} allows to conclude to a glutarate ligand adopting a tri-O-coordination mode or tetradentate with a notable dissymmetry in the coordination. The vibration bands characteristic of C−H and C=C phenyl ligands elongations are present at 725 and 693 cm^{-1}, respectively.

For compound **3**, the glutarate asymmetric and symmetric stretching vibration bands are observed at 1577 cm^{-1} and 1370 cm^{-1}, respectively. In comparison to the free acid, the glutarate asymmetric and symmetric stretching vibration bands are also shifted to lower frequencies resulting from the coordination of glutarate ligands to Sn(IV) atoms forming Sn–O bonds. The $\Delta\nu$(C(O)O) value of 207 cm^{-1} higher than 200 cm^{-1}, is an indicative of the presence of glutarate anion in a monodentate coordination behavior [18–21, 33, 34]. The C−H and C=C elongation vibration bands characteristic of phenyl ligands, are observed at 729 and 694 cm^{-1}, respectively.

3.2. Mössbauer spectroscopy and molecular structures

Compounds **1-3** were also investigated by ^{119}Sn Mössbauer spectroscopy (Table 2).

Table 2. Results of the Mössbauer analyses of compounds 1-3.

Compound	Phases	IS (mm/s) (±0.05)	QS (mm/s) (±0.05)	Γ (mm/s) (±0.04)
1	1	1.21	3.04	0.94
2	1	1.26	3.15	0.94
3	1	1.20	2.90	0.97

* IS: isomer shift; QS: quadrupole splitting; Γ: full width at half-height

The Mössbauer parameters for compound **1** (see Table 2) show one geometry arrangement at Sn center. In the 1970s, Parish and Platt reported the free $SnPh_3Cl$, exhibiting a quadrupole splitting value of 2.48 $mm.s^{-1}$ describing a tetrahedral arrangement [39]. Thus, the value of the quadrupole splitting (Table 2), higher than 2.48 $mm.s^{-1}$, is in accordance with a coordinated $SnPh_3Cl$ moiety. In 2003, Cisse et al. have isolated and investigated the compound $(Me_4N)_2(OOC\text{-}COO)\cdot 4SnPh_3Cl$ whose structure, showing a quadrupole splitting of 3.02 $mm.s^{-1}$, described *trans*-coordinated $SnPh_3$ moieties, linked to monodentate carboxylates affording a trigonal bipyramidal geometry at tin atoms [40]. We suggest, in the solid state, based on FT-IR and Mössbauer data, a discrete structure with a tetra-unidentate anion coordinated to the four $SnPh_3Cl$ molecules (Figure 1), the counter ions interact with the complex-anion through electrostatic interactions.

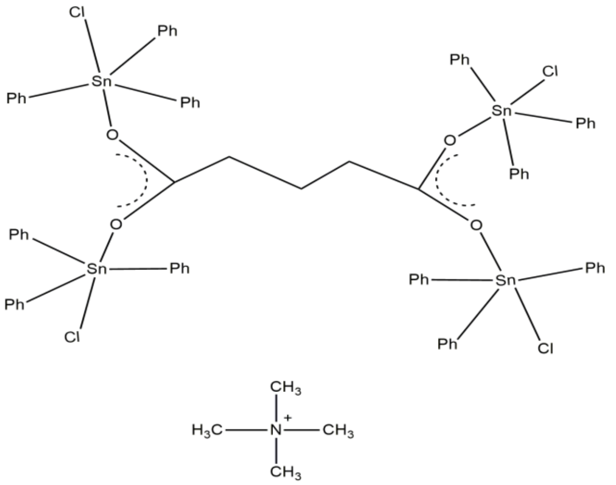

Fig. 1. Proposed structure for **1**.

The Mössbauer parameters for compound **2** (Table 2) evidence the presence of only *trans*-coordinated trigonal bipyramidal geometry at Sn center [40, 41]. Regarding previously reported works published by Cisse et al. [40] and Bancroft and Platt [41], which have a quadrupole splitting higher than 3 $mm.s^{-1}$, we suggest in the solid state, a discrete structure wherein the glutarate ligand, with a pendent no coordinating C=O, describes a tri-O-coordination mode towards Sn metal (Figure 2). The counter cations interact through electrostatic forces with the complex-anion.

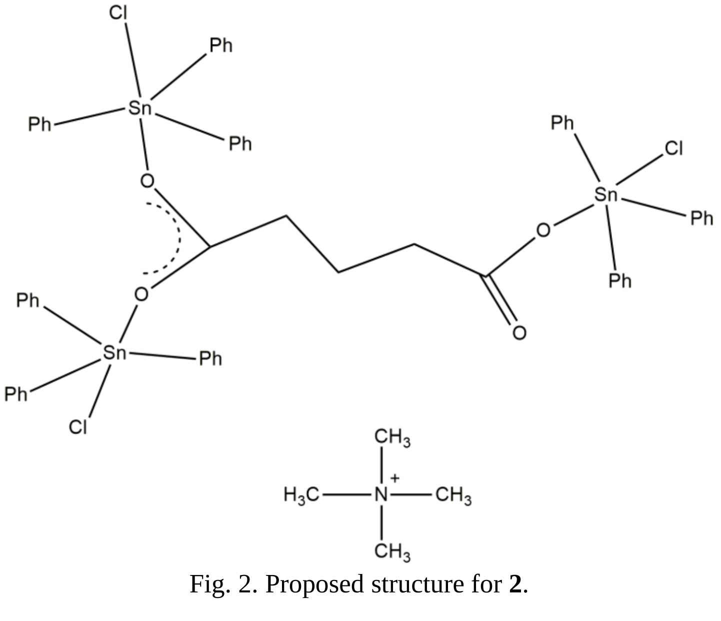

Fig. 2. Proposed structure for **2**.

The absence of Mössbauer parameters for compound **2′** etherizes the choice of the coordination mode of the ligand. Considering only the FT-IR data, and the fact that four metallic fragment are present, we propose for this compound an infinite chain of $[(SnPh_3)O_2C(CH_2)_3CO_2]^-$. To the anionic infinite chain, wherein the carboxylates are bidentate bridging and the $SnPh_3$ moieties *trans*-coordinated describing trigonal bipyramidal geometries at tin centers, are attached two $SnPh_3$ fragments. The trigonal bipyramidal coordination sphere at Sn atom, for one $SnPh_3$ moiety attached to the chain, is completed by a chloride ion while the other Sn atom of the pendent $SnPh_3$ moiety occupies the center of a tetrahedron for which the fourth top was completed by an oxygen atom from a carboxylate C=O group. The glutarate ligand is overall tetradentate but is the site of a dissymmetry in its binding behavior (Figure 3).

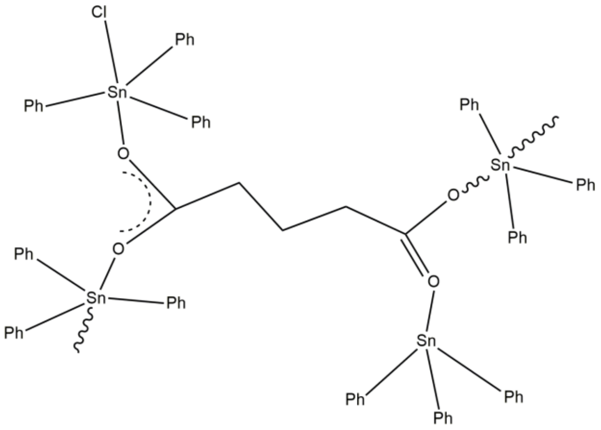

Fig. 3. Proposed structure for **2′**.

The Mössbauer parameters for compound **3** (Table 2) exhibit one arrangement type at Sn atoms *i.e.* trigonal bipyramidal [40, 41]. On the basis of FT-IR and Mössbauer data (Table 2) we suggest in the solid state, a dimeric structure describing monodentate glutarates coordinated to $SnPh_3Cl$ molecules, bridged by the cations through hydrogen bonding patterns giving rise to a cage like-structure (Figure 4).

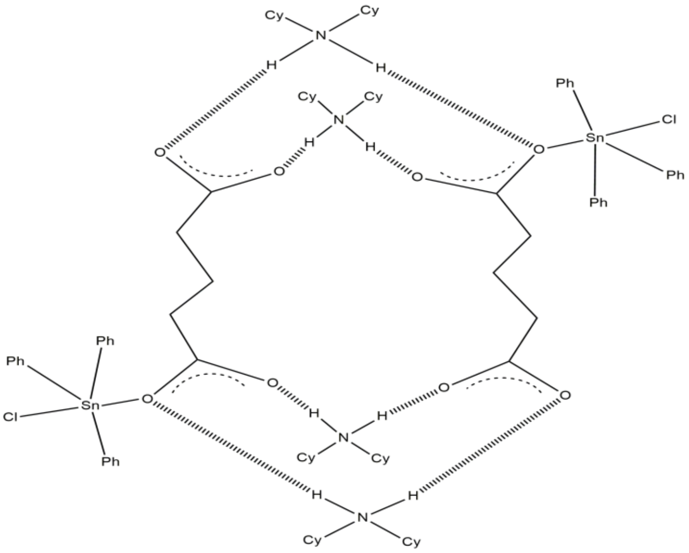

Fig. 4. Proposed structure for **3**.

4. CONCLUSIONS

The reactions, carried out in ethanolic solution, between the glutarate salt, $(Me_4N)_2O_2C(CH_2)_3CO_2{\cdot}5H_2O$ and triphenyltin(IV) chloride organometallic precursor led to the formation of four compounds which were characterized by FT-IR and Mössbauer techniques. The studied adducts have a discrete, monomeric or dimeric, or of infinite chain structure. The glutarate anion behaves as a tetra-, tri- or monodentate ligand. Within the structures, the environment around the tin centers are trigonal bipyramidal or tetrahedral. The use of the dicyclohexylammonium counter ion, which can be involved in hydrogen bonding interactions, has allowed to isolate a structurally interesting complex with hydrogen bonding interactions between the cation and the glutarate resulting in a hydrogen bonded dimeric structure.

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