

Thesis points of dissertation

**Preparation and structural characterization of  
organotin(IV) complexes with ligands containing  
{O,O} and {O,N} donor atoms**

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## Introduction and aim of the work

Tin has a larger number of its organometallic derivatives in commercial use than any other element. Due to this fact considerable amounts of the organotins have entered to the environment. Organotin compounds (OTC) have been known for just the past 150 years, but their wide industrial applications have been started only from 1950 in USA. Recently, organotin compounds have been used in large quantities as polyvinyl chloride (PVC) stabilisers (e.g.  $\text{Bu}_2\text{Sn(IV)-carboxylates}$ ), wood preserver additives (e.g.  $(\text{Bu}_3\text{Sn})_2\text{O}$ ) fungicides (e.g. *Duter*<sup>®</sup> based on  $\text{Ph}_3\text{SnOH}$ ). Organotin compounds have been entered the marine and freshwater environment from their use as antifouling paints on ships and boats. As a result of the above mentioned facts, organotin compounds have become one of the most important pollutant in the aquatic and terrestrial ecosystems. Therefore, they are also present in the human nutrition chain.

Organotin compounds have varying degrees of toxicological properties, depending on the nature and number of organic groups bonded to the tin atom. However, there are other non-pollutive ways for their applications. Organotin(IV) derivatives have been found to possess anticancer effects on different tumour cells. These positive and negative characteristics have significantly furthered the research on the co-ordination chemistry of the organotin(IV) compounds. Therefore, the study of the interaction between organotin(IV) compounds and the building blocks of macro molecules, which appear in a large amount in living organisms, is very important.

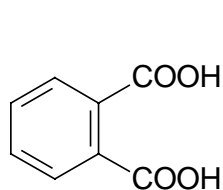
As a part of research initiated in 1988 in the Department of Inorganic and Analytical Chemistry, University of Szeged, the overall aim of my work was to prepare organotin(IV) complexes of some biological active ligands, and study their structure in solid form. Accordingly, the following systems were studied:

1.  ${}^n\text{Bu}_2\text{Sn(IV)}$ , and  $\text{Ph}_3\text{Sn(IV)}$  complexes of five dicarboxylic acids and salicylic acid,
2.  ${}^n\text{Bu}_2\text{Sn(IV)}$  complexes of six (poly)hydroxyalkyl carboxylic acids and two amino dicarboxylic acids,
3.  ${}^n\text{Bu}_2\text{Sn(IV)}$  complexes of three pyruvoyl-amino acid-oximes and 2-hydroxyimino-3-phenyl-propionic acid,
4.  ${}^n\text{Bu}_2\text{Sn(IV)}$ ,  ${}^t\text{Bu}_2\text{Sn(IV)}$  and  $\text{Ph}_3\text{Sn(IV)}$  complexes of three pyridine monocarboxylic acids and six pyridine dicarboxylic acids,
5.  ${}^n\text{Bu}_2\text{Sn(IV)}$ ,  ${}^t\text{Bu}_2\text{Sn(IV)}$  and  $\text{Ph}_3\text{Sn(IV)}$  complexes of seven ligands containing  $-\text{OH}$  ( $-\text{C}=\text{O}$ ) group(s) and aromatic  $\{\text{N}\}$  donor atom.

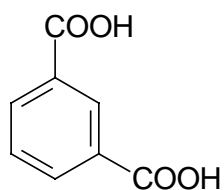
### **The methods applied for the investigations**

The tin content of the studied solid complexes was determined by inductively coupled plasma atomic emission spectrometry (ICP-AES) method. The bonding sites of the ligands were determined by means of FT-IR and Raman spectroscopic measurements. Partial quadrupole splitting calculations were utilized to determine the coordination geometry around the Sn(IV) centre by means of  ${}^{119}\text{Sn}$  Mössbauer measurements. The structures of the complexes were studied in some cases with  ${}^{13}\text{C}$  NMR and extended X-ray absorption fine structure (EXAFS) spectroscopic methods. Six complexes were obtained as single crystals, and their structure was determined with X-ray diffraction method.

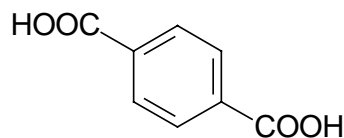
## Structure of the studied ligands



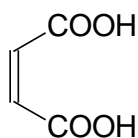
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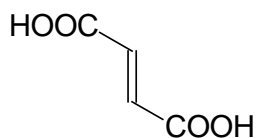
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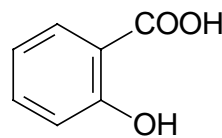
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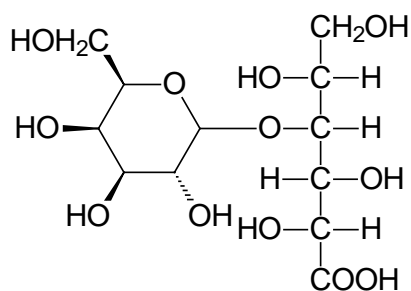
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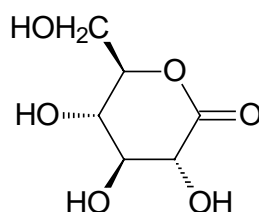
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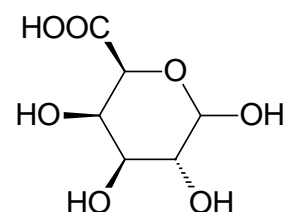
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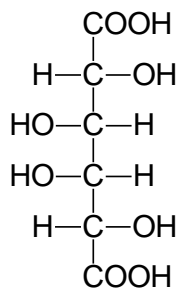
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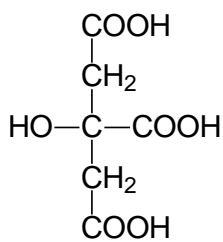
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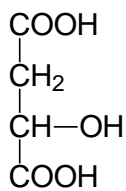
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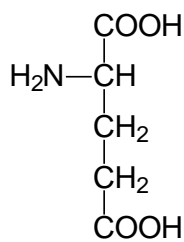
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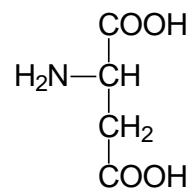
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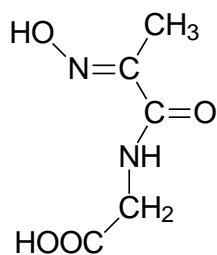
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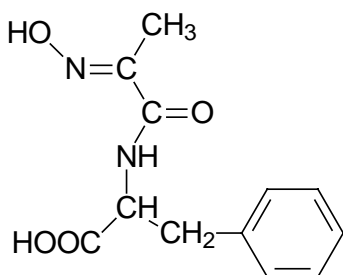
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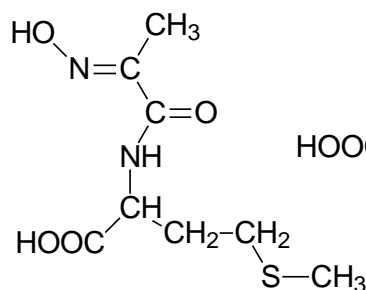
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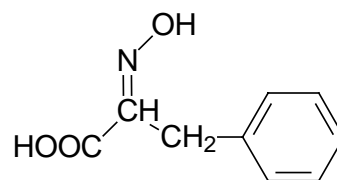
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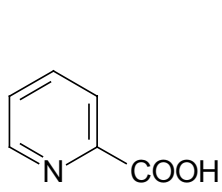
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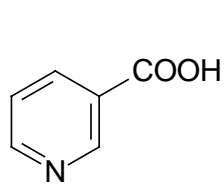
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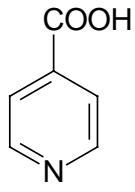
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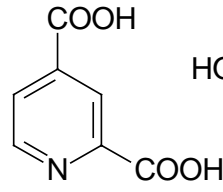
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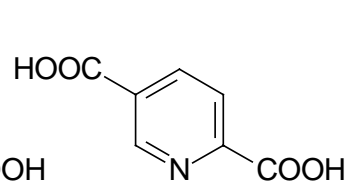
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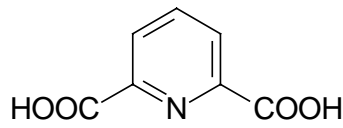
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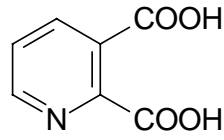
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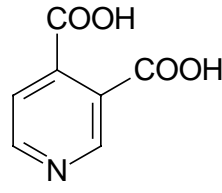
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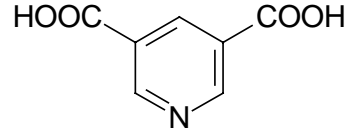
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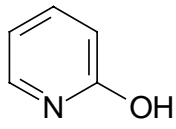
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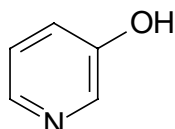
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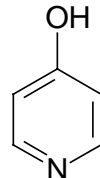
**P35DKS**



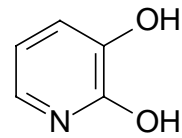
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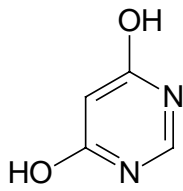
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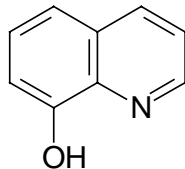
**4HP**



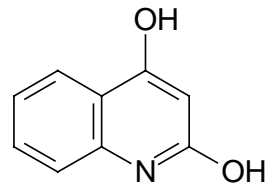
**23DHP**



**46DHPM**



**8HK**



**24DHK**

## New scientific results

The new scientific results presented in the thesis can be summarised as follows:

1.  $^{119}\text{Sn(IV)}$  complexes of phthalic (FS), *i*-phthalic (IFS), *t*-phthalic (TFS), maleic (MAS), fumaric (FUS) and salicylic (SAS) acid have been prepared by two different procedures. On the basis of their tin contents, these compounds have 1:1 metal-to-ligand ratio. The FT-IR and Raman spectral data indicated the deprotonation and co-ordination both of carboxylate groups in the  $^{119}\text{Sn(IV)}$  complexes of dicarboxylic acids. In the complexes of SAS the  $\nu_{\text{as}}\text{COO}^- - \nu_{\text{sym}}\text{COO}^- = 123 \text{ cm}^{-1}$  ( $\Delta\nu$ ) value indicates the bidentate co-ordination mode of the  $-\text{COO}^-$  group. The presence of the  $\nu\text{OH}$  and  $\delta\text{H}_2\text{O}$  bands in the spectra of the dicarboxylic acid complexes strongly suggests the  $\text{H}_2\text{O}$  co-ordination. The results of the Mössbauer spectroscopic investigations suggest the presence of completely equivalent Sn environments in these compounds. The results of the pqs calculations indicate the same trigonal bipyramidal (TBP) environments for the  $^{119}\text{Sn(IV)}$  complexes of the five dicarboxylic acid. The monodentate  $-\text{COO}^-$  groups of different ligands are located in axial (*ax*) positions bridging the individual tin(IV) centres in a polymeric chain. In the  $^{119}\text{Sn(IV)}$ -salicylic acid complex, the  $-\text{COO}^-$  group is bidentately coordinated and the central Sn(IV) atoms are also in TBP environment. The local structure of the  $\text{Bu}_2\text{Sn(IV)}$  complex of maleic acid was determined by EXAFS. On the basis of the magnitude of the non-bonding Sn...Sn distance (410-420 pm), it can be ruled out co-ordination of both maleinic carboxylic groups to same tin(IV) atom, which also verifies the long chain polymeric or cyclic oligomeric structure of the compound.

2.  ${}^n\text{Bu}_2\text{Sn(IV)}$  complexes of six (poly)hydroxyalkyl carboxylic acid [D-lactobionic acid (LBS), D-gluconic acid- $\delta$ -lactone (GLKS), D-galacturonic acid (GAUS), D-galactaric acid (GATS), malic acid (AMS) and citric acid (CITS)] and two amino dicarboxylic acid [glutamic acid (GLAS) and aspartic acid (ASPS)] have been prepared by the above mentioned two methods. The compounds were characterised by elemental analysis, FT-IR, Raman,  ${}^{119}\text{Sn}$  Mössbauer and, in some cases, EXAFS and  ${}^{13}\text{C}$  NMR spectroscopy. Both procedures resulted in compounds with 1:1 metal-to-ligand ratio, except CITS, where we found an additional 2:1 ratio. The results of the FT-IR and Raman spectroscopic investigations suggest in accordance with  ${}^{13}\text{C}$  NMR spectroscopy deprotonation and co-ordination of the alcoholic -OH promoted by  ${}^n\text{Bu}_2\text{Sn(IV)}$  ion in methanolic media. The  $\Delta\nu$  values suggest for the complexes of GAUS and those of amino acids that the  $-\text{COO}^-$  is coordinated in monodentate mode, and for the other in bridging bidentate mode, indicating oligomerization or polymerization of the complexes. The presence of the  $-\text{C}=\text{O}$  stretching vibration in the spectrum of the 2:1  $\text{Bu}_2\text{Sn(IV)}:\text{L}$  complex of CITS indicates that one of the methylene carboxylic groups remained uncoordinated. The presence of the strong  $\nu\text{Sn}-\text{N}$  band suggests the occurrence of dative  $\text{N} \rightarrow \text{Sn}$  bond in the amino acid complexes. The presence of the  $\nu_{\text{as}}\text{Sn}-\text{C}$  and  $\nu_{\text{sym}}\text{Sn}-\text{C}$  absorption bands in the Raman spectra of all the compounds revealed that the C-Sn-C bond angle is non-linear. The broadened or asymmetric shape of the experimental Mössbauer spectra reveals on the presence of two, different tin(IV) centres in compounds. We found that the most preferred geometry by the compounds is TBP. From the ratio of the intensities of the Mössbauer lines assigned to the two types of Sn(IV) atoms we concluded on the degree of oligomerization in complexes. The  ${}^{13}\text{C}$  NMR spectroscopy results confirmed that in DMSO, the oligomers containing bridging carboxylate groups decompose due to the solvent co-ordination, forming monomer species. In



four compounds we determined the bonding Sn–C, Sn–O and the non-bonding Sn...Sn distance by EXAFS method. It is indicative of non-linear – C–O–Sn–O– bonds formation.

3. Five di-*n*-butyltin(IV) complexes with three **Pyruvoyl-Amino acid-Oxime** [pyruvoyl-glycine-oxime (PGO), pyruvoyl-phenylalanine-oxime (PFAO) and pyruvoyl-methionine-oxime (PMO) (1:1)] and with 2-hydroxyimino-3-phenyl-propionic acid (HIFP) (1:1 and 1:2) have been prepared. The FT-IR and Raman spectral data indicated the formation of Sn–O and Sn–N<sub>amid</sub> bonds and the presence of the co-ordinated water molecule. In the PAO and HIFP 1:2 compounds the carboxylate group coordinates in monodentate manner, while in the HIFP 1:1 compound in bridging bidentate form. On the basis of the Mössbauer spectroscopic investigations it was found that tin(IV) centres are in *trans*-O<sub>h</sub> and *mer*-TBP environments in the PAO compounds in 1:1 molar ratio. The non-significant differences between measured  $\Delta$  values suggest that the local structure in the PAO complexes does not depend on the type of the amino acid side-chain. In the HIFP 1:2 compound, the tin(IV) centres are in *trans*-O<sub>h</sub> environment, where the formation of the two five-membered chelate rings stabilizes the monomeric structure.
4. A number of <sup>n</sup>Bu<sub>2</sub>Sn(IV), <sup>t</sup>Bu<sub>2</sub>Sn(IV) and Ph<sub>3</sub>Sn(IV) complexes with three pyridine monocarboxylic acids [2-picolinic acid (PIKS), nicotinic acid (NIKS) and isonicotinic acid (INIKS)] and six pyridine dicarboxylic acids [P23DKS, P24DKS, P25DKS, P26DKS, P34DKS and P35DKS] have been prepared in the solid state by two methods. The analytical data showed different (1:2, 1:1 and 2:1) metal-to-ligand ratios. The structures of three complexes were determined by X-ray diffraction measurements. The units of [<sup>n</sup>Bu<sub>2</sub>Sn(PIKS)<sub>2</sub>]<sub>n</sub> are connected into polymeric chains, where Sn is heptacoordinated in a distorted *trans* pentagonal bipyramidal (*trans*-PBP)

geometry. The donor atoms of the ligands form five-membered  $C_2OSnN$  chelate rings around the tin(IV) centres in the *eq* plane. The units are connected through bridging bidentate carboxylate groups. The structure of  $[{}^{119}\text{Bu}_2\text{Sn}(\text{P26DKS})]$  is also *trans*-PBP. The compound is dinuclear, the two Sn atoms being joined through pairs of  $\text{Sn}-\text{O}_{(\text{carboxylate})}-\text{Sn}$  bridges. In the asymmetric unit of  $[{}^1\text{Bu}_2\text{Sn}(\text{P26DKS})]$  the single crystal X-ray analysis revealed that there are two chemically similar but symmetry independent molecules in sphere pyramidal (SP) co-ordination geometry. The  $-\text{COO}^-$  groups of the tridentate P26DKS anion are unidentate. There are a weak  $\text{C}-\text{H}\cdots\pi$  interactions between the  $\pi$  electrons of the five membered  $C_2OSnN$  chelate rings and the *t*-butyl groups. In the two types of  $\text{R}_2\text{Sn}(\text{IV})\text{P26DKS}$  complexes we interpreted the difference between the behaviour of  ${}^1\text{butyl}$  and  ${}^{119}\text{butyl}$  groups with molecular modelling calculations. On the basis of the FT-IR results,  $\Delta\nu$  values reflect the monodentate coordination mode of the  $-\text{COO}^-$  group for the generality of  $\text{R}_2\text{Sn}(\text{IV})$  pyridine dicarboxylic acid complexes, while for pyridine monocarboxylic acid complexes being indicative of bridging bidentate and monodentate co-ordination mode. In the compounds of  $\text{P2XDKS}$  ( $X = 3, 4, 5$  and  $6$ ) the co-ordination of the pyridine  $\{\text{N}\}$  is proved by the red shift of the  $\beta\text{C}=\text{N}$  band. We revealed that the interaction of  $\text{Ph}_3\text{SnOH}$  and  $\text{PIKS}$  in methanol lead to an interesting “dismutation” reaction followed with the migration of the phenyl group. The products of this reaction were  $\text{Ph}_2\text{Sn}(\text{PIKS})_2$  and  $\text{Ph}_4\text{Sn}$ . Based on the range of  $\Delta_m$  values ( $> 4,00 \text{ mms}^{-1}$ ) it can be concluded that in  ${}^{119}\text{Bu}_2\text{Sn}(\text{IV})$  compounds the central Sn(IV) atoms are in *trans*-PBP surroundings. By means of X-ray crystal data we extended the application of the Sham and Bancroft equation (calculating the  $\text{C}-\text{Sn}-\text{C}$  angle) on heptaco-ordinated  $\text{R}_2\text{Sn}(\text{IV})$  complexes. Using the above mentioned equation the calculated  $\text{C}-\text{Sn}-\text{C}$  angles for compound  $[{}^1\text{Bu}_2\text{Sn}(\text{P26DKS})]$  agree excellently with the X-ray diffraction data, and confirm the validity of the equation. The structural

elucidation based on pqs concept showed that in the  ${}^t\text{Bu}_2\text{Sn(IV)}$  and  $\text{Ph}_3\text{Sn(IV)}$  complexes the Sn centres are in distorted  $\text{O}_h$  and in distorted TBP co-ordination geometry, respectively. The results of the spectroscopic investigations showed that in P34DKS and P35DKS complexes the pyridine {N} atom is not involved in co-ordination.

5.  ${}^n\text{Bu}_2\text{Sn(IV)}$ ,  ${}^t\text{Bu}_2\text{Sn(IV)}$  and  $\text{Ph}_3\text{Sn(IV)}$  complexes with seven ligands (2HP, 3HP, 4HP, 23DHP, 46DHPM, 8HK and 24DHK) containing  $-\text{OH}$  ( $-\text{C}=\text{O}$ ) group(s) and aromatic {N} donor atom have been synthesised. We determined their composition with elemental analysis. The molecular structures of  ${}^n\text{Bu}_2\text{Sn(8HK)}_2$ ,  ${}^t\text{Bu}_2\text{Sn(8HK)}_2$  and  $\text{Ph}_3\text{Sn(23DHP)}$  complexes were measured by X-ray diffraction method. In both  $\text{R}_2\text{Sn(8HK)}_2$  complexes, the tin center is six-coordinated in a *cis*- $\text{O}_h$  coordination geometry. The quinolin-8-olato-N,O anion is bound to the metal centres in a bidentate chelating fashion forming five-membered  $\text{SnONC}_2$  ring. In  $\text{Ph}_3\text{Sn(23DHP)}$  compound the tin(IV) centres are in distorted *cis*-TBP geometry. The 3-hydroxy-2(1H)-pyridinone anion is bound also in a bidentate chelating fashion forming five-membered  $\text{SnO}_2\text{C}_2$  ring. Some ligands exist in different forms (hydroxy-ketone) depending on the physical state or on the pH of their solutions. Based on the FT-IR spectroscopic measurements it was concluded that in organotin(IV) complexes the ligands co-ordinate mostly in hydroxy form. The results of the Mössbauer spectroscopic investigations showed that in  ${}^t\text{Bu}_2\text{Sn(IV)}$  and  $\text{Ph}_3\text{Sn(IV)}$  complexes, which contain bulky organic groups, the tin(IV) centres are in one type, but for  ${}^n\text{Bu}_2\text{Sn(IV)}$  compounds in generality, they are in two different geometries. The pqs calculations suggested that in the monohydroxy pyridine complexes the Sn centres are in distorted TBP co-ordination geometry. The ligands 3HP and 4HP form long-chain polymers with the  $\text{R}_2\text{Sn(IV)}^{2+}$  and  $\text{Ph}_3\text{Sn(IV)}^{2+}$  ions. In complexes, the polymerisation

proceeds through {N,O} donor atoms of the ligands. It was shown in the case of Ph<sub>3</sub>Sn(23DHP) compound that by using two independent methods (X-ray diffraction and pqs method from the Mössbauer spectroscopic results) practically identical results were obtained both for the bonding angles and the co-ordination geometry around the Sn(IV). It is also confirmed by Mössbauer spectroscopic results for two R<sub>2</sub>Sn(8HK)<sub>2</sub> complexes. The pqs calculations indicate that the complex [R<sub>2</sub>Sn(46DHPM)]<sub>n</sub> contains distorted *cis*-O<sub>h</sub> co-ordination sites. In the species the oligomerization is occurred through ligand bridges. According to the Sham-Bancroft equation we determined the C–Sn–C angles in the R<sub>2</sub>Sn(IV) complexes. The range of the values (112-122°) supported the results of the pqs calculations and formation of *mer*-TBP tin(IV) environments.

6. The information collected for the organotin(IV) complexes formed with ligands containing –OH (–C=O) or –COOH group(s) and aromatic {N} donor atom provides the possibility for several comparisons. The analysis of the available data supplied the following statements:
  - (a) For the R<sub>2</sub>Sn(IV) complexes of pyridine carboxylic acids it was found one or two (symmetrical and asymmetrical) νSn–C bands, depending on the type of the butyl group. Such difference does not exist for the R<sub>2</sub>Sn(IV) complexes of hydroxy pyridines and hydroxy quinolines. In their spectra we can assign both Sn–C absorption bands. It strongly suggests that the C–Sn–C bond angle in these compounds is less than 180°.
  - (b) On the basis of their co-ordination chemical properties of ligands 8HK and PIKS are very similar because both of them can form C–O–M–N–C five membered chelate rings. The Δ<sub>m</sub> values of <sup>n</sup>Bu<sub>2</sub>Sn(IV) and <sup>t</sup>Bu<sub>2</sub>Sn(IV)

complex of PIKS are very different (4.26 and 3.31  $\text{mms}^{-1}$ , respectively). Consequently their structures are also different (PBP and  $\text{O}_h$ ). Considering this fact we expected similar differences in case of  $\text{R}_2\text{Sn(IV)}$  complexes of 8HK, but the full agreement of the  $\Delta_m$  values (2.10  $\text{mms}^{-1}$ ) and of the X-ray structural data obtained for these two complexes confirmed their structural identity.

- (c) On the basis of the pqs calculations we showed the variance between the two types of tin(IV) environments [*trans*-TBP ( $|\Delta_{sz}| > 3.00 \text{ mms}^{-1}$ ) or *cis*-TBP ( $|\Delta_{sz}| < 2.00 \text{ mms}^{-1}$ )] in  $\text{Ph}_3\text{Sn(IV)}$  complexes of pyridine carboxylic acids, hydroxy pyridines and hydroxy quinolines.

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