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SHORT COMMUNICATION

$2(Cy_2NH_2)_2SO_4{\cdot}Sn(SO_4)_2Cy_2NH_2Cl$ AND $2(Cy_2NH_2)_2SO_4{\cdot}Sn(SO_4)_22Cy_2NH_2Cl$: SYNTHESIS AND INFRARED STUDY

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Abstract: $2(Cy_2NH_2)_2SO_4{\cdot}Sn(SO_4)_2Cy_2NH_2Cl$ and $2(Cy_2NH_2)_2SO_4{\cdot}Sn(SO_4)_22Cy_2NH_2Cl$ complexes have been obtained on allowing $Cy_2NH_2O_2C\text{-}SO_3H$ and $Cy_2NH_2HSO_4$ to react respectively with $SnBu_2Cl_2$ and $SnBu_3Cl$ in specific ratios.The molecular structures of these compounds have been determined on the basis of the infrared data. The suggested structures are dimeric, the tin atom being octacoordinated by four chelating sulfate anions, the monomeric basic entities being connected by hydrogen bonded cation chloride cation strings.

Keywords: *chelating sulfate, dimer, hexacoordinated, monomer, octacoordinated*

INTRODUCTION

Many applications concerning the organotin (IV) compounds have been displayed by Evans and Karpel [1]. For these reasons and further biological tests, many research groups attempt to synthesize new molecules of tin family [2-7]. Our group which is one of these has yet published many papers during these past decades [8-15].
In this work, we have initiated the study of the interactions between $SnBu_2Cl_2$ and $Cy_2NH_2CO_2SO_3H$ on one hand and, $SnBu_3Cl$ and $Cy_2NH_2HSO_4$ on the other hand, which have yielded $2(Cy_2NH_2)_2SO_4{\cdot}Sn(SO_4)_2Cy_2NH_2Cl$ and $2(Cy_2NH_2)_2SO_4{\cdot}Sn(SO_4)_2$ $2Cy_2NH_2Cl$, infrared studies of which have been carried out, then structures suggested on the basis of spectroscopic data.

EXPERIMENTAL

$2(Cy_2NH_2)_2SO_4{\cdot}Sn(SO_4)_2Cy_2NH_2Cl$ and $2(Cy_2NH_2)_2SO_4{\cdot}Sn(SO_4)_22Cy_2NH_2Cl$ have been prepared in two levels. At the first level imino(amino)methanesulfonic acid and sulfuric acid solutions have been mixed with dicyclohexylamine both in 1/1 ratio in water. After solvent evaporation at 60 °C, $Cy_2NH_2CO_2SO_3H$ and $Cy_2NH_2HSO_4$ were obtained as white crystals. When these two salts as ethanolic solutions are respectively added in $SnBu_2Cl_2$ and $SnBu_3Cl$ ethanolic solutions respectively in 2/1 and 1/2 ratios, clear solutions are obtained, stirred during two hours. When submitted to a slow solvent evaporation, these solutions yield white powders.
Table 1 presents the elemental analysis of **A** and **B** compounds.

Table 1. *Results of the elemental analyses*

Compound	Chemical formula	Elemental analysis (%)					
		C		H		N	
		calc.	found	calc.	found	calc.	found
A	$2(Cy_2NH_2)_2SO_4{\cdot}Sn(SO_4)_2$ Cy_2NH_2Cl	49.89	49.77	8.77	9.17	4.85	4.85
B	$2(Cy_2NH_2)_2SO_4{\cdot}Sn(SO_4)_2$ $2Cy_2NH_2Cl$	51.87	50.62	8.65	9.41	5.04	6.29

The elemental analyses have been performed at the laboratory of Microanalyses at the University of Bath (UK).
The infrared spectra was recorded by a Perkin Elmer 4400 spectrometer (Dakar University), the sample being as Nujol mulls while Csl windows were used.
Infrared data are given in cm^{-1}. IR abbreviations: br (broad), (vs) very strong, (s) strong, (m) medium, (sh) shoulder, (vw) very weak.
All the chemicals are from Aldrich Company and were used without any further purification**.**

RESULTS AND DISCUSSION

Let us consider the infrared data:
A: ν_3: 1114 (vs), 1055 (sh), 1033 (sh); ν_4: 670 (sh), 618 (m), 588 (sh); ν (OH) + δ (OH): 3400 (br) + 1570w;
B: ν_3: 1122 (vs), 1067 (sh); ν_1: 989 (vw); ν (OH) + δ (OH): 3397 (br) + 1565w.
For **A** compound, the absence of ν_1 and the weak splitting of ν_3 band indicate a sulfate of Td symmetry. This allows suggesting a dimeric structure in which each the tin atom is octacoordinated by four chelating sulfate anions. $Cy_2NH_2^+$….Cl…..Cy2NH_2^+ strings connect then the monomeric species (Figure 1).

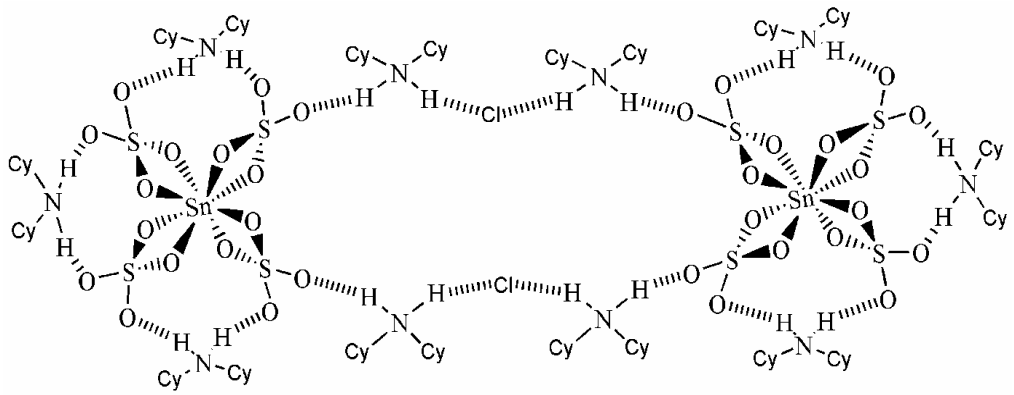

Figure 1. *Proposed structure for the compound* ***A***

The **B** compound derives from **A** on adding Cy_2NH_2Cl. As in **A**, the apparition of ν_1 band as very weak band and the small splitting of ν_3 band indicate Td symmetry for the anion and allow to suggest a structure similar to **A** the string being $Cy_2NH_2^+$….Cl…..$Cy_2NH_2^+$….Cl….Cy_2NH_2 (Figure 2).

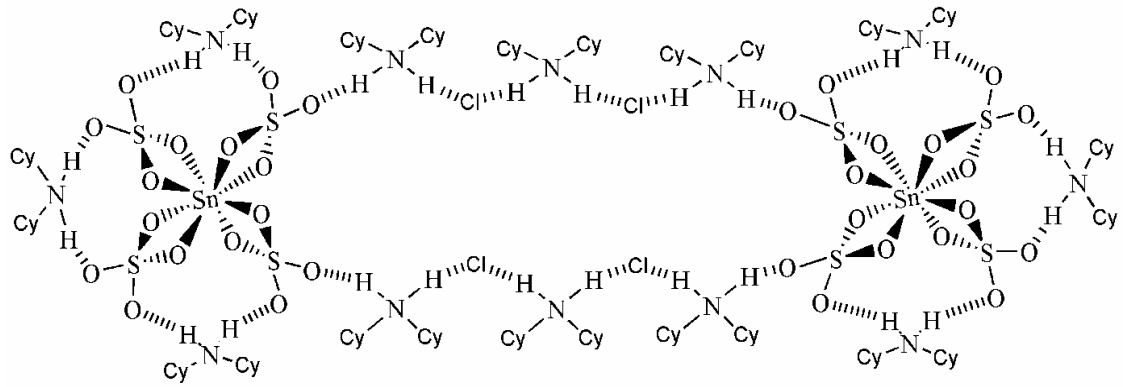

Figure 2. *Proposed structure for the compound* ***B***

CONCLUSIONS

The studied complexes are dimeric, the tin centre being hexacoordinated, strings connecting the the monomeric basic entities.

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