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SHORT COMMUNICATION

ON THE EXISTENCE OF $[SnBu_3Cl_2]^-$ IN $(Cy_2NH_2)_2C_2O_4 \cdot SnBu_3Cl$ AND $Cy_2NH_2O_2PHPh \cdot SnBu_3Cl$: A SPECTROSCOPIC CHARACTERIZATION

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Abstract: On allowing $(Cy_2NH_2)_2C_2O_4 \cdot 2H_2O$ and $Cy_2NH_2PhHPO_2$ to react with $SnBu_3Cl$ in ethanolic media, $(Cy_2NH_2)_2C_2O_4 \cdot SnBu_3Cl$ and $(Cy_2NH_2)O_2PHPh \cdot SnBu_3Cl$ are obtained. Their spectroscopic studies mainly the Mössbauer technique, exhibit the presence of two tin (IV) centres allowing to consider the formulae as $[SnBu_3Cl_2][SnBu_3(C_2O_4)_2][Cy_2NH_2]_4$ or $[SnBu_3Cl_2][(SnBu_3)_2(PhHPO_2)_3][Cy_2NH_2]_2$ containing $[SnBu_3Cl_2]^-$ and $[SnBu_3(C_2O_4)_2]^{3-}$ or $[(SnBu_3)_2(PhHPO_2)_3]^-$ all three being dimeric entities with NH·····Cl and NH·····O hydrogen bonds involving the cations. The spectroscopic characterization of $[SnBu_3Cl_2]^-$ has been done for the first time in this work.

Key words: *hydrogen bonds, IR, Mössbauer, trans coordinated $SnBu_3$ residue*

INTRODUCTION

Many compounds containing organostannic anions such as $[SnMe_3Cl_2]^-$ and $[SnPh_3Cl_2]^-$ have yet been reported [1 – 9]. To the best of our knowledge only $Ph_4PSnBu_3Cl_2$ has been reported by Molloy *et al.* [10] to contain $[SnBu_3Cl_2]^-$. In the dynamic of our seek for new organotin (IV) compounds, we have initiated here, the study of the interactions between $SnBu_3Cl$ and $(Cy_2NH_2)_2C_2O_4{\cdot}2H_2O$ or $Cy_2NH_2PhHPO_2$ which have yielded the title compounds, infrared and Mössbauer studies of which have been carried out, and structures suggested on the basis of the spectroscopic data.

EXPERIMENTAL

The oxalic and $PhHPO_2H$ acid salts $(Cy_2NH_2)_2C_2O_4{\cdot}2H_2O$ and $Cy_2NH_2PhHPO_2$ have been obtained on mixing $H_2C_2O_4{\cdot}2H_2O$ acid or $PhHPO_2H$ with dicyclohexylamine in aqueous solution in 2/1 and 1/1 ratio. The mixture of $(Cy_2NH_2)_2C_2O_4{\cdot}2H_2O$ or $Cy_2NH_2PhHPO_2$ with $SnBu_3Cl$ in EtOH in the ratio 1/2 gives a white precipitate, stirred no less than two hours, filtered and dried under P_2O_5. The analytical data reported below, allow to suggest the following formulae: % calculated (% found)

A $(Cy_2NH_2)_2C_2O_4{\cdot}SnBu_3Cl$: C = 58.24 (58.45); H = 10.87 (09.61); N = 4.00 (3.58)

B $(Cy_2NH_2)O_2PHPh{\cdot}SnBu_3Cl$: C = 55.53 (56.94); H = 8.85 (8.40); N = 2.16 (2.16)

The elemental analyses were performed at the Microanalytical Centre, University of Bath (UK). The infrared spectra were recorded at the University Cheikh Anta Diop, Dakar (Senegal) and the University of Padova (Italy), using a PE 580 (4000 – 200 cm^{-1}) or a FTIR-Brucker FTIR spectrometer, the sample being as Nujol mulls, the windows being CsI. Mössbauer spectra were obtained as described previously [11]. Infrared data are given in cm^{-1} [abbreviations: (vs) very strong, (s) strong, (m) medium, (w) weak)]. Mössbauer parameters are given in $mm{\cdot}s^{-1}$ (abbreviations: Q.S. = quadrupole splitting, I.S. = isomer shift, Γ = full width at half-height). All the chemicals were from Aldrich or Merck Companies and used without any further purification.

RESULTS AND DISCUSSION

Let us consider the infrared in cm^{-1} of:

A: $vasCOO^-$ = 1636vs; $vsCOO^-$ = 1311vs; δCOO^- = 784m.

B: νPH = 2350m; νPO_2 = 1160w, 1140vs, 1060s; ρPO_2 = 365m, 320w; δPO_2 = 560s; $vasSnC_3$ = 640s; $vsSnC_3$ = 617w; $\nu SnCl$ = 210m; νSnO = 300w.

and the Mössbauer data of:

A: $\Delta E_1 = 4.23$; $\Delta E_2 = 3.12$; $\delta_1 = 1.59$; $\delta_2 = 1.48$; $\Gamma_1 = 1.03$; $\Gamma_2 = 1.04$; Sn(1)/Sn(2) = 1.

B: $\Delta E_1 = 3.15$; $\Delta E_2 = 4.22$; $\delta_1 = 1.20$; $\delta_2 = 1.40$; $\Gamma_1 = 1.38$; $\Gamma_2 = 0.93$; Sn(1)/Sn(2) = 2.

The values of the QS indicate two *trans* coordinating $SnBu_3$ residues according to Platt *et al.* [12, 13] allowing to suggest the presence of $[SnBu_3Cl_2]^-$ and $[SnBu_3(C_2O_4)_2]^{3-}$, $[(SnBu_3)_2(PhHPO_2)_3]^-$ all these anions containing *trans* coordinated $SnBu_3$ residues.

Let us notice the similarity of the QS values of $[SnBu_3Cl_2]^-$ (3.15 and 3.12) in both adducts. The same similarity is also observed for the O *trans* coordinated $SnBu_3$

residues in terms of QS values. The complex **A** can be considered as a tetramer involved in a rearrangement process leading to two $[SnBu_3Cl_2]^-$ and one $[(SnBu_3)_2(C_2O_4)_4]^{2-}$ allowing to suggest the structure reported on Figure 1.

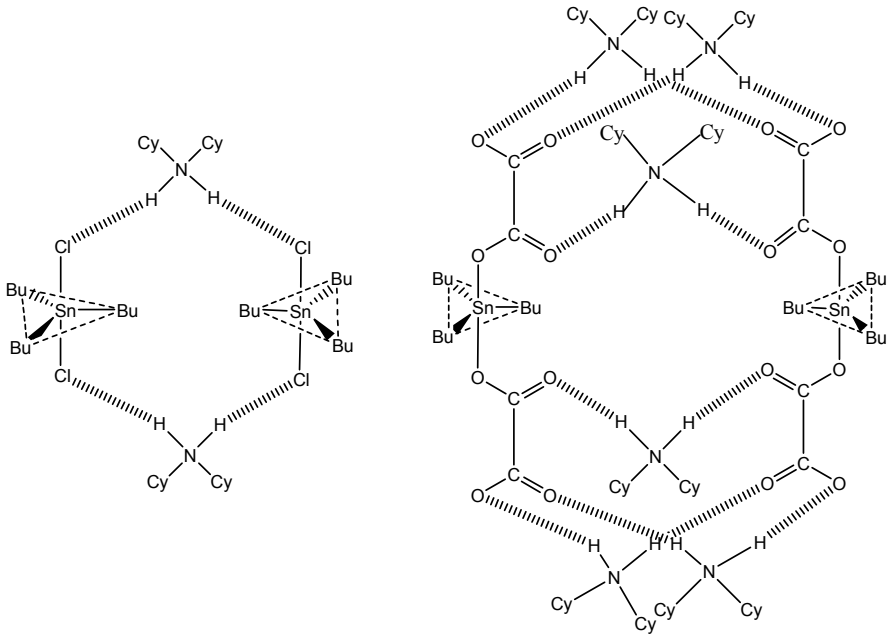

Figure 1. *Suggested structures for* ***A***

The complex **B** can be considered as an hexamer involved in a rearrangement leading to two $[SnBu_3Cl_2]^-$ and one $[(SnBu_3)_4(PhHPO_2)_6]^{4-}$ allowing to suggest the structure reported on Figure 2.

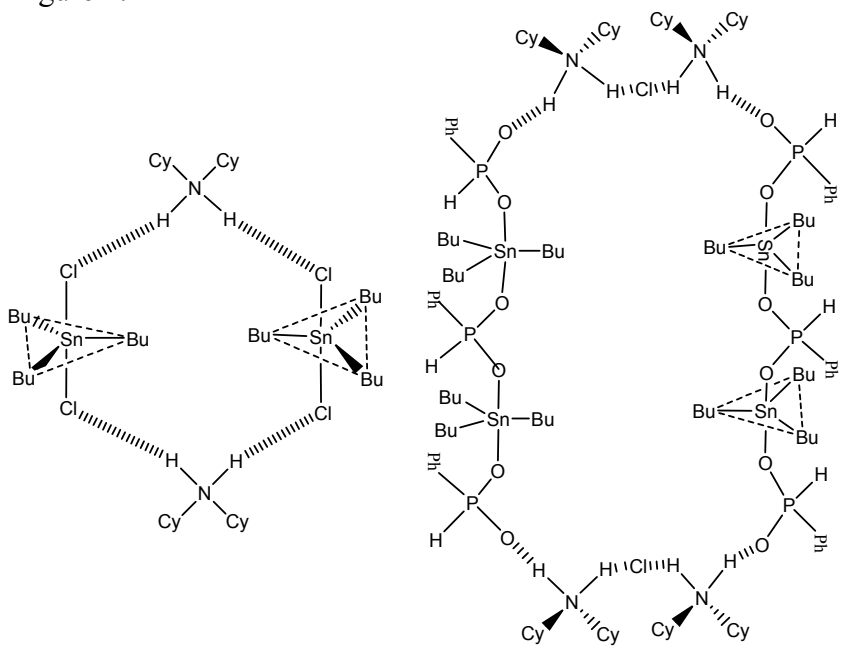

Figure 2. *Suggested structures for* ***B***

CONCLUSION

The title adduct is dimeric with two $[SnBu_3Cl_2]^-$ anions linked as a dimer by through N–H····Cl hydrogen bonds and another pair of $[SnBu_3(C_2O_4)_2]^{3-}$ or $[(SnBu_3)_4(PhHPO_2)_6]^{4-}$ anions connected by cations through N–H····O hydrogen bonds.

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