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SHORT COMMUNICATION

$(Me_4N)_2SO_4 \cdot 4SnPh_3NCS$ AND $(Me_2N)_2CO \cdot 4SnPh_3Cl$ SYNTHESIS AND SPECTROSCOPIC STUDIES

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Abstract: Two tetranuclear $SnPh_3$ residue containing adducts have been synthesized and characterized by infrared and Mössbauer spectroscopies. The structure are both discrete, the sulphate anion and the tetramethyl NN' urea (TMU) acting as tetracoordinating ligands, the environment around the tin IV centres being *trans* trigonal bipyramidal.

Keywords: *tetracoordinating sulphate and tetramethyl NN' urea, tetranuclear adducts, trans coordinated* $SnPh_3$ *residue, Mössbauer, IR*

INTRODUCTION

The antitumor activity of $PhCO_2SnPh_3$ found by Gielen *et al.* [1] has brought many research groups to focus on obtaining new $SnPh_3$ residue compounds [2 – 8]. Interesting results on anti malaria tests and cardio-vascular properties have been reported for some $SnPh_3$ residue containing compounds [9 – 11]. The adduct NN' tetramethyl urea·$SnMe_2Cl_2$ in which the ligand is behaving as a monocoordinating oxygen donor, has been reported [12]. In the framework of our research work on organotin (IV) chemistry, seeking for new $SnPh_3$ residue containing compounds, we have yet published several papers including $SnPh_3NCS$ and $SnPh_3Cl$ adducts [13, 14] and initiate here the study of the interactions between $(Me_4N)_2SO_4{\cdot}H_2O$ and $SnPh_3NCS$ in one side, NN' tetramethyl urea (TMU) and $SnPh_3Cl$ in the other side. These interactions have yielded two new tetranuclear adducts, infrared and Mössbauer studies of which have been carried out, and structures suggested on the basis of spectroscopic data.

EXPERIMENTAL

$(Me_4N)_2SO_4{\cdot}H_2O$ has been obtained on neutralizing completely sulphuric acid with a 20% aqueous solution of Me_4NOH. The powder obtained after water evaporation has been recrystallized from EtOH. % Calculated (% found) C = 36.62 (36.50); H = 9.99 (10.00); N = 10.68 (10.52).

$SnPh_3NCS$ has been obtained as a powder on mixing $SnPh_3Cl$ with KSCN both as ethanolic solutions, filtering off the KCl precipitate and allowing a slow solvent evaporation. On allowing $(Me_4N)_2SO_4{\cdot}H_2O$ and $SnPh_3NCS$ to react both as ethanolic solutions in ratio 1/1 a clear solution is obtained; after a slow solvent evaporation a powder [A] is obtained. When ethanolic solutions of TMU and $SnPh_3Cl$ are mixed in ratio 1/2, [B] is obtained as powder after a slow solvent evaporation. The elemental analyses {% calculated (% found) C = 53.76 (53.83); H = 4.51 (4.85); N = 4.48 (4.42); Sn = 25.30 (25.22) for [A]; C = 55.78 (55.59); H = 4.38 (4.32); N = 1.69 (1.67); Cl = 8.55 (8.45); Sn =28.64 (28.45) for [B]} has allowed to suggest $(Me_4N)_2SO_4{\cdot}4SnPh_3NCS$ and NN'tetramethyl urea·$4SnPh_3Cl$ as formulae. The IR spectra have been obtained at the University of Padova (Italy) on a PE580 and a Bruker FTIR spectrometers, the sample being as Nujol mulls, the windows being CsI or polyethylene while the Mössbauer spectra have been obtained as yet reported in [15].

The IR data are given in cm^{-1}, the Mössbauer ones in $mm{\cdot}s^{-1}$. Infrared abbreviations: strong doublet (sd), very strong (vs), medium (m), weak (w), shoulder (sh). Mössbauer abbreviations: IS = isomer shift; QS = quadrupole splitting; Γ = width at half height.

The elemental analyses were performed by the CNRS "Service Central d'Analyses" Vernaison –France. All the chemicals were obtained from Aldrich or Merck companies and used without any further purification.

RESULTS AND DISCUSSION

Let us consider the main IR bands with their assignments:
[A]: νCN = 2091 vs, $\nu_2 SO_4^{2-}$ = 441 sh, $\nu_3 SO_4^{2-}$ = 1232 s, 1130 vs, $\nu_4 SO_4^{2-}$ = 632 m, 607 m, $\nu_{as}SnC_3$ = 276 vs, $\nu_s SnC_3$ = 212 w, νSnN = 240 m, νSnO = 201 m;
[B]: νCO = 1651sd, νCN_2 =1155 m, $\nu_{as}SnC_3$ =273 vs, $\nu_s SnC_3$ =214 w, νSnO = 202 m and Mössbauer data of the two adducts; [A]: IS = 1.31 $mm \cdot s^{-1}$, QS = 3.36 $mm \cdot s^{-1}$, Γ = 0.93 $mm \cdot s^{-1}$ and [B]: IS = 1.26$mm \cdot s^{-1}$; QS = 2.85 $mm \cdot s^{-1}$; Γ = 0.98 $mm \cdot s^{-1}$. The non splitting of $\nu_3 SO_4^{2-}$ or $\nu_4 SO_4^{2-}$and the absence of $\nu_1 SO_4^{2-}$ are indications of a Td symmetry of the sulphate anion according to Group Theory [16] while the values of the quadrupole splitting indicate the presence of trans coordinated $SnPh_3$ residues in the two adducts according to Platt *et al.* [17]. The structures consistent with these spectroscopic data are discrete and contain both tetracoordinating ligands - sulphate and TMU - (Figures 1 and 2).

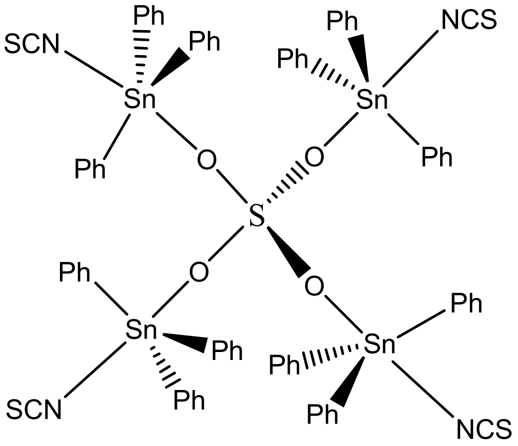

Figure 1.

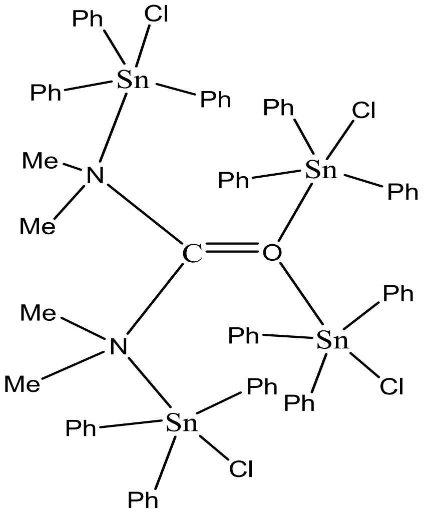

Figure 2.

The coordination of SCN^- in $SnPh_3NCS$ through N bond has been found in the complex-anion $[SnPh_3(NCS)_2]^-$ which have yet been obtained by several authors and in mononuclear $SnPh_3NCS$ adducts reported by Prasad and Smith [18], Kemmer *et al.* [19], Gabe *et al.* [20].

CONCLUSION

The two tetranuclear adducts studied have discrete structure, the sulphate and the NN' tetramethylurea behaving as a tetracoordinating ligand, $SnPh_3Cl$ and $SnPh_3NCS$ being monocoordinated, the environment around the tin centre being in all cases trigonal bipyramidal.

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