

NH₃CH₂CH₂NH₃PhCO₂SnPhCl₃OH, AN UNEXPECTED ADDUCT: SYNTHESIS AND SPECTROSCOPIC STUDIES

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Abstract: On allowing NH₃CH₂CH₂NH₃(PhCO₂)₂ in slightly hydrated methanol to react with SnPh₂Cl₂, NH₃CH₂CH₂NH₃PhCO₂SnPhCl₃OH is obtained as a white precipitate. The IR and Mössbauer studies allow to suggest a moiety obtained by addition of OH⁻ and PhCO₂⁻ to SnPhCl₃, the environment around the tin being octahedral, PhCO₂⁻ behaving as a monocoordinating ligand.

Keywords: *phenyl tin compound, benzoate, infrared spectrum, Mössbauer spectrum, polymeric structure*

INTRODUCTION

In the framework of our seek of new organo-tin compounds because of the antitumor activity of some SnR_2 ($\text{R} = \text{Bu}, \text{Ph}, \text{Bz}$) residue containing compounds [1 – 3] and industrial applications [4] we have yet published various papers [5 – 8] and report here studies on the interactions between $\text{NH}_3\text{CH}_2\text{CH}_2\text{NH}_3(\text{PhCO}_2)_2$ and SnPh_2Cl_2 . The unexpected adduct obtained ($\text{NH}_3\text{CH}_2\text{CH}_2\text{NH}_3\text{PhCO}_2\text{SnPhCl}_3\text{OH}$) is studied by IR and Mössbauer techniques and structures suggested on the basis of spectroscopic data.

EXPERIMENTAL

$\text{NH}_3\text{CH}_2\text{CH}_2\text{NH}_3(\text{PhCO}_2)_2$ (L) is obtained on mixing ethylenediamine (Merck) with PhCO_2H (Merck) in water in 1/2 ratio [analytical data of (L) - calculated (found) - %C = 63.09 (63.13); %H = 6.57 (6.14); %N = 9.20 (9.28)]. When (L) is mixed with SnPh_2Cl_2 (ratio 1/1), both in slightly hydrated methanol, a white precipitate occurs which is stirred around two hours. The analytical data of the adduct - calculated (found) - %C = 35.82 (35.70); %H = 4.18 (4.20); %N = 5.57 (5.69); %Cl = 21.12 (20.92); %Sn = 23.62 (24.01), allow to propose $\text{NH}_3\text{CH}_2\text{CH}_2\text{NH}_3\text{PhCO}_2\text{SnPhCl}_3\text{OH}$ as formula. All the chemicals are from Merck Company and were used without further purification. The elemental analyses were performed by the « Service Central d'Analyses » CNRS, Vernaison, France and the Faculty of Chemistry, University of Konstanz, Germany. The IR spectra were obtained as Nujol mulls with a PE 580 spectrometer ($4000 - 200 \text{ cm}^{-1}$) and a FTIR Nicolet ($600 - 50 \text{ cm}^{-1}$) using CsI or polyethylene windows; the Mössbauer spectrum was obtained as described previously [10]. Infrared data are given in cm^{-1} [abbreviations: (br) broad, (s) strong, (m) medium, (sh) shoulder, (w) weak]. Mössbauer parameters are given in $\text{mm}\cdot\text{s}^{-1}$ (abbreviations: Q.S. = quadrupole splitting, I.S. = isomer shift, Γ = full width at half-height).

RESULTS AND DISCUSSION

Let us consider the more relevant IR bands and their assignments ($\nu_{\text{asCOO}^-} = 1595 \text{ vs}$; $\nu_{\text{sCOO}^-} = 1307 \text{ m}$; $\delta\text{NH}_3 = 1507 \text{ vs}$; $\nu_{\text{SnC}} = 341 \text{ vs}$; $\nu_{\text{SnCl}_3} = 249 \text{ vs}$; $\nu_{\text{SnO}} = 231 \text{ vs}$) and its Mössbauer data (I.S. = $0.55 \text{ mm}\cdot\text{s}^{-1}$; Q.S. = $2.19 \text{ mm}\cdot\text{s}^{-1}$; $\Gamma = 1.06 \text{ mm}\cdot\text{s}^{-1}$).

The quadrupole splitting of $\text{PhSnCl}_3(\text{Ph}_3\text{PO})_2$ is $2.01 \text{ mm}\cdot\text{s}^{-1}$ with an octahedral environment around tin centre [11]. The structure of the compound is built on octahedral moieties $[\text{SnPhCl}_3\text{OHPhCO}_2]^{2-}$ which can be considered as an adduct of SnPhCl_3 , PhCO_2^- and OH^- occupying *cis* or *trans* positions:

- in *cis* positions an hydrogen bond between PCO_2^- and OH^- seems plausible: the deriving structure is an infinite chain as in [12] or an oligomer (a tetramer as in [13] or a hexamer as in [14]) in which the octahedral moieties are connected by cations through $\text{NH}\cdots\text{Cl}$ hydrogen bonds (Figure 1);
- in *trans* positions the structure is also an infinite chain or an oligomer in which the octahedral moieties are connected by cations through $\text{NH}\cdots\text{O}$ hydrogen bonds (Figure 2).

However a rearrangement can involve [SnPhCl₃(OH)₂]²⁻ and [SnPhCl₃(O₂CPh)₂]²⁻ as connected complex anions and gives similar types of structure than above.

N-H---Cl, N-H---O and O-H---O hydrogen bonds appear on the IR spectrum as a wide and strong absorption around 3000 cm⁻¹.

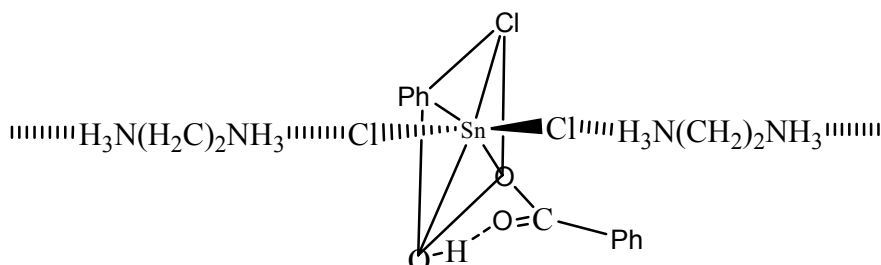


Figure 1. Suggested structure for *cis* adduct

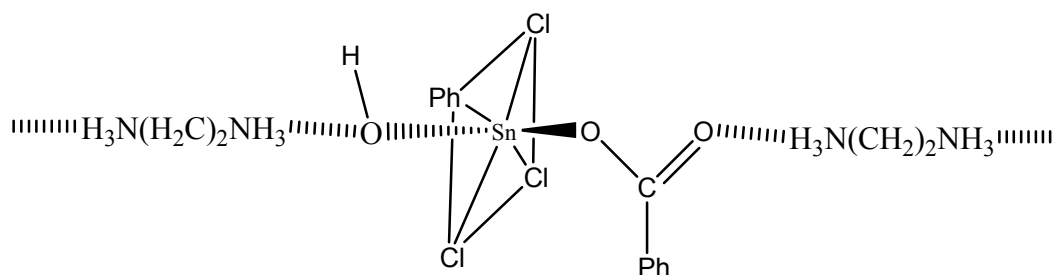
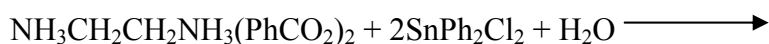


Figure 2. Suggested structure for *trans* adduct

The equation of the reaction is:



SnPh₃OH and PhCO₂H can react to give SnPh₃O₂CPh which remains in solution.

CONCLUSION

In the unexpected SnPhCl₃ adduct obtained, the environment around the tin centre is octahedral, the benzoate anion behaving as a monocoordinating anion; secondary interactions through hydrogen bonds can give an infinite chain or an oligomer as structures.

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