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SHORT COMMUNICATION

$Me_4NOH(SnX_5NMe_4)_2$ (X = Cl, Br): SYNTHESIS, IR AND MÖSSBAUER STUDIES

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Abstract: Allowing Me_4NOH to react with SnX_4 (X = Cl, Br), yields $Me_4NOH(SnX_5NMe_4)_2$. Their IR and Mössbauer studies have allowed suggesting supramolecular structures, the environment around the tin center being octahedral.

Keywords: *IR, Mössbauer, pentahalotin (IV) adducts, spectroscopy, supramolecular structures*

INTRODUCTION

If many $[SnCl_5]^-$ complex-anion containing adducts [1-4] or $SnCl_3OH{\cdot}THF$ [5] were reported, no complex containing $[OH\ldots SnCl_5]^{2-}$ have been found in the literature. We have initiated this study for understanding the behavior of MeNOH towards $SnCl_4$, expecting an addition or a partial substitution of the chloride by OH^- and have obtained to adducts containing $[SnX_5OH]^{2-}$ (X = Cl, Br) what indicates the appearance of Me_4NX *in situ*. The infrared and Mössbauer studies of the compounds have been carried out and structures suggested on the basis of spectroscopic data.

EXPERIMENTAL

On allowing Me_4NOH as ethanolic solution to react with SnX_4 (X = Cl, Br) in benzene a white (X = Cl) or yellow precipitate (X = Br) is obtained, stirred no less than two hours and filtered. Their analytical data have allowed us to suggest the title formulae, $Me_4NOH(SnX_5NMe_4)_2$.
The reaction equation is:

$$3Me_4NOH + 3SnX_4 \rightarrow Me_4NOH{\cdot}2SnX_5NMe_4 + SnX_2(OH)_2$$

Analytical data of synthesized compounds are presented in Table 1.

Table 1. *Analytical data for the synthesized $Me_4NOH(SnX_5NMe_4)_2$*

Compound	C		H		N		X		Sn	
	C	***F***	***C***	***F***	***C***	***F***	***C***	***F***	***C***	***F***
A (X = Cl)	17.32	17.40	4.33	4.35	5.05	5.10	42.70	42.50	28.55	28.32
B (X = Br)	11.28	11.34	2.82	2.91	3.29	3.33	62.67	62.54	18.59	18.70

C - % calculated; F - % found.

All the chemicals are from Aldrich Company and were used without any further purification. The infrared spectra were recorded by a PE 580 (4000 – 200 cm^{-1}) or a FTIR-Nicolet (600 – 50 cm^{-1}) spectrometer, at the University of Padova, Italy, the sample being as Nujol mulls while CsI or polyethylene windows were used. Mössbauer spectra were obtained as described previously [6]. Infrared data are given in cm^{-1}; abbreviations: (vs) very strong, (s) strong, (m) medium, (sh) shoulder. Mössbauer parameters are given in $mm{\cdot}s^{-1}$; abbreviations: Q.S = quadruple 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:
(**A**) νOH = 3545 m; δOH = 1647 m; $\nu SnCl_5$ = 300 s;
(**B**) νOH = 3550 m; δOH = 1650 m; νSn-O = 312 m; $\nu SnBr_5$ = 200 s;
and their Mössbauer data:
(**A**) I.S. = 0.25 mm/s; Q.S. = 0.49 mm/s; Γ =1.07 mm/s;
(**B**) I.S. = 0.64 mm/s; Q.S. = 0.71 mm/s; Γ = 0.93 mm/s.

The intensities and the frequencies of the νSnX_5 are similar to those yet reported in [7]. The I.S. values lower than those reported for SnX_6TMN_2 [8] as expected when and oxygen atom substitutes a halogen.
The presence of the absorption at 3395 cm^{-1} on the two IR spectra are indicative of the presence of O-H....X hydrogen bond that allows to suggest while ignoring the secondary interactions, the structure reported in Figure 1. While considering O-H....X hydrogen bonds, supramolecular architectures are obtained, a string of which is reported on Figure 2.

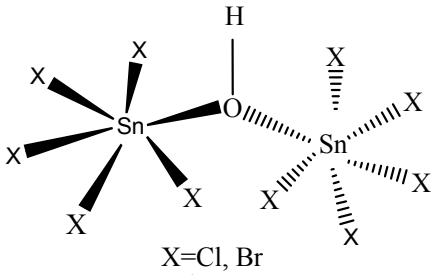

Figure 1.

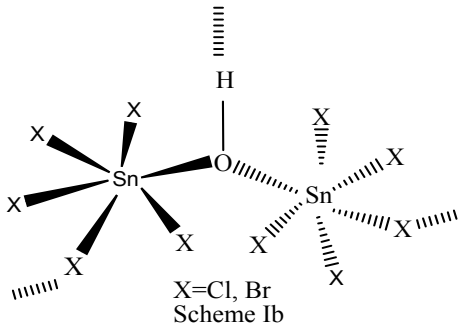

Figure 2.

CONCLUSION

The appearance in situ of Me_4NX leading to the pentahalotin (IV) anion SnX_5^- is noteworthy. When secondary interactions are involved as hydrogen bonds, supramolecular structures are obtained.

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