

SOME NEW SULFONATO ADDUCT: SYNTHESIS AND SPECTROSCOPIC STUDIES

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Abstract: Three new adducts have been synthesized and studied by infrared and NMR spectroscopies. The suggested structures are discrete with a pyridine -3- sulfonate acting as a tri O-chelating and N-donor or as a non σ coordinating ligand, a 4-aminobenzenesulfonate behaving as a monodentate O-donor, the environments around the tin centre being tetrahedral, octahedral or seven coordinated. In all the studied compounds, supramolecular architectures are obtained when hydrogen bonds are considered.

Keywords: *discrete structures, hydrogen bonds, monodentate 4-aminobenzenesulfonate, octahedral, seven coordinated environments, supramolecular architectures, tetrahedral, tri O-chelating and N-donor pyridine -3 - sulfonate*

INTRODUCTION

Substituted and non substituted anions are known to coordinate very well transition and non transition metals as reported by Hathaway [1]. Since several decades our group is focusing in the coordinating ability of oxyanions and has published several papers [2-5]. As a continuation of that research we have initiated in this work the study of the interactions between $(\text{Bu}_2\text{NH}_2)\text{Py-3-SO}_3$ and SnBu_2Cl_2 or $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ which have yielded three new adducts, infrared and NMR studies of which have been carried out then structures suggested on the basis of infrared data.

MATERIALS AND METHODS

The compound **1** is obtained by allowing an ethanolic solution of $(\text{Bu}_2\text{NH}_2)\text{Py-3-SO}_3$ – obtained as a powder on allowing Bu_2NH to react with pyridine-3-sulfonic acid in water and allowing the water to evaporate in the oven at 60°C to react in 1/1 ratio with an ethanolic solution of SnBu_2Cl_2 , while compounds **2** and **3** were obtained by mixing ethanolic solutions of $(\text{Bu}_2\text{NH}_2)\text{Py-3-SO}_3$ and $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ in 2/1 ratio (**2**), ethanolic solutions of $\text{CyNH}_3\text{NH}_2\text{C}_6\text{H}_4\text{SO}_3$ and ZnBr_2 in 1/1 ratio (**3**). All the mixtures were stirred around two hours then filtered before being submitted to a slow solvent evaporation. The analytical data calculated (found) have allowed to suggest the following formulae (Table 1).

Table 1. Suggested formulae of the synthesized compounds

Comp	Suggested formulae	Chemical composition (% mass)					
		C		H		N	
		Calc.	Found	Calc.	Found	Calc.	Found
1	$\text{Py-3-SO}_3\text{Bu}_2\text{NH}_2 \cdot 3/2\text{SnBu}_2\text{Cl}_2$	40.35	41.06	6.86	7.12	3.76	3.84
2	$4\text{Py-3-SO}_3\text{Bu}_2\text{NH}_2 \cdot \text{SnCl}_4 \cdot 2\text{EtOH}$	44.90	45.18	7.21	6.56	49	7.69
3	$\text{CyNH}_3\text{NH}_2\text{C}_6\text{H}_4\text{SO}_3 \cdot \text{ZnBr}_2 \cdot \text{H}_2\text{O}$	27.95	27.29	4.30	4.13	5.43	5.68

The elemental analyses have been obtained from the “Laboratoire de Mesures Physiques” Montpellier II University (France). The IR spectra were performed at the University of Saint Boniface-Winnipeg (Canada). The IR data are given in cm^{-1} [abbreviations: (vs) very strong, (s) strong, (m) medium, (w) weak]. The ^1H NMR analyses were performed at the “Laboratoire de Mesures Physiques” at Montpellier II University. NMR spectra were recorded as saturated CDCl_3 or DMSO at room temperature, using a Bruker 300 MHz spectrometer. The ^1H was measured at 300.13 MHz. ^1H chemical shifts NMR are given in ppm and are referred respectively to TMS [NMR abbreviation: m (multiplet)].

All the chemical were from ALDRICH Company and used as received.

RESULTS AND DISCUSSION

Let us consider the IR data in cm^{-1} of the three adducts.

1: $\nu_{\text{as}}(\text{SO}_3)$: 1257 (s), 1209 (s), 1187 (s), 1143 (s); $\nu_{\text{s}}\text{SO}_3$: 1043 (s), 1022 (s); $\nu(\text{C-S})$: 742 (s); $\delta(\text{SO}_3)$: 875 (s), 821 (w), 777 (s), 683.17 (s), 632 (s); $\nu_{\text{as}}(\text{SnBu}_2)$: 611 (vs);

2: $\nu_{\text{as}}(\text{SO}_3)$: 1200 (s), 1176 (s); $\nu_{\text{s}}(\text{SO}_3)$: 1043 (s); $\nu(\text{C-S})$: 745 (s); $\delta(\text{SO}_3)$: 632 (m);

3: $\nu_{\text{as}}(\text{SO}_3)$: 1243 (vs), 1163 (vs), 1122 (vs); $\nu_{\text{s}}(\text{SO}_3)$: 1085 (vs), 1036 (vs), 1012 (s); $\nu(\text{C-S})$: 694 (s); $\delta(\text{SO}_3)$: 616 (w), 580 (s).

It seems worthy to outline the absence of the bands at 683 cm^{-1} and 611 cm^{-1} due to SnBu_2 on the spectrum of **2**. The intensity of the band at 611 cm^{-1} indicates non linear SnBu_2 group.

Let us consider the ^1H NMR (CDCl_3 or DMSO, ppm) data of adducts **1** and **2**.

1: δ 7.34-7.92 (m, C-H_{Py}), δ 8.44-8.74 (m, NH_2);

2: δ 7.60-7.81 (m, C-H_{Py}), δ 8.39-8.90 (m, NH_2) (the ^{119}Sn NMR spectra being taken in DMSO, the solvent coordinates the complex; so the NMR data cannot be correlated with the suggested structure).

From these spectroscopic data, we suggest:

For **1** we suggest a dimeric structure (Figure 1) with a pyridine-3-sulfonate anion tri O-chelating and N-donor, the environment around the tin centre being octahedral or seven coordinated.

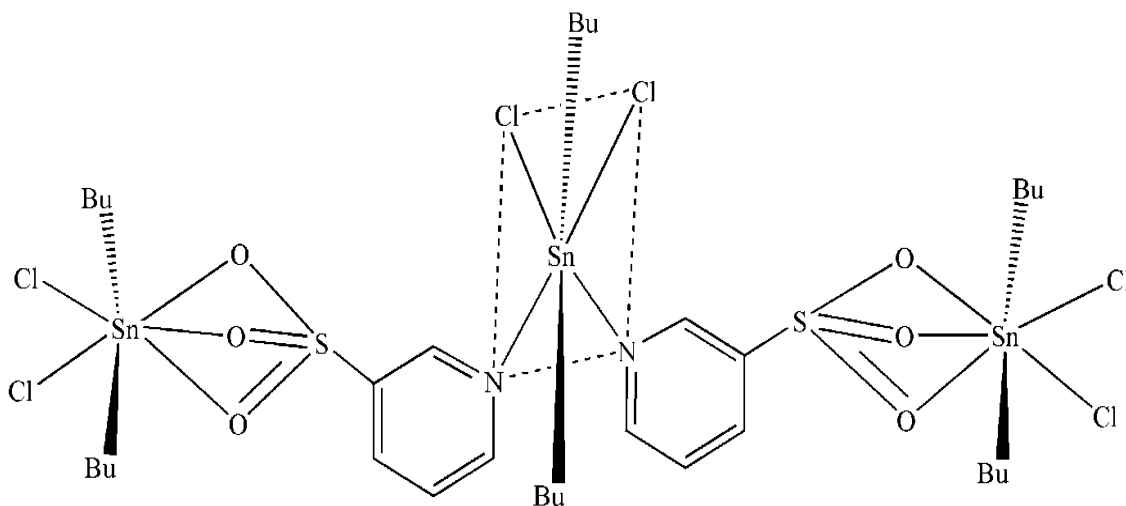


Figure 1. Proposed structure for the compound **1**

For **2** the suggested structures are discrete with a SnCl_4 molecule coordinated by two ethanol molecules conferring an octahedral environment around the tin centre, the $\text{SnCl}_4 \cdot 2\text{EtOH}$ adduct interacting through $\text{OH} \cdots \text{N}$ hydrogen bonds with the dimeric cycles of $\text{Bu}_2\text{NH}_2\text{Py-3-SO}_3$ (Figure 2a) or $\text{NH} \cdots \text{Cl}$ hydrogen bonds with $\text{Bu}_2\text{NH}_2\text{Py-3-SO}_3$ (Figure 2b).

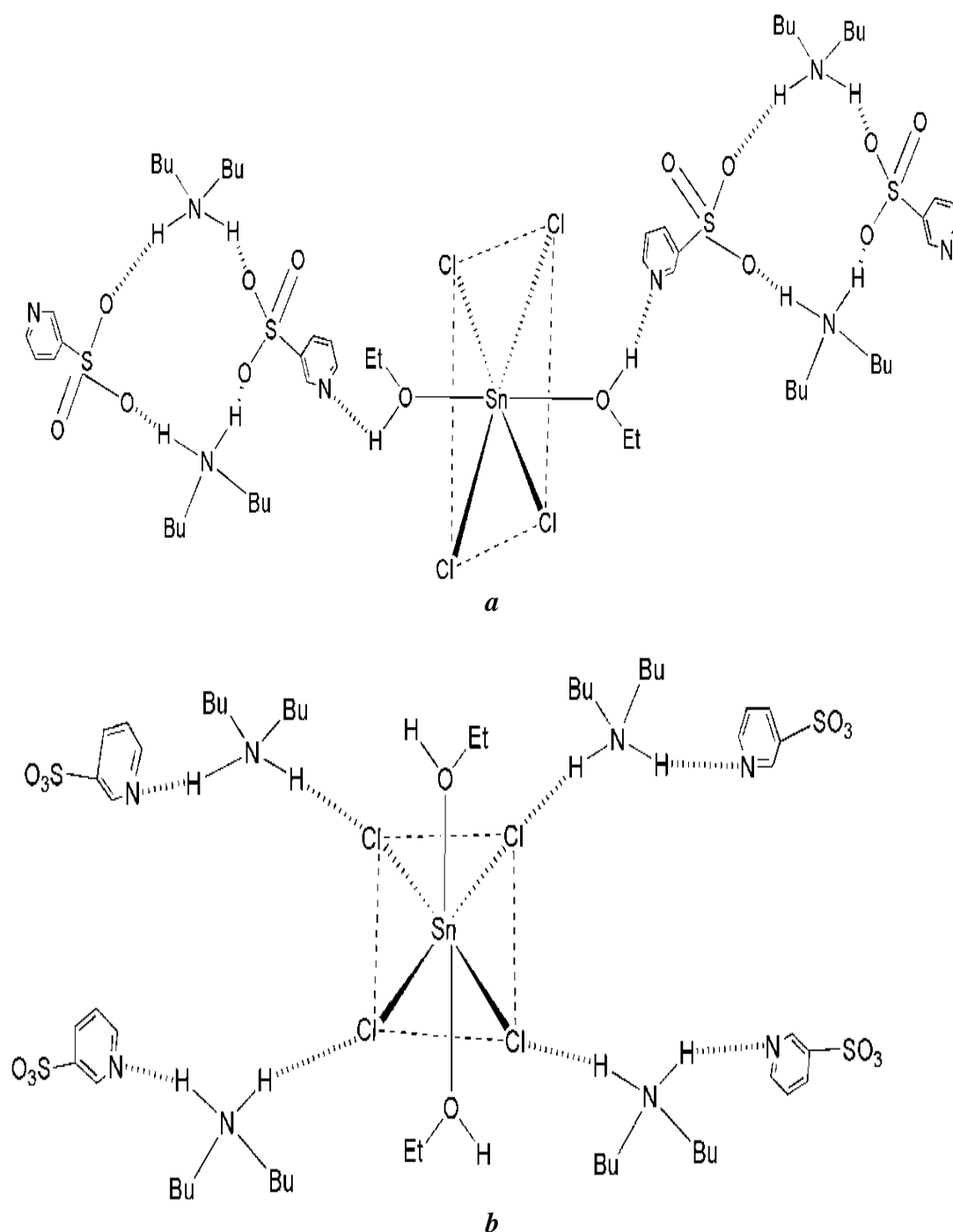


Figure 2. Proposed structure for the compound 2

For **3** we suggest a dimeric structure with a monodentate O-donor, the dimerization involving the cations through NH...O hydrogen bonds, the environment around the zinc being tetrahedral (Figure 3).

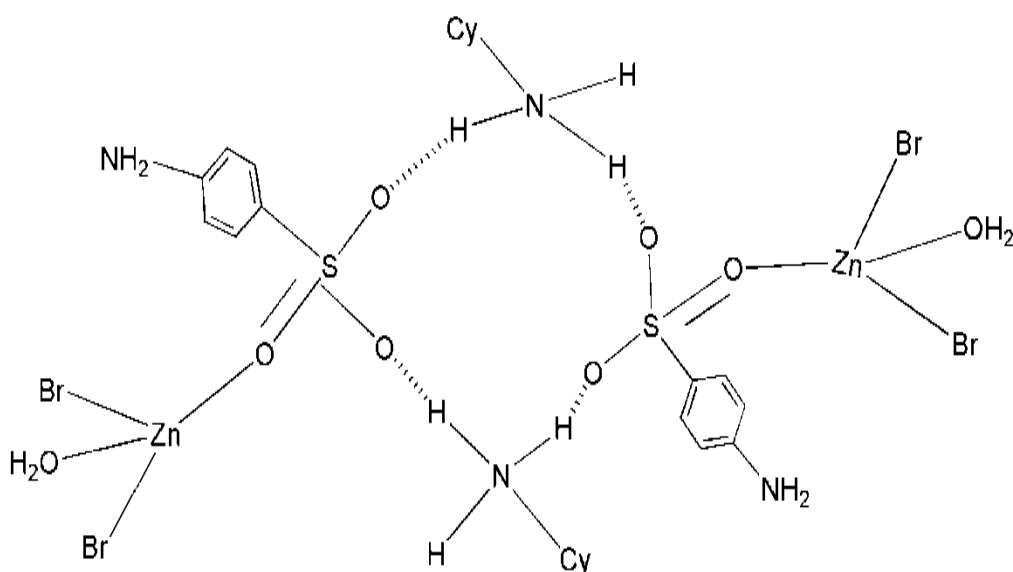


Figure 3. Proposed structure for the compound 3

For all the studied compounds the cations can be involved in extra hydrogen bonds leading to a supramolecular architecture.

CONCLUSION

All the studied adducts have a discrete structure, the sulfonate as a tri O-chelating/N-donor or O-donor ligand, the environment around the metallic centres being tetrahedral, octahedral or seven coordinated. When extra intermolecular hydrogen bonds are considered supramolecular architectures are obtained.

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