

**(Me<sub>4</sub>N)(NO<sub>2</sub>)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>CO<sub>2</sub> NEW MCl<sub>2</sub> (M = Zn, Co, Cd) AND  
SnR<sub>3</sub>Cl (R = Me, Ph) ADDUCTS:  
SYNTHESIS AND INFRARED STUDY**

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**Abstract:** Five new (Me<sub>4</sub>N)(NO<sub>2</sub>)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>CO<sub>2</sub> adducts with MCl<sub>2</sub> (M = Zn, Co, Cd) and SnR<sub>3</sub>Cl (R = Me, Ph) have been synthesized and studied by infrared spectroscopy. The suggested structures are discrete, the anion behaving as mono- and bichelating ligand towards MX<sub>2</sub>, tri- and monodentate towards SnR<sub>3</sub>Cl (R = Me, Ph). The environment around Zn, Co and Cd is tetrahechal, while the tin (IV) centre is trigonal bipyramidal.

**Keywords:** [NO<sub>2</sub>C<sub>6</sub>H<sub>3</sub>CO<sub>2</sub>]<sup>-</sup> ligand, infrared, discrete, structures

## INTRODUCTION

The main results on the coordinating ability of carboxylate anions towards tin have been reported by Tiekink [1]. The coordination ability of carboxylates anions (formiate, acetate and oxalate) has been reported by several authors including our group [2, 3]. We initiate here the study of the interactions between  $(\text{Me}_4\text{N})(\text{NO}_2)_2\text{C}_6\text{H}_3\text{CO}_2$  and  $\text{MCl}_2$  ( $\text{M} = \text{Zn}, \text{Co}, \text{Cd}$ ) or  $\text{SnPh}_3\text{Cl}$  which have yielded five new adducts infrared studies of which have been carried out, then structures suggested on the basis of infrared data.

## MATERIALS AND METHODS

$(\text{Me}_4\text{N})(\text{NO}_2)_2\text{C}_6\text{H}_3\text{CO}_2$  have been obtained as a powder on neutralizing  $(\text{NO}_2)_2\text{C}_6\text{H}_3\text{CO}_2\text{H}$  with a 20% water solution of  $\text{Me}_4\text{NOH}$  and allowing the water to evaporate at the oven at  $60^\circ\text{C}$ .

When ethanolic solutions of  $(\text{Me}_4\text{N})(\text{NO}_2)_2\text{C}_6\text{H}_3\text{CO}_2$  are mixed with ethanolic solutions of  $\text{ZnCl}_2$ ,  $\text{CoCl}_2$ ,  $\text{CdCl}_2$ , precipitation occurs. The precipitates were stirred around two hours and filtered.

For  $\text{SnR}_3\text{Cl}$ ,  $\text{R} = \text{Ph}, \text{Me}$  (both solutions being ethanolic) a powder is collected after a slow solvent evaporation at room temperature.

The analytical data reported below, with the ratio (salt / halide) have allowed to suggest the following formulae (Table 1).

**Table 1.** Suggested formulae of synthesized compounds and the elemental analyses

Compound	Chemical formula	Elemental analysis [%]					
		C		H		N	
		calc.	found	calc.	found	calc.	found
<b>A</b>	$\text{Me}_4\text{N}(\text{NO}_2)_2\text{C}_6\text{H}_3\text{CO}_2 \cdot \text{ZnCl}_2(1/2)$	31.32	31.44	3.55	3.31	9.97	10.05
<b>B</b>	$[\text{Me}_4\text{N}(\text{NO}_2)_2\text{C}_6\text{H}_3\text{CO}_2]_3 \cdot \text{CoBr}_2(1/2)$	36.88	36.95	4.19	3.89	11.73	11.52
<b>C</b>	$\text{Me}_4\text{N}(\text{NO}_2)_2\text{C}_6\text{H}_3\text{CO}_2 \cdot 2\text{CdCl}_2(1/2)$	20.25	20.89	2.30	2.36	6.44	6.70
<b>D</b>	$\text{Me}_4\text{N}(\text{NO}_2)_2\text{C}_6\text{H}_3\text{CO}_2 \cdot 3\text{SnPh}_3\text{Cl} \cdot \text{H}_2\text{O} (1/1)$	54.46	54.80	7.34	7.20	3.20	3.68
<b>E</b>	$\text{Me}_4\text{N}(\text{NO}_2)_2\text{C}_6\text{H}_3\text{CO}_2 \cdot \text{SnMe}_3\text{Cl}(1/1)$	34.67	34.28	4.25	3.84	8.67	8.72

The elemental analyses have been performed by the Laboratory of Microanalyses-University of Montpellier II – France. The infrared spectra have been recorded at the University of Padova -Italy- by means of a Perkin Elmer 180 spectrometer using  $\text{CsI}$ , the sample being as Nujol mulls. Infrared data are given in  $\text{cm}^{-1}$  (IR abbreviations: (vs) very strong, (s) strong, (m) medium, (w) weak, (vw) very weak, (br) broad). All the chemicals were purchased from Aldrich -Germany- and used as such.

## RESULTS AND DISCUSSION

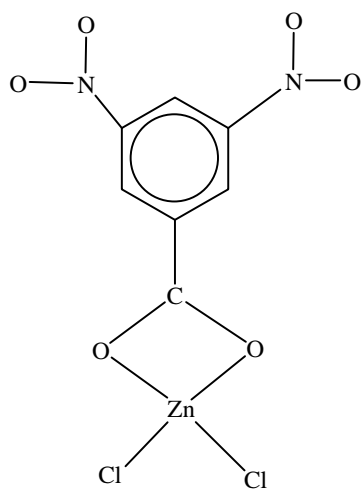
Let we consider the IR data (in  $\text{cm}^{-1}$ ) of the studied adducts:

(A):  $\nu_{\text{OH}} = 3300$  (br);  $\nu_{\text{CO}_2} = 1625$  (vs);  $\nu_{\text{asCO}_2} = 795$  (w);  $\nu_{\text{NO}_2} = 1542$  (vs);  $\nu_{\text{asNO}_2} = 791$  (s);  $\rho_{\text{CO}_2} = 520$  (m);  $\omega_{\text{CO}_2} = 3700$  (m);  $\delta_{\text{NO}_2} = 723$  (m);  $\nu_{\text{MX}_2} = 310$  (vs);  
 (B):  $\nu_{\text{OH}} = 3300$  (br);  $\nu_{\text{CO}_2} = 1600$  (s);  $\nu_{\text{asCO}_2} = 795$  (m);  $\nu_{\text{NO}_2} = 1542$  (vs);  $\nu_{\text{asNO}_2} = 791$  (s);  $\delta_{\text{CO}_2} = 750$  (m);  $\rho_{\text{CO}_2} = 515$  (m);  $\omega_{\text{CO}_2} = 380$  (w);  $\delta_{\text{NO}_2} = 723$  (w);  
 (C):  $\nu_{\text{OH}} = 3400$  (br);  $\nu_{\text{CO}_2} = 1620$  (vs);  $\nu_{\text{asCO}_2} = 795$  (w);  $\nu_{\text{NO}_2} = 1540$  (vs);  $\nu_{\text{asNO}_2} = 791$  (s);  $\rho_{\text{CO}_2} = 520$  (m);  $\omega_{\text{CO}_2} = 380$  (w);  $\delta_{\text{NO}_2} = 723$  (m);  
 (D):  $\nu_{\text{OH}} = 3300$  (br);  $\nu_{\text{CO}_2} = 1638$  (s);  $\nu_{\text{asCO}_2} = 792$  (m);  $\nu_{\text{NO}_2} = 1543$  (s);  $\nu_{\text{asNO}_2} = 791$  (s);  $\delta_{\text{CO}_2} = 744$  (vs);  $\rho_{\text{CO}_2} = 520$  (m);  $\omega_{\text{CO}_2} = 370$  (trace);  $\delta_{\text{NO}_2} = 720$  (trace);  
 (E):  $\nu_{\text{OH}} = 3400$  (br);  $\nu_{\text{CO}_2} = 1638$  (vs);  $\nu_{\text{asCO}_2} = 792$  (m);  $\nu_{\text{NO}_2} = 1542$  (s);  $\nu_{\text{asNO}_2} = 791$  (s);  $\rho_{\text{CO}_2} = 520$  (m);  $\omega_{\text{CO}_2} = 380$  (trace);  $\delta_{\text{NO}_2} = 724$  (w);  $\nu_{\text{asSnCl}_3} = 550$  (s).

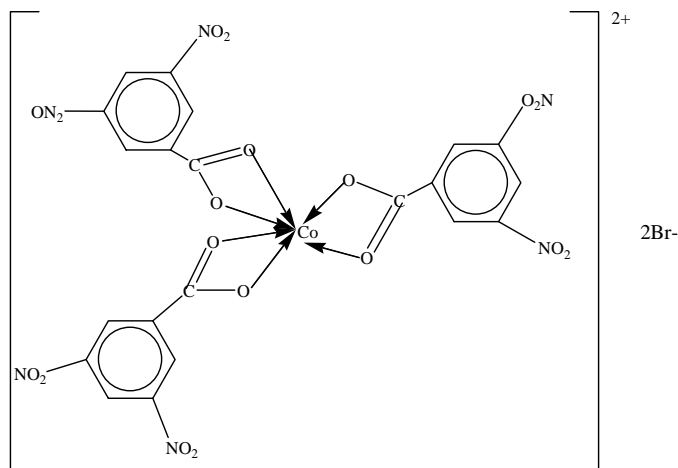
The absence of  $\nu_{\text{SnMe}_3}$  (A') in the spectrum of **E** is in accordance with the monocoordination of the anion.

From these data we suggest the structures reported in Figures 1-5.

For the structures presented in Figures 1 and 2 the anion is monochelating while being bichelating in Figure 3, tricoordinating in Figure 4 and monocoordinating in Figure 5.



**Figure 1.** Compound A



**Figure 2.** Compound B

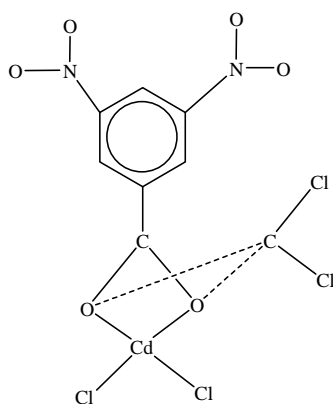


Figure 3. Compound C

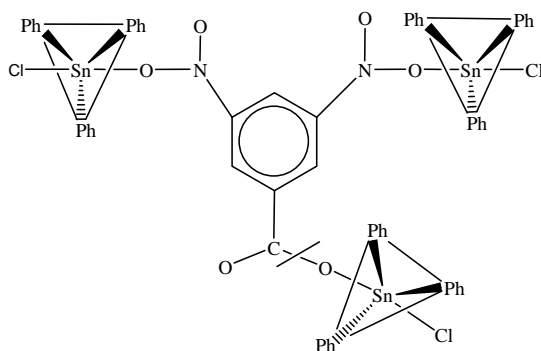


Figure 4. Compound D

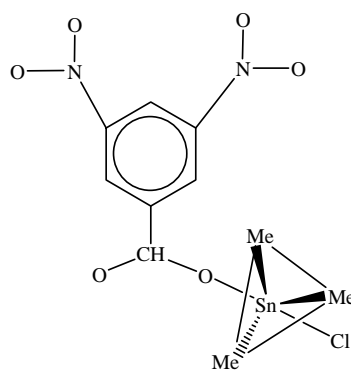


Figure 5. Compound E

## CONCLUSIONS

The studied adducts have discrete structures, the anion being a mono- and bichelating ligand towards  $MX_2$  while being tridentate and monodentate towards organotin compounds.

## ACKNOWLEDGEMENTS

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