

SHORT COMMUNICATION

ANTIMONY HALIDES AND HgX_2 ($X = Cl, Br$) AMINE ADDUCTS: SYNTHESIS AND INFRARED STUDY

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Abstract: Eight new SbF_3 , $SbCl_5$ and HgX_2 ($X = Cl, Br$) amine adducts have been synthesized and their infrared study carried out. Discrete structures have been suggested on the basis of elemental analysis and infrared data, the coordination number of antimony varying from five to nine, while the environment around Hg is tetrahedral.

Keywords: SbF_3 , $SbCl_5$ and HgX_2 ($X = Cl, Br$) amine, discrete structures

INTRODUCTION

Many antimony and mercury halide adducts with amines have yet been reported [1, 2]. We report here the study of the interactions between SbF_3 , SbCl_5 or HgX_2 ($\text{X} = \text{Cl}, \text{Br}$) and amines which have yielded eight new adducts, infrared study of which have been carried out then structures suggested on the basis of infrared data.

EXPERIMENTAL

The studied adducts have been obtained as precipitates – stirred roughly during two hours – on mixing ethanol solutions of the amine and the halide.

The analytical data reported below, allow suggesting the following formulae: % Calculated (% Found).

Table 1. Results of the elemental analyses

Compound	Chemical formula	Elemental analysis (%)					
		C		H		N	
		calc.	found	calc.	found	calc.	found
A	$(\text{iBu}_2\text{NH})_2\text{SbCl}_5 \cdot 2\text{H}_2\text{O}$	32.38	32.27	7.13	7.14	4.92	4.90
B	$\text{iBu}_2\text{NH} \cdot \text{SbCl}_5 \cdot 2\text{H}_2\text{O}$	20.69	20.05	4.99	5.10	3.02	3.02
C	$(\text{Cy}_2\text{NH})_2\text{SbF}_3$	53.24	52.63	8.56	8.44	5.17	5.16
D	$\text{Cy}_2\text{NH} \cdot \text{HgBr}_2$	26.61	26.25	4.28	4.42	2.59	2.56
E	$\text{iBu}_2\text{NH} \cdot \text{HgCl}_2$	23.98	23.76	4.78	4.98	3.50	3.40
F	$\text{Cy}_2\text{NH} \cdot \text{HgCl}_2$	31.83	30.09	5.12	4.53	3.09	3.64
G	$[\text{C}_2\text{H}_5\text{CH}(\text{NH}_2)\text{CH}_2\text{CH}_2\text{NH}_2] \cdot \text{HgCl}_2$	16.07	16.02	3.73	4.00	7.50	7.45
H	$[\text{C}_2\text{H}_5\text{CH}(\text{NH}_2)\text{CH}_2\text{CH}_2\text{NH}_2]_2 \cdot \text{HgCl}_2$	25.24	25.00	5.93	6.01	11.77	11.80

The infrared spectra were recorded at the University College of Saint-Boniface (Winnipeg-Canada). The elemental analyses have been performed at the laboratory of Microanalyses at the University of Bath (UK). Infrared data are given in cm^{-1} – IR abbreviations: br (broad) (vs) very strong, (s) strong, (m) medium, (sh) shoulder, (vw) very weak. All the chemicals were purchased from Aldrich – Germany - and used without any further purification.

RESULTS ET DISCUSSION

Let us consider the IR data of the studied adducts:

- A:** νNH : 3224 cm^{-1} (m); δNH : 1589 cm^{-1} (s); ρNH : 1024 cm^{-1} (s); ωNH : 775 cm^{-1} (s).
B: νNH : 3054 cm^{-1} (m); δNH : 1629 cm^{-1} (vs); ρNH : 1074 cm^{-1} (m); ωNH : 771 cm^{-1} (m).
C: νNH : 1124 cm^{-1} (m); δNH : 1591 cm^{-1} (m); ρNH : 1058 cm^{-1} (s); ωNH : 781 cm^{-1} (s)
D: νNH : 3159 cm^{-1} (m); δNH : 1631 cm^{-1} (s); ρNH : 1049 cm^{-1} (s); ωNH : 771 cm^{-1} (s)
E: νNH : 3249 cm^{-1} (m); δNH : 1560 cm^{-1} (s); ρNH : 1041 cm^{-1} (s); ωNH : 781 cm^{-1} (s)

F: νNH : 3052 cm^{-1} (s); δNH : 1570 cm^{-1} (s); ρNH : 1047 cm^{-1} (s); ωNH : 799 cm^{-1} (s)
G: νNH : 3292-3240 cm^{-1} (m); δNH :1562 cm^{-1} (vs); ρNH :1043 cm^{-1} (s); ωNH :779 cm^{-1} (m)
H: νNH : 3360 cm^{-1} (vs); δNH : 1542 cm^{-1} (vs); ρNH : 1073 cm^{-1} (m); ωNH : 790 cm^{-1} (m)

It is easy from the IR spectra to differentiate the presence of protonated form of the amine from the amine itself by the presence of a strong and wide absorption-protonated form-and its absence but the presence of the non-protonated amine characterized by the appearance of a medium sharp band. The absence of such wide and very strong absorption has allowed concluding to the presence of non-protonated amine in the above formulae.

A: $(\text{iBu}_2\text{NH})_2\text{SbCl}_5 \cdot 2\text{H}_2\text{O}$

This compound contains a seven-coordinated metal center. This allows to consider the complex as $(\text{iBu}_2\text{NH})_2\text{SbCl}_5 \cdot 2\text{H}_2\text{O}$ which structure is reported on Figure 1 - the water molecules are lattice.

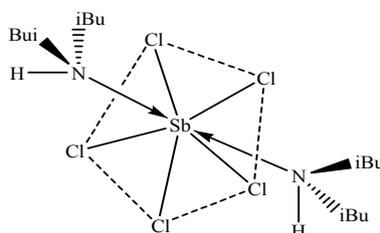


Figure 1.

B: $\text{iBu}_2\text{NH} \cdot \text{SbCl}_5 \cdot 2\text{H}_2\text{O}$

This compound contains a six-coordinated metal center. This allows to consider the complex as $\text{iBu}_2\text{NH} \cdot \text{SbCl}_5 \cdot 2\text{H}_2\text{O}$ which structure is reported on Figure 2 - the water molecules are lattice.

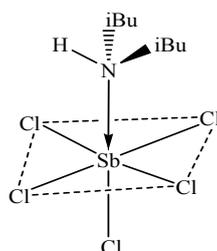


Figure 2.

C: $(\text{Cy}_2\text{NH})_2\text{SbF}_3$

This compound contains a five-coordinated metal center. The structure is reported on Figure 3.

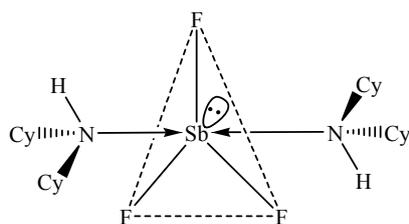
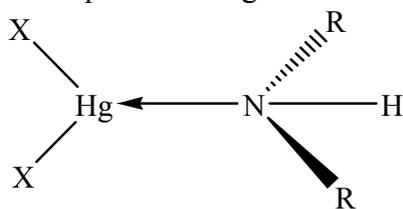


Figure 3.

D: $\text{Cy}_2\text{NH.HgBr}_2$; **E:** $\text{iBu}_2\text{NH.HgCl}_2$; **F:** $\text{Cy}_2\text{NH.HgCl}_2$
The structure of these adducts is reported on Figure 4.



$\text{R} = \text{Cy, iBu; X} = \text{Br, Cl}$

Figure 4.

G: $[\text{C}_2\text{H}_5\text{CH}(\text{NH}_2)\text{CH}_2\text{CH}_2\text{NH}_2]\text{HgCl}_2$

This compound contains a four-coordinated metal center. The structure is reported on Figure 5.

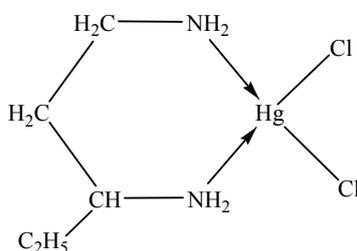


Figure 5.

H: $[\text{C}_2\text{H}_5\text{CH}(\text{NH}_2)\text{CH}_2\text{CH}_2\text{NH}_2]_2\text{HgCl}_2$

This compound contains a six-coordinated metal center. The structure is reported on Figure 6.

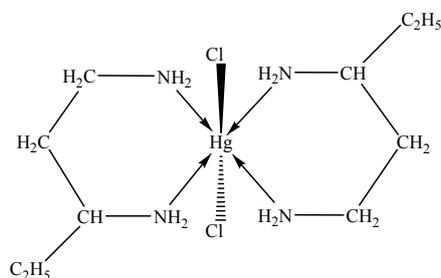


Figure 6.

CONCLUSION

The six studied adducts contain amines. The environment around Sb is heptagonal or pentagonal while the mercury is in all cases tetrahedral.

REFERENCES

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