Studii şi Cercetări Ştiinţifice
Chimie şi Inginerie Chimică, Biotehnologii, Industrie Alimentară
2012, **13** (4), pp. 399 – 403

Scientific Study & Research
Chemistry & Chemical Engineering, Biotechnology, Food Industry
ISSN 1582-540X

ORIGINAL RESEARCH PAPER

NEW R_2NH_2OH (R = Cy, Bu) ADDUCTS OF MX_2, MX_3 OR MX_5: SYNTHESIS AND INFRARED STUDY

Ndongo Gueye[1], Libasse Diop[1]*, Lamine Diop[2]

[1]Université Cheikh Anta Diop, Faculté des Sciences et Technique, Département de Chimie, Laboratoire de Chimie Minérale et Analytique, Dakar, Sénégal
[2]Collège Universitaire de Saint-Boniface, Winnipeg, Canada

*Corresponding author: dlibasse@gmail.com

Received: May, 23, 2012
Accepted: October, 26, 2012

Abstract: When Cy_2NH and Bu_2NH in the presence of a very little amount of water are allowed to react with metal halide $CdBr_2$, $SbCl_3$ or $SbCl_5$, hydroxyl adducts with protonated amines as adverse cations were obtained. Their infrared study allows concluding the presence of OH^- linked to the metallic center.

Keywords: *discrete structure, octahedral environment, Sb adducts*

INTRODUCTION

Amine containing adducts have been reported by several authors [1, 2]. In a previous paper [3] we have reported some amines adducts with $SbCl_5$, SbF_3 and $HgCl_2$. The infrared spectra allow easily distinguishing between the presence of an amine and its protonated form by the presence or not of a very broad and strong absorption related to the presence of the protonated form while a medium band appears in the presence of the amine. In the presence of a small amount of water (2%), Cy_2NH, Bu_2NH and iBu_2NH when allowed to react, with MX_2 (CdX_2, HgX_2), $SbCl_3$ or $SbCl_5$ lead to the ammonium containing adduct. Structures are suggested on the basis of infrared data.

EXPERIMENTAL

The studied adducts have been obtained as precipitates – stirred roughly during two hours – on mixing ethanolic solutions of the amine and the halide.
Table 1 presents the elemental analysis of **A** – **E** compounds.

Table 1. *Results of the elemental analyses*

Compound	Chemical formula	Elemental analysis (%)					
		C		H		N	
		calc.	found	calc.	found	calc.	found
A	$(Bu_2NH_2)SbCl_3OH$	25.60	25.80	5.64	5.94	3.73	3.49
B	$(iBu_2NH_2)SbCl_3OH$	25.60	25.80	5.64	5.94	3.73	3.49
C	$(Cy_2NH_2)_2CdBr_2(OH)_2$	38.39	39.05	6.71	6.85	3.73	3.82
D	$(Cy_2NH_2)_2SbCl_5(OH)_2$	41.32	41.20	7.22	7.37	4.02	3.97
E	$(Cy_2NH_2)_3SbCl_3(OH)_3$	52.34	52.42	9.15	9.25	5.09	5.16

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.

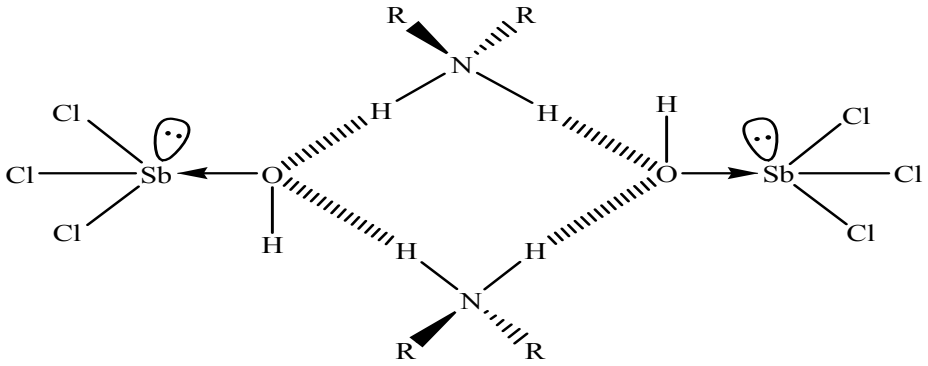
RESULTS AND DISCUSSION

Let us consider the IR data in of the studied compounds:
(A): ν OH = 3400 (m); ν NH = 3200–2600 (l); δ NH = 1629 (s); ρ NH = 1072 (m); ωNH = 773 (s);
(B): ν OH = 3400 (m); ν NH = 3200–2600 (l); δ NH =1629 (s); ρ NH = 1072 (m); ω NH = 773 (s);
(C): ν OH = 3500 (m); ν NH = 3100–2600 (l); δ NH = 1629 (s); ρ NH = 1041 (s); ω NH = 767 (s);

(D): ν OH = 3500 (m); νNH = 3100–2600 (l); δ NH = 1656 (s); ρ NH = 1093 (s); ω NH = 746 (s);
(E): ν OH = 3450 (m); ν NH = 3100–2550 (l); δ NH =1650 (s); ρ NH = 1033 (s); ω NH = 766 (s);

$(Bu_2NH_2)SbCl_3OH$ (*A*), $(iBu_2NH_2)SbCl_3OH$ (*B*)

These adducts contain a four-coordinated metal center. The structure is reported on Figure 1.



R = Bu, iBu

Figure 1. *Proposed structure for the compounds* ***A*** *and* ***B***

$(Cy_2NH_2OH)_2CdBr_2$ (*C*)

The proposed structure for the compound $(Cy_2NH_2OH)_2CdBr_2$ is a dimer as shown in Figure 2. The structure appears as an hydrogen bonded dimeric $[(Cy_2NH_2OH)_2]_2$ to which two $CdBr_2$ molecules connect through oxygen atoms.

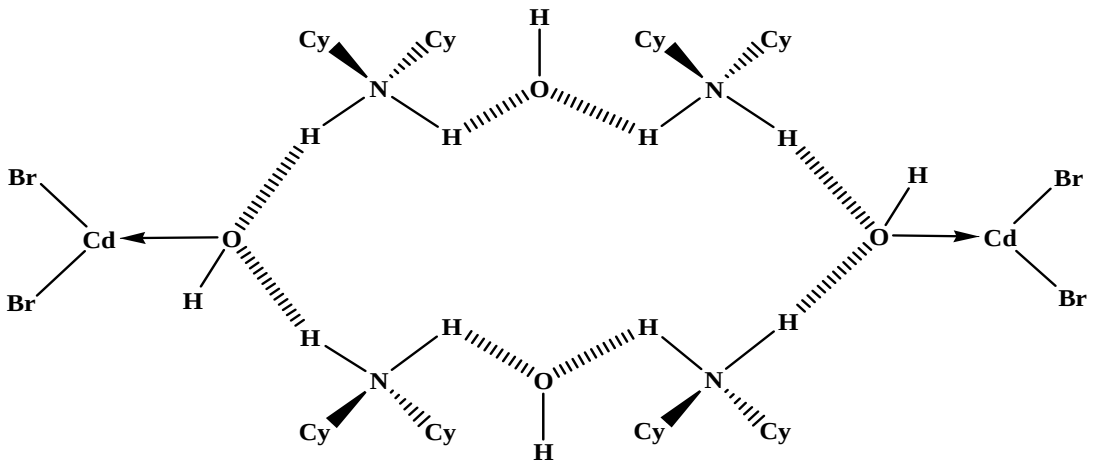


Figure 2. *Proposed structure for the compound* ***C***

$(Cy_2NH_2)_2SbCl_5(OH)_2$ (*D*)

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

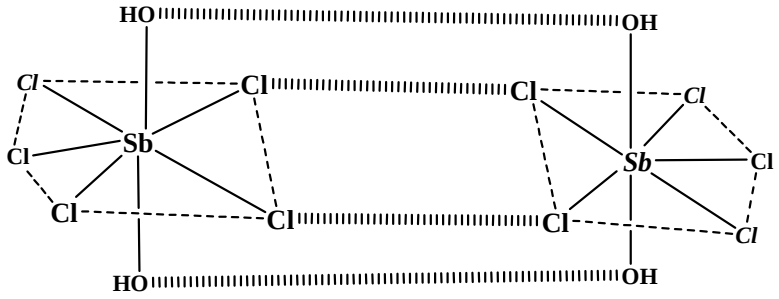


Figure 3. *Proposed structure for the compound* ***D***

$(Cy_2NH_2)_3SbCl_3(OH)_3$ (*E*)

This compound contains a four-coordinated metal center. The structure reported on Figure 4 consists on a cyclic dimeric hydrogen bonded $[(Cy_2NH_2OH)_3]_2$ to which the two molecules of $SbCl_3$ coordinate symmetrically through oxygen atoms.

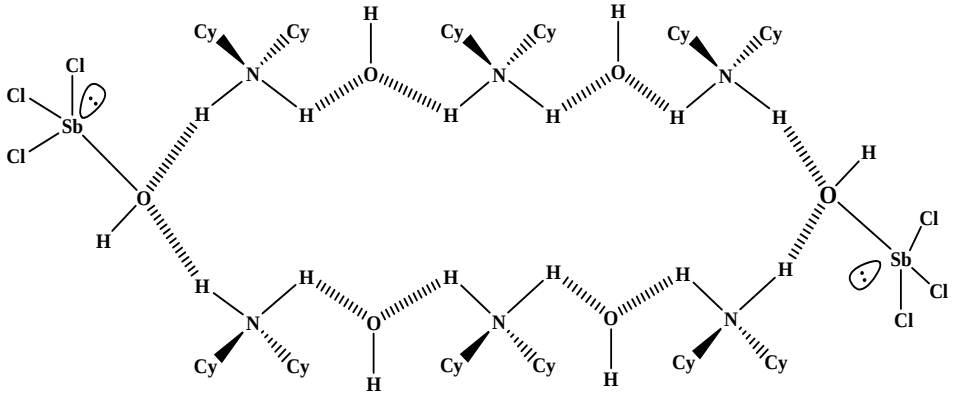


Figure 4. *Proposed structure for the compound* ***E***

In all these four structures when OH----Cl (Br) hydrogen bonds are considered, supra molecular architectures are obtained.

CONCLUSIONS

The structures of protonated form containing adducts are all discrete. The crucial role of the cation and the hydroxyl group is noteworthy. When extra inter hydrogen bonds are considered supramolecular architectures are obtained.

ACKNOWLEDGEMENTS

We thank Professor K. C. Molloy – Bath University (UK) for performing the elemental analyses.

REFERENCES

1. Greenwood, N. N., Earnshaw, A.: *Chemistry of the Elements*, Pergamon Press, Oxford, **1984**;
2. Cotton, F.A., Wilkinson, G.: *Advanced Inorganic Chemistry*, 5th edition, Wiley-Interscience, New York, **1988**;
3. Gueye, N., Diop, L., Diop, L.: Antimony Halides and HgX_2 (X = Cl, Br) Amine Adducts: Synthesis and Infrared Study, *Scientific Study & Research - Chemistry & Chemical Engineering, Biotechnology, Food Industry*, **2012**, **13** (3), 345-348.