Studii şi Cercetări Ştiinţifice 2012, **13** (3), pp. 339 – 344
Chimie şi Inginerie Chimică, Biotehnologii, Industrie Alimentară

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

ORIGINAL RESEARCH PAPER

NEW PERCHLORATO ADDUCTS: SYNTHESIS AND INFRARED STUDY

Omar Gueye, Libasse Diop*

Université Cheikh Anta Diop, Faculté des Sciences et Techniques, Département de Chimie, Laboratoire de Chimie Minérale et Analytique (LACHIMIA), Dakar, Sénégal

*Corresponding author: dlibasse@gmail.com

Received: December, 20, 1211
Accepted: June, 12, 2012

Abstract: Five perchlorato adducts have been synthesized and studied by infrared. The structures are discrete, the perchlorate anion behaving a monodentate or a bidentate ligand.

Keywords: *discrete structure, monodentate or bidentate perchlorate anion*

INTRODUCTION

Hathaway has summarized the data on the coordinating ability of the oxyanions [1].The perchlorate anion has been intensively studied by Potier *et al.* [2]. Many search groups [3-9] including ours [10-17] which have been interested in both the coordination ability of oxyanion and molecules belonging to organotin (IV) family due to their numerous applications. We initiate here the study of the interactions between Et_4NClO_4 and $HgCl_2$, $SbCl_5$, $SnMe_2Cl_2$ or SnX_4 (X = Cl, Br) which have yielded five new adducts, infrared study of which have been carried out then structures suggested on the basis of infrared data.

EXPERIMENTAL

On allowing

- $HgCl_2$ in ethanol to react with Et_4NClO_4 in dichloromethane in 4/1 ratio, a white precipitate is obtained (**A**) which is stirred around two hours then filtered and collected.
- $SnMe_2Cl_2$ in ethanol with Et_4NClO_4 in dichloromethane, in 3/2 ratio a white powder is obtained after a slow solvent evaporation (**B**).
- $SbCl_5$ in benzene and Et_4NClO_4 in dichloromethane a white powder is obtained after a slow solvent evaporation (**C**).
- $SnCl_4$ in benzene and Et_4NClO_4 in dichloromethane, (in 2/1, 3/2 and 1/1 ratio) give a white powder after a slow solvent evaporation (**D**).
- $SnBr_4$ in benzene and Et_4NClO_4 in dichloromethane give a white powder after a slow solvent evaporation (**E**).

The analytical data reported below, have allowed to suggest the following formulas [% calculated (% found)]:

(**A**) $Et_4NClO_4.HgCl_2.H_2O$: C = 31.77 (32.16); H= 6.78 (7.40); N = 4.63 (4.45)

(**B**) $[Et_4NClO_4]_2[SnMe_2Cl_2]_3$: C = 22.87 (22.67); H = 5.37 (5.28); N = 2.42 (2.16)

(**C**) $Et_4NClO_4.SbCl_5.H_2O$: C = 16.99 (16.92); H = 4.24 (4.06); N = 2.47 (2.09)

(**D**) $Et_4NClO_4.SnCl_4.3H_2O$: C = 17.64 (18.00); H = 4.45 (4.80); N = 3.12 (2.38)

(**E**) $[Et_4NClO_4]_2SnBr_4$: C = 21.40 (22.13); H = 4.67 (4.89); N = 3.04 (3.02)

The infrared spectra have been obtained at the University of Padova (Italy) on a PE 580 (4000 - 200 cm^{-1}) and a Bruker FTIR spectrometer, the sample being as Nujol mulls, the window being CsI or polyethylene. The IR data are given in cm^{-1} [abbreviations: (vs) very strong, (s) strong, (m) medium, (sh) shoulder, (w) weak, (vw) very weak].
All the chemicals were purchased from Merck Company and used without any further purification.

RESULTS AND DISCUSSION

Let us consider the infrared data of the studied compounds:

(A): $\nu_1 + \nu_3$: 890w, 1083s, 1187m; $\nu_2 + \nu_4$: 468w, 622s; νHgO: 311sh;

(B): $\nu_1 + \nu_3$: 931m, 1080s, 1173m; $\nu_2 + \nu_4$: 419w, 624s; νSnO: 240w; νSnCl: 316sh, 338vs, 350sh;

(C): $\nu_1 + \nu_3$: 975w, 1079s, 1182m; $\nu_2 + \nu_4$: 468w, 623s; νSnO: 276w; νSnBr: 203vs;

(D): $\nu_1 + \nu_3$: 937w, 1080s, 1170m; $\nu_2 + \nu_4$: 418s, 624s; νSnCl: 334vs;

(E): $\nu_1 + \nu_3$: 937w, 1079s, 1185sh; $\nu_2 + \nu_4$: 467w, 624s; νSnO: 250sh; νSnCl: 308, 325sh, $\nu_s SnC_2$: 517w, $\nu_{as} SnC_2$: 569m.

From the infrared data we can conclude C_{2v} symmetry of $SnCl_4$ in (**D**) and D_{4h} symmetry of $SnBr_4$ in (**E**) because of several bands for $\nu_{as} SnCl_4$ and a sharp band for $\nu_{as} SnBr_4$. The appearance of $\nu_s SnC_2$ (adduct **B**) is consistent with the presence of at least a non linear $SnMe_2$ residue. From these conclusions the following structures are suggested (Figures 1-5).

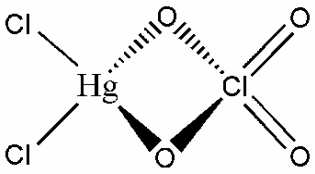

Figure 1

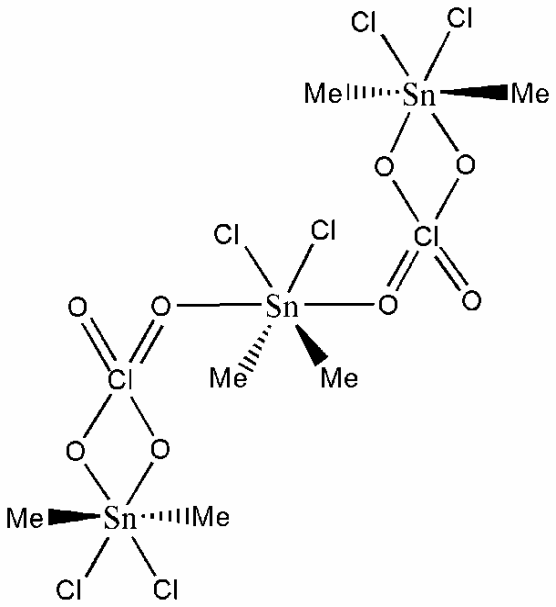

Figure 2

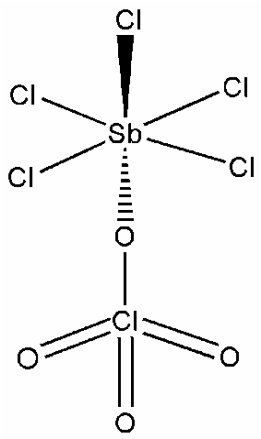

Figure 3

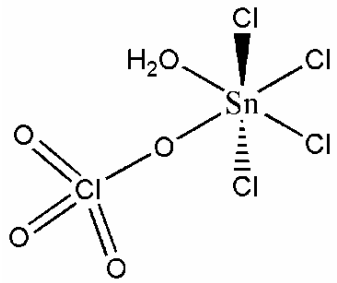

Figure 4

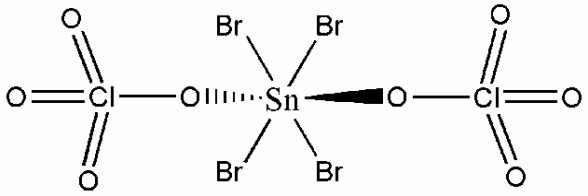

Figure 5

The perchlorate behaves as a monocoordinating, a monochelating, a monocoordinating and chelating ligand. In (**D**) when O-H….O hydrogen bonds are considered a supramolecular architecture is obtained.

CONCLUSIONS

Discrete structures and supramolecular architecture are suggested on the basis of infrared data. The perchlorate behaves as a monocoodinating, monochelating or

monocoordinating and monochelating ligand. A supramolecular architecture is suggested on the $SnCl_4$ adduct.

ACKNOWLEDGMENTS

We thank Professor M. Vidali –Padova University, Italy – for equipment support.

REFERENCES

1. Hathaway, B.J.: *Comprehensive Coordination Chemistry* (Editors: Wilkinson, G., Gillard, R.D., McCleverty, J.A.), 1st ed., volume 5, Chapter 53, Pergamon Press, Oxford, **1987**, 413;
2. Chaabouni, M., Pascal, J.L., Pavia, A.C., Potier, J., Potier, A.: Anhydrous iron perchlorates, *Comptes Rendus hebdomadaires des Séances de l'Académie des Sciences, Série C: Sciences Chimiques*, **1978**, **287**(10), 419-422;
3. Evans, J.C., Karpel, S.: *Organotin Compounds in Modern Technology* (*Journal of organometallic chemistry library 16*), Elsevier Science Ltd., Amsterdam, **1985**;
4. Yin, H.-D., Wang, C.-H.: Crystallographic report: Crystal and molecular structure of triphenyltin thiazole-2-carboxylate, *Applied Organometallic Chemistry*, **2004**, **18**(8), 411-412;
5. Kapoor, R.N., Guillory, P., Schulte, L., Cervantes- Lee, F., Haiduc, I., Parkanyi L., Pannell, K.H.: Di(*p-tert*-butylphenyl)-*N*,*N*-di-(*iso*-butyl)carbamoylmethylphosphine oxide and its organotin and uranyl adducts: structural and spectroscopic characterization, *Applied Organometallic Chemistry*, **2005**, **19**(4), 510-517;
6. Zhang, W.-L., Ma, J.-F., Jiang, H.: μ-Isophthalato-bis[triphenyltin(IV)], Acta Crystallographica Section E: Structure Reports Online, **2006**, **E62**(3), m460-m461;
7. Chandrasekhar, V., Boomishankar, R., Steiner, A., Bickley, J.F.: First Example of a Hydrogen-Bonded Three-Dimensional Pillared Structure Involving an Organotin Motif: Synthesis and X-ray Crystal Structures of $\{[^nBu_2Sn(H_2O)_3(L)Sn(H_2O)_3{}^nBu_2]^{2+}[L]^{2-}\}\cdot 2MeOH\cdot 2H_2O$ and $\{[Ph_3Sn(L)Sn(H_2O)Ph_3]_n\}\cdot THF$ (L = 1,5-Naphthalenedisulfonate), Organometallics, **2003**, **22**(17), 3342-3344;
8. Herntrich,T., Merzveiler, K.:$[(Ph_3Sn)_3VO_4]\cdot CH_3CN$ and $[(Ph_3Sn)_3VO_4]\cdot 2$ DMF, Triphenylzinnvanadate mit neuartigen Kettenstrukturen, *Zeitschrift für anorganische und allgemeine Chemie*, **2006**, **632**(14), 2341-2344;
9. Gielen, M., Bouhdid, A., Kayser, F., Biesemans, M., De Vos, D., Mahieu, B., Willem, R.: Di(*n*-butyl)tin bis(dihydroxybenzoate)s: Synthesis, spectroscopic characterization and *in vitro* antitumour activity, *Applied Organometallic Chemistry*, **1995**, **9**(3), 251-257;
10. Okio, K.Y.A., Diop, L., Russo, U.: $[Cy_2NH_2SO_4(SnPh_3)_2X]_2$ (X = F, Cl): synthesis and spectroscopic studies, *Scientific Study & Research – Chemistry & Chemical Engineering, Biotechnology, Food Industry*, **2009**, **10**(1), 11-14;
11. Diallo, W., Diassé Sarr, A., Diop, L., Mahieu, B., Biesemans, M., Willem, R., Kociok-Köhn, G., Molloy, K.C.: X-Ray structure of tetrabutylammonium chlorotrimethyltin hydrogenosulphate: the first cyclic dimer hydrogenosulphato hydrogen bonded adduct, *Scientific Study & Research – Chemistry & Chemical Engineering, Biotechnology, Food Industry*, **2009**, **10**(3), 207-212;
12. Diallo, W., Okio, K.Y.A., Diop, C.A.K., Diop, L.A., Diop, L., Russo, U.: New selenito $SnPh_3$ residue containing complexes and adducts: Synthesis and spectroscopic studies, *Main Group Metal Chemistry*, **2009**, **32**(2), 93-100;
13. Qamar Kane, H., Okio, K.A., Fall, A., Diop, L., Russo, U., Mahieu, B.: $Et_4NC_2O_4SnPh_3.2SnPh_3Cl$ and $Cy_2NH_2C_2O_4SnPh_3.2SnPh_3Cl$: Synthesis and spectroscopic characterization, *Main Group Metal Chemistry*, **2009**, **32**(4), 229-233;
14. De Barros, D., Diop, L., Mahieu, B.: On the existence of « tetrahedral » $SnMe_2(PhCO_2)_2$ and $[SnMe_2(PhCO_2)_3]^-$ in new benzoato adducts: Synthesis and spectroscopic studies, *Main Group Metal Chemistry*, **2009**, **32**(6), 341-346;

15. De Barros, D., Diop, L., Mahieu, B.: $Me_4NHWO_4SnPh_3X$ (X=Cl, Br), $R_4NWO_4SnPh_3$ (R=Me, Et) and $(Snbu_3)_2WO_4$: Synthesis and spectroscopic studies, *Main Group Metal Chemistry*, **2010**, **33**(1-2), 91-95;
16. Diallo, W. Okio, K.Y.A., Diop, L., Diop, L.A., Russo, U., Wattiaux, A.: Some new sulfato and hydrogenosulfato adducts: synthesis, infrared and Mössbauer studies, *Scientific Study & Research – Chemistry & Chemical Engineering, Biotechnology, Food Industry*, **2010**, **11**(2), 219-226;
17. Diop, C.A.K., Diop, L., Toscano, A.R.: Synthesis, characterization and X-ray structure of polymeric [triphenyltin(IV)][aquatriphenyltin(IV)] sulphate, $[(Ph_3Sn)(H_2OSnPh_3)SO_4]_n$, *Main Group Metal Chemistry*, **2002**, **25**(5), 327-328.