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INTERNATIONAL JOURNAL OF ADVANCED RESEARCH (IJAR)

Article DOI:10.21474/IJAR01/14294
DOI URL: http://dx.doi.org/10.21474/IJAR01/14294

ISSN NO. 2320-5407

RESEARCH ARTICLE

SOME NEW SELENIATO DERIVATIVES AND ADDUCTS ORGANOSTANIC CONTAINING SNR$_2$ RESIDUES (R=ME, BU, ^{T}BU) AND HALOSTANNIC: SYNTHESIS, INFRARED AND ^{119}SN NMR STUDIES

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Manuscript Info

Manuscript History
Received: 18 December 2021
Final Accepted: 20 January 2022
Published: February 2022

Key words:-
Seleniato, Infrared, NMR Spectroscopies,Discrete, Dimeric

Abstract

Seven new selenato and hydrogenoselenatoadducts and derivativesorganostannic containing SnR$_2$ (R=Me, Bu) residues, SnR$_2$Cl$_2$ (R=Bu, ^{t}Bu)andhalostannic containing SnCl$_2$ molecule have been synthesizedin methanol or ethanol solution in room temperature or at reflux. The complexes have been studied by infrared and ^{119}Sn NMR spectroscopies.The suggested structuresfor the organotin (IV) compounds are discrete or dimer in which the seleniateligandsaremonodentate (for one of them) and monochelating or bichelating for the others; SnR$_2$Cl residues aretetrahedral and SnR$_2$ residues are linear.Then, in the case of halostannic adducts, the suggested structuresare based in the process of an oxydation of tin (II) atom to tin (IV) atom. The initial SnCl$_2$molecule become SnCl$_4$. A dimerization of the molecule formular and rearrangementhave been considered, giving an ionic structure with one cation component and one molecule component stabilized by the ammonium cation. When the ammonium cations in hydrogen bonds are involved, supramolecular architectures may be obtained.

Introduction:-

The applications found for many molecules belonging to organotin and halotin family (medicine, agriculture, industry….) explain the focus of several research groups in this field until nowadays (Davies and al.; Evans and al.; Gielen and al.; Zhang and al.; Shang and al.; Willem and al. The coordination between oxyanions and organotin (IV) compounds has been studied since many years (Sougoule and al.; BasuBaul and al.; Yang).Dealing with selenato complexes, some X ray structures of selenato complexes have been reported so far:- Diallo and al has reported an Xray structure of [(Me$_3$Sn)$_3$(SeO$_4$)(OH)]n which structure is polymeric (Diallo and al.); [n-Bu$_2$NH$_2$]$_3$[SnPh$_3$(SeO$_4$)$_2$] which is first triorganotin(IV) complex with terminally coordinated selenato ligands(Diallo and al.).This has prompt us to study the interactions between (R$_2$NH$_2$)$_2$SeO$_4$.xH$_2$O (R=Bu; ^{i}Pr) and (BzEt$_3$)$_2$SeO$_4$.yH$_2$O to react with SnR$_2$Cl$_2$ (R=Me, Bu, ^{t}Bu) and SnCl$_2$. This has yielded seven new adducts and derivateshavebeenstudied by infrared and RMN spectroscopic, then structures suggested.

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Experimental section-

H_2SeO_4 was purchased from Merck Chemicals,Hohenbrunnbei München, Germanywhile $^iPr_2NH$.n-Bu_2NH and SnR_2Cl_2 (R=Bu, Me, ^tBu) were acquired fromSigma-Aldrich ChemieGmbH.Steinheim. Germany and used without any further purification.

Infrared spectra were recorded on a Bruker Vector 22 spectrometer equipped with a Specac Golden Gate^{TM} ATR device. $^{119}Sn\{^1H\}$ NMR spectrum was recorded on a Bruker Avance 400 MHz spectrometer with a wide band sensor BBFO.

$^{119}Sn\{^1H\}$ NMR spectraare reported downfield from Me_4Sn. as the internal standard. Infrared data are given in cm^{-1} and chemical shifts (□ □ inppm[IR abbreviations: (br) broad. (vs) verystrong. (s) strong. (m) medium. (sh) shoulder. (w) weak. (vw) veryweak].

Elemental analyses were performed at the “Institut de ChimieMoléculaire”. Universityof Burgundy. Dijon-France.

Procedure for synthesis of salts

The iso$Pr_2NH_2HSeO_4$.0,5H_2O (**L_1**), (iso$Pr_2NH_2)_2SeO_4$.0,5H_2O (**L_2**), $(Bu_2NH_2)_2SeO_4.H_2O$(**L_3**) salts are synthetized by neutralized the selenic acid $H_2SeO_4$by di-substituted amine R_2NH (R=isoPr, Bu) in water solvent.$(BzEt_3N)_2SeO_4.H_2O$salt(**L_4**)isobtained on reacting$BzEt_3NCl$ and H_2SeO_4in ethanol.After two hours mixed up, colorless solution is obtained, submitted to evaporation at 60° and white powder are obtained after one week.

L_1 (iso$Pr_2NH_2HSeO_4$.0,5H_2O): **1**isoPr_2NH + **1**H_2SeO_4
L_2 (iso$Pr_2NH_2)_2SeO_4$.0,5H_2O :2isoPr_2NH + **1**H_2SeO_4
L_3 $(Bu_2NH_2)_2SeO_4.H_2O$: **2**Bu_2NH + **1**H_2SeO_4
$L_4$$(BzEt_3N)_2SeO_4.H_2O$: **2**$BzEt_3NCl$ +**1**H_2SeO_4

The elemental analysis of all the saltsare showing in table 1.

salts	**Chemical formular**	**C**		**H**		**N**	
		Calc	**Found**	**Calc**	**Found**	**Calc**	**Found**
L_1	**(iso$Pr_2NH_2HSeO_4$.0,5H_2O)**	29.40	29.44	6.43	6.43	5.71	5.54
L_2	**(iso$Pr_2NH_2)_2SeO_4$.0,5H_2O**	39.68	39.45	9.73	9.38	6.12	7.67
L_3	**$(Bu_2NH_2)_2SeO_4.H_2O$**	45.27	45.44	10.68	10.45	6.60	6.25
L_4	**$(BzEt_3N)_2SeO_4.H_2O$**	57.70	57.53	8.07	7.85	5.61	5.33

Table 1:-Analytical data of salts.

a/ Procedure for synthesis ofiso$Pr_2NH_2SeO_4SnMe_2Cl$(**A**).

A mixture of **iso$Pr_2NH_2HSeO_4$.0,5H_2O (L_1)** (0.3 g; 0.782 mmol) with $SnMe_2Cl_2$(1.565 mmol)in 20 mL methanol solutiongives a clear solution and a white powder (**A**) collected after a slow solvent evaporation.

Spectroscopic data:-

1. IR data (cm^{-1}) :(υ$_1$ + υ$_3$)SeO_4= (832vs-721vs); υas$SnMe_2$= 536s; υs$SnMe_2$=510m, υNH_2= (2960-2847-2737)L, δNH_2= 1580s
2. ^{119}Sn NMR data (DMSO): δ= -333.94 ppm

Elemental analysis:-

% found (% calculated): C= 21.98 (22.38); H=5.10 (5.16); N=3.15 (3.26)

b/ Procedure for synthesis of$Bu_2NH_2SeO_4SnBu_2Cl$ (**B**)

On mixing methanol solutions of **$(Bu_2NH_2)_2SeO_4.H_2O$ (L_3)** (0.3 g; 0.782 mmol) with $SnBu_2Cl_2$ (0.3438 g; 1.565 mmol), compounds (**B**) is obtained as white powder after one week in slow solvent evaporation.

Spectroscopic data:-

1. IR data (cm^{-1}) : (υ$_1$ + υ$_3$)SeO_4= (902vs-815vs-786vs-755vs); υas$SnBu_2$ 618tF; υs$SnBu_2$=584f, υNH_2= (2959-2850-2728)L, δNH_2= 1580s

2. ^{119}Sn NMR data : -

Elemental analysis:-% found (% calculated) : C=35.26 (35.48); H=7.32 (7.07); N=2.60 (2.59)

c/ Procedure for synthesis of$SnBu_2$ SeO_4.EtOH (**C**)
When **($isoPr_2NH_2)_2SeO_4$.0,5H_2O** (**L_2**) (0.3 g; 0.782 mmol) is reacted with $SnBu_2Cl_2$ (0.4755 g; 1.565 mmol) in 20 mL of ethanol solution. The solutions are steered up during two hours and white precipitate are obtained (**C**).
Spectroscopic data:-

1. IR data (cm^{-1}) :υ_3SeO_4 = (860vs-840vs-762vs); υas$SnBu_2$=633s; υs$SnBu_2$= 592s; υOH (2957-2939-2429)L, δNH_2 1608s
2. ^{119}Sn NMR data ($CDCl_3$) :δ = -248.53 ppm

Elemental analysis:- -% found (% calculated) : C=28.30 (28.46); H=5.42 (5.73)

d/ Procedure for synthesis of$HSeO_4SnBu_2Cl.SnBu_2Cl_2.H_2O$ (**D**)
When **($isoPr_2NH_2)_2SeO_4$.0,5H_2O** (**L_2**) (0.3 g; 0.782 mmol) is reacted with $SnBu_2Cl_2$ (0.4755 g; 1.565 mmol) in 20 mL of methanol solution at hot temperature (80°C). The solutions are steered up during two hours and white precipitate are obtained (**D**).Spectroscopic data:-

1. IR data (cm^{-1}) : (υ_3+ υ_1)SeO_3= (834vs-775vs); υSe-OH=749s; υas$SnBu_2$ 625vs; υs$SnBu_2$=592w; υOH (2964-2880-2780)L, δOH=1681w
2. ^{119}Sn NMR data ($CDCl_3$) :δ_1= +170.73 ; δ_2 = +162.83 ppm

Elemental Analysis:- -% found (% calculated) : C=26.22 (26.17); H=5.30 (5.35)

e/Procedure for synthesis of$(isoPr_2NH_2)_2SeO_4.2Sn^tBu_2Cl_2$(**E**)
A mixture of **$isoPr_2NH_2HSeO_4$.0,5H_2O** (**L_1**) (0.3 g; 0.782 mmol) with $Sn^tBu_2Cl_2$ (0.7406 g; 2.437 mmol) in 20 mL methanol solution gives a clear solution and a white powder (**E**) collected after a slow solvent evaporation.
Spectroscopic data:-

1. IR data (cm^{-1}) :υ_3SeO_4 =(854vs-843vs); υ_1SeO_4 = 799s; υas$SnBu_2$=633vw; υs$SnBu_2$=544w; υNH_2= (2966-2852-2739)L, δNH_2= 1586s
2. ^{119}Sn NMR data ($CDCl_3$)= -

Elemental analysis:- -% found (% calculated) : C=35.28 (35.10); H=7.95 (7.46); N=3.18 (2.92)

a- f/Procedurefor $^iPr_2NH_2HSeO_4.SnCl_2$ SeO_4**F**
The same salt (**L_1**) (0.3 g; 1.23 mmol) mixing with $SnCl_2.2H_2O$ (1.4619 g; 2.0475 mmol) in 20 mL of methanolat 80°C. A clear solution and a white powder is collected after a slow solvent evaporation (**F**).
Spectroscopic data:-

1. IR data (cm^{-1}): (υ_1 + υ_3)SeO_4^{2-}= 932vs-886s; υSeOH= 808s, υNH_2=(3103-2983-2853)L, δNH_3=1601s
2. ^{119}Sn NMR data ($CDCl_3$) :δ_1= -428.64 ppm ; δ_2=-103 ppm

Elemental analysis:--% found (% calculated): C=12.20 (12.45); H=3.13 (2.96); N=2.36 (2.42)

g/ Procedure for synthesis of$(BzEt_3N)_2SeO_4.SeO_4SnCl_2.3H_2O$ (**G**)
On mixing methanol solutions of **$(BzEt_3N)_2SeO_4.H_2O$** (**L_4**) (0.3 g; 0.5686 mmol) with $SnCl_2.2H_2O$ (0.1078 g; 0.4778 mmol) in hot 20 mL of methanol (60°C), compound (**G**) is obtained as white powder after one week slow solvent evaporation.
Spectroscopic data:-

1. IR data (cm^{-1}) :υ_3SeO_4=(893s-790s); υ_1SeO_4=753s

2. ^{119}Sn NMR data (CDCl3) :δ_1= - 411.59 ppm; δ_2= -103 ppm
Elemental analysis:--% found (% calculated) : C=34.44 (34,46); H=5.16 (4.89); N=3,09 (3,09)

Results And Discussion:-

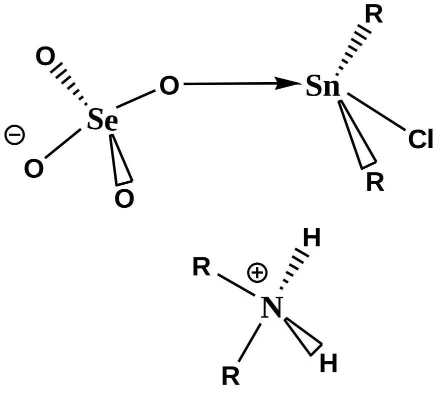
b- $R_2NH_2SeO_4SnR'_2Cl$(R=^{i}Pr, Bu; R'=Me, Bu) **A, B**

The infrared spectra of $^iPr_2NH_2SeO_4SnMe_2Cl$ and $Bu_2NH_2SeO_4SnBu_2Cl$ present many bands in valence zone of SeO_4^{2-} anion showing that this oxyanion is not Td symmetry. In low frequency, it is showing a medium band at 509 cm^{-1} and a weak one at 584 cm^{-1}, respectively in the IR spectra of **A**and**B** corresponding to $\upsilon sSnMe_2$(for **A**) and $\upsilon sSnBu_2$(for **B**). The intensity of these bands is corroborated with a not linear SnC_2residues(Nakamoto and al.).

The proposed structure is discrete with à monodentate seleniate anion. The tin (IV) environment around tin (IV) centre being tetrahedral (Diallo and al.) (figure 1).

The ^{119}Sn NMR spectrumof iso$Pr_2NH_2SeO_4SnMe_2Cl$ complex in DMSO is showing one pic at -333 ppm which is corresponding of a heptacoordinated tin (IV) atom(Holeček and al.). This chemical shift signal reveals a dissociation of the molecular structure and the formation of $[SnMe_2Cl]^+$ cations and the coordination of SeO_4^{2-} anions and DMSO molecules in this complex-cation giving an heptacoordinated tin (IV) atom.

NB: The forms of characteristic absorption bands in the infrared**A**and**B** spectra between 2800 and 3000 cm^{-1} (υNH) and around 1600 cm^{-1}(δNH)areprobably showing the presence of NH----O hydrogen bonds.



R=isoPr Bu; R'=Me, Bu

Figure 1:- Discrete structure of **A**and **B.**

c- $SnBu_2SeO_4$.EtOH**C**

The infrared spectrum of thederivative**C** presents three strong bands in valence zone of SeO_4^{2-}oxyanion. These bands are corresponding to the break-up of T_2 space of υ_3 in three A_1, B_1 and B_2 spaces. This bands show that the SeO_4^{2-} anion is C_{2v} symmetry (Nakamoto and al.). The value of chemical shift in the ^{119}Sn NMRspectrum (δ = -248.53 ppm, almost-260 ppm)shows that the tin (IV) centre is hexacoordinated(Holeček and al.).

The purposed structure is a dimer (figure 2). The seleniate anions are monochelating; the structure contains bicoordinated EtOH molecules with the oxygen atom.

NB:The ABC form of υOH (2957 cm^{-1}-2939 cm^{-1}-2429 cm^{-1}) at high frequency shows the probably presence of hydrogen bonds between the selenate anions and ethanol molecules.When NH---O hydrogen bonds are involved, a supramolecular architecture is considered.

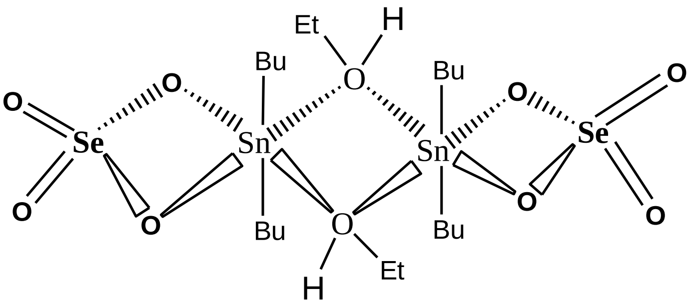


Figure 2:- Dimeric structure of **C.**

d- $HSeO_4SnBu_2Cl.SnBu_2Cl_2.H_2O$**D**

The$HSeO_4SnBu_2Cl.SnBu_2Cl_2.H_2O$compounds (**D**)is a complex-addition containing two molecular components: $HSeO_4SnBu_2Cl$ derivative and one $SnBu_2Cl_2$ molecules. The infrared spectrum of **D**showstwo strong bands at 834 cm^{-1} and 775cm^{-1}in valence zone of the SeO_3 residue corresponding to$\upsilon_3SeO_3$and υ_1SeO_3; The strong band at 749 $cm^{-1}$is corresponding of υSe-OH. In this spectrum, we notetoothe presence of one band at 624 cm^{-1} in weak intensity corresponding to the $\nu SnBu_2$, then the $SnBu_2$residue is almost linear(Nakamoto and al.).

The ^{119}Sn NMR spectrum of **D** present two signals at +170,73 ppm and +162,83ppm. These chemical shifts values are corresponded to tetracoordinated tin (IV) atoms(Holeček and al; Wrackmeyer and al.). These two signals can be explained by a dissociation of $HSeO_4SnBu_2Cl.SnBu_2Cl_2.H_2O$ in $CDCl_3$solventin two molecules: $HSeO_4SnBu_2Cl$ derivative and $SnBu_2Cl_2$ molecule.

When we consider that the water molecule is a coordinated one, the considered structure of $SnBu_2ClHSeO_4.SnBu_2Cl_2.H_2O$is formed by two components: $SnBu_2Cl(H_2O)HSeO_4$ and $SnBu_2Cl_2$molecules. The structure is discrete with a bi-chelating $HSeO_4^-$ and the environment around tin (IV) centre is an octahedral (figure 3).

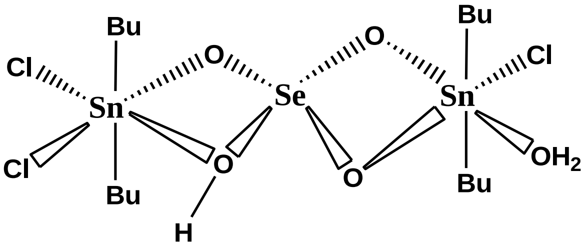


Figure 3:- Discrete structure of **D** with bichelating ligand.

e- $(^iPr_2NH_2)_2SeO_4.2Sn^tBu_2Cl_2$**E**

The$(isoPr_2NH_2)_2SeO_4.2Sn^tBu_2Cl_2$ (**E**) is a complex-addition containing two molecular components: $(^iPr_2NH_2)_2SeO_4$salt and two $Sn^tBu_2Cl_2$ molecules. In the infrared spectrum of **E**, the valence zone of seleniate oxyanion shows the presence of two strong bandsfor $\upsilon_3SeO_4$at 854 cm^{-1} and 843 cm^{-1} and one strong band for $\upsilon_1SeO_4$at 799 cm^{-1}. This observation shows that the symmetry of this oxoanion is Td. The weak band at 633 cm^{-1} is corresponded of the νSn^tBu_2 and can corroborate witha linear Sn^tBu_2 residue (Nakamoto and al.).

The structure is a dimer with a bichelating SeO_4^{2-} oxyanion. The Sn^tBu_2residueare trans coordinated (figure 4).

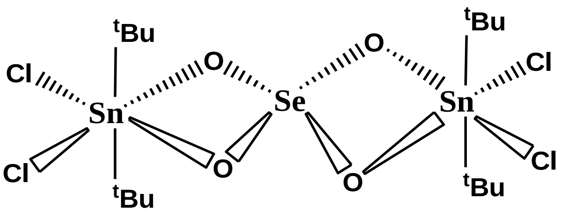


Figure 4:- Discrete structure of **E** with bichelating ligand.

f- $^{i}Pr_2NH_2HSeO_4.SnCl_2$ SeO_4**F**

The infrared spectrum of $^{i}Pr_2NH_2HSeO_4.SeO_4SnCl_2$ (**F**) is showing a lot of strong bands between 932 cm^{-1} and 886 cm^{-1} corroborating with the presence of seleniate oxyanion. The strong band at 808 cm^{-1}corresponds toυSe-OH.

The large absorption around 2900$cm^{-1}$is corresponding of hydroxyd group and can confirmed the presence of $HSeO_4^-$ anion in the complex.

The ^{119}Sn RMN spectrum shows two signals: at -428 ppm in agreement with an octahedral tin atom center and at -103 ppm corresponding to a hexacoordinated tin (IV) atom (Holeček and al; Wrackmeyer and al.).

These NMR data can explain by a dimerization and rearrangement which modified the formular of $^{i}Pr_2NH_2HSeO_4.SeO_4SnCl_2$. In the $CDCl_3$ solvent, the complex is decomposed into two components: $[SeO_4.SnCl_4](iPr_2NH_2^+)_2$complex cation and $[Sn(SeO_4)(HSeO_4)_2]$ complex

Thesignal at -428 ppm corresponds of $[SeO_4.SnCl_4](iPr_2NH_2)_2$ and thesignal at -103 ppm correspondsin $[SeO_4.SnCl_4]^{2-}$.

The structure is ionic with three components; the seleniate and hydrogenoseleniateionsbeingmonochelating ligands. The tin atoms are hexacoordinated in the two complex components(SnO_6 and SnO_2Cl_4) and the environment around the tin (IV) centre being octahedral (figure 5).

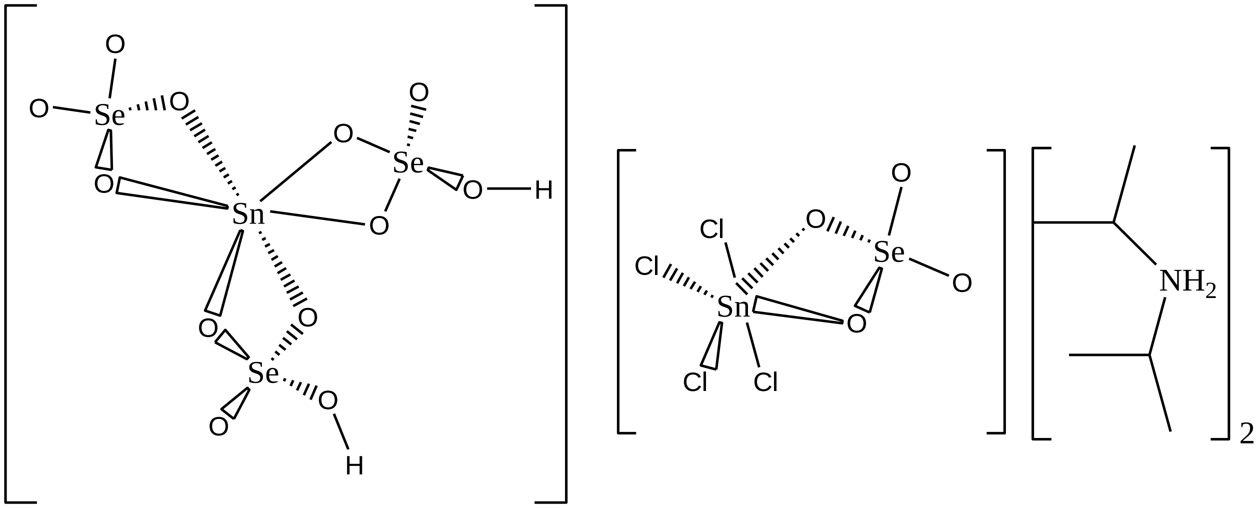


Figure 5:-Ionic structure of F with two complex components.

g- $(BzEt_3N)_2SeO_4.SnCl_2SeO_4.3H_2O$**G**

The threebands in thevalencezoneofseleniateanion in theinfraredspectrumshowthattheseleniateisTdsymmetry(Nakamotoand al.)

The ^{119}Sn RMNspectrumshowstwosignals at -411 ppm and -106 ppm. These values are almost look like those of the previous complex($^{i}Pr_2NH_2HSeO_4.SnCl_2$ SeO_4) in the same solvent. They are showing a dimerization giving $[SeO_4.SnCl_4](BzEt_3N)_4[Sn(SeO_4)_3]$,then a decomposition into two components: $[Sn(SeO_4)_3]^{2-}$ which has an octahedral environment(δ_1= - 411.59 ppm) and $SeO_4.SnCl_4]^{2-}$which has an hexacoordinated environment (δ_2= -103 ppm)around tinenvironment (IV)(Wrackmeyer and al.).

The chemical structure of the compound **G** is almost identical of the previous structure of complex **F**. they are simply differentfrom involved cations and anions.

Conclusion:-

The seleniato complexes studies are discrete and the essential of anions behaving mono- or bichelating ligands. The $SnBu_2$ residue is linear. For the halostannic complexes, a dimerization and resymmetrization in solution give a structure with two components.

Acknowledgments:-

Dakar group thanks are gratefully acknowledged Dr Laurent Plasseraud (University de Bourgogne Dijon-France) for performing elemental analyses and for the equipment support.

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