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RESEARCH ARTICLE

REACTIVITY OF DISUBSTITUED AMMONIUM SELENIATE SALTS WITH $SnMe_3CL$. X-RAY STRUCTURE OF$Cy_2NH_2SeO_4SnMe_3$

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Abstract

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The reaction of $Cy_2NH_2HSeO_4.H_2O$and $SnMe_3Cl$ led to the formation of $Cy_2NH_2SeO_4SnMe_3$ (1), which crystallizes in the orthorhombic space group Pbca with Z = 16, a = 11.59430(10) Å, b = 19.3138(3)Å, c = 36.8673(6)Å, and V = 8255.7(2)Å^3. The asymmetric unit consists of two $[SeO_4SnMe_3]^-$ in which the $SnMe_3$ residue is trans coordinated. The complex-anion are linked via NH----O hydrogen bonds which are involved by $Cy_2NH_2^+$cation. From a supramolecular point of view, the superposition of $[SeO_4SnMe_3]^-$ along a axis are linked by the NH---O of $Cy_2NH_2^+$cation along c axis giving a tridimensional structure. The characterization of 1 is accompanied by infrared and NMR characterization of two seleniate complexes containing $SnMe_3$ residue to complete the study of reactivity between dialkylammonium seleniate salt and $SnMe_3Cl$ molecule.

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Introduction:-

The reactivity of tin trialkyl prompted our research group to synthesize new compounds with oxo anions such as selenate. These compounds find their applications in several fields such as medicine, agriculture and industry. Recently, we studied and published some papers dealing with spectroscopic characterized and X ray structure of new seleniate compounds: $[(Ph_3Sn)_2SeO_4]\cdot CH_3OH$ is isolated fromthe crystallization of $[(Ph_3Sn)_2SeO_4]\cdot 1.5H_2O$ in methanol**:** this Xray structure is polymeric with a planar and none planar $SnPh_3$ residues, the SeO_4^{2-} oxyanion being tri-unidentate [1]. The Xray and spectroscopic analysis of polymericstructure of $[(Me_3Sn)_3(SeO_4)(OH)]_n$ has been reported: This structure contains two $SnMe_3$ residues: an tetrahedral and pendant $SnMe_3$ residue and another $SnMe_3$ residue which is planar[2]**.** In the logic of our research, we have been isolated the complex$Cy_2NH_2SeO_4SnMe_3$ as suitable and colorless crystals from a reaction between H_2SeO_4, $SnMe_3Cl$ and Cy_2NH in "one pot" process. We have isolated too some seleniate complexes as white powder $Bu_2NH_2HSeO_4.SnMe_3Cl$ and $Bu_2NH_2SeO_4SnMe_3.SnMe_3Cl$. The crystal compound $Cy_2NH_2SeO_4SnMe_3$ is studied by X-ray diffraction. It crystallized in orthorhombic system with Pbca space group. The crystal is colorless with 0.36 × 0.2 × 0.18 size. The structure is a 3D supramolecular. We have accompanied of this interesting seleniatoorganostannic complex with two other complexes belonging in the

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same complex family. $Bu_2NH_2HSeO_4.SnMe_3Cl$ and $Bu_2NH_2SeO_4SnMe_3.SnMe_3Cl$ have been isolate as white powder from reaction between $Bu_2NH_2HSeO_4.H_2O$ ligand and $SnMe_3Cl$ molecule in 1: 1 and 1: 2 ratio. These seleniato complexes have been characterized by infrared, ^{119}Sn NMR spectroscopies.

Materials And Methods:-

H_2SeO_4 was purchased from Merck Chemicals. Hohenbrunbei München. Germany while Cy_2NH, n-Bu_2NH and $SnMe_3Cl$ were acquired from Sigma-Aldrich Chemie GmbH. Steinheim. Germany. All chemicals were purchased from Aldrich or Merck chemical companies and were used without 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 spectra are reported downfield from Me_4Sn. as the internal standard. Infrared data are given in cm^{-1} and chemical shifts (□□ in ppm.[IR abbreviations: (br) broad. (vs) very strong. (s) strong. (m) medium. (sh) shoulder. (w) weak. (vw) very weak].

Elemental analyses were performed at the "Institut de Chimie Moléculaire". University of Burgundy. Dijon-France.

The crystallographic data were obtained by a Super Nova diffractometer (Dual source), Rigaku Oxford, 4-circle Goniometer diffraction, double micro-focus Cu and Mo source, 135mm Atlas S2 CCD detector with a binocular magnifier Leica M125 equipped a CCD camera and image acquisition software. The structure was solved by olex2 and refined by SHELXL.

Salts synthesis

The salt $Bu_2NH_2HSeO_4.H_2O$ (**L_1**), $Cy_2NH_2HSeO_4.H_2O$ (**L_2**), are synthetized by neutralized the selenic acid H_2SeO_4 by di-substituted amine R_2NH (R=Bu, Cy) in water solvent. After two hours mixed up, colorless solution is obtained, submitted to evaporation at 60° C and white powder are obtained after one week.

The analytical data of salts are given in the table below

salts	**Chemical formular**	**C**		**H**		**N**	
		Calc	**Found**	**Calc**	**Found**	**Calc**	**Found**
L_1	**$Bu_2NH_2HSeO_4.H_2O$**	32.88	32.54	7.93	8.09	4.79	4.91
L_2	**$Cy_2NH_2HSeO_4.\ H_2O$**	41.86	42.01	7.90	7.85	4.07	4.12

Synthesis of $Cy_2NH_2SeO_4SnMe_3$ By reacting **L_2**salt(m=; n=2.069mmol) with $SnMe_3Cl$ (0,4137g; 2,069mmol) in 20 mL of methanol solvent, a clear solution is obtained after two hours of stirring at room temperature. This clear solution is filtered and the filtrate is evaporated slowly and the suitable crystal are obtained.

Synthesis of $Bu_2NH_2HSeO_4.SnMe_3Cl$

A mixture of **L_1** salt (m=0.5g; n=0.391mmol) and $SnMe_3Cl$ (0,4137g; 2,069mmol), in 20mL of absolute ethanol solvent, gives a limpid solution after two hours steered up. This solution is submitted to slow solvent evaporation. After one week, a white powder is obtained.

Synthesis of $Bu_2NH_2SeO_4SnMe_3.SnMe_3Cl$

When **L_1** salt (0,3g ; 0,782 mmol) is reacted with $SnMe_3Cl$ (0,4137g; 2,069 mmol) in 20 mL of absolute ethanol solvent, a white precipitate is obtained after two hours steered up.

The analytical data are given in the table below.

Adducts	Chemical formulae	C		H		N	
		Calc	Found	Calc	Found	Calc	Found
A	**$Bu_2NH_2HSeO_4.SnMe_3Cl$**	27,88	27.90	6,56	6.39	2,85	2.96
B	**$Bu_2NH_2SeO_4SnMe_3.SnMe_3Cl$**	26,99	26.43	5,78	6.02	2,33	2.20
C	**$Cy_2NH_2SeO_4SnMe_3$**	36.83	36.61	6.80	6.65	2.86	3.05

Results And Discussions:-

Spectroscopic data of $Bu_2NH_2HSeO_4.SnMe_3Cl$

IR data (cm^{-1}): υNH_2 (2961-2872)L, δNH_3 (1591m), $(\upsilon_3+\upsilon_1)SeO_3$= (899vs-815m); υSe-OH=756F; $\upsilon asSnMe_3$=533m, $\upsilon sSnMe_3$= 510vw RMN $^{119}Sn\{^1H\}$ data (CD_3OD): δ = -127,92 ppm

- Spectroscopic data of $Bu_2NH_2SeO_4SnMe_3.SnMe_3Cl$

IR data (cm^{-1}): υNH_2= (2962-2873)L, δNH_2= (1591m), υ_3SeO_4= 846vs; υ_1SeO_4 = 758vs; $\upsilon asSnMe_3$= 548F, $\upsilon sSnMe_3$= 514 tr RMN $^{119}Sn\{^1H\}$data (CH_3OD): δ= +183,07 ppm; δ= -128,20 ppm

- Spectroscopic data of $Cy_2NH_2SeO_4SnMe_3$

IR data: $(\nu_1+\nu_3)SeO_4$ = 874-852s, $\nu asSnMe_3$ = 568vs, $\nu sSnMe_3$= 520m, νNH_2 = (2856-2789-2753) br, δNH_2= 1559s, νOH= 2931br. `

- Crystallography data of $Cy_2NH_2SeO_4SnMe_3$

Formula $C_{15}H_{33}NO_4SeSn$, M= 488.67g/mol, white crystal, a =11.59430(10), b = 19.3138(3), c = 36.8673(6) $\overset{\circ}{A}$, α = 90°, β = 90°, γ = 90°, V = 8255.7(2) $\overset{\circ}{A}$, Z = 16, orthorhombic system, Pbca space group, R_{int} = 0.0825, d_{min}(Mo) = 0.83, R_1 = 0.0550, wR_2 = 0.11.

1- Infrared and ^{119}Sn NMR spectroscopic studies of complexes

- $Bu_2NH_2HSeO_4.SnMe_3Cl$ **A**

This seleniato complex is obtained in-situ at 1: 1 addition reaction between $Bu_2NH_2HSeO_4$ salt and $SnMe_3Cl$.The examination of $Bu_2NH_2HSeO_4.SnMe_3Cl$ by FT-IR spectroscopy in ATR mode shows that intense bands are mainly present in the range 950-500 cm^{-1}. The infrared data reveals an antisymmetric valence vibration as a strong band at 899 cm^{-1} and a symmetric valence vibration at 815 cm^{-1} of seleniate oxyanion. The strong band at 756 cm^{-1} shows that the seleniate anion is substitute one (Se-OH bond) [3, 4]. In low frequency, the $\upsilon asSnMe_3$ appears at 553 cm^{-1} but the presence of $\upsilon sSnMe_3$ as broad is corroborated as a planar $SnMe_3$residu[5, 6].

The $^{119}Sn\{^1H\}$ NMR spectrum of $Bu_2NH_2HSeO_4.SnMe_3Cl$ reveals also only one resonance signal located at -127 ppm corroborated as the presence of tetracoordinated tin species [7]. (we remember that the chemical shift of free $SnMe_3Cl$ is +164 ppm according to Davies and Smith [8]. This chemical value is explained that a mutation of the complex to two components: a free Bu_2NH_2Cl salt and $HSeO_4SnMe_3$ derivative containing a monocoordinated $SnMe_3$ residue. The structure in spectroscopic basis is identical of the crystal structure of $Bu_2NH_2HSO_4.SnMe_3Cl$ reported by Diallo and al is a dimer [9]. The monomer is $[HSeO_4.SnMe_3Cl]^-$ is discrete; there are linking via OH---O hydrogen bonds monodentate seleniate anion. The tin (IV) atom centre is trans bipyramidal trigonal. (figure).

- $Bu_2NH_2SeO_4SnMe_3.SnMe_3Cl$**B**

This complex is obtained in-situ at a succession of substitution and addition reaction.Theinfrared spectroscopy analysis of $Bu_2NH_2SeO_4SnMe_3.SnMe_3Cl$ mainly shows the absorption bands corresponding to νSeO_4 at 846, 758, and 693 cm^{-1}[3,4]. The infrared spectrum shows a burst of the T_2 species of the υ_3SeO_4 oxoanion to an E and A_1 species. This burst of the antisymmetric valence shell reveals a none Td symmetry for the SeO_4^{2-} oxyanion. The large absorption around 2900 cm^{-1} is corresponding of NH bonds of the cation $Bu_2NH_2^+$, then the form of the band shows NH---O hydrogen bonds. The $\upsilon asSnMe_3$ appears as strong bond at 548 cm^{-1} but the $\upsilon sSnMe_3$ at 514 cm^{-1} as a trace shows that $SnMe_3$ residue is planar[10]

In solution, despite the difficulties of solubilization in usual solvents, we were able to obtain the ^{119}Sn NMR fingerprint of $Bu_2NH_2SeO_4SnMe_3.SnMe_3Cl$in CH_3OD showing two resonancesat $\delta=-128.20$ and $\delta=+183.07$ ppm. The first value of chemical shift, are consistent with five-coordinated tin(IV) atoms in a trans trigonal bipyramidal geometry environment [11] and corresponds to a trans coordinated $SnMe_3$ residue. The second value shows a dissociation and liberation of $SnMe_3Cl$ molecule according to Davies and Smith [8]

This complex is considered as 1 : 1 complex of two components $[SeO_4SnMe_3]^-$ complex-anion and $SnMe_3Cl$ molecule. The structure is an infinite chain of $[SeO_4SnMe_3]^-$ anion coordinated with a pendant $SnMe_3Cl$ molecule; The SeO_4^{2-} ligand being bidentate. This structure is identical of $Bu_4NSO_4SnMe_3.SnMe_3Cl$ one published by Diop and al [12].

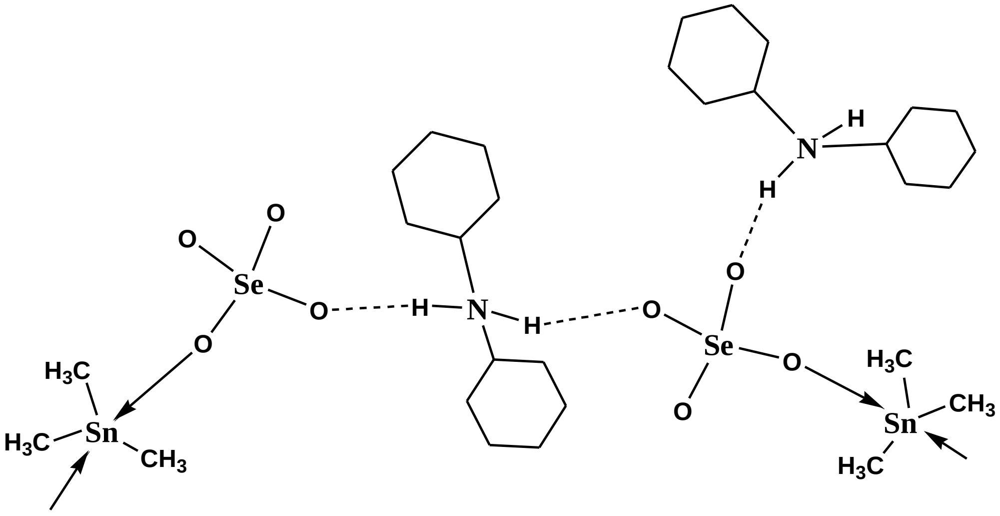
- $Cy_2NH_2SeO_4SnMe_3$**C**From a synthetic point of view, the preparation of **C** requires two steps: In the first time, $(Cy_2NH_2)_2SeO_4$ salt is isolated by reacting selenic acid (H_2SeO_4) and dicyclohexylaminat room temperature (Eq. 1). This synthesis method is similar of those we recently reported to isolate selenato salts bis(di-iso-propylammonium) selenate and di-n-butylammoniumhydrogenoselenate, characterized at the solid state by X-ray crystallographic analysis [13].

The$(Cy_2NH_2)_2SeO_4$ isolated as white powder was then treated with the tin(IV) precursor, $SnMe_3Cl$, in methanol and in a 1:2 molar ratio at room temperature (Eq. 2). Colorless single crystals were collected and were characterized as $Cy_2NH_2SeO_4SnMe_3$(**1**). Together with the crystallization of **1**, it is accompanied with colorless crystals corresponding of Cy_2NH_2Cl which was published previously by Seik [14]. Further work is nevertheless in progress to verify this hypothesis.

$$Cy_2NH + H_2SeO_4 \xrightarrow[\text{eau}]{T^o\text{ amb}} Cy_2NH_2HSeO_4 \qquad \text{Eq1}$$

$$Cy_2NH_2HSeO_4 + SnMe_3Cl \xrightarrow[\text{MeOH}]{60^o\text{ C}} Cy_2NH_2SeO_4SnMe_3 + HCl \qquad \text{Eq2}$$

The infrared spectrum of the compound shows: The presence of two strong bands at 874 and 852 cm^{-1} confirms the presence of the SeO_4^{2-} ion. The bursting of the absorption in the form of several bands in the valence zone of the oxoanion also confirms the presence of selenate ion of not Td symmetry. The bands of the cation appear at 2856, 2789, 2753cm^{-1} and the broad band centered at 2931cm^{-1} confirms the presence of NH---O hydrogen bonds. Indeed, this series of three bands can be explained by the resulting Fermi resonances between υNH and the overtones 2δNH and 2γNH (Sarr and Diop, 1987) [15]. The presence of one medium band at 543 cm^{-1} the medium band corresponds of $\nu asSnMe_3$; then the presence of $\nu sSnMe_3$ at 520 cm^{-1} as very weak band implies that $SnMe_3$ is planar.



Scheme:- Structure of $Cy_2NH_2SeO_4SnMe_3$ basis to the spectroscopic data.

Determination of the structure of the crystalof$Cy_2NH_2SeO_4SnMe_3$

The asymmetric unit of the crystal complex is dimeric $(Cy_2NH_2)_2(SeO_4)_2(SnMe_3)_2$. Each SeO_4^{2-} anion coordinates one $SnMe_3$ residue. Neighboring $[SeO_4SnMe_3]^-$ complex-anion are attached by $Cy_2NH_2^+$ cation via hydrogen bonds between one free oxygen atom of each anion and the hydrogen atoms of $Cy_2NH_2^+$ cation. The asymmetric structure of this complex is showed a monocoordinated SeO_4^{2-} as in the $[n\text{-}Bu_2NH_2]_3[SnPh_3(SeO_4)_2]$ Xray structure reported by Diallo and al. [16]. Crystallographic data and refinement details are summarized in Table 1. An Ortep view, together with selected bonds lengths and angles, is shown in Figure 1.

Table 1:- Crystal data and structure refinement.

Empirical formula	$C_{30}H_{66}N_2O_8Se_2Sn_2$
Formula weight	978.14
Temperature/K	293(2)
Crystal system	orthorhombic
Space group	Pbca
a/Å	11.59430(10)
b/Å	19.3138(3)
c/Å	36.8673(6)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	8255.7(2)
Z	16
ρ_{calc}g/cm^3	3.345
μ/mm^{-1}	6.409
F(000)	8364.0
Crystal size/mm^3	0.36 × 0.2 × 0.18
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	5.6 to 54.526
Index ranges	$-14 \leq h \leq 14$, $-24 \leq k \leq 23$, $-45 \leq l \leq 46$
Reflections collected	115276
Independent reflections	8871 [R_{int} = 0.0815, R_{sigma} = 0.0349]
Data/restraints/parameters	8871/0/397
Goodness-of-fit on F^2	1.695
Final R indexes [I>=2σ (I)]	R_1 = 0.0776, wR_2 = 0.2322
Final R indexes [all data]	R_1 = 0.0961, wR_2 = 0.2421
Largest diff. peak/hole / e Å^{-3}	1.73/-1.88
CCDC Number	2110794

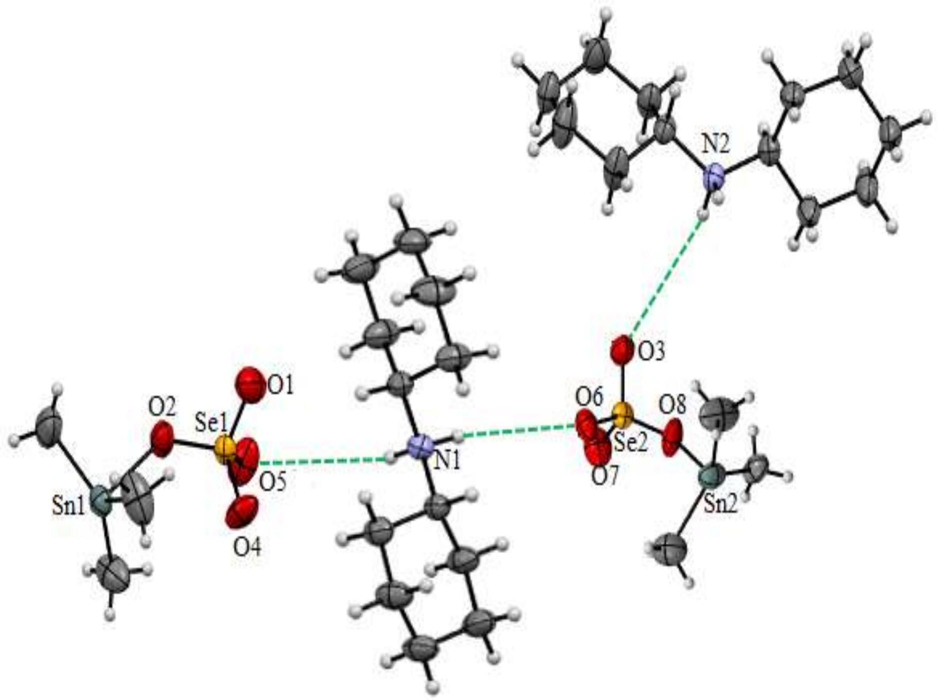


Figure 1:- Asymmetric structure of **$Cy_2NH_2SeO_4SnMe_3$** (**1**).

showing 30% probability ellipsoids and the crystallographic numbering scheme (Ortep view). Selected bond lengths and angles [Å, °]: Sn1 C2 2.103(8), Sn1 C10 2.099(8), and Sn1 C11 2.117(8); Sn2 C9 2.095(9) ; Sn2 C9 2.095(9) ; Sn2 C30 2.165(16) ; Sn2 C37 2.21(2) ; Se1 O1 1.639(4) ; Se1 O3 1.612(4) ; Se1 O4 1.602(5) ; Se1 O5 1.632(5) ; Se2 O2 1.594(5) ; Se2 O6 1.653(8) ; Se2 O7 1.683(9) ; Se2 O8 1.608(11) ; Se2 O9 1.540(10) ; Se2 O10 1.669(11) ; Se2 O11 1.544(10) ; C2 Sn1 C10 117.4(3), C2 Sn1 C11 123.7(3), and C10 Sn1 C11 118.9(3) ; C9 Sn2 C37 113.6(6); C9 Sn2 C30 123.8(8); C30 Sn2 C37 122.4(9) ; O1 Se1 O3 111.7(2) ; O1 Se1 O4 106.4(3) ; O1 Se1 O5 108.7(3) ; O3 Se1 O4 112.2(3) ; O3 Se1 O5 107.8(3) ; O4 Se1 O5 110.2(3)

The equatorial plane is occupied by the three methyl groups [Sn1 C2 2.103(8), Sn1 C10 2.099(8), and Sn1 C11 2.117(8); Sn2 C9 2.095(9) ; Sn2 C9 2.095(9) ; Sn2 C30 2.165(16) ; Sn2 C37 2.21(2) Å]. The Sn-C distances of the complex **1** are in the typical range of Sn-C bonds reported earlier for related compounds [17, 16]. The sum of the angles at tin (1) [C2 Sn1 C10 117.4(3), C2 Sn1 C11 123.7(3), and C10 Sn1 C11 118.9(3)°] is equal to 360°. And the sum of the angles at tin (1) C9 Sn2 C37 113.6(6); C9 Sn2 C30 123.8(8); C30 Sn2 C37 122.4(9) is equal to 359.8°. The apical positions are occupied by two oxygen atoms of two monodentate SeO_4^{2-} terminally coordinated [Sn1 O1 2.252(5); Sn1 O5 2.269(5); Sn2 O8 2.391(11); Sn2 O9 2.304(10) Å]. The Sn-O distances are in the range of those already reported for monodentate by [16]. The O-Sn-O angles around Sn(1) and Sn(2) [O1 Sn1 O5 174.9(2); O9 Sn2 O8 144.5(4) °] indicate a slight deviation from linearity around Sn(1) but a real deviation from linearity around Sn(2). Further information can be obtained by estimating the structural index τ, which represents the relative amount of trigonality [for a square pyramid, $\tau = 0$; for a trigonal pyramid, $\tau = 1$, with $\tau = (\beta-\alpha)/60°$) and where α and β are the two largest angles] . For **Sn(1)**, O1 Sn1 O5 174.9(2)°] is assigned to β and C2 Sn1 C10 117.4(3)°] to α ; For **Sn(2)**, O9 Sn2 O8 144.5(4) °] is assigned to β and C9 Sn2 C37 113.6(6)°]. The calculated value of $\tau1$ (for Sn(1)) is found to be 0.958(6)° and $\tau2$ (for Sn(2)) is found to be 0.515(10)°, which expresses a distorted character for the trigonal bipyramidal geometry, which is consistent with earlier reports [18].

In the crystal lattice, the two SeO_4SnMe_3 frameworks are linked by a SeO_4^{2-} coordinated the two Sn(IV) of distinct $SnMe_3$ residues. Two neighboring $(SeO_4SnMe_3)SeO_4(SeO_4SnMe_3)$ molecule part are linked on both sides by NH…..O hydrogen bonds which are involved by noncoordinated oxygen atoms of the end $SeO4^{2-}$ anions and the hydrogen atom of two distinct surrounding $Cy_2NH_2^+$ cations [N1H1…..O31.910(7); N2H2…..O41.820(7)] giving a macrocyclic building (Figure 2).

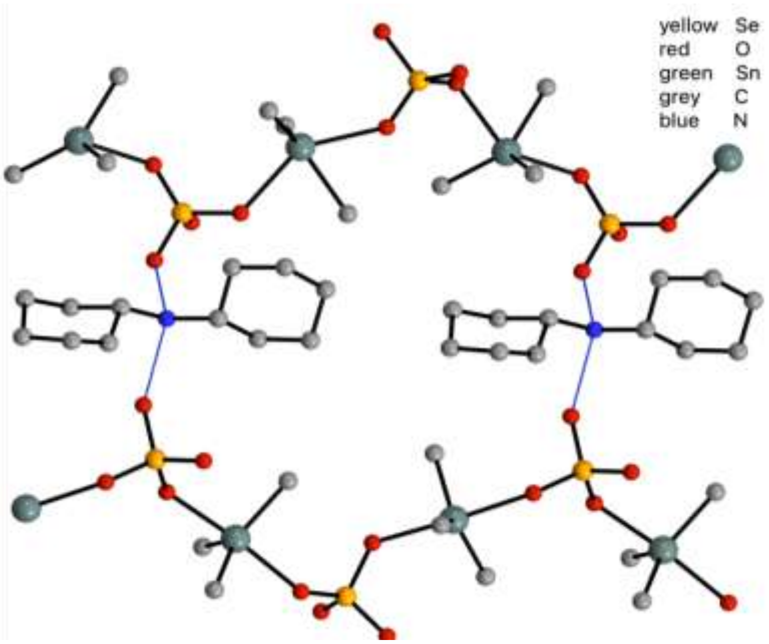


Figure 2:- Crystal packing of 1 through intermolecular NH···O hydrogen bonds.

From a supramolecular point of view, the structure is showed a superposition of complex-anion $[SeO_4SnMe_3]^-$. These anions are alternately linked to each other by a transcoordination of the tin atom (IV) of one of the $[SeO_4SnMe_3]^-$ ion by the noncoordinated oxygen atom of the SeO_4^{2-} oxoanion of the neighboring $[SeO_4SnMe_3]^-$ complex-anion. The superposition of these complex-anion gives an evolution of the structure along a axis.

Along c axis, the $[SeO_4SnMe_3]^-$ anions are linked via NH----O hydrogen bonds which are involved by the hydrogen atom of $Cy_2NH_2^+$ cation and one of free oxygen atom of SeO_4^{2-} oxyanion.

The progress of the structure along a axis by a superposition of $[SeO_4SnMe_3]^-$ anions and along c axis via NH----O hydrogen bonds are involved a tridimensional structure.

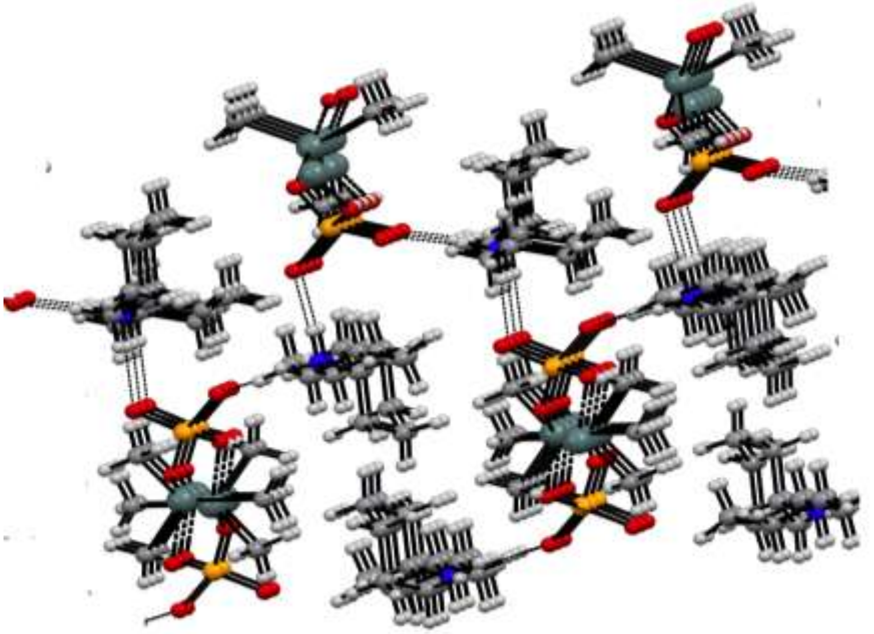


Figure 3:- Representation of the resulting supramolecular structure of 1 highlighting the square-shaped macrocycle organization. **Red=O; yellow=Se; blue=N.**

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