

# Synthesis and Structural Characterization of New Ladder Organostannoxanes Derived from Sulfonic Acid Derivatives

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**Abstract:** Two new ladder n-dibutyltin(IV) compounds with general formulae  $[n\text{-Bu}_2\text{Sn}]_4(\mu_3\text{-O})_2(\mu_2\text{-OH})_2(\text{OSO}_2\text{R})_2$  with  $\text{R}=4\text{-HO-C}_6\text{H}_4\text{-}(1)$  and  $\text{CH}_3\text{-}(2)$  have been synthesized and characterized by elemental analysis, FT-IR,  $^1\text{H}$  NMR and Mass spectroscopies techniques as well as single crystal X-ray diffraction study. The complex 1 crystallizes in the triclinic space group  $P1$  with  $a=12.808$  (8),  $b=15.391$  (9),  $c=15.803$  (10) Å,  $\alpha=87.910$  (5),  $\beta=66.650$  (6),  $\gamma=80.283$  (5)°,  $V=2817.6$  (3) Å<sup>3</sup> and  $Z=1$ . The crystal structure reveal them to be tetranuclear tin (IV) ladder-like structure containing two deprotonated ligands an three-rung-staircase  $\text{Sn}_4\text{O}_4$  core. The structure is a centro-symmetric dimer of an oxoditin (IV) unit having a central four-member ring. In this complex, the central tin (IV) atoms are pentacoordinated, where the sulfonate base acts as monodentate ligands. Supramolecular architecture is formed by extensive hydrogen-bonding interactions,  $\text{O-H}\cdots\text{O}$ .

**Keywords:** Organostannoxane, Pyridine-3-sulfonate, 4-hydroxybenzenesulfonate, Tetranuclear Ladder

## 1. Introduction

Organotin(IV) compounds as an important branch of organometallic compounds, have received the most attention due to their diverse structures including drums [1], cubes [2], O-capped clusters [3], ladders [4], hydroxyl-bridged dimers [5]. In particular, organotin compounds show a wide variety organostannoxanes. The organostannoxanes have received special attention particularly in the view of their immense structural diversity. Several products, such as ladders [6, 7] cubes [8], hexameric [9] and polymeric drums [10] have isolated. Ladder-shaped tetranuclear organotin compounds [11, 6, 12], have been isolated. Many butylstannoxanes were prepared using carboxylates ligands [9, 11, 13, 14]. To the best of our knowledge, there are only a few structurally known examples among the family of organostannoxane-sulfonates  $[(\text{Me}_2\text{Sn})_4(\text{O})_2(\text{OH})_2(\text{O}_3\text{SC}_6\text{H}_4\text{-NH}_2\text{-4})_2]$  (supermolecular architecture),  $[(n\text{-BuSn})_{12}\text{O}_{14}(\text{OH})_6](\text{O}_3\text{SC}_6\text{H}_4\text{-NH}_2\text{-4})_2\cdot 3\text{C}_4\text{H}_8\text{O}_2]$  (dodecanuclear) [6] and  $[(n\text{-C}_4\text{H}_9)_2\text{Sn}\{\text{OSO}_2\text{C}_6\text{H}_3(\text{CH}_3)_2\text{-2,5}\}_2]$  (centrosymmetric dimer) [15]. The aim of the present contribution

was to prepare and characterize organostannoxane-type structures from sulfonates,  $[n\text{-Bu}_2\text{Sn}]_4(\mu_3\text{-O})_2(\mu_2\text{-OH})_2(\text{OSO}_2\text{R})_2$  ( $\text{R}=4\text{-HO-C}_6\text{H}_4\text{-}$  and  $\text{CH}_3\text{-}$ ).

## 2. Experimental Section

### 2.1. Physical Measurements

The melting points were obtained with Kofler micromelting point apparatus and were uncorrected. Elemental analyses (C, H) were performed on a Perkin-Elmer model 2400 CHN elemental analyser. Mass spectrum (EI, 70 eV) was obtained on a VG Analytical 11-2505-70 SMS mass spectrometer. IR spectra in the range  $4000\text{-}400\text{ cm}^{-1}$  were recorded on FT-IR spectrophotometer Nicolet 710 TF-IR operated by the OMNIC software.  $^1\text{H}$  NMR spectra were recorded in  $\text{CDCl}_3$  solution on BRUKER DPX-300 and BRUKER AVANCE II 400 spectrometers with Topspin 2.1 as software. The spectra were acquired at room temperature (298K). The chemical shifts were reported in ppm with respect to the references and were stated relative to external tetramethylsilane (TMS).

## 2.2. X-ray Crystallography

**Table 1.** Crystal's data and structures refinement of complex.

| Empirical formula  | C <sub>44</sub> H <sub>84</sub> S <sub>2</sub> O <sub>12</sub> Sn <sub>4</sub> |
|--|--|
| Formula weight, g·mol <sup>-1</sup>  | 1344.1   |
| Crystal system   | Triclinic  |
| Space group  | P1 <sub>1</sub> -  |
| <i>a</i> , Å; α, °   | 12.8083(8); 87.910 (5)   |
| <i>b</i> , Å; β, °   | 15.3912(9); 66.650 (6)   |
| <i>c</i> , Å; γ, °   | 15.8031(10); 80.283 (5)  |
| <i>V</i> , Å <sup>3</sup>  | 2817.6 (3)   |
| <i>Z</i>   | 32   |
| <i>D</i> <sub>calcd</sub> , g cm <sup>-3</sup>                               | 1.585  |
| Temperature, K   | 293  |
| μ(MoKα), mm <sup>-1</sup>  | 1.88   |
| <i>F</i> (000), <i>e</i>   | 1354   |
| θ range for data collection, deg   | 1.8 – 29.1   |
| Reflections collected  | 30677  |
| Independent reflections  | 13001  |
| <i>R</i> <sub>int</sub>  | 0.032  |
| Data / restraints / parameters   | 9935 / 360 / 324   |
| <i>R</i> <sub>1</sub> / <i>wR</i> <sub>2</sub> [ <i>I</i> > 2 σ( <i>I</i> )] | 0.061  |
| <i>R</i> <sub>1</sub> / <i>wR</i> <sub>2</sub> (all data)                    | 0.056  |
| Goodness-of-fit on <i>F</i> <sup>2</sup>                                     | 1.08   |
| Δρ <sub>fin</sub> (max / min), e Å <sup>-3</sup>                             | 4.33/–5.09   |

The X-ray crystallographic data was collected using a Nonius Kappa CCD diffractometer operating at *T*=293(2) K. Data was measured using φ and ω scans with MoKα radiation (λ=0.71073 Å) and a collection strategy to obtain a hemisphere of unique data as determined by COLLECT [16]. Cell parameters were determined and refined using DIRAX [17]. Data was corrected for absorption correction using EVAL

CCD [18]. The structure was solved by Direct Methods using SHELXS [19]. The structure was refined using least-squares minimization with SHELXL [20]. Programs used for the representation of the molecular and crystal structures: OLEX2 [21] and MERCURY [22]. Crystal data, data collection and structure refinement details for complex are summarized in Table 1.

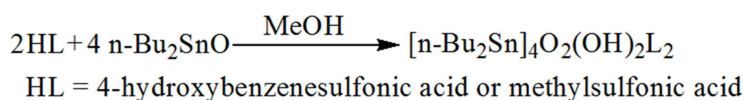
### Accession codes

CCDC 20533059 (1) contain the supplementary crystallographic data for this paper. Copies can be obtained free of charge from the Cambridge Crystallographic Data Centre (CCDC), 12 Union Road, Cambridge CB2 1EZ, UK (fax: int. Code +44 1223 336 033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

## 3. Results and Discussion

### 3.1. Materials and Synthesis

The di-*n*-butyltin(IV) oxide, 4-hydroxylbenzenesulfonic acid and methylsulfonic acid were purchased from Sigma–Aldrich and used as received. Methanolic solutions of 4-hydroxylbenzenesulfonic acid or methylsulfonic acid and di-*n*-butyltin(IV) oxide, n-Bu<sub>2</sub>SnO, in 1/2 molar ratios were mixed and stirred at room temperature for more than 4h (Figure 1). The solution was the left for the solvent to slowly evaporate giving colorless crystals (1) and suitable for x-ray diffraction analysis and powder (2).



**Figure 1.** Synthesis of complexes 1 and 2.

#### 3.1.1. Synthesis of



A methanolic solution containing 4-hydroxybenzenesulfonic acid 0.20 g (1.15 mmol), (4-OH)-C<sub>6</sub>H<sub>4</sub>-SO<sub>3</sub>H, was added to a methanolic solution which contains 0.57 g (2.30 mmol) of di-*n*-butyltin(IV) oxide, n-Bu<sub>2</sub>SnO. The mixture was stirred for around three hours at room temperature and upon slow solvent evaporation gave prismatic crystals suitable for X-ray diffraction analysis. Yield: 71%; m. p: 187°C. – IR (ATR, cm<sup>-1</sup>): 3350m νOH, 1578ν(C=C), 1243, 1153, 1111 ν(SO<sub>3</sub>), 623 vs(Sn-O-Sn) 609 *vas*(SnC<sub>2</sub>), 615 *vs*(SnC<sub>2</sub>), 450 ν(Sn-O). – <sup>1</sup>H NMR (CDCl<sub>3</sub>, ppm): δ(ppm), 0.80 (m), 1.23 (m), 1.30 (m), 1.60 (m) (18H, n-C<sub>4</sub>H<sub>9</sub>Sn); 7.77 – 6.80 (aromatic protons). The following tin containing fragments have been recorded in its mass spectrum *m/z*: 176.83 [Sn–C<sub>4</sub>H<sub>9</sub>]<sup>+</sup>; 256.9 [Sn–C<sub>4</sub>H<sub>9</sub>–SO<sub>3</sub>]<sup>+</sup> (24); 296.92 [Sn–OSO<sub>2</sub>C<sub>6</sub>H<sub>4</sub>(4-OH)]<sup>+</sup>. –Anal. calc. for C<sub>44</sub>H<sub>84</sub>S<sub>2</sub>O<sub>12</sub>Sn<sub>4</sub> (1344.1): C, 39.32 H, 6.30;%. Found C 39.53; H, 6.50.

#### 3.1.2. Synthesis of [n-Bu<sub>2</sub>Sn]<sub>4</sub>(μ<sub>3</sub>-O)<sub>2</sub>(μ<sub>2</sub>-OH)<sub>2</sub>[OSO<sub>2</sub>-CH<sub>3</sub>]<sub>2</sub>

The preparation [n-Bu<sub>2</sub>Sn]<sub>4</sub>(μ<sub>3</sub>-O)<sub>2</sub>(μ<sub>2</sub>-OH)<sub>2</sub>[OSO<sub>2</sub>-CH<sub>3</sub>]<sub>2</sub> is

like that of complex 1 except that methylsulfonic acid was used to replace 4-hydroxybenzenesulfonic acid in 1/2 molar ratios. The mixture was stirred for around four hours at room temperature and upon slow solvent evaporation gave with powder analysis. Yield: 65%; m.p: 165°C. – IR (ATR, cm<sup>-1</sup>): 1174, 1136, 1042 ν(SO<sub>3</sub>), 631 vs(Sn-O-Sn), 619 *vas*(SnC<sub>2</sub>), 614 *vs*(SnC<sub>2</sub>), 453ν(Sn-O). – <sup>1</sup>H NMR (CDCl<sub>3</sub>, ppm): δ(ppm), 1.02 (m), 1.17 (m), 1.30 (m), 1.800 (m) (18H, n-C<sub>4</sub>H<sub>9</sub>Sn); 2.90 (s, SCH<sub>3</sub>). The following tin containing fragments have been recorded in its mass spectrum *m/z*: 842 [Bu<sub>6</sub>Sn<sub>3</sub>O<sub>2</sub>OH-O<sub>3</sub>SMe]<sup>+</sup>, 747.12 [Bu<sub>6</sub>Sn<sub>3</sub>O<sub>2</sub>OH]<sup>+</sup>, 328 [Bu<sub>2</sub>Sn-S(O<sub>3</sub>)Me]<sup>+</sup>, 287 [BuSn(OH)S(O<sub>3</sub>)Me]<sup>+</sup>, 292 [BuSn(OH)<sub>2</sub>S(O<sub>3</sub>)H]<sup>+</sup>. – Anal. calc. for C<sub>34</sub>H<sub>80</sub>S<sub>2</sub>O<sub>10</sub>Sn<sub>4</sub> (1192.13): C, 34.38 H, 6.79. Found C 34.50; H, 6.73.

### 3.2. Spectroscopic Studies

In the IR spectra, the absence of the broad band in the region 3400–2800 cm<sup>-1</sup>, indicates deprotonation and thus, the formation of Sn–O bonds through this site [23]. The band ν(SO<sub>3</sub>) in the I. R spectra is observed at frequencies 1243, 1153, 1111 cm<sup>-1</sup> for complex 1 and 1174, 1136, 1042 cm<sup>-1</sup> for

2. Similar frequencies for  $\nu(\text{SO}_3)$  were observed for organotin (IV) sulfonates [6, 12]. The presence of  $\nu(\text{SnC}_2)_{\text{sym}}$  as a trace band at 614 (1) and 614 (2)  $\text{cm}^{-1}$  are in accordance with a linear  $\text{SnC}_2$  unit [24]. A strong band at 450 (1) and 453 (2)  $\text{cm}^{-1}$  is assigned to  $\nu(\text{Sn-O})$  [25]. This is consistent with the presence of monodentate sulphonates in 1 and 2. The  $^1\text{H}$  data of the three complexes are given in the experimental part and the observed resonances have been assigned on the basis of the chemical shift. The order of the  $^1\text{H}$  chemical shifts of the  $\text{CH}_n$  groups in the Sn-butyl substituents was found to be  $(\alpha, \beta) > (\gamma, \delta)$  for the  $\text{Bu}_2\text{Sn}$  complexes

$(\text{SnCH}_2(\alpha)\text{CH}_2(\beta)\text{CH}_2(\gamma)\text{CH}_3(\delta))$  [23]. Multiplet about 1.50–1.78, 1.30 – 1.23 and 0.76 – 0.87 ppm are attributed to (m,  $\alpha\text{-CH}_2$ ,  $\beta\text{-CH}_2$ ), (m,  $\gamma\text{-CH}_2$ ) and (t,  $\delta\text{-CH}_3$ ), respectively. The terminal methyl groups of Bu unit showed signals between 0.90 – 0.87 ppm for 1 or 2 complexes. The multiplet about 8.12 – 6.77 ppm are assigned to aromatic protons. The suggested structures for 1 and 2 is a tetramer with monodentate aromatic sulfonates (figure 2). The conclusions drawn from the spectroscopic studies data are consistent well with the X-ray crystallography studies.

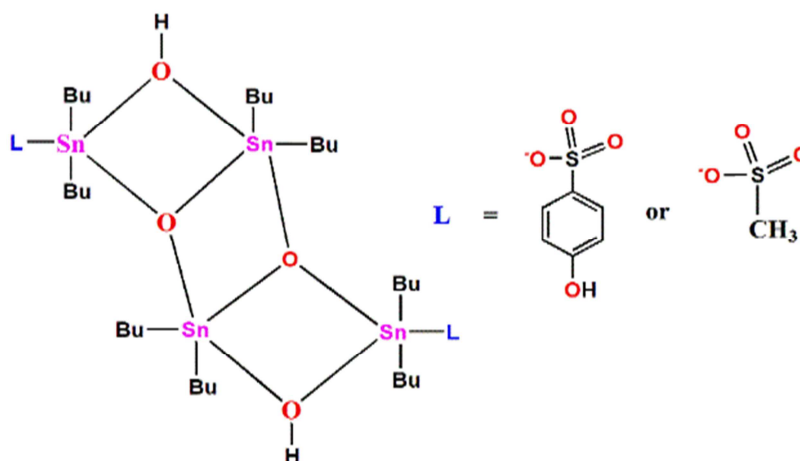


Figure 2. Structures of  $[\text{n-Bu}_2\text{Sn}]_4(\mu_3\text{-O})_2(\mu_2\text{-OH})_2(\text{OSO}_2\text{R})_2$  ( $\text{R} = 4\text{-HO-C}_6\text{H}_4$  and  $\text{CH}_3$ ).

### 3.3. X-ray Crystallography

The ladder structure of complex 1 is presented in figure 3. The 2D supermolecular network and 3D supermolecular architecture of complex 1 are presented in figures 3 and 4. As shown in Figure 1, there are two unique molecules (Molecule

A and Molecule B) in the crystal structure. The conformations of the two independent molecules are almost the same, except for small differences in angles and bond lengths and linked by O-H...O hydrogen bonds. Thus, we choose molecule A to describe in detail (figure 4).

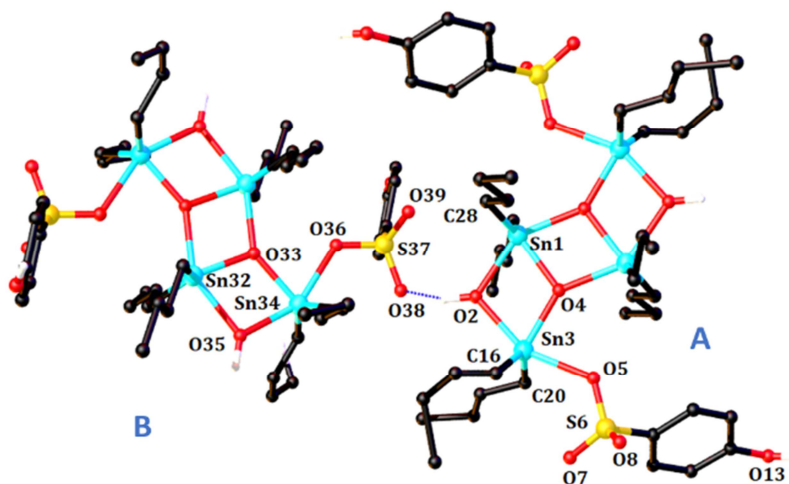


Figure 3. The ladder structure of aggregate complex 1. (Hydrogen atoms bonded to carbon atoms are omitted and only the  $\alpha$ -carbon of the butyl groups has been drawn for clarity. Selected bond lengths and angles ( $\text{\AA}$ ,  $^\circ$ ):  $\text{Sn1-O4}^i$  2.137,  $\text{Sn1-O2}$  2.173,  $\text{Sn1-O4}$  2.064,  $\text{Sn1-C24}$  2.127,  $\text{Sn1-C28}$  2.123,  $\text{Sn3-O2}$  2.127,  $\text{Sn3-O4}$  1.999,  $\text{Sn3-O5}$  2.293,  $\text{Sn3-C16}$  2.126,  $\text{Sn3-C20}$  2.123,  $\text{O5-S6}$  1.176,  $\text{S6-O7}$  1.456,  $\text{S6-O8}$  1.445,  $\text{S6-C9}$  1.744,  $\text{C9-C10}$  1.400,  $\text{C9-C15}$  1.372;  $\text{Sn1-Sn3}$  3.333,  $\text{Sn1-Sn1}^i$  3.392,  $\text{O4}^i\text{-Sn1-O2}$  145.198,  $\text{O4}^i\text{-Sn1-O4}$  72.351,  $\text{C11-Sn1-O1}$  94.6,  $\text{O2-Sn1-O4}$  72.849,  $\text{O4}^i\text{-Sn1-C24}$  96.679,  $\text{O2-Sn1-C24}$  98.613,  $\text{O2-Sn1-C28}$  95.846,  $\text{O4-Sn1-C28}$  113.902,  $\text{C24-Sn1-C28}$  130.088,  $\text{Sn1-O2-Sn3}$  101.666,  $\text{O2-Sn3-O4}$  75.137,  $\text{O2-Sn3-O5}$  154.063,  $\text{O4-Sn3-O5}$  79.462,  $\text{O4-Sn3-C16}$  115.203,  $\text{O5-Sn3-C16}$  88.737,  $\text{O2-Sn3-C20}$  100.037,  $\text{C16-Sn3-C20}$  130.472,  $\text{Sn1}^i\text{-O4-Sn1}$  107.649,  $\text{Sn1-O4-Sn3}$  110.244,  $\text{O5-S6-O7}$  110.615,  $\text{O5-S6-O8}$  111.414,  $\text{O7-S6-O8}$  114.184,  $\text{O5-S6-C9}$  105.362,  $\text{O7-S6-C9}$  107.426,  $\text{O8-S6-C9}$  107.314. Symmetry code: (i)  $-x+2, -y, -z$ ; (ii)  $-x+1, -y+1, -z+1$ .

The molecule A consists of the hydroxide-bridged tetrameric organostannoxane ladder and two deprotonated *p*-hydroxybenzenesulfonic acid ligands. The molecular structures can be considered as a tetramer of Sn(IV) and consist a

centrosymmetric distannoxane dimer, which is built up around the planar cyclic  $\text{Sn}_2\text{O}_2$  unit. The structure is a tetranuclear centro-symmetric dimer of an oxoditin(IV) unit having a central four-member ring composed of  $\text{Sn}(3)\text{--O}(2)\text{--Sn}(1)\text{--O}(4)$ .

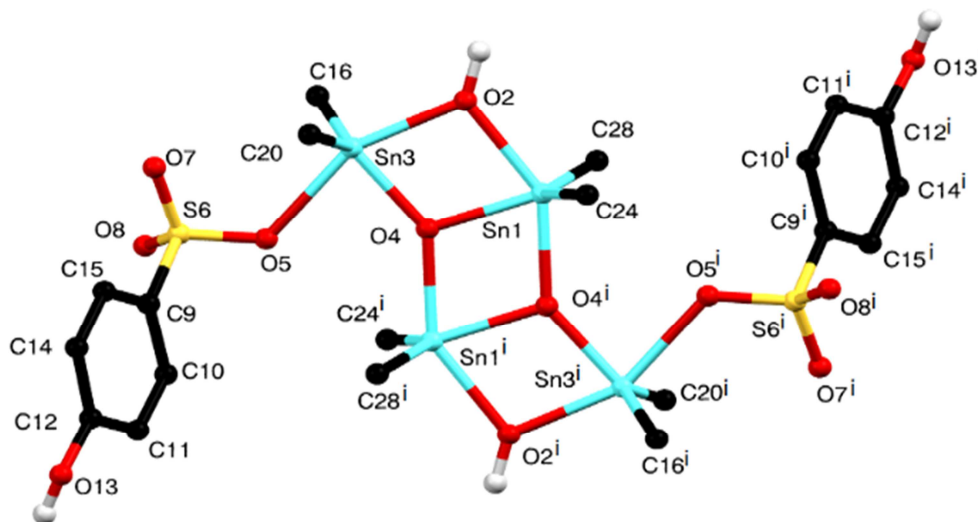


Figure 4. Tetranuclear structure of  $[n\text{-Bu}_2\text{Sn}]_4(\mu_3\text{-O})_2(\mu_2\text{-OH})_2[\text{OSO}_2\text{-C}_6\text{H}_4\text{-(4-OH)}]_2$ .

Both two oxygen atoms of this unit can be considered as tridentate, where they link three different tin centres, two endocyclic and one exocyclic. Tridentate oxygen atom, which shares its electrons with three tin centers, has a distorted tetrahedral configuration. The hydroxyl groups are bidentate bridging. Therefore, all of them are three-coordinated and adopt distorted trigonal geometry. All Sn(IV) atoms are five-coordinated, showing a distorted trigonal bipyramid configuration but having two chemical environments:  $\text{SnBu}_2\text{O}_2(\text{OH})$  and  $\text{SnBu}_2\text{O}(\text{OH})\text{L}$  with  $\text{L} = 4\text{-hydroxylbenzenesulfonate}$ . The  $\text{SnBu}_2\text{O}_2(\text{OH})$  are coordinated with tridentate O atoms and two C atoms from butyl groups, while  $\text{SnBu}_2\text{O}(\text{OH})\text{L}$ , one of the coordinated O atoms is from the sulfonate group. A supramolecular structure

is formed by  $\text{O-H}\cdots\text{O}$  interactions ( $\text{H21}\cdots\text{O38}$  2.00 Å,  $\text{H351}\cdots\text{O7}^{\text{iii}}$  1.93 Å) between the hydroxide hydrogen of the tetrameric organostannoxane ladder and the sulfonate oxygen (figure 3), and by the interactions  $\text{O-H}\cdots\text{O}$  ( $\text{H131}\cdots\text{O8}^{\text{i}}$  2.37 Å,  $\text{H441}\cdots\text{O8}^{\text{iii}}$  2.11 Å) (table 2) between the hydroxide of sulfonate and sulfonate oxygen. The endocyclic Sn(1) atom, the basal plane defined by C(24), C(28) and O(4), and the axial sites are occupied by the O(4i) and O(2) atoms, which form an angle of  $145.198^\circ$  (2), deviating from a linear arrangement. The exocyclic Sn(3) atom, the trigonal plan is defined by two butyl groups and the O(4) atom, and the axial positions defined by O(2) and O(5) which form an angle of  $154.063^\circ$  (3).

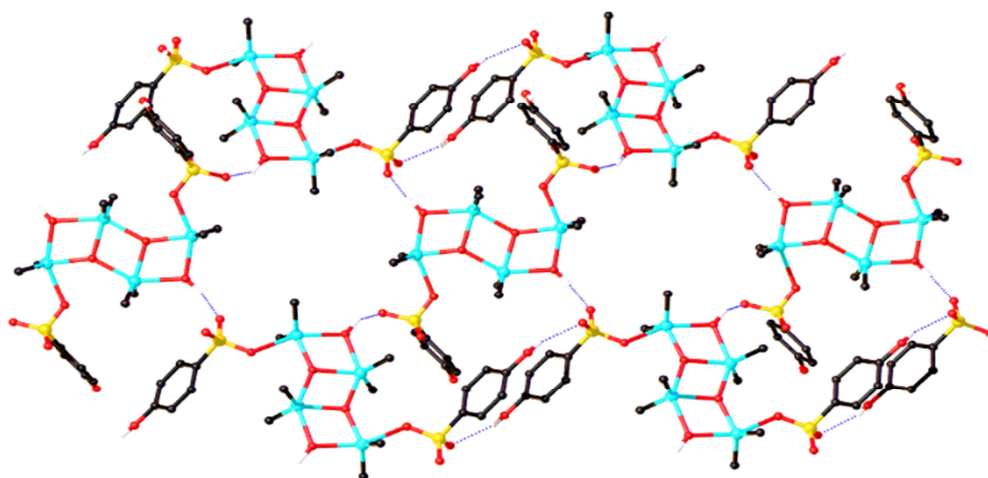


Figure 5. 3D supermolecular architecture constructed by the intermolecular  $\text{O-H}\cdots\text{O}$  hydrogen bond (bleu dashes). Atom color code: Sn, greenish; C, black; H, white; N, blue; O, red; Cl, green. Only Cipso of the *n*-butyl groups are drawn; hydrogen atoms except for those of the water and chloroform molecules are omitted for clarity.

**Table 2.** Hydrogen bonds in crystals of **1** (Symmetry codes: (i)  $-x+3, -y, -z-1$ ; (ii)  $-x+2, -y+1, -z$ ; (iii)  $x, y, z+1$ ).

| $D-H\cdots A$                       | $d(D-H)$ | $d(H\cdots A)$ | $d(D\cdots A)$ | $\angle(D-H\cdots A)$ |
|-------------------------------------|----------|----------------|----------------|-----------------------|
| O2—H21 $\cdots$ O38                 | 0.95     | 2.00           | 2.7992 (2)     | 141                   |
| O13—H131 $\cdots$ O8 <sup>i</sup>   | 0.83     | 2.37           | 3.1633 (2)     | 162                   |
| O35—H351 $\cdots$ O7 <sup>ii</sup>  | 0.95     | 1.93           | 2.7446 (2)     | 142                   |
| O44—H441 $\cdots$ O8 <sup>iii</sup> | 0.83     | 2.11           | 2.9319 (2)     | 169                   |

## 4. Conclusion

The one-pot reactions at room temperature between 4-hydroxylbenzenesulfonic acid or methylsulfonic with di-*n*-butyltin(IV) oxide gave ladder organostannoxanes. The conclusions drawn from the IR and <sup>1</sup>H NMR studies are consistent well with the X-ray crystallography study. The molecule, [n-Bu<sub>2</sub>Sn]<sub>4</sub>(μ<sub>3</sub>-O)<sub>2</sub>(μ<sub>2</sub>-OH)<sub>2</sub>(OSO<sub>2</sub>R)<sub>2</sub> (R=4-HO-C<sub>6</sub>H<sub>4</sub>- and CH<sub>3</sub>-), structures consist of the hydroxide-bridged tetrameric organostannoxanes ladder and two deprotonated sulfonic acid. In the structure, the centre Sn(IV) atoms of complexes adopt five-coordination mode, display a distorted trigonal bipyramid geometry, and reveal rich supramolecular structures by intermolecular hydrogen bonding interactions.

## Author Contributions

All the authors have accepted responsibility for entire content of this submitted manuscript and approved submission.

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None declared.

## Conflict of Interest Statement

The authors declare no conflicts of interest regarding this article.

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