

## Synthesis, spectroscopic studies and crystal structure of $(\text{Et}_4\text{N})(\text{SnMe}_3)_7(\text{HAsO}_4)_4 \cdot 2\text{H}_2\text{O}$ <sup>☆</sup>

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### Abstract

On allowing tetraethylammonium dihydrogen arsenate dihydrate to react with trimethyltin chloride, the title compound has been obtained and characterized by infrared, Mössbauer and NMR techniques. Its crystal structure has been determined and consists of layers containing both corner sharing  $\text{AsO}_4\text{H}$  tetrahedra and *trans*- $\text{O}_2\text{SnC}_3$  entities. It contains large cavities in which tetraethylammonium cations are located.

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### 1. Introduction

The structures of organotin derivatives of common inorganic anions have not been widely studied [1–5]. In the course of our research work on the coordination ability of oxyanions we have recently reported the crystal structures of  $(\text{SnMe}_3)_2\text{SeO}_3 \cdot \text{H}_2\text{O}$  [6] and  $\text{SnMe}_3\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$  [7] which compliment the known structures of  $\text{R}_3\text{SnCO}_3$  (R = Me, Bu) [8,9],  $\text{SnMe}_3\text{SO}_4 \cdot 2\text{H}_2\text{O}$  [10] and  $\text{SnMe}_3\text{NO}_3 \cdot \text{H}_2\text{O}$  [11]. In addition to their inherent interest as novel organotin species, such formulations also have a wider implication for supramolecular organometallic chemistry [12]. Supramolecular structures are usually assembled by using

rigid connectors between flexible vertices. In the main, it is a metal fragment that acts as a fulcrum for the growing lattice by virtue of its flexible coordination environment, while the rigid interconnects are chosen from organic fragments, such as 4,4'-bipyridine. In our work on organotin and organothallium tetrazoles ([13, and references therein]), we have shown that such a scenario can be reversed, e.g., five-coordinate triorganotin species can effectively act as the rigid rod via the electronegative groups in apical positions, provided flexibility can be offered by the coordinated ligands. Thus, an understanding of the synthetic and structural chemistry of organotin derivatives of simple anions has wide implications and should stimulate the use of other organometallic fragments as part of the supramolecular toolbox.

In this paper, we report the crystal structure and spectroscopic studies of  $(\text{Et}_4\text{N})(\text{SnMe}_3)_7(\text{HAsO}_4)_4 \cdot 2\text{H}_2\text{O}$  which incorporates the features described above and shows the potential wealth of structures that these simple organotin compounds can offer.

<sup>☆</sup> Supplementary data associated with this article can be found, in the online version, at [doi:10.1016/j.jorganchem.2004.03.033](https://doi.org/10.1016/j.jorganchem.2004.03.033).

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## 2. Experimental

Details of the instruments used for measuring IR [14], NMR [5] and Mössbauer [15] spectra are given elsewhere. The elemental analyses of C, H, N and Sn, were performed at the Department of Inorganic Metallorganic and Analytical Chemistry (University of Padua, Italy) and by the “Service Central d’Analyses” CNRS, Vernaison France.

### 2.1. Synthesis

Et<sub>4</sub>NOH (10% in water), H<sub>3</sub>AsO<sub>4</sub> and SnMe<sub>3</sub>Cl are Aldrich chemicals and were used without further purification.

An ethanolic solution containing (Et<sub>4</sub>N)(H<sub>2</sub>AsO<sub>4</sub>)·5H<sub>2</sub>O (1.13 g, 2.84 mmol) [obtained from a solution mixture of Et<sub>4</sub>NOH and H<sub>3</sub>AsO<sub>4</sub> in 1:1 ratio] and SnMe<sub>3</sub>Cl (1.12 g, 5.68 mmol) stirred at room temperature for around one hour gives colourless crystals of the title compound after slow solvent evaporation; yield: 80%; m.p. 174 °C: Anal. Found (calculated for C<sub>29</sub>H<sub>91</sub>As<sub>4</sub>NO<sub>18</sub>Sn<sub>7</sub>): C: 18.6 (18.6); H: 4.79 (4.86); N: 0.80 (0.78); Sn: 44.4 (44.4)%. Infrared data (cm<sup>-1</sup>): 2924m, 2359m ν OH<sub>2</sub>; 1685m δ OH<sub>2</sub>; 897vs, 777vs ν AsO<sub>4</sub>; 553vs ν<sub>as</sub> SnMe<sub>3</sub>; 447s, 422s δ AsO<sub>4</sub>, 291m ν SnO (vs = very strong, s = strong, m = medium). Mössbauer (mm s<sup>-1</sup>) I.S. = 1.25; Q.S. = 3.50; Γ = 0.86; NMR [CD<sub>3</sub>OD; δ (ppm); J (Hz): <sup>1</sup>H:0.52 (s + satellites) (63H) [SnMe<sub>3</sub>; <sup>2</sup>J<sup>119</sup>SnH = 68.5 and <sup>2</sup>J<sup>117</sup>SnH = 65.6]; 1.30(12H) CH<sub>3</sub>(Et<sub>4</sub>N); 3.29(8H) CH<sub>2</sub>(Et<sub>4</sub>N). <sup>13</sup>C: -0.15(q) [SnMe<sub>3</sub>; <sup>1</sup>J<sup>119</sup>SnC = 503.3 and <sup>1</sup>J<sup>117</sup>SnC = 481.4]; 7.56(s) [CH<sub>3</sub>]; 53.2(t) [CH<sub>2</sub>]; <sup>119</sup>Sn: 40.5.

### 2.2. Structure determination

The crystal data and details of data collection are given in Table 1. The diffraction intensities of colourless crystal were collected at 150(2) K on an Nonius Kappa CCD diffractometer using monochromatic Mo Kα radiation; data were corrected for absorption.

The structure was solved by heavy atom method using the SHELXS-86 program [16] and refined by full-matrix least-squares method with SHELXL-97 program [17]. The residual electron density is in the region of ammonium cation, which shows considerable disorder, but no attempt has been made to model this. Methyl hydrogen atoms were placed at calculated positions (C–H: 0.98 Å) and refined with a riding model with isotropic thermal parameter fixed at 1.5 times the value of the corresponding carbon atom. Hydrogen atoms attached to O(9) of the water were located in the difference maps and refined in the usual manner. Hydrogens associated with the disordered ethyl groups were refined over two sites each with site occupancy of 0.5.

Table 1

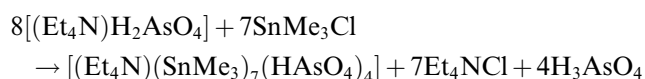
Crystal and structure refinement data

Empirical formula	C <sub>29</sub> H <sub>91</sub> As <sub>4</sub> NO <sub>18</sub> Sn <sub>7</sub>
Formula weight	1872.54
Crystal system	Monoclinic
Space group	C2/c
<i>Unit cell dimensions</i>	
<i>a</i> (Å)	14.0470(1)
<i>b</i> (Å)	20.6890(2)
<i>c</i> (Å)	21.0830(2)
β (°)	97.14(1)
<i>V</i> (Å <sup>3</sup> )	6079.62(9)
<i>Z</i>	4
μ (Mo Kα) (mm <sup>-1</sup> )	5.043
Reflections collected	58115
Independent reflections <i>R</i> <sub>int</sub>	8859 (0.0666)
Goodness-of-fit on <i>F</i> <sup>2</sup>	1.105
<i>R</i> , <i>R</i> <sub>w</sub> [ <i>I</i> > 2σ( <i>I</i> )]	0.0299, 0.0738
<i>R</i> , <i>R</i> <sub>w</sub> (all data)	0.0359, 0.0765
Largest difference peak and hole (e Å <sup>-3</sup> )	2.872 and -1.910

Crystallographic data for the structural analysis has been deposited with the Cambridge Crystallographic Data Centre, CCDC No. 184816. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ UK; fax (int code): +44-1223-336-033 or e-mail: [deposit@ccdc.cam.ac.uk](mailto:deposit@ccdc.cam.ac.uk) or www: <http://www.ccdc.cam.ac.uk>.

## 3. Result and discussion

The title compound was isolated as stable colourless crystals after slow solvent evaporation of an aqueous ethanolic mixture of (Et<sub>4</sub>N)H<sub>2</sub>AsO<sub>4</sub>·5H<sub>2</sub>O and SnMe<sub>3</sub>Cl in 1:2 ratio, according to the following reaction:



The product was isolated as the dihydrate [(Et<sub>4</sub>N)-(SnMe<sub>3</sub>)<sub>7</sub>(HAsO<sub>4</sub>)<sub>4</sub>]·2H<sub>2</sub>O.

## 4. Spectroscopic study

The absence of a band around 520–515 cm<sup>-1</sup> in the infrared spectrum of the title compound attributable to ν<sub>s</sub> SnC<sub>3</sub> is indicative of planar SnC<sub>3</sub> groups. The Mössbauer spectrum consists of a simple doublet Q.S. (3.50 mm s<sup>-1</sup>) consistent with tin atoms in equivalent environments (Γ = 0.86) and *trans*-O<sub>2</sub>SnC<sub>3</sub> coordination about the metal [18]. The value of the coupling constants [<sup>1</sup>J<sup>119</sup>SnC = 503 and <sup>1</sup>J<sup>117</sup>SnC = 481; <sup>2</sup>J<sup>119</sup>SnH = 68.5 Hz and <sup>2</sup>J<sup>117</sup>SnH = 65.6 Hz] and of the <sup>119</sup>Sn resonance (40.5 ppm) indicate that the five coordination of the tin atoms is maintained in solution [19].

## 5. Crystallographic study

An ORTEX [20] view including the asymmetric unit with atomic labelling scheme of the title compound is shown in Fig. 1. One tin atom [Sn(3)] and the carbon C(8) of its associated methyl group lie on a crystallographic twofold axis inherent in the  $C2/c$  space group; the  $\text{Me}_3\text{Sn}(3)$  unit is completed by C(7) and C(7<sup>(i)</sup>) (symmetry code:  $1-x, y, 1/2-z$ ). All the remaining atoms associated with the complex anion are in general positions, such that the asymmetric unit of the  $(\text{SnMe}_3)_7(\text{HAsO}_4)_4 \cdot 2\text{H}_2\text{O}$  component of the structure consists, in total, of four trimethyltin units, two hydrogen arsenate anions and one water molecule. The asymmetric unit of the tetraethylammonium cation also lies on a crystallographic twofold axis [N(1)] with two ethyl groups [C(12)–C(15)] on general positions.

The structure contains two types of  $\text{trans-O}_2\text{SnC}_3$  moieties, one involving the two oxygens of different monohydrogen arsenates [Sn(2), Sn(3) and Sn(4)], the other [Sn(1)] involving one oxygen of an monohydrogen arsenate and a water molecule of crystallisation; in all cases the tin atoms adopt a trigonal bipyramidal environment with oxygens in apical positions. The value of the  $\text{AsO-Sn-OH}_2$  angle [O(1)–Sn(1)–O(9):  $175.48(8)^\circ$ ] is between those of  $\text{AsO-Sn-OAs}$  [O(2)–Sn(2)–O(7):

$174.62(8)^\circ$  and O(5)–Sn(3)–O(5<sup>(i)</sup>):  $177.62(13)^\circ$ ]; the values indicate the near linearity of O–Sn–O axial units and are similar to those reported for selenito and oxalate trimethyltin derivatives [6,7] (see Table 2).

The Sn(1) atom completes its axial coordination rather weakly with a water molecule of crystallisation [Sn(1)–O(9):  $2.367(2) \text{ \AA}$ ] and as a consequence forms the strongest Sn–O bonds [Sn(1)–O(1):  $2.172(2) \text{ \AA}$ ].

The Sn–OAs bonds are in the expected range [ $2.172(2)$ – $2.357(2) \text{ \AA}$ ] while Sn(1)–O(9) bond is in the range of typical  $\text{H}_2\text{O-Sn}$  bonds [ $2.14$ – $2.47 \text{ \AA}$ ] [6]. Sn–C bonds are in the range  $2.117(4)$ – $2.128(4) \text{ \AA}$  which is expected from literature precedent. The C–Sn–C angles within the equatorial plane for the four tin atoms spread in the relatively wide range [ $115.20(16)$ – $126.84(18)^\circ$ ].

While considering the monohydrogen arsenate anion, each As atom is linked to three different tin atoms via As–O–Sn bridges with As–O distances ranging from  $1.655(2)$  to  $1.696(2) \text{ \AA}$ ; the remaining oxygen atom, being protonated, is involved in a much longer As–O(H) interaction [As(2)–O(8):  $1.743(2)$ , As(1)–O(4):  $1.726(2) \text{ \AA}$ ]. The OH groups are directed toward the cavities containing the tetraethylammonium cations.

The overall structure is a complex three-dimensional lattice which is constructed using both bridging arsenate groups and a network of hydrogen bonds (Figs. 2

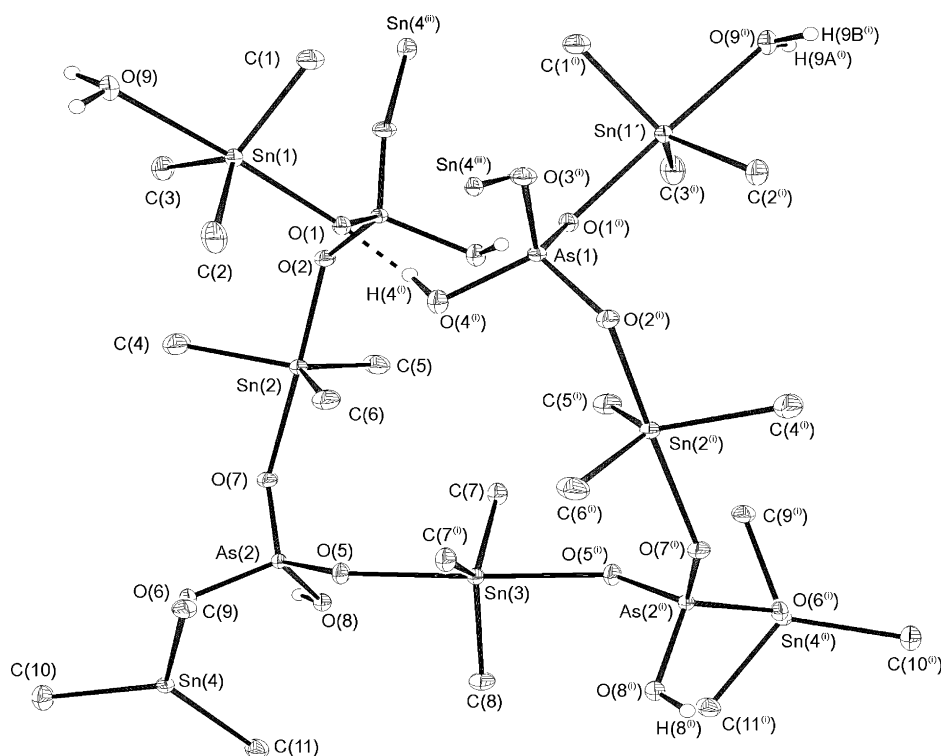


Fig. 1. The structure of the title compound showing part of the three-dimensional structure and incorporating the asymmetric unit, along with atomic labelling; ellipsoids are at the 30% level. The asymmetric unit is indicated by unprimed atoms; other symmetry operations: (i)  $1-x, y, 1/2-z$ ; (ii)  $1/2+x, 1/2+y, z$ ; (iii)  $1/2-x, 1/2+y, 1/2-z$ . The tetraethylammonium cations have been omitted for clarity.

Table 2  
Bond lengths (Å) and angles (°)

Sn(1)–O(1)	2.172(2)	Sn(2)–O(2)	2.172(2)
Sn(1)–O(9)	2.367(2)	Sn(2)–O(7)	2.357(2)
Sn(3)–O(5 <sup>(ii)</sup> )	2.215(2)	Sn(4)–O(3 <sup>(iii)</sup> )	2.195(2)
Sn(3)–O(5)	2.215(2)	Sn(4)–O(6)	2.276(2)
As(1)–O(3)	1.660(2)	As(1)–O(2)	1.662(2)
As(1)–O(1)	1.696(2)	As(1)–O(4)	1.726(2)
As(2)–O(5)	1.655(2)	As(2)–O(7)	1.679(2)
As(2)–O(6)	1.682(2)	As(2)–O(8)	1.743(2)
O(3)–Sn(4 <sup>(iii)</sup> )	2.195(2)		
C(3)–Sn(1)–C(1)	117.80(19)	C(3)–Sn(1)–C(2)	120.02(18)
C(1)–Sn(1)–C(2)	121.24(17)	C(3)–Sn(1)–O(1)	88.54(13)
C(1)–Sn(1)–O(1)	95.45(12)	C(2)–Sn(1)–O(1)	95.55(12)
C(3)–Sn(1)–O(9)	86.95(13)	C(1)–Sn(1)–O(9)	86.85(12)
C(2)–Sn(1)–O(9)	86.54(12)	O(1)–Sn(1)–O(9)	175.48(8)
C(5)–Sn(2)–C(6)	126.84(18)	C(5)–Sn(2)–C(4)	116.1(2)
C(6)–Sn(2)–C(4)	116.81(19)	C(5)–Sn(2)–O(2)	94.03(12)
C(6)–Sn(2)–O(2)	89.23(13)	C(4)–Sn(2)–O(2)	91.31(13)
C(5)–Sn(2)–O(7)	91.35(12)	C(6)–Sn(2)–O(7)	87.68(13)
C(4)–Sn(2)–O(7)	86.16(13)	O(2)–Sn(2)–O(7)	174.62(8)
C(7)–Sn(3)–C(7 <sup>(i)</sup> )	123.3(2)	C(7)–Sn(3)–C(8)	118.33(11)
C(7 <sup>(i)</sup> )–Sn(3)–C(8)	118.33(11)	C(7)–Sn(3)–O(5)	85.67(11)
C(7 <sup>(i)</sup> )–Sn(3)–O(5)	93.20(11)	C(8)–Sn(3)–O(5)	91.19(7)
C(7)–Sn(3)–O(5 <sup>(ii)</sup> )	93.20(11)	C(7 <sup>(i)</sup> )–Sn(3)–O(5 <sup>(ii)</sup> )	85.67(11)
C(8)–Sn(3)–O(5 <sup>(ii)</sup> )	91.19(7)	O(5)–Sn(3)–O(5 <sup>(ii)</sup> )	177.62(13)
C(10)–Sn(4)–C(9)	118.23(16)	C(10)–Sn(4)–C(11)	115.20(16)
C(9)–Sn(4)–C(11)	126.57(15)	C(10)–Sn(4)–O(3 <sup>(iii)</sup> )	94.16(13)
C(9)–Sn(4)–O(3 <sup>(iii)</sup> )	84.38(11)	C(11)–Sn(4)–O(3 <sup>(iii)</sup> )	91.42(12)
C(10)–Sn(4)–O(6)	88.81(12)	C(9)–Sn(4)–O(6)	90.48(11)
C(11)–Sn(4)–O(6)	91.13(11)	O(3 <sup>(iii)</sup> )–Sn(4)–O(6)	174.83(9)
O(3)–As(1)–O(2)	111.81(12)	O(3)–As(1)–O(1)	108.10(12)
O(2)–As(1)–O(1)	113.28(12)	O(3)–As(1)–O(4)	111.93(13)
O(2)–As(1)–O(4)	106.04(12)	O(1)–As(1)–O(4)	105.55(11)
O(5)–As(2)–O(7)	115.47(12)	O(5)–As(2)–O(6)	110.46(11)
O(7)–As(2)–O(6)	110.26(12)	O(5)–As(2)–O(8)	106.15(11)
O(7)–As(2)–O(8)	106.18(12)	O(6)–As(2)–O(8)	107.90(11)

Symmetry transformations used to generate equivalent atoms:

- (i)  $-x+1, y, -z+1/2$ ; (ii)  $x-1/2, y-1/2, z$ ; (iii)  $x+1/2, y+1/2, z$ ; (iv)  $-x, y, -z+1/2$ .

and 3). Three tin centres [Sn(2), Sn(3), Sn(2<sup>(i)</sup>)] (Fig. 1) are held in a chain by bridging arsenate units. The chains are then brought into a closed loop by hydrogen bonding between pairs of O(1)···O(4) (2.692(3) Å) (Fig. 1). These rings, containing three tin and four arsenic centres, propagate into columns by virtue of coordination of Sn(4) to O(6) on one side supported by a hydrogen bond between O(8)···H–O(9) [O(8)···O(9) 2.829(3) Å], i.e., involving the coordinated water, on the other (Fig. 1).

These columns are then cross-linked into a network via pairs of hydrogen bonds between O(8)–H···O(7) [O(8)···O(7) 2.617(3) Å] with reinforcement by a hydrogen bond between O(6) and H–O(9) [O(6)···O(9) 2.676(3) Å]. (Figs. 2 and 3) The two arsenate moieties based on As(1) and As(2) are thus differentiated by the fact that As(1) is associated with O(4) which forms only one hydrogen bond while O(8), bonded to As(2) is both a hydrogen bond donor and acceptor. The coordinated water also forms pairs of hydrogen bonds. The cavities

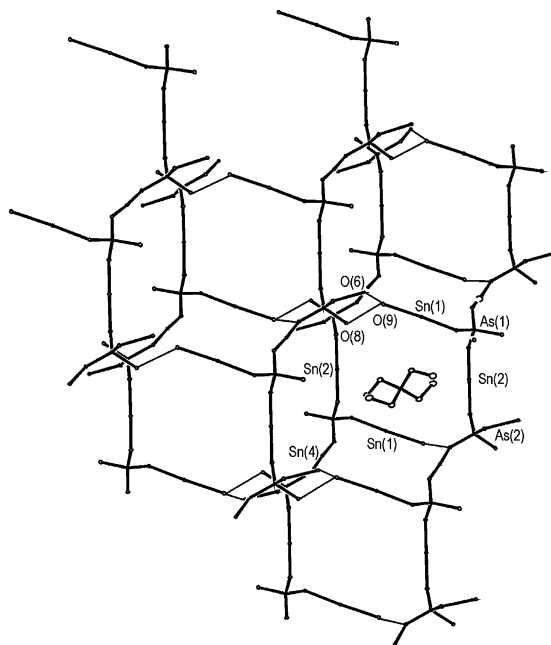


Fig. 2. The lattice structure of the title compound showing the formation of sheets approximately parallel to 001. Carbon atoms have been omitted for clarity and only one [Et<sub>4</sub>N]<sup>+</sup> cation is shown.

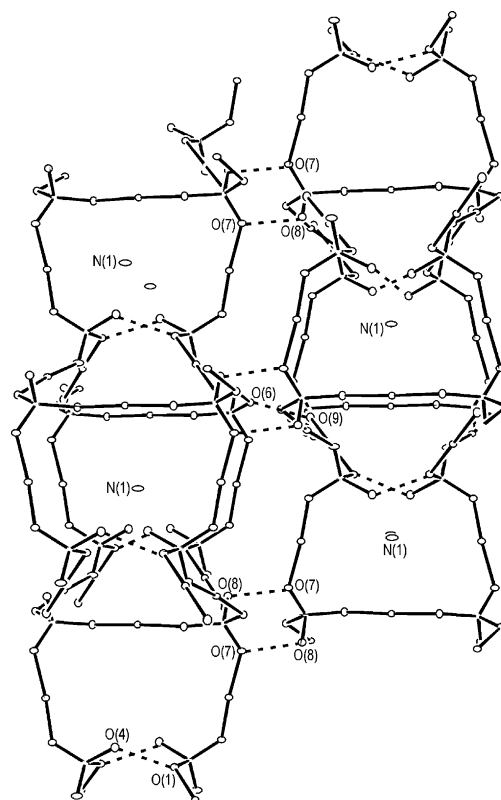


Fig. 3. An alternative view of the lattice structure of the title compound, orthogonal to that shown in Fig. 2; carbon atoms have been omitted for clarity.

thus generated in the structure are occupied by the tetraethylammonium cations (Figs. 2 and 3).

The structure is in some ways related to that of  $\text{Me}_3\text{SnO}_2\text{P}(\text{OH})\text{Ph}$  [21], which forms a helical polymer reminiscent of the structural fragment shown in Fig. 1. However, the phosphonate structure is less complex due to the lack of coordinated water, thus permitting association of helices into chains but precluding formation of a 3D network.

## 6. Conclusion

The title compound forms a polymeric structure with two types of *trans*- $\text{O}_2\text{SnC}_3$  moieties, one involving two different hydrogenoarsenates, the other involving one oxygen of hydrogenoarsenate and the water molecule of crystallisation. A network of hydrogen bond and bridging hydrogenoarsenate groups results in a complex three-dimensional architecture containing large cavities in which tetraethylammonium cations are located. The versatility of rigid, five-coordinated *trans*- $\text{X}_2\text{SnC}_3$  ( $\text{X}=\text{O}, \text{N}$ ) as a key component of supramolecular structures is thus further demonstrated.

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