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Waly Diallo*, Libasse Diop, Kieran C. Molloy, Mary F. Mahon and Laurent Plasseraud

Reactivity of bis(cyclohexylammonium) 4-nitrophenylphosphate with SnMe_3Cl . X-ray structure of $4\text{-NO}_2\text{C}_6\text{H}_4\text{PO}_4(\text{SnMe}_3)_2\cdot\text{H}_2\text{O}$

Abstract: The reaction of bis(cyclohexylammonium) 4-nitrophenylphosphate with Me_3SnCl (1:2) under reflux in ethanol yielded the title compound $4\text{-NO}_2\text{C}_6\text{H}_4\text{PO}_4(\text{SnMe}_3)_2\cdot\text{H}_2\text{O}$ (**1**). The X-ray crystallographic analysis achieved on single crystals obtained by slow evaporation at room temperature revealed the formation of an unexpected supramolecular coordination network. The elementary building block can be viewed as two Me_3Sn moieties linked by a bridging 4-nitrophenylphosphate ligand. The two tin atoms are five-coordinated and describe a *trans*- Me_3SnO_2 geometry in a trigonal bipyramidal arrangement. However, the Sn atoms are distinct, exhibiting two different environments. Thus, one is linked to two axial oxygen atoms from two distinct phosphato groups, whereas the other is bound to one phosphato oxygen atom and to a water molecule. From a supramolecular point of view, the combination of the tridentate binding mode of $4\text{-NO}_2\text{C}_6\text{H}_4\text{PO}_4$ and the additional formation of intermolecular hydrogen bonding interactions between $\text{NO}_2/\text{H}_2\text{O}$ and $\text{PO}_4/\text{H}_2\text{O}$ groups gives rise to a three-dimensional lattice network.

Keywords: coordination-driven self-assembly; hydrogen bonding; trimethyltin(IV), 4-nitrophenylphosphato; X-ray crystallography.

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Introduction

As some organotin(IV) compounds have been found to be the subject of potential applications – medicine, agriculture,

industry – many research groups around the world are still involved in the quest for new organotin compounds (Evans and Karpel, 1985; Yin and Wang, 2004; Zhang et al., 2006; Hanif et al., 2007). Our group is also strongly involved in this field, focusing in particular on the reactivity of organotin(IV) precursors with oxyanions in order to structurally characterize new derivatives and unexpected molecular architectures (Diallo et al., 2007, 2009; Gueye et al., 2011).

4-Nitrophenylphosphoric acid is a colorless, endogenous or exogenous pigment precursor that may be transformed by biological mechanisms into colored compounds used in many fields of application. In enzymology, it is particularly employed as a substrate for the phosphatase assay, acting as a phosphoryl donor for several phosphotransferases (Yamazaki et al., 1994; Hengge et al., 1996). Its dianion is notably traded in the form of its bis(cyclohexylammonium) salt. From a crystallographic point of view, the dihydrate form, $2[\text{C}_6\text{H}_{11}\text{NH}_3]^+[\text{4-O}_2\text{-NC}_6\text{H}_4\text{OP}(\text{O})_2]^{2-}\cdot 2\text{H}_2\text{O}$, was structurally characterized for the first time in the 80s by Jones and coworkers (Jones et al., 1984). Since then, several transition metal coordination complexes based on the use of 4-nitrophenylphosphate as ligand have been structurally isolated (Rawji et al., 2000; Gajda et al., 2001; Hassana et al., 2012). However, and surprisingly, to the best of our knowledge, only two crystallographic reports showing the coordination of bis(4-nitrophenyl)phosphate ligand to a *p*-block metal atom (Pb) have been deposited to date in the Cambridge Chemical Data Centre (Yamami et al., 1998a,b). In this context, it seemed worthwhile and challenging to undertake a study of the reactivity of bis(cyclohexylammonium) 4-nitrophenylphosphate toward organotin compounds. In this paper, we report the results gained from the use of Me_3SnCl as tin precursor.

Results and discussion

When bis(cyclohexylammonium) 4-nitrophenylphosphate reacts with SnMe_3Cl in EtOH, in 1:2 molar ratio under reflux, the green crystals characterized were obtained by slow solvent evaporation at room temperature (yield, 27%).

The crystals are air-stable and poorly soluble in common organic solvents. The infrared (IR) spectrum exhibits characteristic absorption bands, which can be assigned to OH (3514 cm⁻¹), PO₄ (1259 and 1109 cm⁻¹) and NO₂ (1340 and 1172 cm⁻¹) functions. In comparison to the IR fingerprint of the bis(cyclohexylammonium) 4-nitrophenylphosphate itself, some modifications affecting the PO₄ and NO₂ absorption bands were observed, suggesting the interaction between the tin(IV) complex and the organic ligand.

The X-ray crystallographic analysis revealed a three-dimensional (3-D) supramolecular coordination network built on the self-assembly of elementary building blocks, which can be defined as 4-NO₂C₆H₄PO₄(SnMe₃)₂·H₂O (**1**). An ORTEP diagram showing the crystal structure of **1**, together with selected bond lengths and angles, is given in Figure 1. The asymmetric unit consists of two independent molecules of similar metrical data (see figure caption), and the following discussion is given for the molecule based on Sn(1,2) only for brevity. **1** can be viewed as two Me₃Sn moieties connected by a bridging 4-nitrophenylphosphate ligand (L) via two P-O-Sn linkages. The two tin atoms are five-coordinated and describe a *trans*-Me₃SnO₂ geometry in a trigonal

bipyramidal (TBP) arrangement. The equatorial plane of each Sn atom contains the carbon atoms of the three methyl groups [C-Sn=2.110(9) Å (mean), C-Sn-C=119.6(4)° (mean)]. However, the Sn atoms are distinct, exhibiting two different environments with regard to the ligands in axial positions. Thus, one (Sn2) is linked to two axial oxygen atoms from two distinct phosphato groups [Sn2-O4=2.239(5) and Sn2-O5=2.231(4) Å], whereas the other (Sn1) is bound to one phosphato oxygen atom [Sn1-O2=2.174(4)] and to a coordinated water molecule [Sn1-O1=2.391(5)], thus corroborating the presence of broad absorption band observed at 3514 cm⁻¹ in the IR spectrum. Both TBP environments are slightly distorted [O1-Sn1-O2=177.5(2)°, O4-Sn2-O5=174.2(2)°]. The Sn-O bond to the coordinated water is the longest of this type in the molecule [Sn(1)-O(1) 2.391(5) Å], whereas the Sn-O bond trans to this one is the shortest [Sn(1)-O(2) 2.174(4) Å]; the two Sn(2)-O bonds lie between these two values and are equal within experimental error.

From a supramolecular point of view, the phosphato group of L exhibits a tridentate coordination mode. Thus, the phosphato oxygen atom [P=O] not implicated in bonding to tin in **1** (O5, O12) is also bonded to a tin atom of

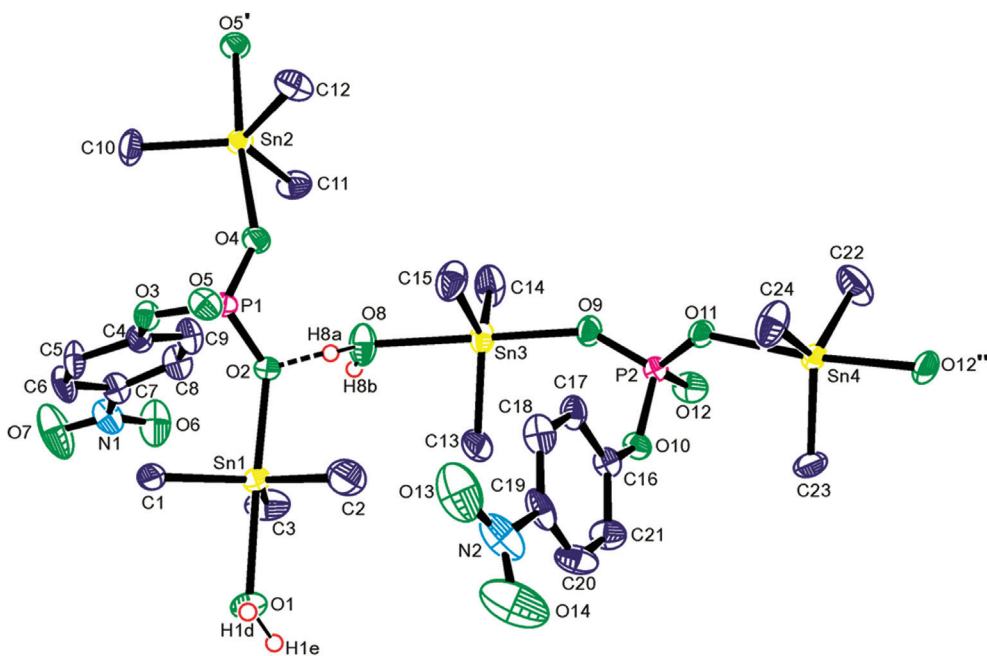


Figure 1 The asymmetric unit of **1** showing the labeling scheme (ORTEP view).

Displacement ellipsoids are drawn at 50% probability level [H atoms non-implicating in hydrogen bonding were omitted for clarity. Color code: Sn, yellow; C, violet; O, green; P, pink; N, light blue]. Selected bond lengths and angles (Å, °): Sn1-C2 2.092(9), Sn1-C1 2.097(7), Sn1-C3 2.110(7), Sn1-O2 2.174(4), Sn1-O1 2.391(5), Sn2-C11 2.120(7), Sn2-C10 2.121(7), Sn2-C12 2.129(7), Sn2-O5 2.231(4), Sn2-O4 2.239(4), Sn3-C14 2.119(8), Sn3-C15 2.119(8), Sn3-C13 2.134(8), Sn3-O9 2.136(5), Sn3-O8 2.401(6), Sn4-C23 2.105(8), Sn4-C24 2.109(8), Sn4-C22 2.113(8), Sn4-O12ⁱⁱ 2.214(5), Sn4-O11 2.306(4); C2-Sn1-C1 120.6(4), C2-Sn1-C3 121.1(4), C1-Sn1-C3 117.2(3), C2-Sn1O2 91.2(3), C1-Sn1-O2 97.3(3), C3-Sn1-O2 92.3(3), C2-Sn1-O1 88.3(3), C1-Sn1-O1 85.0(3), C3-Sn1-O1 85.9(3), O2-Sn1-O1 177.5(2), P1-O2-Sn1 130.5(3), P1-O4-Sn2ⁱⁱ 139.8(3), C10-Sn2-C11 122.6(3), C10-Sn2-C12 117.6(3), C11-Sn2-C12 119.8(3). Symmetry transformations used to generate equivalent atoms (i): -x+2, y-1/2, -z; (ii): -x+2, y+1/2, -z; (iii): -x+3, y+1/2, -z+1; (iv): -x+3, y-1/2, -z+1.

another unit, leading finally to the propagation of a polymeric architecture. In addition, the formation of intermolecular hydrogen bonding interactions between NO₂/H₂O and PO₄/H₂O groups gives rise to a 3-D lattice network. The geometry of hydrogen bonds is detailed in Table 1. Interestingly, the coordination-driven self-assembly leads to the creation of 19-membered (3Sn, 3P, 8O, 4C, 1N) square-shaped macrocycles joined to each other in a grid-like arrangement and in which three corners are occupied by phosphorus atoms (Figure 2). The resulting network topology can be described by the {4,4} Schläfli symbol.

Hydrogen bonds are shown by orange dashed lines. H atoms non-implicated in hydrogen bonding have been omitted for clarity (color code: Sn, green; P, pink; O, red; N, light blue; C, gray; H, yellow).

Table 1 Geometry of hydrogen bonds.

Interaction	D ^a -H (Å)	H...A ^b (Å)	D...A (Å)	D-H...A (°)
O-H...O(NO)	0.92(4)	1.84(4)	2.736(7)	166(5)
O-H...O(NO)	0.90(7)	2.23(7)	2.862(10)	127(6)
O-H...O(PO ₃)	0.91(3)	1.84(3)	2.733(6)	168(6)
O-H...O(PO ₃)	0.90(7)	1.85(7)	2.747(7)	174(5)

^aHydrogen bond donor. ^bHydrogen bond acceptor.

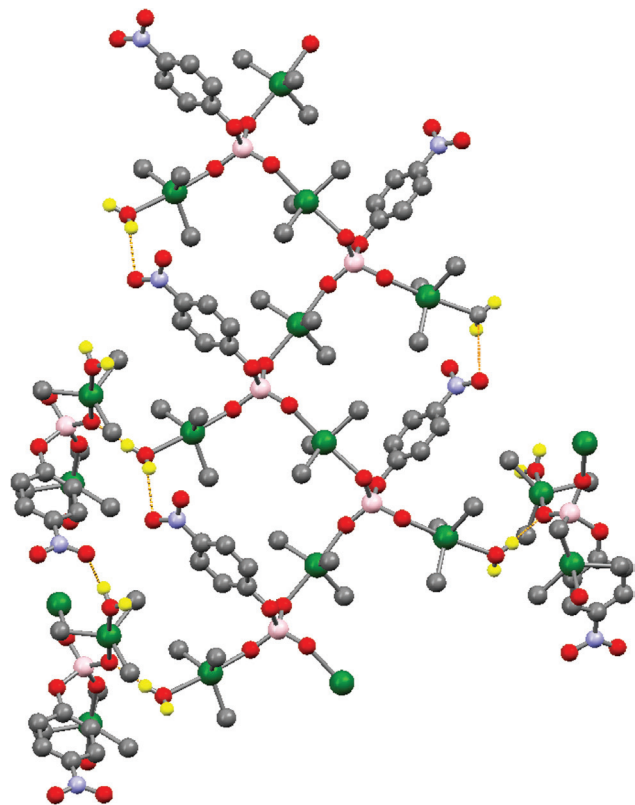


Figure 2 Representation of the resulting supramolecular structure of **1** highlighting the square-shaped macrocycle organization.

When evaporation conditions were speeded up by heating in a double boiler at 40°C, a yellow powder (**2**) was obtained instead of green crystals. The microanalysis of **2** suggests the formation of monosubstituted [C₆H₁₁NH₃]⁺[(SnMe₃)L]⁻. The IR spectrum recorded on this powder showed significant differences with the IR fingerprint of **1**. We observed in particular the disappearance of the broad band located at 3514 cm⁻¹ assigned to ν(OH). Moreover, the collected powder was very soluble in organic solvents. The ¹¹⁹Sn{¹H} nuclear magnetic resonance (NMR) spectrum recorded in solution (C₆D₆) exhibits only one very broad signal at δ=-43 ppm. Such a value of chemical shift, in the range of -40 and -120 ppm, suggests, for a methyltin(IV) derivative, tetracoordination (Wrackmeyer, 1985, 2008). Moreover, Holecek and Lycka showed in the past that the coupling constant ¹J(^{119,117}Sn-¹³C) can be used as a tool to determine the type of geometry around a tin(IV) atom (Nadvornik et al., 1984). In our case, values of ¹J(^{119,117}Sn-¹³C) coupling constants (522, 546 Hz) are in agreement with a quasi-tetrahedral geometry for Sn. These spectroscopic data suggest in solution a discrete structure that could correspond to a SnMe₃ moiety associated to a monodentate L ligand, with the negative charge on the resulting [(SnMe₃)L]⁻ anion compensated for by a cyclohexylammonium cation. In comparison to compound **1**, we assume that the speeding up of the evaporation process by heating at 40°C has reduced the presence and implication of water (confirmed by the IR spectrum of **2**). At the solid state, a polymeric organization seems very plausible, involving additional electronic donations of uncoordinated phosphato oxygen atoms to neighboring tin atoms. Currently, attempts of crystallization are under way in our laboratory to get single crystals from this powder in order to confirm unambiguously the proposed structure.

Finally, and from a mechanistic point of view, we suppose in solution an equilibrium implicating the three tin species (Me₃Sn)₂L (nonhydrated form of **1**), [CyNH₃]⁺L⁻ and [(Me₃Sn)L]⁻[CyNH₃]⁺ (**2**), and Me₃SnCl. Taking into account the reaction stoichiometry (L/Me₃SnCl=1:2), the neutral nonhydrated species **1** should be formed preferably, being also more soluble than charged **2**. With slow evaporation, **1** hydrates (this lowers its solubility as hydrogen bonds form) and crystallizes. With a faster evaporation, **2** becomes the first to precipitate.

Conclusion

The reactivity between bis(cyclohexylammonium) 4-nitrophenylphosphate and Me₃SnCl was studied, leading to an unexpected supramolecular coordination 3-D network

based on 4-NO₂C₆H₄PO₄(SnMe₃)₂·H₂O building blocks (1). To our knowledge, such a self-assembly organization is unusual, and the complex 1 constitutes the first example of a 4-nitrophenylphosphatotin(IV) derivative. Further works are in progress to extend this study to other organotin(IV) precursors, focusing in particular on the nature and number of alkyl substituents bound to Sn.

Experimental

General

Bis(cyclohexylammonium) 4-nitrophenylphosphate was purchased from Sigma-Aldrich Chemie GmbH, Steinheim, Germany and SnMe₃Cl was purchased from Merck Chemicals, Hohenbrunn bei München, Germany; both were used without any further purification. The elemental analyses (C, H, N) were performed at the Microanalyses Laboratory of the University of Bath (UK) and at the Institut de Chimie Moléculaire de l'Université de Bourgogne (Dijon, France). IR spectra were recorded on a Bruker FTIR Vector 22 spectrometer (Wissembourg, France) equipped with a Specac Golden Gate™ attenuated total reflectance (ATR) device and a Perkin-Elmer BX FTIR spectrometer (Waltham, MA, USA), using KBr pellets or Nujol mulls. Multinuclear NMR spectra (¹H, ¹³C{¹H}, ³¹P, ¹¹⁹Sn{¹H}) were recorded on a 300 MHz Bruker Avance III NanoBay NMR spectrometer. Samples were dissolved in C₆D₆ and the chemical shift scale of the spectrum was referenced to internal (CH₃)₄Sn for ¹¹⁹Sn{¹H} NMR spectra.

Synthesis of 4-NO₂C₆H₄PO₄(SnMe₃)₂·H₂O (1)

An ethanolic solution of bis(cyclohexylammonium) 4-nitrophenylphosphate (0.300 g, 0.7187 mmol, 15 mL) was added dropwise to an ethanolic solution of SnMe₃Cl (0.286 g, 1.4373 mmol, 10 mL). After stirring at room temperature, the mixture was heated under reflux for 2 h. The resulting colorless solution was then filtered and submitted to a slow solvent evaporation at room temperature (25°C in Dakar). After 2 weeks, green single crystals were collected and characterized as 4-NO₂C₆H₄PO₄(SnMe₃)₂·H₂O (1) (0.056 g, yield 27%). IR (KBr, cm⁻¹): 3514 (m, br, νOH), 2935 (m, νCH), 1606 (m), 1591 (s), 1509 (s), 1494 (s), 1340 (s, ν_{as}NO₂), 1259 (s, νP-O-C), 1172 (s, ν_sNO₂), 1095 (vs, νP=O), 1007 (s), 896 (s), 773 (s), 741 (s), 692 (m). Anal. calculated for C₁₂H₂₄NO₇PSn₂ (562.67): C 25.61; H 4.30; N 2.49; found: C 25.80; H 3.81; N 2.31%.

Synthesis of (CyNH₃)[4-NO₂C₆H₄PO₄(SnMe₃)₂] (2)

The procedure is similar to that used for 1 and differs only by the evaporation condition of the solution obtained after heating under reflux. The evaporation process is speeded up by heating in a double boiler at 40°C. After 2 weeks, a yellow powder was collected. IR (ATR, cm⁻¹): 2936 (s), 2859 (s), 1590 (s), 1509 (s), 1512 (s), 1491 (s), 1388 (m), 1341 (s, ν_{as}NO₂), 1259 (s, νP-O-C), 1139 (s, ν_sNO₂), 1109 (vs, νP=O),

984 (s), 879 (s), 896 (s), 777 (m), 756 (m), 724 (m), 693 (m), 645 (m), 618 (m), 572 (s), 550 (s). ¹H NMR (ppm, C₆D₆): 0.62 (s, H₃C-Sn), ³J(¹H-^{119,117}Sn)=70 Hz), 1.00 (m, Cy), 1.43 (m, Cy), 1.92 (m, Cy), 2.23 (m, Cy), 2.81 (m, Cy), 6.40 (m, br, Cy-NH₃), 6.87 (m, Ph), 7.27 (m, Ph), 7.98 (m, Ph). ¹³C{¹H} NMR (ppm, C₆D₆): 3.2 (CH₃-Sn), ¹J(^{119,117}Sn-¹³C)=546, 522 Hz), 24.4 (CH₂-Cy), 25.1 (CH₂-Cy), 30.9 (CH₂-Cy), 50.0 (CH-Cy), 116.8 (CH-Ph), 120.3 (CH-Ph), 120.4 (CH-Ph), 139.0 (C-Ph), 141.7 (C-Ph), 161.1 (C-Ph), 161.2 (C-Ph), 167.0 (C-Ph). ¹¹⁹Sn{¹H} NMR (ppm, C₆D₆): -43 (br). ³¹P NMR (ppm, C₆D₆): -0.5 (s, integral 60%), -6.2 (s, integral 20%), -8.0 (br, ^{117,119}Sn satellites observed but not resolved, integral 18%) -18.2 (s, integral 2%). Anal. calculated for C₁₅H₂₇N₂O₆PSn (481.07): C 37.45; H 5.66; N 5.82; found: C 37.20; H 5.11; N 5.96%.

Crystal structure determination

Formula C₁₂H₂₄NO₇PSn₂, *M*=562.67, green crystal: 0.4 mm×0.4 mm×0.4 mm, *a*=10.0219(2), *b*=10.3360(2), *c*=19.7286(4) Å, α=90°, β=90.201(2)°, γ=90°, *V*=2043.60(7) Å³, *D*_{calcd}=1.829 g/cm³, μ=2.550 mm⁻¹, *Z*=4, monoclinic, space group *P*2₁, λ=0.71073 Å, *T*=150(2) K, 37 930 reflections collected on a Nonius Kappa CCD diffractometer (index ranges: *h*: -13, 13; *k*: -14, 14; *l*: -27, 27), 10 866 independent (*R*_{int}=0.0558) and 10 866 observed reflections [*I*≥2σ(*I*)], 440 refined parameters, 7 restraints, *R* indices for observed reflections: *R*₁=0.0358, *wR*₂=0.0706, *R* indices for all data: *R*₁=0.0470, *wR*₂=0.0747, goodness of fit=1.034, maximum residual electron density 2.037 and -1.464 e/Å³. The structure was solved by SHELXS and refined by SHELXL (Sheldrick, 1986, 1997) using a full-matrix least-squares method based on *F*². The asymmetric unit consists of two independent but essentially similar molecules. The absorption correction applied was semiempirical from equivalents. In the final cycles of least-squares refinement, all non-hydrogen atoms were allowed to vibrate anisotropically. Hydrogen atoms when included at calculated positions were relevant. The structural model converged successfully once 180° pseudo-merohedral twinning about the 001 direct lattice direction was taken into account. The fractional contribution for the twin components was 77:23. Hydrogen atoms attached to O(1) and O(8) were located in the difference Fourier map and were refined using geometric restraints.

Supporting information

Crystallographic data for the structure reported in this paper have been deposited at the Cambridge Crystallographic Data Centre (CCDC), No. CCDC 789750 for 1. Copies of the data may be obtained free of charge from the Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44 1223 336 033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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