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Synthesis, spectroscopic characterization and crystal and molecular structures of phenylphosphonato SnR_3 (R=Ph, Me) derivatives

Abstract: Four new phenylphosphonato SnR_3 (R=Ph, Me) derivatives have been synthesized and characterized by infrared and Mössbauer spectroscopy. The structure of catena-poly$[PhPO_3HSnMe_3]_n$ has been determined by single-crystal X-ray diffraction analysis. The Sn^{IV} atoms are five-coordinated in all compounds, with the SnC_3O_2 framework in a trans trigonal bipyramidal arrangement and the $PhPO_3H^-$ anions being in axial positions. The molecular structure of $[PhPO_3HSnMe_3]_n$ is arranged as a one-dimensional coordination polymer in which planar $SnMe_3$ groups are axially bridged by -O-P-O- linkages of the $PhPO_3H^-$ ligand. Neighboring chains are linked via O-H•••O hydrogen bond interactions, generating a layered structure. In the $R_2NH_2(PhPO_3H)_2SnR'_3$ (R=Cy, Bu; R′=Ph, Me), the $SnPh_3$ or $SnMe_3$ residue is axially coordinated by two monodentate $PhPO_3H^-$. The role of the dialkylammonium cation, $R_2NH_2^+$, is crucial in the lattice building via a hydrogen bond network. These hydrogen bonds contribute to the crystal stability and compactness and result in a three-dimensional arrangement. The aqua complex $PhPO_3(SnPh_3)_2{\cdot}2H_2O$ has a discrete structure and the anion $PhPO_3^{2-}$ behaves as a bidentate ligand.

Keywords: coordination polymer; phenylphosphonato derivatives; tin; trigonal bipyramidal.

Introduction

Organotin(IV) complexes are extensively studied due to their applications in industry as well as their biocidal properties (Willem et al., 1997; Gielen, 2002; Davies et al., 2008). Numerous studies on organotin(IV) complexes have been carried out in order to determine its biological properties against bacteria, fungi and cancer cell lines (Teoh et al., 1997; Crouse et al., 2004). Reports on structure determinations or spectroscopic characterizations of trimethyl- and triphenyltin(IV) derivatives with mono- and polybasic oxyanions (XO_m^{n-}; X=Cr, Se, S, As; m=3, 4; n=1, 2, 3) show that the oxyanions behave mainly as polydentate ligands involving a one-dimensional polymeric, bi- or tridimensional network structure (Molloy et al., 1989; Diop et al., 2002; Diassé-Sarr et al., 2004; Fall et al., 2010; Boye et al., 2012). These triphenyltin(IV) or trimethyltin(IV) derivatives or complexes exist as monomers or polymers with a five-coordinated tin(IV) atom. Several papers dealing with organotin(IV) chemistry and the coordinating ability of phosphonates have been reported (Raymond et al., 1992; Song et al., 2007; Chunlin et al., 2008; De Barros et al., 2010; Shankar et al., 2011). The X-ray structure of a tetragonal form of $[SnMe_3PhPO_3H]_n$ has been reported by Molloy et al. (1981). Our group has conducted research on $SnMe_3$ and $SnPh_3$ residues containing derivatives with mono- and polybasic oxyanions (SO_4^{2-}, $C_2O_4^{2-}$, $PhP(H)O_2^-$, $HAsO_4^{2-}$) (Diassé-Sarr et al., 2004; Diallo et al., 2009; Diop et al., 2011; Gueye et al., 2011; Sow et al., 2012). In this paper, we have initiated the study of interactions between $R_2NH_2PhPO_3H$ (R=Bu, Cy) or $PhPO(OH)_2$ and SnR_3Cl (R=Ph, Me) or $SnPh_3OH$ that have yielded the studied derivative and adducts, of which one structure has been determined by X-ray crystallography and which have all been characterized by Mössbauer and infrared spectroscopy.

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Results and discussion

X-ray structure and spectroscopic characterization of a monoclinic form of catena-poly[$PhPO_3HSnMe_3$]$_n$ (1)

The structure of catena-poly[$PhPO_3HSnMe_3$]$_n$ consists of a zigzag infinite chain with a *trans*-SnC_3O_2 framework, the bidentate $PhPO_3H^-$ moiety σ bonded to the $SnMe_3$ group (Figure 1). A similar chain arrangement around the tin atom has been observed in the crystal structure of $SnMe_3O_2P(OPh)_2$ (Newton et al., 1993) and {[$(CH_3)_3Sn$]$_4$(O_3PPh)$_2$}$_n$ (Chunlin et al., 2008). The Sn-O distances [2.182(3), 2.374(3) Å] and the O(1)-Sn-O(3) angle of 171.47(12)° indicate unsymmetrical coordination around the tin center and a significant deviation from linearity when compared to the value in the tetragonal form variety (Molloy et al., 1981) in which Sn-O bond distances are 2.240(6) and 2.319(5) Å and the O-Sn-O axial angle is 178.0°. The Sn-O distances of the studied derivative, 2.216(11), 2.359(10) Å, compared to the value in the tetragonal form, indicate the strengthening of one bond and the weakening of the other in the monoclinic polymorph. These variations are accompanied by a movement of the phenyl group of $PhPO_3H^-$, which moves from a cis position (tetragonal form) to a trans position (monoclinic form). In the title compound, the Sn-O bond lengths are 2.182(3) and 2.374(3) Å, which are very close to those observed in $SnMe_3MePO_3H$ (Diop et al., 2002) [2.165(4) and 2.434(4) Å] . The angles around the phosphorus atoms are approximately tetrahedral except those including bridging oxygen atoms [O(1)-P-O(3)], which are significantly larger [115.0(2)°]. The two P-O distances of the bridging O(1)-P-O(3) moieties are also essentially equal, P-O(1) 1.507(3) Å and P-O(3) 1.512(3) Å, indicating the presence of extensive π delocalization of the P=O double bond. Although the virtual linearity of the O(1)-Sn-O(3) bonds requires linearity of the coordination polymer in the vicinity of the tin atoms, the chains are bent through the tetrahedrally coordinated phosphorus atoms of the phenylphosphonate ligand at an O-P-O angle of 115.0(2)°. The parallel polymer adjacent chains are linked together by a network of O-H···O hydrogen bond interactions [O-H, 0.83 Å; H···O, 1.76 Å; O(3)···O(2), 2.61 Å] (Table 1) between the free O-H groups of the phenylphosphonato ligand and the coordinated oxygen, generating a three-dimensional network (Figure 2).

The presence of a weak infrared band due to $\nu_s SnC_3$ at 515 cm^{-1} for [$SnMe_3PhPO_3H$]$_n$ is an indication of almost planar SnC_3 groups (D_3h) according to group theory. The Mössbauer spectrum shows a slightly asymmetric quadrupole split doublet with an isomer shift (IS) value (1.34 mm/s) in the normal range for organotin(IV) derivatives (Flinn et al., 1978). The quadrupole splitting value (3.78 mm/s) is consistent with the presence of trans coordinated Me_3Sn residues (Davies and Smith, 1982). The crystallographic study confirms the spectroscopic conclusions.

Selected bond distances (Å)

Sn-O(1) 2.182(3); Sn-O(3′) 2.374(3); Sn-C(2) 2.096(6); Sn-C(3) 2.110(5); Sn-C(1) 2.123(6); P-O(1)1.507(3); P-O(3) 1.512(3); P-O(2) 1.563(4); P-C(4A) 1.744(11); P-C(4) 1.866(19); O(2)-H(2) 0.85(2); O(3″)-Sn 2.374(3); C(4)-C(5) 1.3900; C(4)-C(9) 1.3900; C(5)-C(6) 1.3900.

Selected bond angles (°)

O(1)-Sn-O(3′) 171.47(12); C(2)-Sn-C(3) 124.1(3); C(2)-Sn-C(1) 121.9(3); C(3)-Sn-C(1) 113.7(3); C(2)-Sn-O(1) 90.3(2); C(3)-Sn-O(1) 90.40(19); C(1)-Sn-O(1) 94.82(18); C(2)-Sn-O(3′) 85.0(2); C(3)-Sn-O(3′) 86.4(2); C(1)-Sn-O(3′) 93.71(18); O(1)-P-O(3) 115.0(2); O(1)-P-O(2) 107.7(2); O(3)-P-O(2) 109.1(2); O(1)-P-C(4) 109.1(7); O(3)-P-C(4)105.5(7); O(2)-P-C(4) 110.4(7); P-O(1)-Sn 141.4(2); P-O(2)-H(2) 116(5); P-O(3)-Sn#2131.30(18).

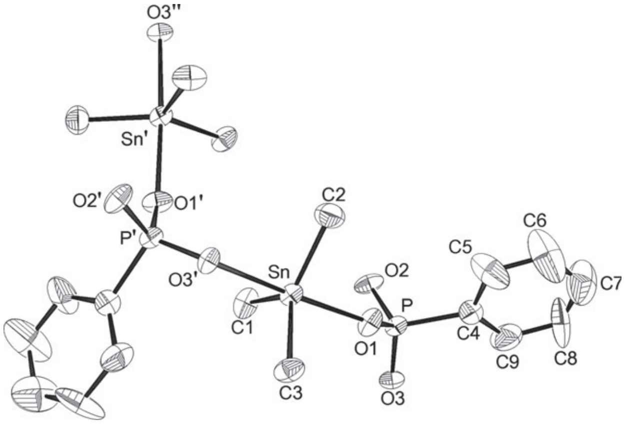


Figure 1 Zigzag infinite chain of [$SnMe_3PhPO_3H$]$_n$.
H atoms and the second component of the disordered phenyl ring have been omitted for clarity. Symmetry codes: (′) 1/2-*x*, *y*-1/2, 1/2-*z* (″) *x*, *y*-1/2; displacement ellipsoids are drawn at the 50% probability level.

Table 1 Hydrogen bond geometry (Å, °).

D-H···A	D-H	H···A	D···A	D-H···A
O(2)-H(2)...O(2)	0.85	1.76(3)	2.610(5)	172(5)

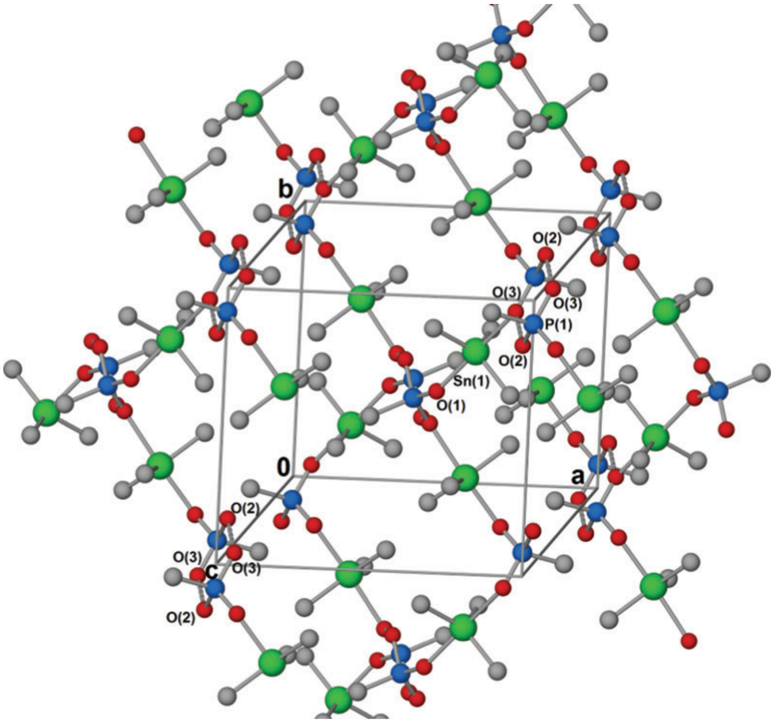


Figure 2 Unit cell of $[SnMe_3PhPO_3H]_n$ showing intermolecular P=O···OH hydrogen bonds. The phenyl groups have been omitted for clarity.

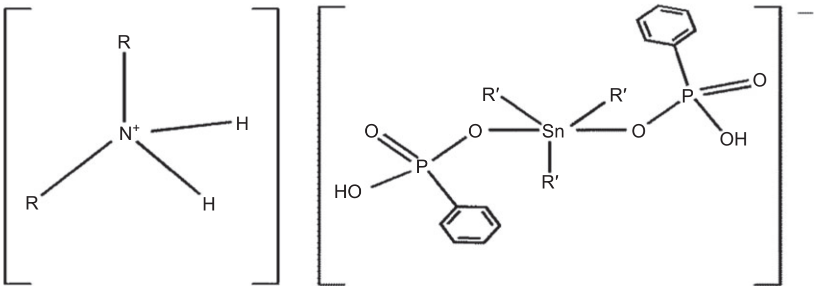
X-ray crystallographic data of $[PhPO_3HSnMe_3]_n$

A crystal of approximate dimensions 0.20 mm×0.10 mm ×0.10 mm was used for data collection. Program data were collected on a Nonius Kappa CCD diffractometer at 150(2) K using Mo-Kα radiation (λ=0.71073 Å); refinement was full-matrix least squares based on F^2; the absorption correction was semiempirical from equivalents. In the final cycles of least-squares refinement all nonhydrogen atoms were allowed to vibrate anisotropically. The C-bound H atoms were included in calculated positions and treated as riding atoms: C-H=0.95 and 0.98 Å for CH and CH_3 H atoms, respectively, with $U_{iso}(H)=K\times U_{eq}(C)$, where K=1.2 for CH H atoms, and K=1.5 for CH_3 H atoms. However, H binding to O(2) had to be restrained with O-H=0.82 Å. The molecule includes a phenyl ligand disordered over two sites in the ratio 40:60; C atoms in the rings have been restrained to form an ideal hexagon, and C4 was refined isotropically.

The following programs were used: SIR97 to solve structure (Altomare et al., 1999), SHELXL97 to refine structure (Sheldrick, 2008), and ORTEP-3 for Windows for molecular graphics (Farrugia, 1997, 1999). Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre, CCDC No. 852507. Crystal data and structure refinement: empirical formula, $C_9H_{15}O_3PSn$; formula weight, 320.87; crystal system, monoclinic; space group, $P12_1/n1$; a=11.0675(8) Å, b=10.4180(6) Å, c=12.0670(8) Å; α=90°, β=103.584(3)°, γ=90°; V=1352.42(15) $Å^3$; Z=4; ρ_{calc}=1.576 mg/m; μ (Mo-Kα)=1.991 mm^{-1}; F(000), 632; reflections collected, 11,929; independent reflections, [R(int)] 2986 [0.1207]; reflections observed, (>2σ) 1808; absorption correction: semi-empirical from equivalents; maximum, minimum transmission, 0.8258, 0.6916; refinement method, full-matrix least squares on F^2; goodness of fit, 0.991; final R indices: [I>2σ(I)]=0.0475, 0.0988; R indices (all data): 0.1041, 0.1185; largest difference peak and hole: 0.537, -1.853 $eÅ^3$.

Spectroscopic characterization of $Cy_2NH_2(PhPO_3H)_2SnMe_3$ (2) and $Bu_2NH_2(PhPO_3H)_2SnPh_3$ (3)

In comparison with the data reported for other phenylphosphonates adducts or derivatives (Adair et al., 2003; Song et al., 2007; Chunlin et al., 2008; De Barros et al., 2010; Shankar et al., 2011), we suggest the following infrared band assignments to be made for the compound. The bands located at 1170 s, 1120 s and 1083 s for $Cy_2NH_2(PhPO_3H)_2SnMe_3$ (**2**) and 1135 s, 1102 s and 1056 s for $Bu_2NH_2(PhPO_3H)_2SnPh_3$ (**3**) are assigned to the stretching vibrations of the PO_3 groups. The bands at 948 s cm^{-1} (**2**) and 947 s cm^{-1} (**3**) are assigned to δPO_3. The presence

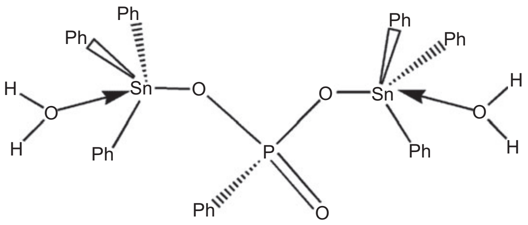


Scheme 1: Structure of $R_2NH_2(PhPO_3H)_2SnR'_3$ (R=Cy, Bu; R′=Me, Ph) (**2** and **3**).

of ν_sSnC_3 on the infrared spectra of (**2**) as a weak band is an indication of D_3h symmetry for the SnC_3 residue. The value of the quadrupole splitting (QS) of (**3**) (QS=3.57 mm/s) is consistent with the presence of a trans coordinated $SnPh_3$ group according to Bancroft and Platt (1972). The suggested structure for (**2**) and (**3**) consists of a five-coordinate trigonal bipyramidal geometry around the tin(IV) atom with two axial oxygen atoms of monodentate $PhPO_3H^-$ and three equatorial phenyl groups (Scheme 1). N-H...O hydrogen bonds via the cation $Bu_2NH_2^+$ or O-H...O hydrogen bonds via hydrogenophenylphosphonate are forming a supramolecular architecture.

Spectroscopic characterization of $PhPO_3(SnPh_3)_2{\cdot}2H_2O$ (4)

The QS value (=2.95 mm/s) of this dinuclear compound is consistent with a dissymmetrical trans O_2SnC_3 stereochemistry about the tin center according to Bancroft and Platt (1972). This allows suggestion of a monomeric structure, the environment around the tin center being dissymmetrical trans trigonal bipyramidal, and the anion $PhPO_3^{2-}$ bi-unidentate:



Experimental

Materials and spectroscopic methods

$SnMe_3Cl$, $SnPh_3Cl$, $SnPh_3OH$, the $PhPO(OH)_2$, and R_2NH (R=Bu, Cy) were purchased from Aldrich Chemical Company, Inc (product of Germany) and used without further purification. The infrared spectra were recorded at the laboratory of control medicine (Dakar) by means of a Bruker FT-IR type spectrometer. The samples were prepared as KBr pellets. Elemental analyses were performed at the University of Bath (UK) using an Exeter Analytical CE440 analyzer. Infrared data are given in cm^{-1} (abbreviations: vs, very strong; s, strong; m, medium; w, weak). ^{119}Sn Mössbauer spectra were obtained from a constant-acceleration spectrometer moving a $CaSnO_3$ source at room temperature. The samples were analyzed at liquid N_2 temperature, and the IS values are given with respect to that source. All Mössbauer spectra were computer-fitted assuming Lorentzian line shapes. Mössbauer parameters are given in mm/s.

Synthesis of the ligands

$R_2NH_2R'PO_3H$ (R=Bu, Cy) was obtained as a white precipitate on mixing an aqueous solution of R_2NH (R=Bu, Cy; R′=Ph, Me) with $PhPO_3H_2$ at 1:1 ratio. Analytical data: % found (% calc. for ligand): $Bu_2NH_2PhPO_3H$ or $C_{14}H_{26}NO_3P$ (L_1): % C=58.61 (58.52); % H=9.20 (9.12); % N=4.80 (4.87). $Cy_2NH_2PhPO_3H$ or $C_{18}H_{30}NO_3P$ (L_2): % C=63.66 (63.70); % H=9.00(8. 89); % N=4.17(4.13)

Synthesis of $[PhPO_3HSnMe_3]_n$

A methanolic solution containing 0.2 g (0.69 mmol) of $Bu_2NH_2PhPO_3H$ (L_1) and 0.13 g (0.69 mmol) of trimethyltin(IV) chloride ($SnMe_3Cl$) was stirred at room temperature for more than 1 h. After 72 h of slow evaporation of the solution, colorless crystals of $PhPO_3HSnMe_3$ [yield: 62%; melting point (mp): 115°C] suitable for X-ray structure determination were collected within the solvent. The powder obtained after complete solvent evaporation has the formula Bu_2NH_2Cl according to its infrared spectrum.

The chemical substitution reaction is:

$$Bu_2NH_2PhPO_2(OH)+SnMe_3Cl \rightarrow PhPO_3HSnMe_3+Bu_2NH_2Cl$$

Analytical data: % found (% calc. for $C_9H_{15}O_3PSn$, 320.87 g/mol): % C, 33.69 (33.80); % H, 4.71 (4.02). Infrared data (cm^{-1}): 1176 s, 1110 s, 1062 vs νPO_3; 927 s δPO_3; 749 m νPC 556 s $\nu_{as}SnC_3$; 515 w ν_sSnC_3. Mössbauer data (mm/s): IS=1.34, QS=3.78, Γ (full width at half-height)/2=0.43.

Synthesis of $Cy_2NH_2(PhPO_3H)_2SnMe_3$

$Cy_2NH_2(PhPO_3H)_2SnMe_3$ (B) has been obtained by reacting 0.25 g (0.73 mmol) of L_2 with 0.073 g (0.36 mmol) of trimethyltin(IV) chloride

($SnMe_3Cl$) in methanol. After a slow solvent evaporation, a white powder was collected in the solvent (75%, mp 170°C).

The chemical substitution reaction is:

$$2Cy_2NH_2PhPO_2(OH)+SnMe_3Cl \rightarrow Cy_2NH_2(PhPO_3H)_2SnMe_3+Cy_2NH_2Cl$$

Analytical data: % found (% calc. for $C_{27}H_{45}NO_6P_2Sn$, 660.27 g/mol): % C, 48.88 (49.11); % H, 6.93 (6.87); % N, 2.23 (2.12). Infrared data (cm^{-1}): 3289 vs, 3227 s, 2759 br, νNH_2; 1284 s νOH; 1170 s, 1083 vs νPO_3; 948 s νPC; 558 s $\nu_{as}SnC_3$; 517 w ν_sSnC_3.

Synthesis of $Bu_2NH_2(PhPO_3H)_2SnPh_3$

An ethanolic solution containing 0.30 g (1.04 mmol) of L_1 and 0.20 g (0.52 mmol) of triphenyltin(IV) chloride ($SnPh_3Cl$) was stirred at room temperature for more than 1 h. After a slow solvent evaporation of the solution a white powder was obtained (89%, mp 187°C).

The chemical substitution reaction is:

$$2Bu_2NH_2PhPO_2(OH)+SnPh_3Cl \rightarrow Bu_2NH_2(PhPO_3H)_2SnPh_3+Bu_2NH_2Cl$$

Analytical data: % found (% calcd. for $C_{38}H_{47}NO_6P_2Sn$, 795.19 g/mol): % C, 57.37 (57.45); % H: 5.92 (5.96); % N, 1.12 (2.76). Infrared data (cm^{-1}): 3287 vs, 2954 br, νNH_2; 1282 s νOH; 1135 s, 1102 vs νPO_3; 947 s νPC. Mössbauer data (mm/s): IS=1.25, QS=3.57, Γ=0.85.

Synthesis of $PhPO_3(SnPh_3)_2{\cdot}2H_2O$

The dinuclear compound was obtained by reacting 0.06 g (0.40 mmol) of phenylphosphonic acid with 0.08 g (0.23 mmol) of triphenyltin(IV) hydroxide ($SnPh_3OH$) in methanol. After a slow solvent evaporation a white powder was collected in the solvent (80%, mp 120°C).

The chemical substitution reaction is:

$$PhPO(OH)_2+2SnPh_3OH \rightarrow PhPO_3(SnPh_3)_2{\cdot}2H_2O$$

Analytical data: % calc. (% found for $C_{42}H_{39}O_5PSn_2$, 891.4 g/mol): % C, 56.54 (56.50); % H, 4.37 (4.31). Infrared data (cm^{-1}): 1160 s, 1109 vs, 1009 s νPO_3; 928 s δPO_3; 746 m νPC. Mössbauer data (mm/s): IS=1.23, QS=2.95, Γ=0.98±0.04.

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