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Crystal and Molecular Structures of Some Six-Coordinate Tin(IV) Halogeno Complexes with Phosphorus-Containing Ligands

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ABSTRACT: *Six new complexes of tin(IV) halides with phosphorus-containing ligands have been fully characterized by single-crystal X-ray diffraction at low temperature. Three of the compounds, derived from the diphosphanes bis-(diphenylphosphino)methane or bis-(dicyclohexylphosphino)methane, have a novel zwitterionic structure, with five Cl ligands and one unidentate phosphorus-containing ligand on tin, together with a proton on the second phosphorus atom of the potentially bidentate ligand; these are* $Cl_5Sn^-P(Ph_2)CH_2PPh_2H^+$ (**1**), $Cl_5Sn^-OP(Ph_2)CH_2PPh_2H^+$ (**2**), *and* $Cl_5Sn^-OP(cy_2)CH_2Pcy_2H^+$ (**3**). *The other three complexes have a bidentate donor attached to the* SnX_4 *moiety; they comprise* $Cl_4SnOP(Ph_2)(CH_2)_2PPh_2O$ (**4**), *a derivative of bis-(diphenylphosphino)ethane dioxide,* $I_4SnOP(Ph_2)CH_2PPh_2O$ (**5**), *a similar derivative of bis-(diphenylphosphino)methane dioxide, and the very unusual* $Br_4SnAs(Ph_2)(CH_2)_2PPh_2O$ (**6**), *with coordination to tin by As and O. Since the starting material for the last compound was* $Ph_2As(CH_2)_2PPh_2$, *this result illustrates well the more facile oxidation of P(III) than As(III).* Heteroatom Chem 20:136–143, 2009; DOI 10.1002/hc.20525

INTRODUCTION

During our investigations into new phosphorus heterocycles such as cyclic triphosphenium ions [1–7] and their P-alkyl or -aryl derivatives [8–11], we have often used tin(II) halides SnX_2 (X=Cl or Br) as reducing agents, in an attempt to obtain crystalline products as hexahalogenostannates(IV). Occasionally, we have instead obtained crystalline tin-containing coordination complexes, sometimes in the presence of adventitious oxygen and/or moisture, resulting in the coordination by O donors and/or protonation of one of the phosphorus atoms in a diphosphane. The most interesting compound formed in this way resulted from the reaction of $Ph_2P(CH_2)_2AsPh_2$ with PCl_3 in the presence of $SnCl_2$, where, instead of the desired (and unknown) heterocycle containing the As—P—P grouping, a six-coordinate complex **6** of tin(IV) chloride and the bidentate ligand $Ph_2P(O)(CH_2)_2AsPh_2$ was obtained. This product clearly indicates that P(III) is more easily oxidized than As(III). An attempt to obtain a complex between SnI_4 and bis-(diphenylphosphino)methane (dppm) also resulted in a crystalline complex **5** of the diphosphane dioxide as a bidentate ligand (6-membered ring) rather than

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the desired four-membered ring with the diphosphane coordinating directly to tin. While several halogeno complexes of tin(IV) with a —P, —OP, or —As donor atom are known, as discussed in the following section, four of the six new species described represent new types of coordination, including three zwitterionic structures $Cl_5Sn^-P(Ph_2)CH_2PPh_2H^+$ (**1**), $Cl_5Sn^-OP(Ph_2)CH_2PPh_2H^+$ (**2**), and $Cl_5Sn^-OP(cy_2)$-$CH_2Pcy_2H^+$ (**3**), where cy = cyclohexyl. In these complexes, the $SnCl_5^-$ moiety is further coordinated to a P or O donor atom and the charge is balanced by the protonation of the second P in a potentially bidentate ligand. Compound **4** involves more conventional coordination of bis-(diphenylphosphino)ethane dioxide to $SnCl_4$. Complexes **1–6** have all been fully characterized by single-crystal X-ray diffraction at low temperature.

RESULTS AND DISCUSSION

The molecular structure of compound **1** is shown in Fig. 1, whereas selected bond lengths and angles are listed in Table 1. There are two molecules in the unit cell, with slightly differing bond distances and angles, as indicated in the table. This complex corresponds to unidentate coordination of the potentially bidentate diphosphane bis-(diphenylphosphino)methane to $SnCl_4$, with the addition of HCl to form the zwitterionic structure, as shown. The arrangement around Sn, as in all the complexes studied here, is a slightly distorted octahedron, with bond angles between trans pairs of ligands from 171.5° to 175.3°. The crystal and molecular structures have been previously reported for *trans*-$SnCl_4(dppm)_2$ [12], in which both phosphorus-containing ligands coordinate in a unidentate fashion, possibly indicating that a four-membered ring containing a large atom such as Sn is energetically unfavorable. The Sn(1)-P(1) and Sn(2)-P(3) distances of 2.7312(7) and 2.7573(6) Å in **1** are within the normal range for six-coordinate complexes of tin(IV) [13–18], where values from 2.532(1) to 2.7578(5) Å have been ascertained. A longer Sn—P distance of 2.862(2) Å was found in $BuSnCl_3{\cdot}PPh_3$, which has a trigonal bipyramidal structure with the phosphorus ligand in an axial position [19]. The Sn—Cl bond lengths in **1** are also entirely as expected. Although a search of the Cambridge Structural Database (December 2008 release) [20,21] revealed several complexes with the $SnCl_5^-$ moiety coordinated to other groups, such as R (R = Et, *n*-Bu, or Ph), H_2O, THF, MeCN, or MeOH, compound **1** appears to be the first example of such a species with a zwitterionic structure to be characterized by X-ray diffraction. The zwitterionic nature of this structure is paralleled by compounds **2** and **3**. The formulae of complexes **1**–**6** are shown in Scheme 1.

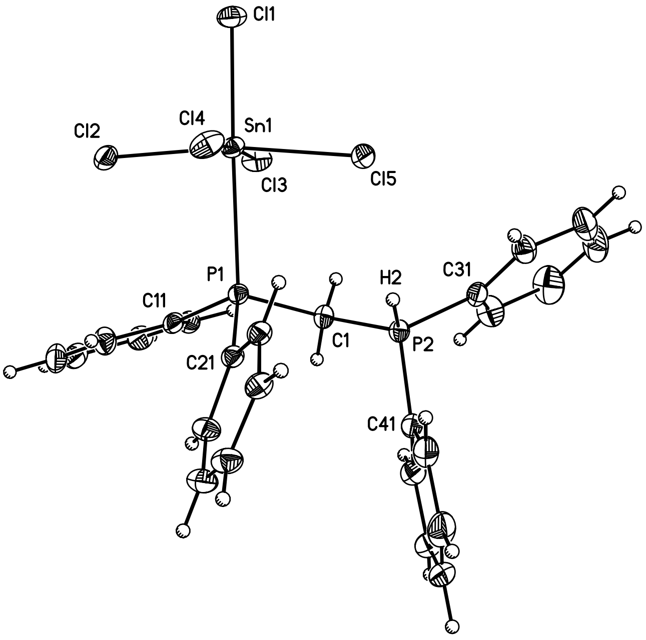
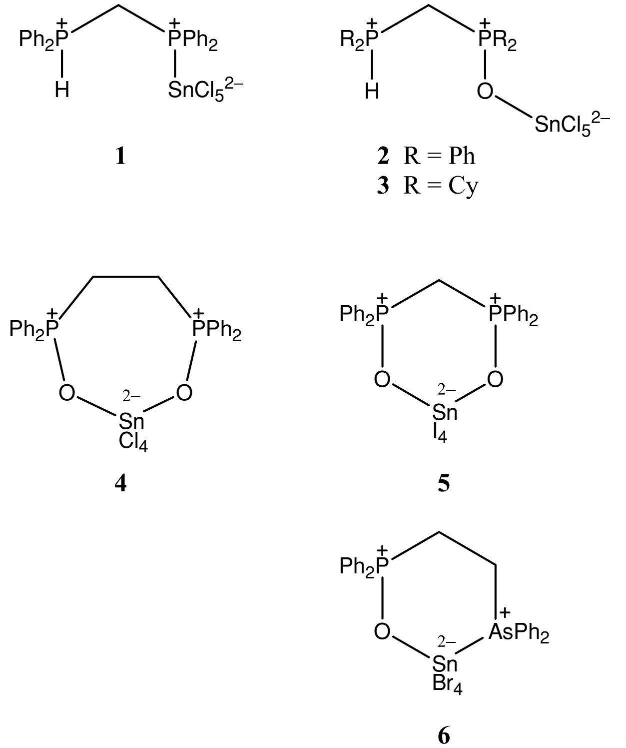


FIGURE 1 Molecular structure of **1**. Thermal ellipsoids are shown at 50% probability.

The molecular structures of complexes **2** and **3** are shown in Figs. 2 and 3, respectively; selected bond distances and angles are listed in

TABLE 1 Selected Bond Distances (Å) and Angles (°) in Compound **1**

Sn(1)—P(1)	2.7312(7)	Sn(2)—P(3)	2.7573(6)
Sn(1)—Cl(1)	2.3986(6)	Sn(2)—Cl(6)	2.4028(6)
Sn(1)—Cl(2)	2.4120(6)	Sn(2)—Cl(7)	2.4205(5)
Sn(1)—Cl(3)	2.4368(6)	Sn(2)—Cl(8)	2.4235(6)
Sn(1)—Cl(4)	2.4369(6)	Sn(2)—Cl(9)	2.4182(6)
Sn(1)—Cl(5)	2.4251(6)	Sn(2)—Cl(10)	2.4398(5)
P(1)—Sn(1)—Cl(1)	175.205(17)	P(3)—Sn(2)—Cl(6)	175.130(17)
Cl(3)—Sn(1)—Cl(4)	175.216(18)	Cl(7)—Sn(2)—Cl(10)	173.185(17)
Cl(2)—Sn(1)—Cl(5)	171.502(18)	Cl(8)—Sn(2)—Cl(9)	175.296(17)



SCHEME 1

Table 2. [Compound **3** is a bis-($CHCl_3$) solvate.] In both instances, the starting diphosphane (dppm or bis-(dicyclohexylphosphino)methane) has been oxidized at one of its phosphorus atoms to the oxide, which has then coordinated to the Cl_5Sn^- group, the balance of charges being maintained by the protonation of the second phosphorus atom. (Alternatively, they could arise from the coordination of the diphosphane monoxide to $SnCl_4$, followed by the addition of HCl.) The only previous literature report of a species with the X_5SnOP^- structure (X=Cl, Br, or I) is from our work, in the centrosymmetric dianion [$Cl_5SnOP(Ph_2)CH{=}CHP(Ph_2)OSnCl_5$], which has a P—O bond length of 1.507(4) Å, the Sn—O distance is 2.131(4) Å, and Sn—Cl values lie between 2.385(2) and 2.434(2) Å [3]. There have also been several structural determinations for complexes of the type $X_4Sn(OP\cdots PO)$ (a chelating diphosphane dioxide), or $X_4Sn(OP\text{-})_2$, with both *cis* and *trans* isomers known for some unidentate phosphane monoxides [22–32]. Representative distances for chloro compounds [3,22–26] make it abundantly clear that the values for compounds **2** and **3** are within the normal ranges, apart from the P—O bond in **3**, which is very slightly longer than is usual.

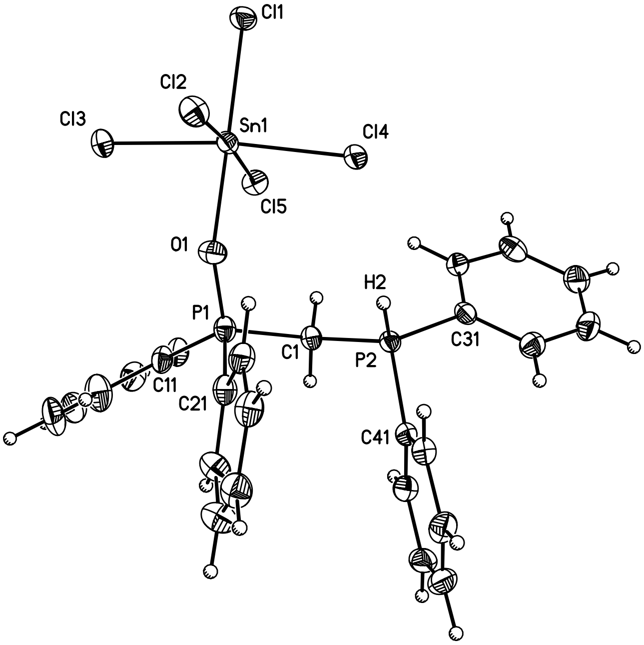


FIGURE 2 Molecular structure of **2**. Thermal ellipsoids are shown at 50% probability.

In zwitterions **1** and **2**, there are contacts between the hydrogen atom on phosphorus and one or more chlorine atoms on the tin atom of an adjacent zwitterion at less than the calculated sum of the van der Waals radii, possibly corresponding to weak H-bonding interactions (Fig. 4). This gives rise to weak dimerization. In zwitterion **3**, this weak dimerization motif is still apparent, although the distance

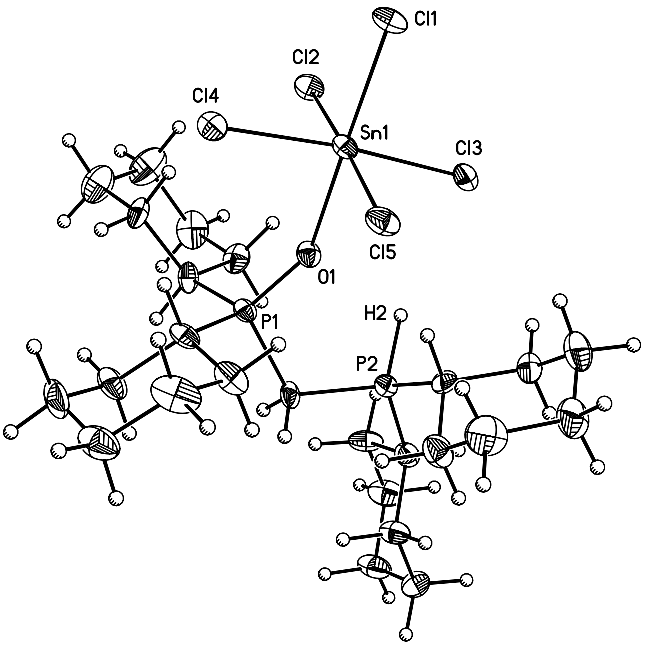


FIGURE 3 Molecular structure of **3**. Thermal ellipsoids are shown at 50% probability.

TABLE 2 Selected Bond Distances (Å) and Angles (°) in Compounds **2** and **3**

	2	*3*
Sn(1)—O(1)	2.108(2)	2.131(4)
O(1)—P(1)	1.492(2)	1.528(5)
Sn(1)—Cl(1)	2.404(1)	2.408(2)
Sn(1)—Cl(2)	2.409(1)	2.420(2)
Sn(1)—Cl(3)	2.410(1)	2.401(2)
Sn(1)—Cl(4)	2.413(1)	2.399(2)
Sn(1)—Cl(5)	2.438(1)	2.423(2)
O(1)—Sn(1)—Cl(1)	177.50(6)	178.50(14)
Cl(3)—Sn(1)—Cl(4)	173.41(3)	173.86(6)
Cl(2)—Sn(1)—Cl(5)	174.16(3)	176.26(6)

between the chlorine and hydrogen atoms is somewhat longer than the sum of the van der Waals radii.

Compounds **4** and **5** are complexes of SnX_4 (X=Cl and I, respectively) and a diphosphane dioxide, giving seven- (**4**) and six- (**5**) membered rings. The molecular structures are shown in Figs. 5 and 6, while selected bond distances and angles are listed in Table 3. (Compound **4** is an acetone solvate.) As for the other species studied here, there is a slightly distorted octahedral geometry around Sn, with the angles between *trans* pairs of ligands varying from 171.7° to 176.3° in **4** and from 167.2° to 175.3° in **5**. The only minor difference is that the smallest of these angles is X—Sn—X for compounds **1–4** (and **6**), whereas in **5** both O—Sn—I angles are smaller than the *trans* I—Sn—I angle. This may result from steric effects from the more bulky iodine ligands. The Sn—O, Sn—Cl, and P—O bond lengths in **4** may again be compared with literature data [3,22–26] and confirm that these are as expected for such a compound. Some previous results from coordination compounds of SnI_4 [13,27,28] show a similar, consistent pattern.

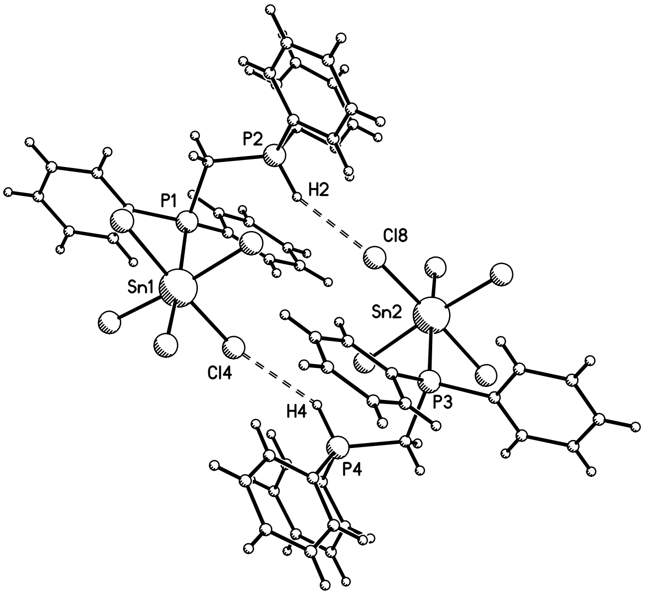


FIGURE 4 Short H···Cl contacts in **1** giving a weak dimeric structure.

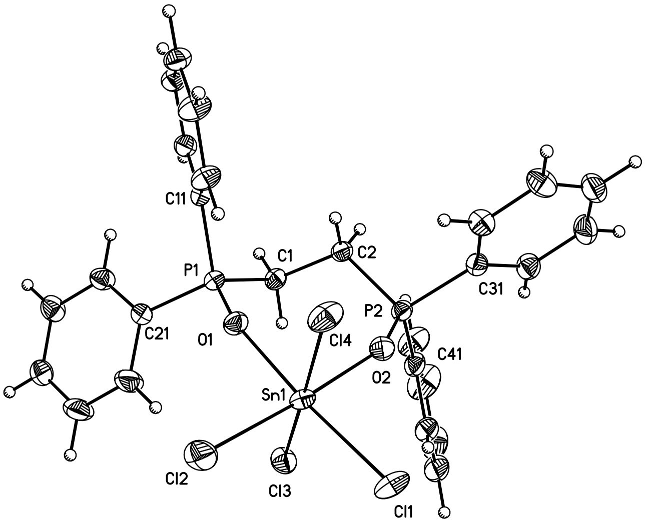


FIGURE 5 Molecular structure of **4**. Thermal ellipsoids are shown at 50% probability.

Complex **6** is in some ways the most interesting of all because it arose from the use of the starting material $Ph_2P(CH_2)_2AsPh_2$ in an attempt to prepare a novel (unknown) heterocyclic cation with a —P—P—As— grouping as its hexabromostannate(IV) salt. (Similar cyclic derivatives containing —P—P—P— or —P—As—P— units are well established [1–3,5,7,33]. Instead, the new heterocycle **6** was obtained, with the Br_4Sn moiety coordinated by As and O(P), giving a six-membered ring. This result

TABLE 3 Selected Bond Distances (Å) and Angles (°) in Compounds **4** and **5**

	4 (X=Cl)	*5 (X=I)*
Sn(1)—O(1)	2.099(1)	2.127(4)
Sn(1)—O(2)	2.102(1)	2.163(4)
O(1)—P(1)	1.519(1)	1.522(4)
O(2)—P(2)	1.519(1)	1.516(4)
Sn(1)—X(1)	2.407(1)	2.795(1)
Sn(1)—X(2)	2.389(1)	2.778(1)
Sn(1)—X(3)	2.377(1)	2.783(1)
Sn(1)—X(4)	2.402(1)	2.795(1)
O(1)—Sn(1)—X(1)	174.80(4)	170.93(11)
O(2)—Sn(1)—X(2)	176.25(4)	167.21(10)
X(3)—Sn(1)—X(4)	171.73(2)	175.28(2)

TABLE 4 Selected Bond Distances (Å) and Angles (°) in Compound **6**

Sn(1)—As(1)	2.7519(4)	Sn(1)—Br(2)	2.5418(4)
Sn(1)—O(1)	2.146(2)	Sn(1)—Br(3)	2.5508(4)
O(1)—PI(1)	1.527(2)	Sn(1)—Br(4)	2.5856(4)
Sn(1)—Br(1)	2.5392(4)		
As(1)—Sn(1)—Br(2)	169.908(12)	Br(3)—Sn(1)—Br(4)	168.951(13)
O(1)—Sn(1)—Br(1)	172.96(5)		

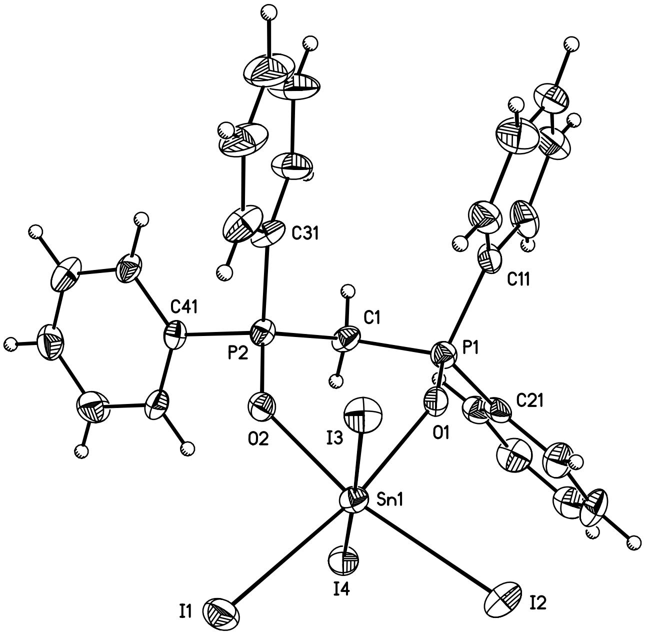


FIGURE 6 Molecular structure of **5**. Thermal ellipsoids are shown at 50% probability.

clearly shows that P(III) is more easily oxidized than As(III) in the original arsaphosphane. Its molecular structure is shown in Fig. 7, while selected bond distances and angles are listed in Table 4. The structure is a slightly distorted octahedron around the tin atom, with bond angles between *trans* pairs of ligands of 172.96(5)°, 168.951(13)°, and 169.908(12)°. The Br—Sn, Sn—O, and O—P bond distances when compared with literature data for six-coordinate Sn(IV) complexes [18,29–32] are in keeping with the expected ranges. There are comparatively few reports for As(III) bonded to Sn(IV) in six-coordinate species (Sn—As bond lengths: 2.716(2), 2.752(2) [28], 2.7623(3) [34], 2.6932(5), 2.7095(6) [18]), and the present result is again within this range of values. A slightly longer distance of 2.780(1) Å was found in a five-coordinate Sn complex, in which the As ligand occupies an axial site in a trigonal bipyramidal structure [34].

In conclusion, we have established the crystal and molecular structures at low (100 K or 120 K)

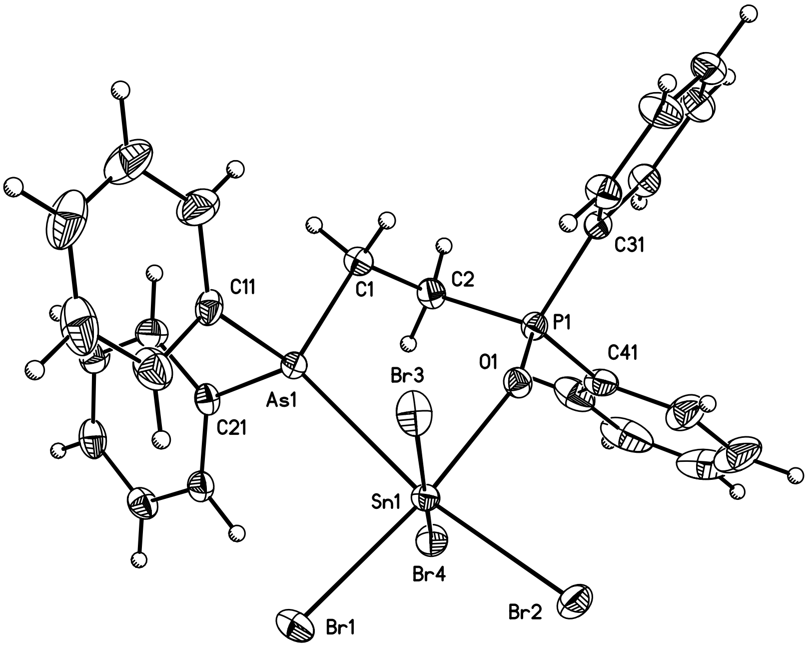


FIGURE 7 Molecular structure of **6**. Thermal ellipsoids are shown at 50% probability.

TABLE 5 Crystal Data for Compounds **1–6**

	1	*2*	*3*	*4*	*5*	*6*
Empirical formula	$C_{25}H_{23}Cl_5P_2Sn$	$C_{25}H_{23}Cl_5OP_2Sn$	$C_{25}H_{47}Cl_5OP_2Sn$ $\cdot 2(CHCl_3)$	$C_{26}H_{24}Cl_4O_2P_2Sn$ $\cdot C_3H_6O$	$C_{25}H_{22}I_4O_2P_2Sn$ $\cdot {}^1/_2\,(CHCl_3),{}^1/_2(CH_2Cl_2)$	$C_{26}H_{24}AsBr_4OPSn$
Formula weight	681.31	697.31	960.24	748.96	1,144.80	896.67
Temperature (K)	100(2)	100(2)	120(2)	120(2)	120(2)	120(2)
Crystal system	Monoclinic	Monoclinic	Monoclinic	Triclinic	Monoclinic	Monoclinic
Space group	$P2_1/n$	$P2_1/c$	$P2_1/n$	P-1	$P2_1$	$P2_1/n$
a (Å)	15.222(3)	9.8089(4)	15.948(2)	9.796(2)	9.2198(6)	10.886(2)
b (Å)	19.794(3)	18.6540(6)	16.284(2)	11.497(1)	19.027(1)	16.455(3)
c (Å)	18.420(3)	15.6379(6)	16.138(2)	16.171(3)	10.2615(6)	16.723(3)
α (°)				99.91(1)		
β (°)	96.34(1)	104.912(1)	105.952(2)	102.85(1)	109.704(1)	106.937(2)
γ (°)				110.81(1)		
Volume (Å^3)	5,516.1(16)	2,764.98(18)	4,029.6(7)	1,595.6(5)	1,694.7(2)	2,865.5(9)
Z	8	4	4	2	2	4
ρ_{calc} (mg/mm^3)	1.641	1.675	1.583	1.559	2.243	2.078
μ (mm^{-1})	1.540	1.541	1.465	1.265	4.712	7.690
F (000)	2,704	1,384	1,944	752	1,060	1,704
Crystal size (mm)	0.33 × 0.32 × 0.09	0.36 × 0.14 × 0.14	0.21 × 0.18 × 0.11	0.52 × 0.21 × 0.18	0.1 × 0.08 × 0.02	0.4 × 0.4 × 0.28
θ range for data collection	1.52° to 29.00°	1.73° to 25.03°	1.59° to 25.02°	1.34° to 29.00°	2.11° to 27.01°	1.78° to 28.29°
Index ranges	$-19 \le h \le 20$ $-26 \le k \le 24$ $-25 \le l \le 25$	$-11 \le h \le 11$ $-22 \le k \le 22$ $-18 \le l \le 18$	$-18 \le h \le 14$ $-19 \le k \le 19$ $-17 \le l \le 19$	$-13 \le h \le 13$ $-15 \le k \le 14$ $-21 \le l \le 22$	$-11 \le h \le 11$ $-24 \le k \le 24$ $-13 \le l \le 13$	$-14 \le h \le 14$ $-21 \le k \le 21$ $-21 \le l \le 22$
Reflections collected	67,331	24,161	18,990	19,296	17,886	36,028
Independent reflections	14,546 [R(int) = 0.0369]	4,886 [R(int) = 0.0370]	7,068 [R(int) = 0.0831]	8,175 [R(int) = 0.0308]	7,365 [R(int) = 0.0367]	7,109 [R(int) = 0.0605]
Data/restraints/ parameters	14,546/0/603	4,886/0/311	7,068/12/383	8,175/94/367	7,365/33/350	7,109/0/307
Goodness-of-fit on F^2	1.078	0.997	1.028	1.045	0.913	1.078
Final R indexes [$I > 2\sigma(I)$]	$R_1 = 0.0257$ $wR_2 = 0.0577$	$R_1 = 0.0267$ $wR_2 = 0.0611$	$R_1 = 0.0618$ $wR_2 = 0.1407$	$R_1 = 0.0286$ $wR_2 = 0.0709$	$R_1 = 0.0282$ $wR_2 = 0.0515$	$R_1 = 0.0278$ $wR_2 = 0.0686$
Final R indexes (all data)	$R_1 = 0.0337$ $wR_2 = 0.0625$	$R_1 = 0.0330$ $wR_2 = 0.0638$	$R_1 = 0.1084$ $wR_2 = 0.1595$	$R_1 = 0.0317$ $wR_2 = 0.0730$	$R_1 = 0.0346$ $wR_2 = 0.0534$	$R_1 = 0.0326$ $wR_2 = 0.0706$
Largest difference peak/hole	0.668/−0.282	0.912/−0.702	1.328/−0.816	0.975/−0.770	1.131/−1.123	0.845/−1.119
Flack parameter					0.011(19)	

temperature of six new slightly distorted octahedral complexes of tin(IV). Three of these (**1–3**) have a novel zwitterionic structure, with the formal negative charge on tin balanced by the protonation of a phosphorus atom elsewhere in the molecule. Another (**6**) shows the formation of a new six-membered heterocycle, with the —As—C—C—P—O— group coordinated to the $SnBr_4$ moiety.

EXPERIMENTAL

X-ray Crystallography

Single-crystal structure determinations were carried out from X-ray data collected using graphite monochromated Mo Kα radiation ($\lambda = 0.71073$ Å) on a Bruker SMART-CCD 1 K diffractometer at 100 K or 120 K (Table 5). The temperature was controlled with a Cryostream N_2 flow cooling device [35]. In each case, a series of narrow ω-scans (0.3°) was performed at several ϕ-settings in such a way as to cover a sphere of data to a maximum resolution of 0.70 Å. Cell parameters were determined and refined with SMART software [36] and raw frame data were integrated with the SAINT program [37]. The structures were solved by direct methods [38] and refined by full-matrix least-squares on F^2 with SHELXL-97 [39] and the graphical user interface Olex2 [40]. Crystallographic data for structures **1**–**6** have been deposited with the Cambridge Crystallographic Data Centre (CCDC) as CCDC 720150–720155, respectively. These data can be obtained free of charge from the CCDC at: www.ccdc.cam.ac.uk/data_request/cif.

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