Crystal and molecular structures of the adducts of tri-*p*-tolyltin chloride, bromide and iodide with 4,4′-bipyridine

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Abstract

The structures of the three adducts $[(p\text{-tolyl})_3SnX]_2 \cdot 4,4'$-bipyridine (X = Cl, Br, I), have been established by X-ray diffraction techniques. The complexes are monoclinic, space group $P2_1/c$ and $Z = 2$. The cell parameters are: $a = 13.262(2)$, $b = 11.382(2)$, $c = 17.029(2)$ Å and $\beta = 110.050(11)°$ for X = Cl; $a = 13.103(3)$, $b = 11.351(2)$, $c = 17.607(4)$ Å and $\beta = 110.050(16)°$ for X = Br; $a = 12.867(3)$, $b = 11.389(4)$, $c = 18.496(4)$ Å and $\beta = 110.050(11)°$ for X = I. The complexes are binuclear with a bridging 4,4′-bipyridine. The tin is in a trigonal bipyramidal environment with the *p*-tolyl groups on the equator and the X atom and an N of the bipyridine on the axis. The Sn–N bond distances are 2.668(3), 2.653(3) and 2.655(7) Å, for X = Cl, Br and I, respectively. The Sn–X bond distances are 2.452(2), 2.691(1) and 2.830(1) Å, for X = Cl, Br and I, respectively.

Introduction

There has been much interest in complexes of 4,4′-bipyridine (4,4′-bpy) mainly as a result of the electronic structure and bifunctional nature of the ligand. Consequently, the ligand provides a mechanism for magnetic superexchange [1] and facilitates electron transfer, as for example, between iron(II) pentacyano and cobalt(III) complexes [2]. The structural chemistry of 4,4′-bpy complexes is also of interest and crystallographic data have revealed several structural roles for the ligand. Thus, it acts as a mono-functional ligand in the cation $[Cu(dien)(4,4'\text{-bpy})(H_2O)]^{2+}$ [1] (dien = diethylenetriamine). However, its more common role is as a bifunctional ligand whereby it may link two Lewis acid moieties, as in the ions $[Cu(dien)(ClO_4)]_2 \cdot 4,4'\text{-bpy}^{2+}$ [1], $[Nd(H_2O)_2(NO_3)_4]_2 \cdot 4,4'\text{-bpy}^{1-}$ [3] and $[Gd(H_2O)_8]_2 \cdot 4,4'\text{-bpy}^{3+}$ [4], in $[Co(ida)(H_2O)_2]_2 \cdot 4,4'\text{-bpy} \cdot 4H_2O$ [5] and $[Cu(ida)]_2 \cdot 4,4'$-bpy [6] (ida = iminodiacetate). The ligand may generate infinite chain structures, e.g. in $Co(dmg)_2 \cdot 4,4'$-bpy [7], $Ni(S_2COC_2H_5)_2 \cdot 4,4'\text{-bpy} \cdot 2CCl_4$ [8], $Cu(acac)_2 \cdot 4,4'$-bpy [9] and $Ni(BF_2dmg)_2 \cdot 4,4'$-bpy [10] (dmg = dimethylglyoxinate). Among the main group elements, the only crystallographic investigation reported, as far as we know, is for $[Te(S_2COC_2H_5)_2]_2 \cdot 4,4'$-bpy, whose structure consists of molecular binuclear species with a bridging 4,4′-bpy molecule [11]. Some preparative and spectroscopic investigations of tin(IV)–4,4′-bpy adducts have been reported [12, 13], but no crystallographic data on such compounds have appeared. As part of our interest in the chemistry of the Group 14 elements, we wish to report here the detailed structural features of $[(p\text{-tolyl})_3SnCl]_2 \cdot 4,4'$-bpy, $[(p\text{-tolyl})_3SnBr]_2 \cdot 4,4'$-bpy and $[(p\text{-tolyl})_3SnI]_2 \cdot 4,4'$-bpy.

Experimental

Preparations

Tin tetrachloride, tin tetrabromide and 4,4′-bpy were obtained from Aldrich Chemical Co. Tin tetraiodide was prepared by a published procedure [14]. The tri-*p*-tolyltin halides were prepared using the redistribution reaction between tetra-*p*-tolyltin and the appropriate stannic halide [15].

$[(p\text{-tolyl})_3SnCl]_2 \cdot 4,4'$-bpy (I)

To a hot solution of 1.98 g (4.6 mmol) of $(p\text{-tolyl})_3SnCl$ in 20 ml of toluene was added slowly a hot solution of 0.36 g (2.3 mmol) of 4,4′-bpy in 10 ml of toluene. The mixture was stirred for 0.5 h, cooled in ice and

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the product collected and recrystallized from petroleum ether. Yield 1.3 g (55%), m.p. 138–140 °C.

[(p-tolyl)$_3$SnBr]$_2$·4,4′-bpy (II)

An identical method to that described above using 2.40 g (4.6 mmol) of (*p*-tolyl)$_3$SnBr and 0.36 g (2.3 mmol) of 4,4′-bpy gave, after recrystallization from petroleum ether, 1.1 g (40%) of the adduct, m.p. 132–134 °C.

[(p-tolyl)$_3$SnI]$_2$·4,4′-bpy (III)

To a hot solution of 2.6 g (5.0 mmol) of (*p*-tolyl)$_3$SnI in 25 ml of benzene was added slowly a hot solution of 0.39 g (2.5 mmol) of 4,4′-bpy in 20 ml of benzene while stirring. The solution was filtered to remove a small amount of an unidentified yellow solid and the filtrate concentrated to around 15 ml. The solution was left in the freezer for 2 days and the crystals which formed were filtered off, washed with cold benzene and dried in air. Yield 0.6 g (20%), m.p. 105–107 °C.

Anal. **I**. Found: C, 61.96; H, 5.19; N, 2.58. Calc.: C, 61.80; H, 5.00; N, 2.77%. **II**. Found: C, 56.74; H, 4.70; N, 2.46. Calc.: C, 56.80; H, 4.50; N, 2.55%. **III**. Found: C, 52.46; H, 4.19, N, 2.17. Calc.: C, 52.31; H, 4.42; N, 2.23%.

X-ray crystallography

Crystals suitable for X-ray structure determination were obtained by slow evaporation of a toluene solution of **I**, a benzene solution of **II** and a petroleum ether solution of **III**. Crystallographic measurements were carried out using a Siemens R3m/V diffractometer and graphite monochromatized Mo Kα radiation ($\lambda = 0.71073$ Å) at an ambient laboratory temperature of 25 °C. The ω–2θ scan technique was used with a 2θ range of 3.5–45° and a scan range (ω) of 1.5°. Backgrounds were measured at the end of each scan range for 25% of the total scan time. For **I** and **II**, three standard reflections were measured at intervals of 50 reflections; for **III** four standard reflections were measured at intervals of 100 reflections. The structures were solved by the heavy atom method and refined by full-matrix least-squares techniques. The function minimized was $\Sigma\omega(F_o-F_c)^2$. Calculations were performed using the Siemens SHELXTL PLUS (MicroVax II) software package. Crystal data and other details of the X-ray structure determinations are in Table 1.

Results and discussion

In 1953, Poller and Tooley [12] reported the formation of 1:1 adducts between Ph_3SnCl, Ph_2SnCl_2, (octyl)$_2$SnCl$_2$ and 4,4′-bpy, and a 1:2 adduct (Sn:ligand) with $SnBr_4$. On the other hand, the elemental analyses for the compounds reported here (**I**, **II**, **III**) indicate [(*p*-tolyl)$_3$SnX]$_2$·4,4′-bpy species (X = Cl, Br, I). The compounds are isostructural and the structure of **I**, with atomic labelling is shown in Fig. 1. Selected bond lengths and bond angles for the three compounds are given in Table 2 and atomic coordinates for **I** in Table 3. The complex molecules possess crystallographic C_i symmetry and the ligand is bonded to two symmetry related tin atoms to form a bridged species. The unique tin atom is in a distorted trigonal bipyramidal environment and is bonded to three *p*-tolyl groups in the equatorial plane. The halogen atom and a nitrogen of the bpy ligand are in the axial positions. The angles subtended at tin by the *ipso*-carbon atoms are 116.0, 118.6 and 122.4° for **I**, 116.1, 117.5 and 123.4° for **II**, and 115.6, 116.9 and 124.0° for **III**. Thus, for each compound, two of the angles are lower than the 120° normally expected for a regular structure, while one is slightly larger. However, the sum of these equatorial angles is the same for **I** and **II** (357.0°), while for **III** it is 356.5°. The distortion from regular geometry is indicated also by the N–Sn–X bond angles, which are 176.0(1)° for **I**, 176.6(1)° for **II** and 176.8(1)° for **III**. In addition, the Sn atom in each adduct is displaced from the plane formed by C(7), C(13) and C(19) towards the halide atom by 0.161 Å for **I**, 0.270 Å for **II** and 0.230 Å for **III**.

The average Sn–C (*p*-tolyl) bond lengths are 2.124, 2.120 and 2.133 Å, for **I**, **II** and **III**, respectively, in good agreement with published values [16, 17]. These values correspond well with the sum of the covalent radii (2.15 Å) of tin and carbon [18]. The Sn–N bond lengths for **II** and **III** are nearly the same, 2.653(3) and 2.655(7) Å, respectively. For **I**, this bond is only slightly longer, 2.668(3) Å. However, it should be noted that packing effects can alter the Sn–N bond lengths in the range observed for the three compounds. Actually, on the basis of electronegativity alone, the Sn–N bond length is expected to be in the order $Cl < Br < I$: the more electronegative chloride should render the tin more acidic thereby enhancing the nucleophilic attack of the nitrogen. However, bonding effects [19] may operate so as to cause a higher Lewis acidity at tin for X = Br and even higher for X = I. It should be pointed out also that the Sn–N bond lengths for these 4,4′-bpy complexes are among the longest yet to be reported. Values usually range from about 2.3–2.5 Å for various di- and triorganotin compounds [17, 19, 20–30], but abnormally long values have been reported for some pyrazine adducts of Ph_2SnCl_2 [31] and also for Me_2SnBr_2·AZP [32], a seven-coordinate compound (AZP = 2,2′-azopyridine). In all cases reported the Sn–N bond lengths exceed the sum of the atomic radii of Sn and N (2.10 Å), and this has been interpreted as indicating a low degree of covalent character in the

TABLE 1. Crystallographic data

	Chloro adduct	Bromo adduct	Iodo adduct
Crystal data			
Empirical formula	$C_{52}H_{50}N_2Cl_2Sn_2$	$C_{52}H_{50}Br_2N_2Sn_2$	$C_{52}H_{50}N_2I_2Sn_2$
Crystal size (mm)	0.36×0.60×0.20	0.26×0.28×0.12	0.20×0.40×0.50
Crystal system	monoclinic	monoclinic	monoclinic
Space group	$P2_1/c$	$P2_1/c$	$P2_1/c$
Unit cell dimensions			
a (Å)	13.362(2)	13.103(3)	12.867(3)
b (Å)	11.382(2)	11.351(2)	11.389(4)
c (Å)	17.029(2)	17.607(4)	18.496(4)
β (°)	110.050(11)	110.050(16)	110.050(11)
Volume (Å^3)	2481.8(6)	2460.0(9)	2547.0(12)
Z	2	2	2
Formula weight	1011.2	1100.2	1194.2
Density (calc.) (Mg/m^3)	1.391	1.485	1.557
Absorption coefficient (mm^{-1})	1.183	2.661	2.216
F(000)	1020	1092	1164
Data collection			
Scan speed	variable; 3.00 to 15.00°/min. in ω	variable; 3.00 to 15.00°/min. in ω	variable; 3.00 to 20.00°/min. in ω
Index Ranges	$-4 \leqslant h \leqslant 14$	$-4 \leqslant h \leqslant 14$	$0 \leqslant h \leqslant 13$
	$-2 \leqslant k \leqslant 12$	$-4 \leqslant k \leqslant 12$	$0 \leqslant k \leqslant 12$
	$-18 \leqslant l \leqslant 17$	$-18 \leqslant l \leqslant 17$	$-19 \leqslant l \leqslant 18$
Reflections collected	3512	3583	3746
Independent reflections	2935 ($R_{int}=0.95\%$)	3009 ($R_{int}=0.95\%$)	3354 ($R_{int}=3.95\%$)
Observed reflections	2766 ($F>3.0\sigma(F)$)	2472 ($F>3.0\sigma(F)$)	2527 ($F>3.0\sigma(F)$)
Absorption correction	Semi-empirical	Semi-empirical	Semi-empirical
Min./max. transmission	0.6264/0.9799	0.6132/0.8893	0.1485/0.2685
Solution and refinement			
Absolute configuration	N/A	N/A	N/A
Extinction correction	$c=0.00086(14)$	$c=0.00029(3)$	$c=0.00001(5)$
		where $F^*=F[1+0.002cF^2/\sin(2\theta)]^{-1/4}$	
Hydrogen atoms	riding model, fixed isotropic U	riding model, fixed isotropic U	riding model, fixed isotropic U
Weighting scheme, w^{-1}	$\sigma^2(F)+0.0025F^2$	$\sigma^2(F)+0.0001F^2$	$\sigma^2(F)+0.0027F^2$
Parameters refined	272	272	272
Final R indices (obs. data) (%)			
R	3.10	3.75	4.26
R_w	5.42	3.50	5.82
R indices (all data) (%)			
R	3.30	4.91	5.93
R_w	5.66	3.61	6.69
Goodness-of-fit	1.00	1.46	0.94
Largest and mean Δ/σ	0.097, 0.024	1.119, 0.003	0.328, 0.006
Data-to-parameter ratio	10.5:1	9.4:1	9.6:1
Largest difference peak (e Å^{-3})	0.52	0.33	0.65
Largest difference hole (e Å^{-3})	−0.31	−0.40	−0.81

Sn–N bond [33]. However, in organotin adducts containing N-heterocycles, differences in Sn–N bond lengths may be related to the steric requirements and donor abilities of the heterocyclic ligand as estimated from their basicities. Allowance should also be made for the group *trans* to the heterocyclic ligand in five-coordinate complexes, and in the case of octahedral complexes, for the presence of different pairs of the *trans* ligand.

The Sn–Cl, Sn–Br and Sn–I bond lengths are 2.452(2), 2.619(1) and 2.830(1) Å, respectively. The Sn–Cl and Sn–Br bond lengths are in the ranges reported in the literature for five-coordinated organotin(IV) chloride (2.432–2.613 Å [19, 22, 28, 34–38]) and bromide (2.576–2.739 Å [28, 38–43]) complexes. The d(Sn–I) for **III** is identical to that reported for five-coordinate (2-carbomethoxy-1,4-cyclohexadien-1-yl)-dimethyltin iodide [19], but shorter than the two axial Sn–I bonds in six-coordinate $C_2H_5SnI_3\cdot$(diphenyl sulfoxide)$_2$ [25]. For **I** and **II** the shortest intermolecular distance is between C(4) and C(16) of adjacent mol-

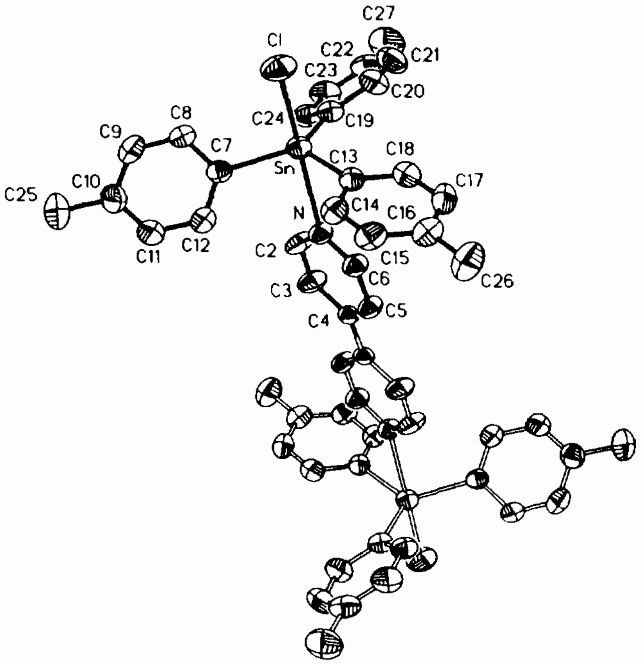

Fig. 1. Structure of $[(p\text{-tolyl})_3SnCl]_2 \cdot 4,4'\text{-bpy}$.

TABLE 2. Selected bond lengths (Å) and bond angles (°) for **I**, **II** and **III** with e.s.d.s in parentheses

	X=Cl	X=Br	X=I
Bond lengths			
Sn–X	2.452(2)	2.619(1)	2.830(1)
Sn–N	2.668(3)	2.653(5)	2.655(7)
Sn–C(7)	2.136(4)	2.128(5)	2.139(7)
Sn–C(13)	2.115(4)	2.115(5)	2.133(8)
Sn–C(19)	2.121(4)	2.117(6)	2.128(8)
Bond angles			
X–Sn–N	176.0(1)	176.6(1)	176.8(1)
N–Sn–C(7)	84.1(1)	83.4(2)	82.5(3)
N–Sn–C(13)	81.0(1)	81.9(2)	82.3(3)
N–Sn–C(19)	87.7(1)	87.5(2)	86.9(3)
X–Sn–C(7)	96.0(1)	95.4(2)	96.4(2)
X–Sn–C(13)	95.6(1)	96.2(2)	95.9(2)
X–Sn–C(19)	95.8(1)	95.9(2)	96.3(2)
C(7)–Sn–C(13)	122.4(2)	123.4(2)	124.0(3)
C(7)–Sn–C(19)	116.0(1)	116.1(2)	116.9(3)
C(13)–Sn–C(19)	118.6(1)	117.5(2)	115.6(3)

ecules. This distance is 3.534±0.005 Å for **I** and 3.519±0.005 Å for **II**. In the case of **III**, the shortest internuclear distance (3.492±0.005 Å) is between C(2) and C(11) of adjacent molecules.

Supplementary materials

Tables of hydrogen parameters, anisotropic temperature factors, observed and calculated structure factors, complete lists of bond lengths and bond angles, and atomic coordinates for **II** and **III** are available from author S.B.

TABLE 3. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement coefficients ($Å^2 \times 10^3$) for $[(p\text{-tolyl})_3SnCl]_2 \cdot 4,4'\text{-bpy}$ with e.s.d.s in parentheses

	x	*y*	*z*	U_{eq} [a]
Sn	2934(1)	1092(1)	1505(1)	49(1)
Cl	2191(1)	−431(1)	2169(1)	83(1)
N	3875(3)	2674(3)	828(2)	49(1)
C(2)	3344(3)	3169(4)	83(3)	67(2)
C(3)	3740(3)	4067(3)	−259(3)	62(2)
C(4)	4762(3)	4501(3)	167(2)	41(1)
C(5)	5313(4)	3979(3)	913(3)	52(2)
C(6)	4841(3)	3082(3)	1218(2)	54(2)
C(7)	2622(3)	189(3)	347(2)	50(1)
C(8)	1625(3)	−319(4)	−32(3)	67(2)
C(9)	1402(5)	−945(4)	−747(4)	79(2)
C(10)	2185(5)	−1125(3)	−1134(4)	71(2)
C(11)	3152(4)	−621(4)	−750(3)	70(2)
C(12)	3378(3)	45(3)	−34(3)	64(2)
C(13)	4470(3)	1118(3)	2443(2)	48(2)
C(14)	5272(3)	352(3)	2430(3)	57(2)
C(15)	6291(4)	422(4)	3001(3)	68(2)
C(16)	6572(4)	1268(3)	3622(3)	63(2)
C(17)	5768(4)	2019(4)	3653(3)	67(2)
C(18)	4738(3)	1959(3)	3073(3)	59(2)
C(19)	1813(3)	2413(3)	1522(3)	54(2)
C(20)	1737(4)	2832(4)	2270(3)	69(2)
C(21)	1001(5)	3690(4)	2265(4)	84(3)
C(22)	293(4)	4154(4)	1504(4)	75(2)
C(23)	357(4)	3727(4)	775(3)	72(2)
C(24)	1114(3)	2867(4)	784(3)	62(2)
C(25)	1930(5)	−1847(5)	−1907(4)	104(3)
C(26)	7674(5)	1353(5)	4226(4)	101(3)
C(27)	−524(5)	5097(5)	1503(4)	105(3)

[a] Equivalent isotropic *U* defined as one third of the trace of the orthogonalized U_{ij} tensor.

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