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CRYSTAL AND MOLECULAR STRUCTURE OF 2,2′-BIPYRIDYLDICHLORODI(*p*-TOLYL)TIN(IV)

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Summary

2,2′-Bipyridyldichlorodi(*p*-tolyl)tin(IV), $C_{24}H_{22}N_2Cl_2Sn$, crystallizes in the monoclinic space group $P2_1/c$ with a 9.488(2), b 9.895(4), c 18.884(5) Å, β 101.71(2)°, V 2290(1) Å^3, and $Z = 4$. The structure was determined by direct methods and refined to a final R value of 0.031 for 3736 observed Mo-K_α reflections. The tin atom is in a distorted octahedral environment, with the *cis*-tolyl rings (angle C–Sn–C 108.7(1)°) making a dihedral angle of 76.2(8)° to minimize steric repulsion, and the chlorine atoms in an approximately *trans* disposition (angle Cl–Sn–Cl 161.4(1)°); the chelating bipyridine ligand is forced into slight non-planarity, resulting in inequivalence of the Sn–N bonds (Sn–N(1) 2.306(3); Sn–N(2) 2.374(3) Å).

Introduction

Diorganotin(IV) dihalides and bis-pseudohalides are known to form stable octahedral complexes with several monodentate and bidentate nitrogen bases. A number of such complexes involving the α-diimine ligands, 2,2′-bipyridine (bipy), 1,10-phenanthroline (phen) and 3-(2-pyridyl)-5,6-diphenyl-1,2,4-triazine (pdt) have recently been shown to exhibit reproducible therapeutic activity in vivo towards P-388 lymphocytic leukaemia in mice [1–3]. There has also been an attempt [3b] to relate the observed anti-tumour activity of these complexes to some intrinsic molecular dimensions, such as the bond distances and angles linking tin to halogen and ligand residues. However, X-ray diffraction studies, on which such information is based, were conducted only on a few such compounds; complete or partial structural determinations were reported in the literature for the following: Et_2SnCl_2L

(L = bipy [4], phen [3b], pdt [5]), n-Bu_2SnCl_2L (L = bipy [3b], phen [3a]), Ph_2SnX_2bipy (X = Cl [6], NCS [7]). Of these, all but the diisothiocyanate adduct possess *trans*- or distorted *trans*-[SnR_2] structures. Indeed, the *trans*-geometry, which is commonly indicated by infrared (and Raman) and tin-119m Mössbauer evidence, appears to be general for dialkyltin(IV) dihalide and bis-pseudohalide complexes [8,9], while for the corresponding diaryltin(IV) diisothiocyanate complexes only *cis*-[SnR_2] geometries have so far been encountered [10]. This change in preferred geometry from *trans* to *cis* for the diaryltin(IV) systems consequent upon the replacement of halide by the NCS group has been attributed to steric effects [11]. Mixed alkylaryl complexes such as n-BuPhSn$(NCS)_2L$ (L = bipy, phen), however, appear to retain the *trans*-[SnR_2] skeletal geometry [12].

The literature additionally contains a few isolated examples of spectroscopically characterized *cis*-[SnR_2] structures, such as $(CH_2)_5SnBr_2L$ (L = bipy, phen) [13] and $(Ph)\overline{C{=}C(Ph)\text{-}C(Ph){=}C(Ph)Sn}X_2$bipy (X = Br, I) [14] and also, but with less convincing evidence, $Ph_2SnCl_2(5\text{-}NO_2\text{-phen})$ [11] and the bis(heteroaryl)tin(IV) dihalide adducts $(3\text{-}C_4H_3O)_2SnX_2$bipy (X = Cl, Br) and $(3\text{-}C_4H_3S)_2SnCl_2$bipy [15]. To this list, we now add our title complex, 2,2′-bipyridyldichlorodi(*p*-tolyl)tin(IV), and describe its crystal and molecular structure. As far as we are aware this complex constitutes the first crystallographically authenticated example of a *cis*-[SnR_2] octahedral structure observed for a diaryltin(IV) dihalide chelate complex.

Experimental

Preparation of complex

Equimolar quantities (2.7 mmol) of (*p*-tolyl)$_2SnCl_2$ (prepared by comproportionation of (*p*-tolyl)$_4$Sn with $SnCl_4$ by the Kocheshkov method [16]) and 2,2′-bipyridine were separately dissolved in ethanol and the solutions mixed to give a precipitate of the complex in near-quantitative yield (1.36 g). Recrystallization from methanol afforded colorless crystals, m.p. 220°C (with decomposition), suitable for X-ray crystallography. Microanalyses were carried out by the Australian Microanalytical Service, CSIRO, Melbourne. (Found: C, 53.81; H, 4.00; N, 5.01. $C_{24}H_{22}N_2Cl_2Sn$ calcd.: C, 54.59; H, 4.17; N, 5.31%).

Mössbauer spectra

The tin-119m Mössbauer spectra of the title complex, the analogous *p*-chloro- and *p*-thiomethyl-substituted derivatives and the corresponding diisothiocyanate adduct (Table 1) were recorded at 80 K using a constant acceleration microprocessor spectrometer (Cryophysics model) with a 512-channel data store, and a 15 mCi $Ca^{119m}SnO_3$ source which was kept at room temperature. The spectra, recorded on a Hewlett–Packard 7225B plotter, were fitted as Lorentzian doublets by a least-squares fitting programme [17].

X-ray analysis

A selected crystal was mounted on a Nicolet R3m automatic four-circle diffractometer. Standard centering and auto-indexing procedures [18] indicated a primitive monoclinic cell. The orientation matrix and accurate cell dimensions were determined from a least-squares fit of 19 reflections. Intensity data were collected at 295 K; the details are summarized in Table 2. Two standard reflections were used to

TABLE 1

TIN-119m MÖSSBAUER DATA[a] FOR 2,2′-BIPYRIDYLDICHLORODI(*p*-TOLYL)TIN(IV) AND RELATED COMPLEXES AT 80 K

Complex	*IS*[b]	*QS*	Γ_1	Γ_2	Ref.
Ph_2SnCl_2bipy	1.25	3.52	[c]	[c]	29
$Ph_2Sn(NCS)_2bipy$	0.80	2.26	[c]	[c]	10
$(p\text{-}MeC_6H_4)_2SnCl_2bipy$	0.91	2.25	0.91	0.92	[d]
$(p\text{-}MeC_6H_4)_2Sn(NCS)_2bipy$	0.70	2.42	0.93	0.91	[e]
$(p\text{-}ClC_6H_4)_2SnCl_2bipy$	1.20	3.53	1.10	1.12	[e]
$(p\text{-}MeSC_6H_4)_2SnCl_2bipy$	1.08	3.57	0.99	0.98	[e]
$(3\text{-}C_4H_3O)_2SnCl_2bipy$	0.76	2.02	[c]	[c]	15
$(3\text{-}C_4H_3O)_2SnBr_2bipy$	0.83	2.13	[c]	[c]	15
$(3\text{-}C_4H_3S)_2SnCl_2bipy$	0.86	2.00	[c]	[c]	15

[a] Error ±0.03 mm s^{-1} for unpublished data cited, including those for the title complex. [b] Relative to $CaSnO_3$ or $BaSnO_3$. [c] Not reported. [d] This work. [e] V.G. Kumar Das, C.K. Yap and P.J. Smith, unpublished results.

monitor every 125 reflections. Empirical absorption corrections were applied by a pseudo-ellipsoidal treatment of the intensities of selected strong reflections measured at different azimuthal (ψ) angles [19–21]. Redundant and equivalent reflections were averaged and converted into unscaled $|F_0|$ values after corrections for Lorentz

TABLE 2

DATA COLLECTION AND PROCESSING PARAMETERS

Molecular formula	$(p\text{-tolyl})_2SnCl_2bipy$, $C_{24}H_{22}N_2Cl_2Sn$
Molecular weight	528.05
Cell constants	*a* 9.488(2), *b* 13.056(4), *c* 18.884(5) Å, β 101.71(2)°, *V* 2290(1) Å^3, $Z = 4$
Density (calcd)	1.532 g cm^{-3}
Space group	$P2_1/c$
Radiation	graphite-monochromatized Mo-K_α, λ 0.71069 Å
Absorption coefficient	13.66 cm^{-1}
Crystal size	0.24×0.20×0.10 mm
Mean μr	0.08
Transmission factors	0.769–0.856
Scan type and speed	ω-2θ; 2.02–8.37 deg min^{-1}
Scan range	1° below $K_{\alpha1}$ to 1° above $K_{\alpha2}$
Background counting	stationary counts for one-half of scan time at each end of scan
Collection range	h, k, $\pm l$; $2\theta_{max}$ 52°
Unique data measured	4517
Observed data with $\lvert F_0\rvert > 3\sigma(\lvert F_0\rvert)$, n	3736
Number of variables, p	268
$R_F = \Sigma\lVert F_0\rvert - \lvert F_c\rVert/\Sigma\lvert F_0\rvert$	0.031
Weighting scheme	$w = [\sigma^2(F_0)+0.0010\lvert F_0\rvert^2]^{-1}$
$R_G = [\Sigma w(\lvert F_0\rvert - \lvert F_c\rvert)^2/\Sigma w\lvert F_0\rvert^2]^{\frac{1}{2}}$	0.041
$S = [\Sigma w(\lvert F_0\rvert - \lvert F_c\rvert)^2/(n-p)]^{\frac{1}{2}}$	0.913
Residual extrema in final difference map	+0.44 to −0.38 eÅ^{-3} in the neighbourhood of the Sn atom

TABLE 3

FRACTIONAL ATOMIC COORDINATES ($\times 10^5$ for Sn and Cl; $\times 10^4$ for other atoms)

Atom	*x*	*y*	*z*
Sn	35254(2)	9190(2)	17839(1)
Cl(1)	42585(10)	−4787(7)	10345(5)
Cl(2)	21721(10)	19085(8)	25688(5)
N(1)	2711(3)	−456(2)	2354(2)
N(2)	1118(3)	503(3)	1231(2)
C(1)	3579(4)	923(3)	2909(2)
C(2)	3125(4)	−1730(3)	3281(2)
C(3)	1743(5)	−2074(4)	3069(2)
C(4)	846(5)	−1598(3)	2491(2)
C(5)	1352(4)	−792(3)	2142(2)
C(6)	453(4)	−241(3)	1528(2)
C(7)	−1002(4)	−442(4)	1275(2)
C(8)	−1776(4)	138(4)	723(2)
C(9)	−1090(4)	897(4)	423(2)
C(10)	335(4)	1053(3)	684(2)
C(11)	3473(4)	2104(3)	981(2)
C(12)	3993(4)	1932(3)	353(2)
C(13)	3984(5)	2698(4)	156(2)
C(14)	3483(4)	3668(3)	−58(2)
C(15)	2996(5)	3856(3)	573(2)
C(16)	2975(4)	3087(3)	1086(2)
C(17)	3472(6)	4504(4)	−609(3)
C(18)	5543(4)	1019(3)	2554(2)
C(19)	5753(4)	1786(3)	3076(2)
C(20)	7063(4)	1898(3)	3553(2)
C(21)	8222(4)	1284(4)	3515(2)
C(22)	8031(4)	527(4)	2984(2)
C(23)	6722(4)	384(3)	2519(2)
C(24)	9642(5)	1409(6)	4031(3)

and polarization effects. Direct phase determination revealed the position of the Sn atom; the remaining non-hydrogen atoms were located from subsequent difference-Fourier maps. All non-hydrogen atoms were refined anisotropically. The 16 ring hydrogen atoms were placed in positions calculated on the basis of sp^2-hybridization (d(C–H) 0.96 Å) of their respective parent C atoms and were assigned a common isotropic temperature factor of 0.06 Å^2. Difference-Fourier maps yielded approximate orientations of the methyl groups, which were refined as rigid groups with d(C–H) 0.98 Å and a common temperature factor. All computations were performed on a Data General Corporation Nova 3/12 minicomputer with the SHELXTL program package [19]. Analytical expressions [22] of neutral atom scattering factors [23] were employed, and anomalous dispersion corrections [24] were applied. Blocked-cascade least-squares refinement [19,25] of 268 parameters converged to the R indices listed in Table 2. Positional and thermal parameters of the non-hydrogen atoms are given in Table 3, bond distances and angles in Table 4, and least-squares planes in Table 5; tables of hydrogen coordinates, temperature factors, structure factors, and anisotropic thermal parameters are available (from TCWM) on request.

TABLE 4

MOLECULAR DIMENSIONS OF 2,2'-BIPYRIDYLDICHLORODI(*p*-TOLYL)TIN(IV), WITH ESTIMATED STANDARD DEVIATIONS IN PARENTHESES

Bond distances (Å)			
Sn–Cl(1)	2.493(1)	Sn–Cl(2)	2.507(1)
Sn–N(1)	2.306(3)	Sn–N(2)	2.374(3)
Sn–C(11)	2.159(3)	Sn–C(18)	2.161(3)
N(1)–C(1)	1.341(4)	N(2)–C(10)	1.343(5)
C(1)–C(2)	1.382(6)	C(9)–C(10)	1.375(6)
C(2)–C(3)	1.366(6)	C(8)–C(9)	1.370(7)
C(3)–C(4)	1.387(6)	C(7)–C(8)	1.374(6)
C(4)–C(5)	1.379(6)	C(6)–C(7)	1.391(5)
N(1)–C(5)	1.344(4)	N(2)–C(6)	1.342(5)
C(5)–C(6)	1.479(5)		
C(11)–C(12)	1.394(6)	C(18)–C(19)	1.390(5)
C(12)–C(13)	1.386(6)	C(19)–C(20)	1.387(5)
C(13)–C(14)	1.378(6)	C(20)–C(21)	1.374(6)
C(14)–C(15)	1.384(6)	C(21)–C(22)	1.392(7)
C(15)–C(16)	1.399(6)	C(22)–C(23)	1.381(5)
C(16)–C(11)	1.395(5)	C(23)–C(18)	1.405(5)
C(14)–C(17)	1.507(7)	C(21)–C(24)	1.503(6)
Bond angles (deg)			
Cl(1)–Sn–Cl(2)	161.4(1)	N(1)–Sn–N(2)	69.1(1)
Cl(1)–Sn–N(1)	81.6(1)	Cl(2)–Sn–N(1)	82.2(1)
Cl(1)–Sn–N(2)	86.4(1)	Cl(2)–Sn–N(2)	79.4(1)
Cl(1)–Sn–C(11)	95.5(1)	Cl(2)–Sn–C(11)	96.0(1)
Cl(1)–Sn–C(18)	96.6(1)	Cl(2)–Sn–C(18)	93.5(1)
N(1)–Sn–C(18)	93.9(1)	N(2)–Sn–C(11)	88.4(1)
N(1)–Sn–C(11)	157.4(1)	N(2)–Sn–C(18)	162.2(1)
C(11)–Sn–C(18)	108.7(1)		
Sn–N(1)–C(1)	120.5(2)	Sn–N(2)–C(10)	122.4(3)
Sn–N(1)–C(5)	120.5(2)	Sn–N(2)–C(6)	118.3(2)
N(1)–C(5)–C(6)	116.1(3)	N(2)–C(6)–C(5)	115.8(3)
N(1)–C(1)–C(2)	122.5(3)	N(2)–C(10)–C(9)	122.6(4)
C(1)–C(2)–C(3)	118.8(4)	C(10)–C(9)–C(8)	118.8(4)
C(2)–C(3)–C(4)	118.9(4)	C(9)–C(8)–C(7)	119.1(4)
C(3)–C(4)–C(5)	119.9(4)	C(8)–C(7)–C(6)	119.9(4)
C(4)–C(5)–N(1)	120.9(3)	C(7)–C(6)–N(2)	120.7(3)
C(5)–N(1)–C(1)	119.0(3)	C(6)–N(2)–C(10)	118.9(3)
C(4)–C(5)–C(6)	123.0(3)	C(7)–C(6)–C(5)	123.5(4)
Sn–C(11)–C(12)	121.4(3)	Sn–C(18)–C(19)	120.1(2)
Sn–C(11)–C(16)	121.3(3)	Sn–C(18)–C(23)	122.5(3)
C(16)–C(11)–C(12)	117.2(3)	C(23)–C(18)–C(19)	117.2(3)
C(11)–C(12)–C(13)	121.4(4)	C(18)–C(19)–C(20)	121.2(4)
C(12)–C(13)–C(14)	121.7(4)	C(19)–C(20)–C(21)	121.6(4)
C(13)–C(14)–C(15)	117.5(4)	C(20)–C(21)–C(22)	117.6(3)
C(14)–C(15)–C(16)	121.5(4)	C(21)–C(22)–C(23)	121.5(4)
C(15)–C(16)–C(11)	120.7(4)	C(22)–C(23)–C(18)	120.8(4)
C(13)–C(14)–C(17)	121.8(4)	C(20)–C(21)–C(24)	121.6(4)
C(15)–C(14)–C(17)	120.6(4)	C(22)–C(21)–C(24)	120.8(4)

Results and discussion

The Mössbauer spectrum of the title complex reveals isomer shift (*IS*) and quadrupole splitting (*QS*) values of 0.91 and 2.25 mm s^{-1} respectively (Table 1),

TABLE 5

LEAST-SQUARES PLANES [a]

Plane	Atoms fitted	l	m	n	d	rms Δ (Å × 10^{4})
1	N(1), C(1) to C(5)	−0.2974	0.6471	0.7070	1.6395	28
2	N(2), C(6) to C(10)	−0.2238	0.6542	0.7224	1.7175	55
3	C(11) to C(17)	0.9195	0.2727	0.2830	4.0622	78
4	C(18) to C(24)	0.2998	0.6373	−0.7141	−0.3335	95

Angles between planes (standard deviation ~ 0.8°): 1–2, 4.4°; 1–3, 84.2°; 1–4, 100.5°; 2–3, 79.8°; 2–4, 99.4°; 3–4, 76.2°

[a] Plane equation is of the form $lx_0 + my_0 + nz_0 = d$, where x_0, y_0, z_0 are orthogonal coordinates in Å referred to axes a_0, b_0, c_0 respectively, with a_0 parallel to a, b_0 to b, and c_0 to c.

which are rather similar to those observed for the corresponding diisothiocyanate adduct and also those reported for bis(3-thienyl)- and bis(3-furyl)tin(IV) dihalide complexes [15], and for $Ph_2Sn(NCS)_2bipy$ which has a crystallographically authenticated *cis*-[SnR_2] structure [7]. A *cis*-geometry is thus inferred for the title complex as well as for the diisothiocyanate analogue, which is in accord with the point-charge model [26–28], which specifies for *trans*-[SnR_2] configurations *QS* values of ca. 4 mm s^{-1} and for *cis*-geometries *QS* values only half as large. On the other hand, Ph_2SnCl_2bipy, which has a distorted *trans*-[SnR_2] structure [6], shows a *QS* value of 3.51 mm s^{-1} [29]. The C–Sn–C angle in the title complex can be calculated from a point-charge approximation [30] which assumes that the partial *QS* contributions from the ligands are small compared to that from the organic groups, and the correlation is supported by data from compounds for which both Mössbauer and diaryltin(IV) C–Sn–C angles are available [30,31]. On this basis, an angle of 111° is predicted for the angle in the title complex. This is closer to the idealized octahedral *cis*-angle of 90° than the *trans*-angle of 180°. An alternative possibility is, of course, a trigonal bipyramidal geometry with basal R groups, which might be envisaged if bipy were unidentate. However, this seems unlikely upon comparison with the following data from the recent literature on the discrete five-coordinated adduct complexes: Ph_2SnCl_2(benzthiazole) [32] (angle C–Sn–C (X-ray) 132.5°; *IS* 1.36, *QS* 2.92 mm s^{-1}) and Ph_2SnCl_2(2,6-lutidine *N*-oxide) [33] (angle C–Sn–C (X-ray) 124.1°; *IS* 1.01, *QS* 2.94 mm s^{-1}). Nevertheless, it was rather surprising that Mössbauer evidence favoured a *cis*-octahedral alignment for the title complex when for the unsubstituted complex, Ph_2SnCl_2bipy, as well as for the *p*-chloro and *p*-thiomethyl substituted analogues (Table 1), *trans*-[SnR_2] geometries seem to be indicated. Mössbauer data also seem to support a *trans*-[SnR_2] geometry for (*p*-tolyl)$_2SnCl_2$phen (*IS* 1.24, *QS* 3.58 mm s^{-1}). A crystal structure verification was therefore sought for the title complex, and the molecular geometry and labelling scheme are shown in Fig. 1. A stereoview of the molecular packing in the unit cell is given in Fig. 2.

As shown in Fig. 1, di(*p*-tolyl)tin(IV) dichloride forms a discrete hexacoordinated complex with the bipyridine ligand in a chelating mode. The complex is, however, distorted from idealized octahedral geometry, as revealed by the Cl(1)–Sn–Cl(2) and C(11)–Sn–C(18) angles (161.4(1) and 108.7(1)°, respectively). The chlorine atoms

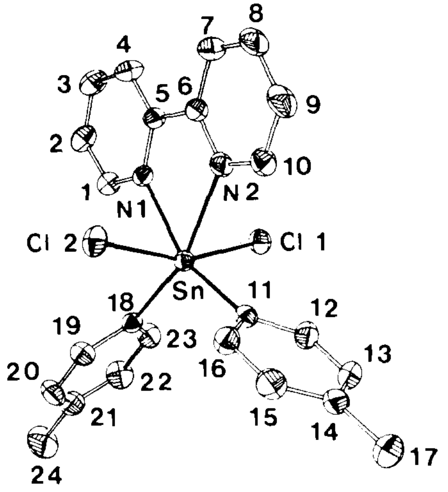

Fig. 1. Perspective view of the 2,2'-bipyridyldichlorodi(p-tolyl)tin(IV) molecule with atom labelling. The thermal ellipsoids are drawn at the 35% probability level.

subtend the largest angle at tin and, with respect to the tolyl groups and the bipyridyl residue, are considered to occupy the *trans* "axial" positions of the distorted octahedron. The sum of the "equatorial" angles is 360.1°. The data agree well with those reported for $Ph_2Sn(NCS)_2$bipy, but some differences (see Table 6) are evident. Thus, while the bipy ligand is essentially planar in the diisothiocyanate

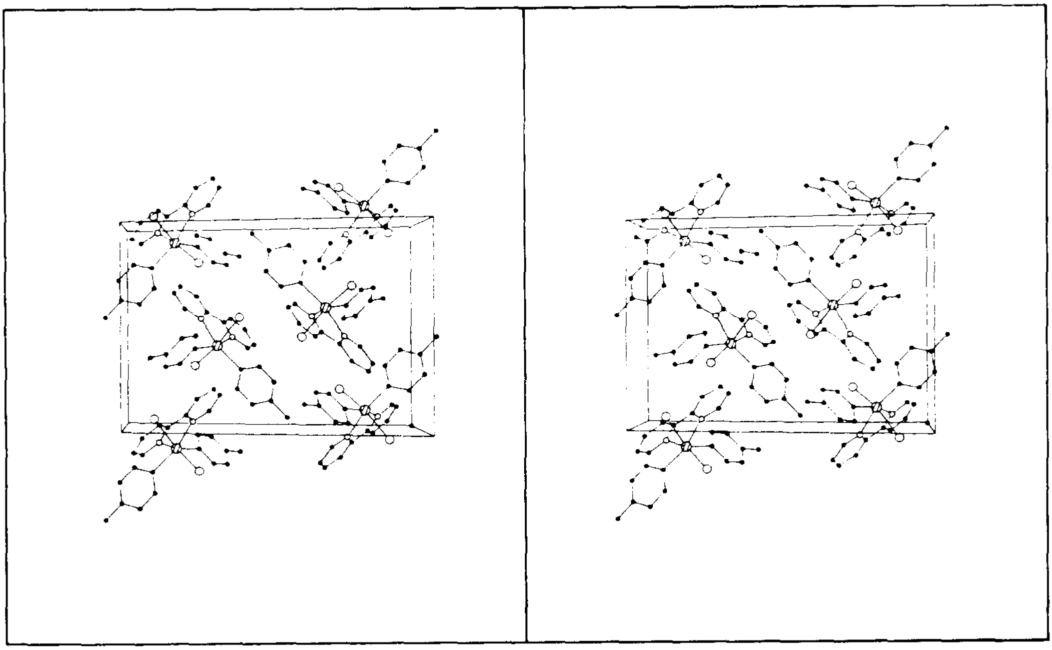

Fig. 2. Stereo-drawing showing the molecular packing. The origin of the unit cell lies at the upper left corner, with a pointing towards the reader, b downwards, and c from left to right.

TABLE 6

COMPARISON OF SELECTED MOLECULAR DIMENSIONS OF 2,2'-BIPYRIDYL-DICHLORODI(*p*-TOLYL)TIN(IV) WITH THOSE OF RELATED COMPLEXES

	$(p\text{-Tolyl})_2SnCl_2bipy$ ^{a}	$Ph_2Sn(NCS)_2bipy$ ^{b}	Ph_2SnCl_2bipy ^{c}
Bond distances (Å)			
Sn–C	2.159(3), 2.161(3)	2.161, 2.183	2.151, 2.153
Sn–N	2.306(3), 2.374(3)	2.329, 2.338	2.344, 2.378
Sn–Cl	2.493(1), 2.507(1)		2.511, 2.508
Bond angles (°)			
Cl–Sn–Cl	161.4(1)		103.5
SCN–Sn–NCS		161.1	
N–Sn–N	69.1(1)	69.7	69.0
C–Sn–C	108.7(1)	106.3	173.5
Dihedral angle between planes (°)			
C_5H_4N rings of bipy ligand	4.4(8)	1.7	4.2
Aryl groups	76.2(8)	ca. 90	79.5

^{a} This work. ^{b} Ref. 7. ^{c} Ref. 6.

complex, in both the title complex and Ph_2SnCl_2bipy, the C_5H_4N rings of the ligand are twisted by about 4°. However, in the title complex the slight inequivalence of the two Sn–N bonds is more conspicuous. This can be attributed to the co-accomodation of the bulky tolyl groups in the "equatorial" plane of the octahedron. The "bite" angle of the ligand is lowered by 0.6° relative to the diisothiocyanate case, but there are no discernible dimensional changes in the rest of the ligand. The *cis*-occupancy of the two tolyl groups in the octahedral structure also leads to an increased C–Sn–C angle opening (relative to the angle between phenyl groups in the diisothiocyanate complex) as well as to an incipient twist of the tolyl rings. Thus the dihedral angle between the planes of the tolyl rings is 76.2(8)°, which is closer to the conformation adopted in *trans*-Ph_2SnCl_2bipy than in $Ph_2Sn(NCS)_2$-bipy. An examination of the unit cell (Fig. 2) indicates that one of the tolyl rings (C(18) to C(24)) is positioned virtually parallel (dihedral angle 0.6°) and in overlapping proximity to the C_5H_4N ring (N(1), C(1) to C(5)) of a neighboring chelated bipyridine ligand (symmetry transformation: $1-x$, $0.5+y$, $0.5-z$). The interplanar distance is 3.61(1) Å, which is outside the expected contact range (3.23–3.38 Å) for any significant π-interaction [34], so that our description of the crystal structure as discrete molecular units held together by Van der Waals forces appears secure. The Sn–C and Sn–Cl bond distances (Table 4) are within the range of values commonly encountered for six-coordinated diorganotin(IV) dichloride complexes.

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