

Journal of Organometallic Chemistry 515 (1996) 119-130

Tin(IV) and organotin(IV) complexes containing mono or bidentate N-donor ligands II. ¹ 4-Phenylimidazole derivatives. Crystal and molecular structure of [bis(4-phenylimidazole) trimethyltin(IV)] chloride

Claudio Pettinari ^{a, *}, Fabio Marchetti ^a, Maura Pellei ^a, Augusto Cingolani ^a, Luisa Barba ^b, Alberto Cassetta ^b

^a Dipartimento di Scienze Chimiche, Università degli Studi, via S. Agostino 1, 62032 Camerino, Macerata, Italy ^b Istituto di Strutturistica Chimica ''G. Giacomello'', Consiglio Nazionale delle Ricerche, Padriciano 99, 30412 Trieste, Italy

Received 2 October 1995

Abstract

A series of 2:1 adducts of the type $[(L')_2R_nSN_{4-n}] \cdot zH_2O(L' = 4$ -phenylimidazole, R = Me, Et, ⁿBu, Ph or Cy, X = I, Br or Cl, n = 0, 1, 2 or 3, z = 0 or 1) has been characterized in the solid state and in solution by analyses, spectral (IR, far-IR, ¹H and ¹³C) data and conductivity measurements. The derivatives $[(L')_2(Me)_3Sn]Cl(1)$ and $[(L')_2(Me)_2SnCl_2]$ (3) react with NaClO₄ in THF giving the ionic complexes $[(L')_2(Me)_3Sn]ClO_4$ and $(L')_3[(Me)_2Sn(ClO_4)_2] \cdot (H_2O)_4$ respectively. Whereas the triorganotin(IV) derivatives are completely dissociated in acetone solution, the diorganotin(IV) derivatives dissociate only partly and the tri- and tetrahalidetin(IV) complexes probably retain the hexacoordinate configuration. The crystal structure of $[(L')_2(Me)_3Sn]Cl(1)$ has been determined by X-ray analysis. The tin atom is coordinated to three methyl groups and two 4-phenylimidazole donors in a substantially regular trigonal bipyramidal geometry. The ionic chloride group and the two N-H moieties are involved in a hydrogen bond network.

Keywords: Sn; X-ray structure; Trimethyltin chloride; Imidazole adducts; H-bonding

1. Introduction

Since the first report on the antitumor activity of diorganotin compounds appeared in 1980 [1], there has been increased interest in the synthesis of tin-based antitumor drugs [2,3]. It seems that the coordinated organic ligand facilitates the transport of the complex across cell membranes, while the antitumor activity is exerted by the dissociated diorganotin(IV) moieties. The action mechanism of these derivatives requires a complete understanding of their structure. The correlation

between X-ray crystallographic data and antitumor activity may facilitate the design and synthesis of even more active complexes [4–7]. The study of complexes with monodentate nitrogen donor ligands, such as imi-



Fig. 1. 4-phenylimidazole.

* Corresponding author.

For Part I See Ref. [10].

dazole which, as a part of histidine, plays an important role in many biological processes, could be very useful in increasing the understanding of interactions of these derivatives with DNA, where N-atoms of nucleic acid bases could be involved [8,9].

As an extension of our research on tin(IV) and organotin(IV) complexes of imidazoles [10], we now describe the synthesis and spectroscopic characterization of new adducts between $R_n SnX_{4-n}$ (R = Me, Et, ⁿBu, Ph or Cy, n = 0, 1, 2 or 3, X = Cl, Br, I or ClO_4) acceptors and the 4-phenylimidazole donor (L') (Fig. 1). We also report the X-ray crystal structure of one of

Table 1

Physical, analytical and conductivity data for derivatives 1–2
--

these complexes, which is the first structurally characterized ionic triorganotin(IV) derivative containing two N-donor monodentate ligands.

2. Results and discussion

The reaction between 4-phenylimidazole (L') (Fig. 1) and several $R_n SnX_{4-n}$ acceptors (R = Me, X = Cl, n = 0, 1, 2 or 3; R = Me, Et or ⁿBu, X = Br, n = 2; R = Et, n = 2, X = Cl; $R = {}^{n}Bu$, X = Cl, n = 1 or 2;

Compound	No.	Yield	M.p. (°C)	Elemental analysis (%); Found (Calc.)			Conductivities ^b		
and formula ^a		(%)		C	Н	N	Solvent	Conc.	$\overline{\Lambda}$
[(L') ₂ (CH ₃) ₃ Sn]Cl	1	51	198-202	51.6	5.4	11.1	Acetone	2.8	3.6
$C_{21}H_{25}ClN_4Sn$				(51.7)	(5.2)	(11.5)			
$[(L')_2(C_6H_5)_3Sn]Cl$	2	40	131-135	63.6	4.9	8.0	Acetone	2.3	1.3
$C_{36}H_{31}CIN_4Sn$				(64.2)	(4.6)	(8.3)			
$[(L')_2(CH_3)_2SnCl_2]$	3	99	212-214	47.2	4.6	11.1	Acetone	5.3	4.1
$C_{20}H_{22}Cl_2N_4Sn$				(47.2)	(4.4)	(11.0)			
$[(L')_2(CH_3)_2SnBr_2]$	4	97	207-210	40.4	3.8	9.0	Acetone	2.2	17.3
$C_{20}H_{22}Br_2N_4Sn$				(40.2)	(3.7)	(9.4)			
$[(L')_2(CH_3)_2SnI_2]$	5	25	166–170	35.0	3.3	8.3	Acetone	2.5	84.0
$C_{20}H_{22}I_{2}N_{4}Sn$				(34.8)	(3.2)	(8.1)			
$[(L')_2(C_2H_5)_2SnCl_2]$	6	99	197-201	49.5	5.1	10.0	Acetone	2.5	6.0
$C_{22}H_{26}Cl_2N_4Sn$				(49.3)	(4.9)	(10.4)			
$[(L')_2(C_2H_5)SnBr_2]$	7	92	185-190	42.2	4.5	9.0	Acetone	2.6	12.7
$C_{22}H_{26}Br_2N_4Sn$				(42.3)	(4.2)	(9.0)			
$[(L')_2(C_2H_5)_2SnI_2]$	8	56	135-138	36.9	4.1	8.0	Acetone	1.6	63.1
$C_{22}H_{26}I_2N_4Sn$				(36.7)	(3.6)	(7.8)			
$[(L')_2(C_4H_9)_2SnCl_2]$	9	92	147-148	52.8	6.1	9.1	Acetone	2.2	6.4
$C_{26}H_{34}Cl_2N_4Sn$				(52.7)	(5.7)	(9.5)			
$[(L')_{2}(C_{4}H_{9})_{2}SnBr_{2}]$	10	99	150-151	46.0	5.2	8.4	Acetone	2.0	11.0
$C_{26}H_{34}Br_2N_4Sn$				(45.8)	(5.0)	(8.2)			
$[(L')_2(C_6H_5)_2SnCl_2]$	11	99	172-174	55.7	4.1	8.4	Acetone	1.9	33.7
$C_{30}H_{26}Cl_2N_4Sn$				(57.0)	(4.1)	(8.9)			
$[(L')_2(C_6H_5)_2SnBr_2]$	12	34	170–176	50.0	3.9	8.1	Acetone	1.8	38.9
$C_{30}H_{26}Br_2N_4Sn$				(50.0)	(3.6)	(7.8)			
$[(L')_2(cyclo-C_6H_{11})_2SnBr_2]$	13	93	157-159	49.6	5.7	7.0	Acetone	1.7	3.5
$C_{30}H_{38}Br_2N_4Sn$				(49.1)	(5.2)	(7.6)			
$[(L')_2(cyclo-C_6H_{11})_2SnI_2]$	14	33	111-115	44.4	5.1	6.7	Acetone	1.4	35.7
$C_{30}H_{38}I_2N_4Sn$				(43.6)	(4.6)	(6.8)			
$[(L')_2 CH_3 SnCl_3]$	15	53	220 dec.	44.1	4.3	10.8	Acetone	2.8	45.0
$C_{19}H_{19}Cl_3N_4Sn$				(43.2)	(3.6)	(10.6)			
$[(L')_3(C_4H_9SnCl_3)_2]$	16	50	117-121	42.4	4.6	8.8	Acetone	2.9	166.5
$C_{35}H_{42}Cl_6N_6Sn_2$				(42.2)	(4.2)	(8.4)			
$[(L')_3(C_6H_5SnCl_3)_2] \cdot H_2O$	17	61	290 dec.	44.2	3.5	8.1	Acetone	1.5	109.3
C ₃₉ H ₃₆ Cl ₆ N ₆ OSn				(44.4)	(3.4)	(8.0)			
$[(L')_2 SnCl_4] \cdot H_2O$	18	90	52 dec.	38.3	3.7	9.9	Acetone	2.3	41.3
$C_{18}H_{18}Cl_4N_4OSn$				(38.1)	(3.2)	(9.9)			
$[(L')_2(CH_3)_3Sn]ClO_4$	19	83	183-187	47.4	5.3	10.8	Acetone	3.2	130.6
$C_{21}H_{25}ClN_4O_4Sn$				(45.7)	(4.6)	(10.2)			
$(L')_{3}[(CH_{3})_{2}Sn(ClO_{4})_{2}]_{2} \cdot (H_{2}O)_{4}$	20	26	204-206	31.6	3.7	6.5	Acetone	1.5	375.5
$C_{31}H_{44}Cl_4N_6O_{20}Sn_2$				(31.0)	(3.7)	(7.0)			

^a L' is 4-phenylimidazole.

^b In Ω^{-1} cm² mol⁻¹ at room temperature; conc. is molar concentration (×10³).

R = Ph, n = 1, 2 or 3, X = Cl; R = Cy, X = Br, n = 2) was carried out in diethyl ether or benzene or dichloromethane solutions from which the colorless complexes 1-4, 6, 7, 9-11, 13, and 15-18 (Table 1) were isolated as insoluble or sparingly soluble precipitates, in accordance with Eq. (1).

$$x(L') + R_n SnX_{4-n} \cdot zH_2O$$

$$\xrightarrow{S} [(L')_x (R_n SnX_{4-n})] \cdot zH_2O \qquad (1)$$

The reactions are rapid at room temperature, except those involving triorganotin(IV) derivatives which require a longer reaction time. This is in accordance with the low acidity of these organotin species [11].

The $[(L')_2R_2SnCl_2]$ type compounds 3, 6, and 13 are converted into the analogous di-iodide complexes 5, 8, and 14 respectively (Table 1), and the compound $[(L')_2Ph_2SnCl_2]$ (11) into the bromide $[(L')_2Ph_2SnBr_2]$ (12) on treatment with excess sodium halide in tetrahydrofuran suspension (Eq. (2)).

$$[(L')_2 R_2 \text{SnCl}_2] + 2\text{NaX}$$

$$\xrightarrow{\text{THF}} [(L')_2 R_2 \text{SnX}_2] + 2\text{NaCl}$$
(2)

This conversion did not occur when $[(L')_2^n Bu_2 SnCl_2]$ was employed as starting material, whereas an incomplete substitution was observed when the reaction between sodium iodide and $[(L')_2 Ph_2 SnCl_2]$ was carried out under the same conditions.

The perchlorato complexes 19 and 20 were obtained when an ethanol solution of sodium perchlorate was added to a THF or ethanol solution of compounds 1 and 3 respectively.

All the complexes are generally air- and thermallystable, soluble in acetone, moderately soluble in chlorinated solvents and insoluble in diethyl ether, ethanol, methanol, and water. Upon prolonged standing in acetone solution they are found to decompose somewhat, producing the starting materials or insoluble products for which the IR spectra and analytical data suggest an oxide nature.

The electrical conductivities were measured for all the compounds listed in acetone solution, in which a typical 1:1 electrolyte such as tetra-n-butylammonium bromide has a specific conductivity of 137 Ω^{-1} cm² mol⁻¹. Some of the compounds are non-electrolites (1-4, 6, 7, 9, 10, and 13), other electrolytes (16, 17, 19, and 20) and the remaining complexes are partly ionized.

2.1. IR data

In Table 2 we report the most relevant infrared data for the ligand and its tin(IV) and organotin(IV) complexes 1-20 in the range 4000-100 cm⁻¹. The spectra

were recorded both in Nujol mull and in chloroform solution. By comparison with the data reported for other tin(IV) and organotin(IV) adducts containing N-donor ligands [12], we suggest for 1-20 the following assignments.

2.1.1. Ligand absorptions

In the 3200–3000 cm⁻¹ region, the ligand exhibits weak bands typical of C–H stretching due to a pseudoaromatic ring, and in the region 1650–1500 cm⁻¹, some more intense absorptions due to ring breathing mode [13]. These bands do not shift markedly upon coordination to tin, suggesting a weak influence of the complexation on the bands within the donor. In the 2800–2600 cm⁻¹ region, the ligand exhibits a broad band typical of N–H stretching. In the tin(IV) and organotin(IV) adducts this band shifts in the 3600–2800 cm⁻¹ region, suggesting an important influence of the coordination to tin. The position and the broadening of the N–H stretching band are consistent with the presence of a hydrogen bond between the N–H moiety and the halide groups [14].

2.1.2. Sn-C stretching frequencies

Compound 1 shows a medium band at 546 cm^{-1} due to the Sn-C stretching vibrations. The appearance of only a single band due to ν_{asym} is taken to imply a C_{3v} symmetry of the C₃-Sn skeleton and a trans-pyramidal configuration of the two ligands [15]. In the triphenvltin(IV) derivative 2, the v_{asym} and v_{sym} Sn-C are observed as medium bands at 282 and 274 cm⁻¹, respectively; these absorptions are consistent with an essentially trigonal pyramidal arrangement of phenyl groups with a marked deviation from planarity. Only a single Sn-C band appeared in the spectra of dimethyl-(3, 4), diethyl- (6-8), di-n-butyl- (9, 10), and dicyclohexyltin(IV) complexes (13, 14), in accordance with a trans-octahedral configuration of the two alkyl groups [16]. Instead, two strong absorptions at 289 and 279 cm⁻¹ were observed in the spectra of the diphenyltin(IV) complex 12. These bands are similar to those previously assigned [17] to v_{asym} and v_{sym} Sn-C in markedly distorted trans-R2 octahedral diphenyltin-(IV) complexes containing N-donor ligands.

2.1.3. Sn-Cl stretching frequencies

In the triorganotin(IV) derivatives 1 and 2, the tin(IV) chloride stretching frequencies are absent. On the basis of previous reports [18] on triorganotin(IV) complexes, we have hypothesized an ionic formulation of the type $[(L')_2R_3Sn]^+[Cl]^-$. The X-ray crystal structure determination of compound 1 (see Section 2.3) confirms our hypothesis. In the di- and mono-organotin complexes,

the ν (Sn-Cl) are observed in the region 230-200 cm⁻¹ and 260-245 cm⁻¹, respectively. These absorptions are downfield with respect to those observed for the 1-ben-

zylimidazole tin(IV) adducts [10]: this is likely to be due to the presence of hydrogen bonds between the N-H moieties of imidazole and halide groups [14].

Table 2

Selected IR data (4000-100 cm⁻¹) ^a

L 3117w. 303w. 300 2800–2600br 1606w. 1576w. 1544w. 521m. 435m. 435m. 345w. 308m. 325w. 315w. 305w. 305w. 3150w. 3150w. 310w. 3200–2600br 1611w. 1587w. 1572w. 305m. 320m. 282m. 320m. 282m. 320m. 282m. 320m. 282m. 345w. 305m. 201m. 210m. 2000r. 1610w. 1585w. 1580w. 1500. 3016w. 3100v. 3100r. 1611m. 1590m. 1574m. 450m. 450m. 3044w. 523m. 210m. 3146w. 523m. 210m. 3146w. 520m. 3100w. 300- 3000br 1610w. 1587w. 1572w. 496m. 445m. 364m. 534m. 217s. 194m. 162w. 155w. 300m. 200m. 1610w. 1587w. 1578w. 457m. 300m. 200m. 1610w. 1587w. 1578w. 457m. 300m. 200m. 1610w. 1587w. 1574w. 496m. 444m. 397sh. 300m. 200br 1610w. 1587w. 1574w. 496m. 445m. 364w. 523m. 218w. 175M. 175M. 200m. 175M. 247W. 300m. 200m. 175M. 247W. 300m. 200m. 175M. 247W. 300M. 200m. 1610w. 1588w. 1569w. 467m. 210m. 3124w. 3200br 1610w. 1588w. 1569w. 457m. 300M. 200m. 175M. 200m. 1334 9 3126w. 3120w. 3100br 1610w. 1588w. 1569w. 1572w. 366m. 300M. 200m. 155W. 300M. 200m. 155W. 300M. 200Mr. 1610W. 1588W. 1569W. 300M. 200Mr. 1610W. 1588W. 1569W. 300W. 200Mr. 200Mr. 1610W. 1588W. 1569W. 300W. 200Mr. 200Mr. 200Mr. 200Mr. 175W. 155W. 300Mr. 200Mr. 200M	Compound	(C-H)azolo	ν(N-H)	1650-1500	< 600	$\nu(Sn-C)$	$\nu(\text{Sn-X})$	$\frac{\delta(C-Sn-C)}{+\delta(Cl-Sn-Cl)}$
3666sh 1516w, 1508w 352w, 345w, 306m, 280w, 179w, 159m 352w, 345w, 306m, 280w, 179w, 159m 1 3154w, 3060w 3194s br 1611w, 1587w, 1572w 500m, 446m, 438m, 361m, 192m, 164m 54em 2 3126w, 301w 320-2600br 1608w, 1586w, 1573m 99m, 440m, 367m 57m 229s 178m, 152s, 145s, 366m 3 3127w, 3113w 3214s br 1609m, 1585w, 1574m 50m, 428w, 3385w, 200m 56m, 57m 229s 178m, 152s, 145s, 366m, 3056w 3 3170w 3200br 1610m, 1585w, 1574w, 3056a 209m, 428w, 3385w, 207m, 278w, 213ah 563m 176m, 161m 6 3126w, 3076w, 3018w 3170br 1610m, 1585w, 1558w, 3016w 207m, 278w, 213ah 503m 217s 194m, 162w, 155w, 134s 7 3156w, 3120w, 3016w 3200-2700br 1610w, 1587w, 157w 406m, 445m, 364m, 230m, 27m, 27m, 21m, 26m 523m 206m 185m, 154h, 128s 8 126w 3200-2700br 1610w, 158w, 157w, 158w, 157w, 158w 457m, 147m, 357m, 300m, 220m, 200m 228bn 178w, 163w 9 3126w 3200-2700br 1612w, 1590w, 1572w, 158w	Ľ	3117w, 3083w,	2800-2600br	1606w, 1576w, 1544w,	521m, 435m, 428m,			
14 3154w 3014b br 1611w 1587w 1500m 460m 46m		3060sh		1516w, 1508w	352w, 345w, 308m,			
1 3154w. 3060w 3194 br 1611w. 1587w. 1572w 500m. 436m. 438m. 546m. 2 3126w. 3051w. 3200-2600br 1608w. 1586w. 1500h 552w. 543w. 509m. 282m 274m 3 3127w. 3113w 3214 br 1609m. 1589m. 1573m 493m. 440m. 367m 575m 229 s 178m. 152s. 145s. 136s 4 3125w. 3110w 32010br 1611m. 1587m. 1574w 493m. 430m. 367m 575m 219 sh 136m. 150m. 129br 5 3170w 3200br 1610w. 1585w. 1558w 50m. 428w. 358w. 563m 176m. 161m 5 3170w 3200br 1610w. 1580m. 1574m 996m. 435m. 364w. 534m. 534m. 547w. 200m 523m. 535m 176m. 161m 6 3126w. 3076w. 3170br 1610w. 1587m. 1578w. 437w. 296m. 435m. 364w. 534m. 217s 194m. 162w. 155w 134s 7 3156w. 3120w 3300- 3000br 1610w. 1587w. 1578w. 364m. 397m 523m. 210m 178w. 163w 9 3126w 3200br 1610w. 1587w. 1574w. 366m. 423m. 364w. 329m. 217m. 316m 218 m. 150b. 140br 178w. 163w 11 3145w 3300br 1609w. 1587w. 1574w. 568w. 36					280w, 179w, 159m			
2 3126w, 3051w 3200-2600br 668w, 1586w, 1500b 365w, 543w, 500w, 227w, 274w, 215w, 165m, 152w, 136s, 125w, 136s, 125w, 3100w, 3120w, 3056w 3125w, 3110w, 3210br, 3210br, 1587m, 157w, 157w, 1587w, 157w, 1587w, 157w, 1587w, 157w, 157w, 1398w, 3050w 567m 215sh 165m, 150m, 129tr 439sh, 300w, 217w, 278w, 213sh, 305m 3 3126w, 3076w, 3170br 1610w, 1587w, 1588w, 1578w, 505m, 428w, 358w, 505m 563m, 428w, 358w, 505m 534m 217s 194m, 162w, 155w, 136w, 200m 3 3156w, 3120w, 3190br 1610w, 1587w, 1588w, 1578w, 505m, 200m 534m 217s 194m, 162w, 155w, 136w, 200m 3 3156w, 3120w, 3100br 3195br 1610w, 1587w, 1558w 487m, 441m, 397sh, 305m, 304w, 200br, 200w, 200br, 200br, 200br, 1578w, 1558w 495m, 444m, 995w, 621m, 21m, 201m, 200br, 1610w, 1587w, 158W, 160w, 210br, 200br, 200br, 200br, 1578w, 1578w, 1578w, 163m, 200br, 200br, 200br, 1578w, 1578w, 163m, 200br, 200br, 200br, 1578w, 1578w, 200br, 200b	1	3154w, 3060w	3194s br	1611w, 1587w, 1572w	500m, 446m, 438m,	546m		
2 3126w, 3051w, 3200-2500br 1608w, 1586w, 1500b, 1587, 449w, 219m, 274m, 210m 274m, 210m, 200m, 1306, 1306, 1300, 217w, 200m, 200m, 1611m, 1587m, 1574w, 200m, 200m, 217w, 200m, 200m, 1510w, 1370w, 3100br, 1611m, 1587m, 1574w, 200m, 200m, 2370w, 218w, 213b, 200m, 217w, 200m, 217w, 200m, 217w, 213b, 200m, 217w, 213b, 200m, 1574w, 200m, 200m, 450m, 450m					361m, 192m, 164m			
3031v 465v, 449v, 219m, 210m 274m, 210m 3 3127v, 3113w 3214s br 1609m, 1589m, 1573m 93m, 440m, 367m 575m 229s 178m, 152s, 145s, 215sh 136 4 3125v, 3110w, 3056w 3200br 1611m, 1587m, 157w 908m, 432m, 361m, 439sh, 300w, 217v, 200m 567m 176m, 161m 5 3170w 3200br 1610w, 1585w, 1558w, 150sh 505m, 200m 534m 217s 194m, 162w, 155w, 134s 6 3126w, 3170br 317br 1610w, 1587w, 157w 496m, 445m, 364w, 296m 523m 213m 178w, 163w 7 3156w, 3120w, 3166w 3200-2700br 1612w, 1590w, 1572w, 1558w 495m, 444m, 395w, 63m, 21m, 22m, 21m, 299m, 288b, 247w 323m 178w, 163w 9 3126w 3200-2700br 1612w, 1590w, 1572w, 1558w 495m, 444m, 395w, 63m, 21m, 22m, 208w, 23w, 22w 631m 22sh, 138 br 185s, 154s, 128s 11 3145w 3300br 1609w, 1587w, 1574w 508m, 433, 439m, 300br 218 br 179m, 156w, 140br, 155w, 300m, 22v, 22w 13 3115w, 3060w, 3130br 3130br 1609w, 1587w, 158w, 300br	2	3126w, 3051w,	3200-2600br	1608w, 1586w, 1500sh	552w, 543w, 509m,	282m,		
3 3127w, 3113w 3214s br 1609m, 1589m, 1573m 93m, 440m, 367m 575m 229s 178m, 152s, 145s, 136s 4 3125w, 3110w 3210br 1611m, 1587m, 1574w 93m, 440m, 367m 575m 229s 165m, 150m, 129br 5 3170w 3200br 1610w, 1585w, 1558w, 1558w, 1500sh 505m, 428w, 358w, 505m, 428w, 358w, 505m 563m, 127m, 161m, 1590m, 1574w 966m, 445m, 364w, 534m 217s 194m, 162w, 155w, 134s 7 3156w, 3120w, 3195br 1610w, 1587w, 1578w 966m, 445m, 364w, 534m 217s 194m, 162w, 155w, 134s 8 3149w 3000-3000br 1610w, 1587w, 1558w 563m, 310w, 220br, 206m 528m 178w, 163w 9 3126w 3200-2700br 1612w, 1590w, 1572w, 158w, 569w 528m, 128b r, 138b r, 150b r, 148m, 397m, 327m, 248m, 398m, 310m, 249w 218 br 138br, 150b r, 148m, 150b r, 148m, 307m, 129b r, 138b r, 150m, 157w, 158W, 569w 528m, 127m, 261m, 310r, 148m, 397m, 148m, 310m, 249w 205b r, 138br, 150h, 148m, 307m, 249w 218b r, 160w, 158m, 150m, 157m, 156W, 300m, 320m, 250m, 327m, 225m, 300m 139m, 148m, 398m, 433m, 398w, 310m, 249m, 310w, 310w, 310w, 3130br 1609w, 1587w, 156w, 300m, 320m, 250m, 327m, 325m, 327m, 250m, 300m, 327m, 325m,		3031w			465w, 449w, 219m,	274m		
3 3127w, 3113w 3214s br 1609m, 1539m, 1573m 493m, 440m, 367m 57m 229s 178m, 152s, 145s, 155s 4 3125w, 3110w, 3210br 3210br 1611m, 1587m, 1574w 508m, 432m, 361m, 507m 57m 229s 178m, 152s, 145s, 155k 5 3170w 3200br 1610w, 1585w, 1558w, 505m, 428w, 358w, 505m 505m 176m, 161m 6 3126w, 3076w, 3170br 3170br 1611m, 1590m, 1574w 496m, 445m, 364m, 534m 217s 194m, 162w, 155w, 154w, 296m 7 3156w, 3120w, 3195br 1610w, 1587w, 1558w 300m, 301w, 200m, 201m, 280m, 378h, 247w 124k 188m, 150sh, 126br 8 3149w 3300-3000br 1610w, 1587w, 1558w 487m, 441m, 397sh, 362m, 210m, 210s br, 178w, 163w 229w, 208w 228m, 201m, 210s br, 178w, 163w 9 3126w 3200br 1610w, 1587w, 157w 558w 63m, 30m, 220hr, 308w, 278s, 229s 220m, 178m, 158w, 150w, 140br, 36m, 310m, 218s br 178w, 163w 11 3145w 3300br 1609w, 1587w, 157w 50m, 437m, 355m, 320w, 278s, 230m 199m, 155m 12 313w 3300br 1609w, 1587w, 158					210m			
4 3125w, 3110w, 3210br 1611m, 1587m, 1574w 508m, 432m, 361m, 439h, 300w, 217w, 200m 507m 557m 165m, 150m, 129br 5 3170w 3200br 1610w, 1585w, 1558w, 1508w, 1508w, 1508w, 1508w, 1508w, 1508w, 1508w, 1508w, 3018w 505m 215m 166m, 161m 6 3126w, 3076w, 3170br, 3195br 1610w, 1587w, 1578w, 1588w, 1507w, 3016w 3146w 217s 194m, 162w, 155w, 1348 8 3149w 3300-2700br 1612w, 1590w, 1572w, 1588w, 1507w, 1558w 497m, 441m, 396m, 301w, 280br, 209m, 278sh, 247w 528m 178w, 163w 9 3126w 3200-2700br 1612w, 1590w, 1572w, 1578w 497m, 444m, 396m, 301w, 280br, 210w, 209m, 278sh, 247w 528m 178w, 163w 10 3124w 3200br 1610w, 1588w, 1507w, 1572w, 1558w 497m, 441m, 395w, 361m, 270w, 210m, 210s br, 179m, 156w, 140br, 361m, 270w, 210m, 208s, 133br 1218s br 179m, 156w, 140br, 363m, 313br 11 3145w, 3300br 1609w, 1587w, 1574w 508m, 443x, 364m, 398w, 327w, 326w, 320w, 313br 1609w, 1587w, 1568w 507m, 443m, 398w, 320w, 310w, 3130br 1609w, 1587w, 1568w 506m, 443w, 332w, 326w, 320w, 1597w, 1568w 506m, 443m, 398w, 32m, 258m, 230m 199m, 195m 13 3115w, 3060w, 3136br 1609w, 1587w, 1568w 506m, 443m, 3	3	3127w, 3113w	3214s br	1609m, 1589m, 1573m	493m, 440m, 367m	575m	229s	178m, 152s, 145s,
4 3125w, 3110w, 3056w 3210br 1611m, 1587m, 1574w 500m, 432m, 361m, 398h, 300w, 217w, 200m 507m 165m, 150m, 129br 5 3170w 3200br 1610w, 1585w, 1558w, 1500sh 505m, 1500sh 176m, 161m 6 3126w, 3076w, 3018w 3170br 1611m, 199m, 1574m 905m, 428w, 358w, 296m 534m 217s 194m, 162w, 155w, 134s 7 3156w, 3120w, 3016w 3100br 1610w, 1587w, 1558w 495m, 445m, 364m, 293m, 278s, 247w 534m 217s 194m, 162w, 155w, 134s 8 3149w 3300-3000br 1610w, 1587w, 1558w 495m, 444m, 396m, 627m, 228w, 208w 631m 225sh, 228m 178w, 163w 9 3126w 3200-2700br 1610w, 1587w, 1572w, 1558w 495m, 462m, 211m, 21m, 21m 218 br 178w, 163w 10 3124w 3200br 1609w, 1587w, 1574w 508m, 443x, 398m, 507m, 444m, 398w, 423m, 200m 287s 229s 206s, 205s, 133 br 133br 11 3145w 3300br 1609w, 1587w, 1578w 506m, 443x, 398w, 307m, 2079s 279s 205s 205s, 205s, 205s 205s, 205s 205s, 205s, 205s,	-						215sh	136s
3056w 439sh, 300v, 217w, 200m 439sh, 300v, 217w, 200m 5 3170w 3200br 1610w, 1585w, 1558w, 503m, 200m, 428w, 358w, 503m, 200m, 157m, 3018w 176m, 161m 6 3126w, 3076w, 3170br, 3170br 1611m, 1590m, 157m, 498w, 445m, 364m, 534m, 234m, 213sh 505m, 134sh 178w, 163w, 134sh 7 3156w, 3120w, 3195br 1610v, 1587w, 1558w 496m, 445m, 364m, 374m, 374m, 134sh 528m 188m, 150sh, 126br 8 3149w 3300-3000br 1610w, 1587w, 1558w 497m, 441m, 397ch, 360m, 310u, 280br, 528m 178w, 163w 9 3126w 3200-2700br 1612w, 1590w, 1572w 495m, 444m, 396m, 631m 218s br 10 3124w 3200br 1610w, 1588w, 1567w 493m, 444m, 395w, 627m 210s br, 179m, 156w, 140br, 364m, 310w, 280w, 279s 11 3145w 3300br 1609w, 1587w, 1574w 508m, 443m, 398w, 327s, 229s 200s, 201s, 177m, 156w, 140br, 302m, 187w, 158w, 1507w, 302m, 143m, 398w, 327w, 278s, 326m, 320m, 143m, 328w, 327w, 326m, 320m, 143m, 328w, 320w, 3130br 1609w, 1587w, 1568w 506m, 443m, 398w, 327w, 278s, 320m, 199m, 148m 14 3106w, 3120w 3130br 1610w, 1587w, 1568w 506m, 443m, 398w, 31m, 328m, 217w, 325m, 247w, 320m, 197m, 355m, 3	4	3125w, 3110w,	3210br	1611m, 1587m, 1574w	508m, 432m, 361m,	567m		165m, 150m, 129br
5 3170w 3200br 1610w, 1585w, 155w, 157w, 126w, 213h 563m, 428w, 358w, 563m, 505m 176m, 161m 6 3126w, 3076w, 3170br 3170br 1611m, 1590m, 1574m 498w, 445m, 364m, 534m 217s 194m, 162w, 155w, 134w 7 3156w, 3120w, 3195br 3195br 1610w, 1587m, 1572w 498m, 445m, 364m, 534m 523m 206m 188m, 150kh, 126br 8 3149w 3300-3000br 1610w, 1587w, 1558w 47m, 441m, 397sh, 364m, 528m 528m 178w, 163w 9 3126w 3200-2700br 1612w, 1590w, 1572w, 158w 487m, 441m, 397sh, 364m, 522m, 205s, 128s, 154s, 128s 367m, 210m, 210m 2188 br 179m, 156w, 140br, 361m, 225 sh, 133br 10 3124w 3200br 1610w, 1587w, 1574w 508m, 463s, 439m, 287w, 208w, 201m, 179m, 156w, 140br, 361m, 249w, 3		3056w			439sh, 300w, 217w,			
5 3170w 3200br 1610w, 1985w, 1528w, 305m, 428w, 358w, 505m, 176m, 161m 6 3126w, 3076w, 3170br 1611m, 1590m, 1574m 297w, 278w, 213sh 505m 194m, 162w, 155w, 134s 7 3156w, 3120w, 3195br 1610w, 1589m, 1572w 296m, 445m, 364w, 524m 205m 134s 8 3149w 3300-3000br 1610w, 1587w, 157ew, 447m, 364w, 528m 528m 178w, 163w 9 3126w 3200-2700br 1612w, 1590w, 1572w, 157ew, 1558w, 444m, 395m, 631m 225sh, 185s, 154s, 128s 10 3124w 3200br 1610w, 1587w, 157ew 495m, 444m, 395m, 631m 225sh, 185s, 154s, 128s 11 3145w 3300br 1609w, 1587w, 1574w 508m, 463w, 427m, 210s br, 177m, 156w, 140br, 361m, 270w, 210m 205sh, 133br 12 3132w 3300br 1609w, 1587w, 1574w 508m, 443m, 398w, 287s, 229s 220m, 197m, 156w, 149br, 307w, 307m, 444m, 395w, 627m, 210s br, 177m, 364m, 310m, 249w 159m, 148m 12 3132w 3300br 1609w, 1587w, 1574w 506m, 444w, 395w, 627m, 210s br, 177m, 364m, 310m, 249w 159m, 148m 13 3115w, 3060w, 3130br 1609w, 1587w, 1574w 506m, 440w, 398m, 423m 218m 200m, 193m 3011w <td< td=""><td>-</td><td></td><td></td><td></td><td>200m</td><td></td><td></td><td></td></td<>	-				200m			
6 3126w, 3076w, 3170br 1500sh 29/w, 278w, 123sh 500m 948w, 445m, 364m, 534m 217s 194m, 162w, 155w, 134s 7 3156w, 3120w, 3192bv, 3195br 1610w, 1589m, 1572w 496m, 445m, 364m, 523m 20m 188m, 150sh, 126br 8 3149w 3300-3000br 1610w, 1587w, 158w 487m, 441m, 397sh, 360m, 611m 225sh, 185s, 154s, 128s 9 3126w 3200-2700br 1610w, 1587w, 1590w, 1572w, 1590w 495m, 444m, 396m, 611m 225sh, 185s, 154s, 128s 10 3124w 3200br 1610w, 1588w, 1509w 493m, 444m, 395w, 627m 210s br, 179m, 156w, 140br, 200sh 133br 11 3145w 3300br 1609w, 1587w, 1574w 508m, 463s, 439m, 287s 229s 206s, 201s, 177m, 159m, 148m 12 3132w 3300br 1609w, 1587w, 158w 507m, 329m, 256m 199m, 195m 14 3106w, 3120br 1612w, 1590w, 1572w 509m, 443m, 398w, 320w, 320w 218m 200m, 193m 3011w 3132w, 3060w 3350br, 3180br 1609w, 1587w, 1568w 506m, 437m, 355m, 492m, 264m 180br, 160s, 149s 3132w, 3060w 335	5	3170w	3200br	1610w, 1585w, 1558w,	505m, 428w, 358w,	563m,		176m, 161m
6 3120w, 3070w, 3170br 161 Im, 1590m, 1574m 495w, 445m, 364m, 534m 217s 194m, 162w, 1530w, 134 7 3156w, 3120w, 3195br 1610w, 1589m, 1572w 496m, 445m, 364w, 523m 206m 188m, 150sh, 126br 8 3149w 3300–3000br 1610w, 1587w, 1558w 496m, 445m, 364w, 523m 206m 188m, 150sh, 126br 9 3126w 3200–2700br 1610w, 1587w, 1558w 366m, 301w, 2080br 528m 728m, 247m, 210s 9 3126w 3200br 1610w, 1588w, 1569w 495m, 444m, 395w, 627m 210s br, 179m, 156w, 140br, 218s br 10 3124w 3200br 1610w, 1588w, 1569w 362m, 271m, 261m 218s br 11 3145w 3300br 1609w, 1587w, 1574w 508m, 463s, 439m, 287s, 289s, 210s, 201s, 177m, 364m, 310m, 249w 159m, 144m 12 3132w 3300br 1612w, 1590w, 1572w 506m, 442m, 395w, 423m, 279s, 364w, 352w, 326w, 300m, 257w, 364w, 352w, 326w, 300m, 257w, 364w, 352w, 326w, 300m, 279s, 364w, 352w, 326w, 300m, 279s, 364w, 352w, 326w, 300m, 279s, 364w, 352w, 326w, 300m, 225w, 300w 148m 1069w, 1587w, 1568w 506m, 437m, 355m, 429m, 424m, 180br, 160s, 149s, 303m, 225m, 2458 br 131m, 200w, 3100w, 3380br, 3200br		2126 2026	2170	1500sh	29/w, 2/8w, 213sh	505m		104 160 155
3018w 3120w, 3120w, 3195br 1610w, 1589m, 1572w 290m 205m 188m, 150sh, 126br 8 3149w 3300-3000br 1610w, 1587w, 1558w 487m, 441m, 397sh, 360m, 301w, 280br, 528m 178w, 163w 9 3126w 3200-2700br 1612w, 1590w, 1572w, 1558w 487m, 444m, 396m, 631m 225sh, 185s, 154s, 128s 10 3124w 3200br 1610w, 1587w, 158w, 1569w 493m, 444m, 395w, 627m 210s br, 179m, 156w, 140br, 205sh 11 3145w 3300br 1609w, 1587w, 1574w 508m, 463s, 439m, 287s 229s 206s, 201s, 177m, 364m, 310m, 249m, 352w, 320w 12 3132w 3300br 1609w, 1588w, 1507w, 507m, 494m, 458s, 289s, 230m 199m, 195m 1558w 13 3115w, 3060w, 3130br 1612w, 1590w, 1572w 507m, 494m, 398w, 320w, 320w, 302w 205m, 200m, 193m 14 3106w, 3020w 3130br 1612w, 1590w, 1572w 506m, 437m, 355m, 320m, 245m, 320m, 247m, 225m, 200m, 370m, 320m, 320m, 250m, 327m, 325m, 300w 1617w, 1594w, 1576w 506m, 437m, 355m, 3200m, 419m, 352w, 320m, 247m, 225m, 200m, 370w, 225m, 200m, 370w, 225m, 200m, 247m, 225m, 200m, 171m, 303m 16 3135w 3528br, 3200br 1617w, 1594w, 1576w 509m, 454m, 352	6	3126w, 3076w,	3170br	1611m, 1590m, 1574m	498w, 445m, 364m,	534m	21/s	194m, 162w, 155w,
7 3150w, 3120w, 3120w, 31950r 1610w, 1587w, 152w 490m, 443m, 364w, 523m 200m 188m, 150sh, 1250r 8 3149w 3300-3000br 1610w, 1587w, 1558w 487m, 441m, 397sh, 360m, 301w, 325sh, 185s, 154s, 128s 9 3126w 3200-2700br 1612w, 1590w, 1572w, 1578w, 229w, 208w 631m 225sh, 185s, 154s, 128s 10 3124w 3200br 1610w, 1588w, 1569w 493m, 444m, 395w, 627m 210s br, 179m, 156w, 140br, 205sh 11 3145w 300br 1609w, 1587w, 1574w 508m, 463s, 439m, 287s 229s 206s, 201s, 171m, 364m, 129m, 157m, 364m, 310m, 249w 12 3132w 3300br 1609w, 1587w, 1574w 509m, 443m, 398w, 423m, 287s 229s 206s, 201s, 171m, 364m, 129m, 157m, 364m, 310m, 249w 13 3115w, 3060w, 3130br 1612w, 1590w, 1572w 509m, 443m, 398w, 423m, 218m 200m, 193m 3011w 300hr 1609w, 1587w, 1568w 506m, 437m, 355m, 326m, 280m, 177m, 307m, 325m, 326m, 280m, 177m, 25m, 326m, 280m, 177m, 307m, 325m, 326m, 280m, 177m, 307w, 307w, 325w, 300w 2464m 180br, 160s, 149s 16 3132w, 300br 1609w, 1587w, 1568w 506m, 437m, 355m, 326m, 280m, 177m, 307w, 156w, 303w, 225w, 300w 501m, 454m, 352w, 242s 266s br 200m, 171m <t< td=""><td>-</td><td>3018w</td><td>21051</td><td>1/10 1500 1570</td><td>296m</td><td>500</td><td>007</td><td>1348</td></t<>	-	3018w	21051	1/10 1500 1570	296m	500	007	1348
3016W 2930, 2783, 2783, 247W 8 3149w 3300-3000br 1610w, 1587w, 1558w 497m, 441m, 397sh, 360m, 301w, 280br, 528m 178w, 163w 9 3126w 3200-2700br 1612w, 1590w, 1572w, 1558w 360m, 301w, 280br, 229w, 208w 631m 225sh, 218s br 178w, 163w 9 3126w 3200-2700br 1612w, 1590w, 1572w, 1558w 362m, 271m, 261m 218s br 179m, 156w, 140br, 133br 10 3124w 3200br 1609w, 1587w, 1574w S08m, 444m, 395w, 627m 2108 br 179m, 156w, 140br, 139br 11 3145w 3300br 1609w, 1587w, 1574w S08m, 433m, 349m, 42w 287s 229s 206s, 201s, 177m, 159m, 143m 12 3132w 3300br 1609w, 1587w, 1574w S07m, 494m, 458s, 289s, 290m, 437m, 398w, 423m 218m 200m, 193m 3011w 3015w 1330br 1612w, 1590w, 1572w 509m, 433m, 398w, 423m 218m 200m, 193m 3011w 3136br 1609w, 1587w, 1568w 506w, 440w, 398m, 419m 325m, 236m, 2300w 218m 200m, 193m 15 3132w, 3360br, 3180br 1609w,	7	3156w, 3120w,	3195br	1610w, 1589m, 1572w	496m, 445m, 364w,	523m	206m	188m, 150sh, 126br
8 3149w 3300-300007 1610w, 1587w, 1538w 497m, 441m, 397sh, 229w, 208w 178w, 163w 9 3126w 3200-2700br 1612w, 1590w, 1572w, 305m, 326m, 210m 218s br 178w, 163w 10 3124w 3200br 1610w, 1588w, 1569w 493m, 444m, 396m, 631m 221sh, 185s, 154s, 128s 11 3145w 3200br 1610w, 1588w, 1569w 493m, 444m, 395w, 627m 210s br, 179m, 156w, 140br, 364m, 310m, 249w 12 3132w 3300br 1609w, 1587w, 1574w 508m, 463s, 435m, 28r, 229s 206s, 201s, 177m, 159m, 148m 12 3132w 3300br 1609w, 1588w, 1507w, 507m, 494m, 458s, 289s, 230m 199m, 195m 13 3115w, 3060w, 3130br 1612w, 1590w, 1572w 509m, 443m, 398w, 423m 218m 200m, 193m 3011w 3106w, 3020w 3136br 1609w, 1587w, 1568w 506m, 437m, 355m, 492m, 264m 180br, 160s, 149s 3011w 3132w, 3060w 3350br, 3180br 1609w, 1587w, 1568w 506m, 437m, 355m, 492m, 264m 180br, 160s, 149s 3011w 3132w, 3060w 3350br, 3180br 1609w, 1587w, 1568w 506m, 437m, 355m, 492m, 264m 180br, 160s, 149s 3115w 3228br, 3200br	0	3016W	2200 2000h-	1610 1597 1559	293m, 278sn, 247w			
9 3126w 3200-2700br 1612w, 1590w, 1572w, 1558w 495m, 444m, 396m, 631m 225sh, 218s 185s, 154s, 128s 10 3124w 3200br 1610w, 1588w, 1569w 39m, 444m, 396m, 627m 210s br, 179m, 156w, 140br, 205sh 133br 11 3145w 3300br 1609w, 1587w, 1574w 508m, 463s, 439m, 220s, 133br 200s, 201s, 177m, 364m, 310m, 249w 159m, 148m 12 3132w 3300br 1609w, 1588w, 1507w, 507m, 494m, 458s, 289s, 230m 199m, 195m 195m, 148m 13 3115w, 3060w, 3130br 1612w, 1590w, 1572w 509m, 443m, 398w, 423m 218m 200m, 193m 3011w 3115w, 3060w, 3130br 1612w, 1590w, 1572w 509m, 443m, 398w, 423m 218m 200m, 193m 3011w 3130br, 3180br 1609w, 1587w, 1568w 506w, 440w, 398m, 325m, 226m 218m 200m, 193m 14 3106w, 3020w 3136br 1609w, 1587w, 1568w 506w, 440w, 398m, 423m 218m 200m, 193m 15 3132w, 3060w 3350br, 3180br 1609w, 1587w, 1568w 506m, 447m, 325m, 240m 418m, 398w, 31m, 315w, 325m, 24m, 247m 316w, 160s, 149s 316 3135w 3528br, 3200br 1617w, 1594w, 1576w	8	3149W	3300-3000br	1610w, 158/w, 1558w	48/m, 441m, 39/sn,	520		170 167
9 3126w 3200-2700br 1612w, 1590w, 1572w, 1578w, 1631m, 220sh, 210s br, 179m, 156w, 140br, 362m, 271m, 261m 218s br 218s br 10 3124w 3200br 1610w, 1588w, 1569w 495m, 444m, 395m, 621m, 270m, 210s br, 179m, 156w, 140br, 205sh, 133br 130sr, 177m, 156w, 140br, 205sh, 133br 11 3145w 3300br 1609w, 1587w, 1574w 508m, 463s, 439m, 270w, 210m 205sh, 133br 135m, 148m 12 3132w 3300br 1609w, 1587w, 1574w 507m, 494m, 458s, 289s, 230m 195m, 148m 13 3115w, 3060w, 3130br 1612w, 1590w, 1572w, 302w 507m, 444m, 395w, 423m, 388w, 279s, 302w 230m 195m, 148m 3011w 3011w 1699w, 1587w, 1568w 506m, 4470w, 398m, 423m, 326m, 26m 218m 200m, 193m 3011w 313br 1609w, 1587w, 1568w 506m, 437m, 335m, 492m, 26m 204m, 180br, 160s, 149s 3011w 3132w, 3060w 3350br, 3180br 1609w, 1587w, 1568w 506m, 437m, 335m, 492m, 26dm 180br, 160s, 149s 3135w 3528br, 3200br 1617w, 1594w, 1576w 506m, 437m, 355m, 245m 245m 16 3135w 3200br 1623w,					300m, 301w, 2800r,	528m		178w, 103w
9 3126w 3200-27000r 1012w, 1390v, 1372w, 14950, 1441m, 3901m 2123s1r, 123s 123s1, 123s 10 3124w 3200br 1610w, 1588w, 1569w 493m, 444m, 395w, 627m 210s br, 179m, 156w, 140br, 205sh 11 3145w 3300br 1609w, 1587w, 1574w 508m, 463s, 439m, 287s 229s 205s, 201s, 177m, 364m, 139v, 159m, 148m 12 3132w 3300br 1609w, 1588w, 1507w, 507m, 494m, 458s, 289s, 230m 29m, 195m, 148m 13 3115w, 3060w, 3130br 1612w, 1590w, 1572w 509m, 443m, 398w, 423m 218m 200m, 193m 3011w 3011w 1609w, 1587w, 1568w 506w, 440w, 398m, 419m 325m, 226m 218m 200m, 193m 14 3106w, 3020w 3130br 1612w, 1590w, 1572w 509m, 443m, 395w, 423m 218m 200m, 193m 15 3132w, 3060w 3350br, 3180br 1609w, 1587w, 1568w 506w, 440w, 398m, 419m 325m, 2264m 180br, 160s, 149s 303a, 225w 530w 1617w, 1594w, 1576w 506m, 437m, 355m, 492m, 264m 180br, 160s, 149s 313bw 3200br 1617w, 1594w, 1576w 496m, 452w, 432m, 615s br 245s br 264m 180br, 160s, 149s	0	2126	2200 27004-	1612 1500 1572	229W, 208W	621-	225ah	1950 1540 1390
10 3124w 3200br 1610w, 1588w, 1569w 493m, 444m, 395w, 627m 2108 br, 179m, 156w, 140br, 205sh 11 3145w 3300br 1609w, 1587w, 1574w 508m, 463x, 439m, 287s 229s 206s, 201s, 177m, 159m, 148m 12 3132w 3300br 1609w, 1588w, 1507w, 507m, 494m, 458s, 328w, 279s 230m 199m, 195m 159m, 148m 12 3132w 3300br 1612w, 1590w, 1572w 507m, 494m, 458s, 289s, 230m 230m 199m, 195m 13 3115w, 3060w, 3130br 1612w, 1590w, 1572w 509m, 443m, 398w, 320w, 302w 218m 200m, 193m 14 3106w, 3020w 3136br 1609w, 1587w, 1568w 506m, 437m, 355m, 422m, 264m 180br, 160s, 149s 15 3132w, 3060w 3350br, 3180br 1609w, 1587w, 1568w 506m, 437m, 355m, 492m, 264m 180br, 160s, 149s 16 3135w 3528br, 3200br 1617w, 1594w, 1576w 506m, 437m, 355m, 492m, 264m 180br, 160s, 149s 17 3156w, 3138w 3200br 1623w, 1597w, 1576w 509m, 454m, 352w, 282s 260s br 200m, 171m 18 3130w, 3110w 3380br, 3200br 1623w, 1596w, 1576w 418w, 398w, 351w, 226m, 203m, 177m, 303m	9	3120W	3200-27000f	1012W, 1390W, 1372W,	495111, 444111, 590111,	031111	22380, 218e br	1858, 1548, 1288
10 3124w 32001 10104, 138w, 1503w 4907, 1303w, 1203w 210s 01, 1731w, 1730w, 1400, 1331r 11 3145w 3300br 1609w, 1587w, 1574w 508m, 463s, 439m, 310m, 249w 287s 229s 206s, 201s, 177m, 159m, 148m 12 3132w 3300br 1609w, 1587w, 1574w 508m, 463s, 439m, 30m, 249w 230m 199m, 148m 12 3132w 3300br 1609w, 1588w, 1507w, 507m, 494m, 458s, 289s, 230m 230m 199m, 195m 13 3115w, 3060w, 3130br 1612w, 1590w, 1572w 509m, 443m, 398w, 423m 218m 200m, 193m 3011w 3106w, 3020w 3136br 1609w, 1587w, 1568w 506m, 437m, 355m, 492m, 264m 180br, 160s, 149s 3011w 313bv 352br, 3200br 1617w, 1594w, 1576w 506m, 452w, 432m, 615s br 245s br 14 3106w, 3138w 3200br 1617w, 1594w, 1576w 506m, 452w, 432m, 615s br 245s br 15 3132w, 3060w 3350br, 3180br 1609w, 1587w, 1568w 506m, 452w, 432m, 615s br 245s br 16 3135w 3528br, 3200br 1617w, 1594w, 1576w 498w, 398w, 351w, 320w 245s br 17 3156w, 3138w <td>10</td> <td>3124</td> <td>2200br</td> <td>1550w 1610w 1588w 1560w</td> <td>403m 444m 305w</td> <td>627m</td> <td>2108 Di 210e br</td> <td>170m 156w 140br</td>	10	3124	2200br	1550w 1610w 1588w 1560w	403m 444m 305w	627m	2108 Di 210e br	170m 156w 140br
11 3145w 3300br 1609w, 1587w, 1574w 508m, 4638, 439m, 364m, 310m, 249w 229s 229s 206s, 201s, 177m, 159m, 148m 12 3132w 3300br 1609w, 1588w, 1507w, 507m, 494m, 458s, 289s, 279s 230m 199m, 195m 13 3115w, 3060w, 3130br 1612w, 1590w, 1572w 509m, 443m, 398w, 423m 218m 200m, 193m 14 3106w, 3020w 3130br 1612w, 1590w, 1587w, 1568w 506w, 440w, 398m, 320m, 256m 419m 325m, 326m, 280m, 247m, 225m, 200m, 176m 15 3132w, 3060w 3350br, 3180br 1609w, 1587w, 1568w 506w, 440w, 398m, 320m, 247m, 225m, 200m, 176m 264m 180br, 160s, 149s 16 3135w 3528br, 3200br 1617w, 1594w, 1576w 492m, 458 br 245s br 200m, 171m 17 3156w, 3138w 3200br 1623w, 1597w, 1576w 509m, 454m, 352w, 282s 260s br 200m, 171m 18 3130w, 3110w 3380br, 3200br 1623w, 1596w, 1576w 488w, 398w, 351w, 325m, 226m, 226m, 203m, 177m 303m 19 s 3139w, 3110w 3265br 1612m, 1590m, 1575m, 504m, 441m, 363m 551s 20 ^{bd} 3139w, 3110w 3265br 161	10	5124w	520001	1010w, 1588w, 1509w	361m 270w 210m	02711	2103 DI, 205sb	133hr
11 5185 w 1600 w, 1500 w,	11	3145w	3300br	1609w 1587w 1574w	508m 463s 439m	287s	200311	206s 201s 177m
12 3132w 3300br 1609w, 1588w, 1507w, 1507m, 494m, 458s, 289s, 230m 230m 199m, 195m 13 3115w, 3060w, 3130br 1612w, 1590w, 1572w 507m, 494m, 458s, 289s, 279s 218m 200m, 193m 14 3106w, 3020w 3136br 1609w, 1587w, 1568w 506w, 443w, 398w, 326m, 243m, 218m 200m, 193m 15 3132w, 3060w 3350br, 3180br 1609w, 1587w, 1568w 506w, 440w, 398m, 247m, 225m, 200m, 247m, 225w 530w 180br, 160s, 149s 16 3135w 3528br, 3200br 1617w, 1594w, 1576w 496m, 452w, 432m, 615s br 245s br 17 3156w, 3138w 3200br 1623w, 1597w, 1576w 509m, 443m, 352w, 282s 260s br 200m, 171m 18 3130w, 3110w 3380br, 3200br 1623w, 1597w, 1576w 418w, 398w, 351w, 325m, 225m, 200m, 171m 303m 224m 19 3139w, 3110w 3265br 1612m, 1590m, 1575m, 504m, 441m, 363m 551s 551s 20 ^{bd} 3147w 3230br 1622m, 1598m, 1579m 517w, 498w, 476w, 567m 567m 20 ^{bd} 3147w 32		5145₩	550001	10074, 15074, 15744	364m 310m 249w	2073	2275	159m 148m
12 5152m 5050m 1558m 1558m 439m 2053m 205m 155m 1558m 13 3115w, 3060w, 3130br 1612w, 1590w, 1572w 509m, 443m, 398w, 302w 302w 218m 200m, 193m 14 3106w, 3020w 3136br 1609w, 1587w, 1568w 506m, 440w, 398m, 419m 419m 15 3132w, 3060w 3350br, 3180br 1609w, 1587w, 1568w 506m, 437m, 355m, 492m, 264m 180br, 160s, 149s 16 3135w 3528br, 3200br 1617w, 1594w, 1576w 496m, 452w, 432m, 615s br 245s br 17 3156w, 3138w 3200br 1623w, 1597w, 1576w 496m, 452w, 432m, 351w, 325m, 245s br 200m, 171m 18 3130w, 3110w 3380br, 3200br 1623w, 1597w, 1576w 418w, 398w, 351w, 325m, 225m, 200m, 171m 224m 19 3139w, 3110w 3265br 1612m, 1590m, 1575m, 1500w, 1576w 504m, 441m, 363m 551s 20 bd 3147w 3230br 1622m, 1598m, 1579m 517w, 498w, 476w, 567m 507m, 244m, 247w, 244m, 247w, 244m, 244m, 247m, 244m, 247m, 244m, 2	12	3132w	3300br	1609w, 1588w, 1507w,	507m, 494m, 458s.	289s.	230m	199m, 195m
13 3115w, 3060w, 3130br 1612w, 1590w, 1572w 509m, 443m, 398w, 302w 423m 218m 200m, 193m 14 3106w, 3020w 3136br 1609w, 1587w, 1568w 506w, 440w, 398m, 419m 419m 15 3132w, 3060w 3350br, 3180br 1609w, 1587w, 1568w 506m, 437m, 355m, 3200m, 176m 492m, 264m 180br, 160s, 149s 16 3135w 3528br, 3200br 1617w, 1594w, 1576w 496m, 452w, 432m, 615s br 245s br 17 3156w, 3138w 3200br 1623w, 1597w, 1576w 509m, 444m, 352w 282s 260s br 200m, 171m 18 b 3130w, 3110w 3380br, 3200br 1623w, 1596w, 1576w 418w, 398w, 351w, 325m, 303m 325m, 325m, 303m 303m 19 c 3139w, 3110w 3265br 1612m, 1590m, 1575m, 1500sh 504m, 441m, 363m 551s 20 b.d 3147w 3230br 1622m, 1598m, 1579m 517w, 498w, 476w, 567m 567m 20 b.d 3147w 3230br 1622m, 1598m, 1579m 517w, 498w, 476w, 567m 567m		01020	500001	1558w	439m, 420w, 398w.	279s		.,,.,.,.
13 3115w, 3060w, 3130br 1612w, 1590w, 1572w 509m, 443m, 398w, 329m, 256m 218m 200m, 193m 14 3106w, 3020w 3136br 1609w, 1587w, 1568w 506w, 440w, 398m, 419m 419m 15 3132w, 3060w 3350br, 3180br 1609w, 1587w, 1568w 506m, 437m, 355m, 200m, 176m 492m, 264m 180br, 160s, 149s 16 3135w 3528br, 3200br 1617w, 1594w, 1576w 506m, 437m, 355m, 492m, 264m 180br, 160s, 149s 303w, 225w 530w 530w 167m 157m, 3156br, 3180br 1609w, 1587w, 1568w 506m, 437m, 355m, 492m, 264m 180br, 160s, 149s 303w, 225w 530w 530w 1617w, 1594w, 1576w 496m, 452w, 432m, 615s br 245s br 17 3156w, 3138w 3200br 1623w, 1597w, 1576w 509m, 454m, 352w 282s 260s br 200m, 171m 224m 18 3130w, 3110w 3380br, 3200br 1623w, 1596w, 1576w 418w, 398w, 351w, 325m, 225m, 226m, 237m, 315w, 226m, 237m, 315w, 226m, 203m, 177m 303m 19 c 3139w, 3110w 3265br 1612m, 1590m, 1575m, 1500w, 1575m, 1500w, 1575w, 438w, 351w, 323m, 225m, 226m, 233m, 177m 303m 20 b.d 3147w 3230br <				1000	364w, 352w, 326w,			
13 3115w, 3060w, 3020w 3130br 1612w, 1590w, 1572w 509m, 443m, 398w, 329m, 256m 218m 200m, 193m 14 3106w, 3020w 3136br 1609w, 1587w, 1568w 506w, 440w, 398m, 325m, 225m, 220m, 176m 419m 352m, 326m, 280m, 247m, 225m, 200m, 176m 15 3132w, 3060w 3350br, 3180br 1609w, 1587w, 1568w 506m, 437m, 355m, 303w, 225w 492m, 530w 264m 180br, 160s, 149s 16 3135w 3528br, 3200br 1617w, 1594w, 1576w 496m, 452w, 432m, 351m, 311m, 290w 615s br 245s br 17 3156w, 3138w 3200br 1623w, 1597w, 1576w 509m, 441m, 352w, 260s br 200m, 171m 18 ^b 3130w, 3110w 3380br, 3200br 1623w, 1596w, 1576w 418w, 398w, 351w, 282m, 26m, 230m 325m, 25m, 200m, 171m 19 ^c 3139w, 3110w 3265br 1612m, 1590m, 1575m, 1500w, 1576w 504m, 441m, 363m 551s 20 ^{b,d} 3147w 3230br 1622m, 1598m, 1579m 517w, 498w, 476w, 567m 567m 20 ^{b,d} 3147w 3230br 1622m, 1598m, 1579m 517w, 498w, 476w, 567m 567m					302w			
3011w 360m, 329m, 256m 14 3106w, 3020w 3136br 1609w, 1587w, 1568w 506w, 440w, 398m, 352m, 326m, 280m, 247m, 225m, 200m, 176m 15 3132w, 3060w 3350br, 3180br 1609w, 1587w, 1568w 506m, 437m, 355m, 300w, 256w 492m, 264m 180br, 160s, 149s 16 3135w 3528br, 3200br 1617w, 1594w, 1576w 496m, 452w, 432m, 303w, 225w 530w 245s br 17 3156w, 3138w 3200br 1617w, 1597w, 1576w 509m, 454m, 352w 282s 260s br 200m, 171m 18 b 3130w, 3110w 3380br, 3200br 1623w, 1597w, 1576w 509m, 454m, 352w 282s 260s br 200m, 171m 19 c 3139w, 3110w 3265br 1612m, 1596w, 1576w 418w, 398w, 351w, 225m, 315w, 225m, 303m 315w, 226m, 203m, 177m 303m 19 c 3139w, 3110w 3265br 1612m, 1590m, 1575m, 1500sh 504m, 441m, 363m 551s 51s 20 b.d 3147w 3230br 1622m, 1598m, 1579m 517w, 498w, 476w, 434w, 353m, 282m, 240m, 181w, 167w 567m	13	3115w, 3060w,	3130br	1612w, 1590w, 1572w	509m, 443m, 398w,	423m	218m	200m, 193m
14 3106w, 3020w 3136br 1609w, 1587w, 1568w 506w, 440w, 398m, 320m, 247m, 225m, 200m, 247m, 225m, 200m, 176m 15 3132w, 3060w 3350br, 3180br 1609w, 1587w, 1568w 506m, 437m, 355m, 492m, 264m 180br, 160s, 149s 16 3135w 3528br, 3200br 1617w, 1594w, 1576w 496m, 452w, 432m, 318w 615s br 245s br 17 3156w, 3138w 3200br 1623w, 1597w, 1576w 509m, 454m, 352w 282s 260s br 200m, 171m 18 b 3130w, 3110w 3380br, 3200br 1612m, 1596w, 1576w 418w, 398w, 351w, 226m, 225m, 200m, 171m 325m, 226m, 203m, 177m 303m 19 c 3139w, 3110w 3265br 1612m, 1590m, 1575m, 1500w, 1576w 504m, 441m, 363m 551s 20 b.d 3147w 3230br 1622m, 1598m, 1579m 517w, 498w, 476w, 434w, 353m, 282m, 240m, 181w, 167w 567m		3011w			360m, 329m, 256m			
15 3132w, 3060w 3350br, 3180br 1609w, 1587w, 1568w 506m, 437m, 355m, 392m, 264m 180br, 160s, 149s 16 3135w 3528br, 3200br 1617w, 1594w, 1576w 496m, 452w, 432m, 615s br 245s br 17 3156w, 3138w 3200br 1623w, 1597w, 1576w 509m, 454m, 352w 282s 260s br 200m, 171m 18 b 3130w, 3110w 3380br, 3200br 1623w, 1596w, 1576w 418w, 398w, 351w, 224m 325m, 225m, 200m, 171m 18 b 3130w, 3110w 3380br, 3200br 1623w, 1596w, 1576w 418w, 398w, 351w, 226m, 230m, 315w, 226m, 230m, 1575w, 266m, 203m, 177m 303m 19 c 3139w, 3110w 3265br 1612m, 1590m, 1575m, 1500w, 1576w 504m, 441m, 363m 551s 20 b.d 3147w 3230br 1622m, 1598m, 1579m 517w, 498w, 476w, 567m 567m 434w, 353m, 282m, 240m, 181w, 167w 517w, 498w, 476w, 567m 567m 434w, 353m, 282m, 240m, 181w, 167w	14	3106w, 3020w	3136br	1609w, 1587w, 1568w	506w, 440w, 398m,	419m		
15 3132w, 3060w 3350br, 3180br 1609w, 1587w, 1568w 506m, 437m, 355m, 492m, 264m 180br, 160s, 149s 16 3135w 3528br, 3200br 1617w, 1594w, 1576w 496m, 452w, 432m, 351w, 351m, 311m, 290w 245s br 17 3156w, 3138w 3200br 1617w, 1594w, 1576w 509m, 454m, 352w 282s 260s br 200m, 171m 18 3130w, 3110w 3380br, 3200br 1623w, 1596w, 1576w 418w, 398w, 351w, 224m 325m, 315w, 315w, 224m 19 c 3139w, 3110w 3265br 1612m, 1590m, 1575m, 1504m 504m, 441m, 363m 551s 20 bd 3147w 3230br 1622m, 1598m, 1579m 517w, 498w, 476w, 434w, 353m, 282m, 240m, 181w, 167w 567m					352m, 326m, 280m,			
15 3132w, 3060w 3350br, 3180br 1609w, 1587w, 1568w 506m, 437m, 355m, 300w 492m, 530w 264m 180br, 160s, 149s 16 3135w 3528br, 3200br 1617w, 1594w, 1576w 496m, 452w, 432m, 31m, 290w 615s br 245s br 17 3156w, 3138w 3200br 1623w, 1597w, 1576w 509m, 454m, 352w 248s 260s br 200m, 171m 18 3130w, 3110w 3380br, 3200br 1623w, 1596w, 1576w 418w, 398w, 351w, 260m, 254m, 247m, 315w, 260m, 254m, 247m, 226m, 203m, 177m 303m 19 c 3139w, 3110w 3265br 1612m, 1590m, 1575m, 1500w, 1576w, 1500w, 441w, 363m, 551s 551s 20 b.d 3147w 3230br 1622m, 1598m, 1579m 517w, 498w, 476w, 434w, 353m, 282m, 240m, 181w, 167w 567m					247m, 225m, 200m,			
15 3132w, 3060w 3350br, 3180br 1609w, 1587w, 1568w 506m, 437m, 355m, 492m, 303w, 225w 264m 180br, 160s, 149s 16 3135w 3528br, 3200br 1617w, 1594w, 1576w 496m, 452w, 432m, 351m, 311m, 290w 615s br 245s br 17 3156w, 3138w 3200br 1623w, 1597w, 1576w 509m, 454m, 352w 282s 260s br 200m, 171m 18 b 3130w, 3110w 3380br, 3200br 1623w, 1596w, 1576w 418w, 398w, 351w, 226m, 203m, 177m 325m, 315w, 226m, 203m, 177m 303m 19 c 3139w, 3110w 3265br 1612m, 1590m, 1575m, 1500w, 1576w 504m, 441m, 363m 551s 20 b.d 3147w 3230br 1622m, 1598m, 1579m 517w, 498w, 476w, 367m, 240m, 181w, 167w 567m					176m			
16 3135w 3528br, 3200br 1617w, 1594w, 1576w 303w, 225w 530w 17 3156w, 3138w 3200br 1623w, 1597w, 1576w 509m, 454m, 352w 282s 260s br 200m, 171m 18 b 3130w, 3110w 3380br, 3200br 1623w, 1596w, 1576w 418w, 398w, 351w, 282m, 245m, 315w, 224m 325m, 315w, 200m, 171m 18 b 3130w, 3110w 3265br 1612m, 1596w, 1576w 418w, 398w, 351w, 226m, 203m, 177m 303m 19 c 3139w, 3110w 3265br 1612m, 1590m, 1575m, 1500w, 1576w 504m, 441m, 363m 551s 20 b.d 3147w 3230br 1622m, 1598m, 1579m 517w, 498w, 476w, 367m 567m 20 b.d 3147w 3230br 1622m, 1598m, 1579m 517w, 498w, 476w, 367m 567m	15	3132w, 3060w	3350br, 3180br	1609w, 1587w, 1568w	506m, 437m, 355m,	492m,	264m	180br, 160s, 149s
16 3135w 3528br, 3200br 1617w, 1594w, 1576w 496m, 452w, 432m, 615s br 245s br 17 3156w, 3138w 3200br 1623w, 1597w, 1576w 509m, 454m, 352w 282s 260s br 200m, 171m 18 b 3130w, 3110w 3380br, 3200br 1623w, 1596w, 1576w 418w, 398w, 351w, 282m, 254m, 247m, 315w, 226m, 203m, 177m 303m 19 c 3139w, 3110w 3265br 1612m, 1590m, 1575m, 1500sh 504m, 441m, 363m 551s 20 b.d 3147w 3230br 1622m, 1598m, 1579m 517w, 498w, 476w, 567m 567m 20 b.d 3147w 3230br 1622m, 1598m, 1579m 517w, 498w, 476w, 567m 567m					303w, 225w	530w		
17 3156w, 3138w 3200br 1623w, 1597w, 1576w 509m, 454m, 352w 282s 260s br 200m, 171m 18 b 3130w, 3110w 3380br, 3200br 1623w, 1596w, 1576w 418w, 398w, 351w, 282m, 224m 325m, 315w, 315w, 280m, 254m, 247m, 315w, 226m, 203m, 177m 19 c 3139w, 3110w 3265br 1612m, 1590m, 1575m, 1500sh 504m, 441m, 363m 551s 20 b.d 3147w 3230br 1622m, 1598m, 1579m 517w, 498w, 476w, 567m 567m 20 b.d 3147w 3230br 1622m, 1598m, 1579m 517w, 498w, 476w, 567m 567m	16	3135w	3528br, 3200br	1617w, 1594w, 1576w	496m, 452w, 432m,	615s br	245s br	
17 3156w, 3138w 3200br 1623w, 1597w, 1576w 509m, 454m, 352w 282s 260s br 200m, 171m 18 b 3130w, 3110w 3380br, 3200br 1623w, 1596w, 1576w 418w, 398w, 351w, 224m 325m, 315w, 280m, 254m, 247m, 315w, 226m, 203m, 177m 19 c 3139w, 3110w 3265br 1612m, 1590m, 1575m, 1500w, 1576w 504m, 441m, 363m 551s 20 b.d 3147w 3230br 1622m, 1598m, 1579m 517w, 498w, 476w, 567m 567m 20 b.d 3147w 3230br 1622m, 1598m, 1579m 517w, 498w, 476w, 567m 567m 20 b.d 3147w 3230br 1622m, 1598m, 1579m 517w, 498w, 476w, 567m 567m					351m, 311m, 290w			
18 b 3130w, 3110w 3380br, 3200br 1623w, 1596w, 1576w 224m 325m, 325m, 325m, 315w, 315w, 328m, 226m, 203m, 177m, 303m 19 c 3139w, 3110w 3265br 1612m, 1590m, 1575m, 1500sh 504m, 441m, 363m, 551s 20 b.d 3147w 3230br 1622m, 1598m, 1579m 517w, 498w, 476w, 567m 20 b.d 3147w 3230br 1622m, 1598m, 1579m 517w, 498w, 476w, 567m	17	3156w, 3138w	3200br	1623w, 1597w, 1576w	509m, 454m, 352w	282s	260s br	200m, 171m
18 3130w, 3110w 3380br, 3200br 1623w, 1596w, 1576w 418w, 398w, 351w, 225m, 280m, 254m, 247m, 315w, 226m, 203m, 177m 303m 19 3139w, 3110w 3265br 1612m, 1590m, 1575m, 504m, 441m, 363m 551s 20 ^{b,d} 3147w 3230br 1622m, 1598m, 1579m 517w, 498w, 476w, 567m 434w, 353m, 282m, 240m, 181w, 167w 240m, 181w, 167w 167w	10 h			1/22 1/2/ 1/27/	224m		225	
19 ° 3139w, 3110w 3265br 1612m, 1590m, 1575m, 504m, 441m, 363m 551s 20 ^{b,d} 3147w 3230br 1622m, 1598m, 1579m 517w, 498w, 476w, 567m 434w, 353m, 282m, 240m, 181w, 167w	18 °	3130w, 3110w	3380br, 3200br	1623w, 1590w, 1570w	418w, 398w, 351w,		325m,	
19 c 3139w, 3110w 3265br 1612m, 1590m, 1575m, 504m, 441m, 363m 551s 20 b.d 3147w 3230br 1622m, 1598m, 1579m 517w, 498w, 476w, 567m 434w, 353m, 282m, 240m, 181w, 167w					280m, 254m, 247m,		315W,	
19 5159w, 5110w 52050r 101211, 1590m, 1575m, 504m, 441m, 305m 5518 20 b.d 3147w 3230br 1622m, 1598m, 1579m 517w, 498w, 476w, 567m 434w, 353m, 282m, 240m, 181w, 167w	10 \$	2120	2765h-	1612m 1500m 1575	220m, $203m$, $17/m$	551c	303m	
20 ^{b,d} 3147w 3230br 1622m, 1598m, 1579m 517w, 498w, 476w, 567m 434w, 353m, 282m, 240m, 181w, 167w	19 -	5159W, 5110W	32030F	1012III, 1390m, 1373m,	504m, 441m, 505m	5518		
434w, 353m, 282m, 240m. 181w. 167w	20 b.d	31/711	3230br	1500811 1677m 1508m 1570m	517w 108w 176m	567m		
240m. 181w. 167w	<i>4</i> 0	514/W	525001	102211, 137011, 137911	434w 353m 282m	50/11		
					240m, 181w, 167w			

^a Nujol mull and/or CHCl₃ solution. ^b ν (H₂O): 3300 cm⁻¹ br. ^c ν (ClO₄): 1100s br, 625s br.

^d $\nu(ClO_4)$: 1100s br, 622s.

Table 3					
¹ H NMR data ^a	for the li	gand and it	s tin(IV) and	organotin(IV) de	rivatives 1-20

Com- pound	$H_2 + H_5$	N-H	H aromat.	R-Sn	$J(^{119}\mathrm{Sn}^{-1}\mathrm{H})$	$J(^{117}\text{Sn}-^{1}\text{H})$	
Ľ	7.71d	7.55d	9-10br	7.19t, 7.35t, 7.84d		······	
1	7.78d	7.55d		7.21t, 7.36t, 7.82d	0.65s	67.4	64.5
2	7.71d	7.59d		7.22t, 7.35-7.50m,	7.30-7.40m, 7.40-7.50m,		
				7.75d	7.85–7.90m		
3	7.95br	7.64br	5.29br	7.25t, 7.39t, 7.82d	1.20s	91.5	87.1
4	8.12br	7.72d	6.40br	7.28t, 7.41t, 7.82d	1.48s	93.5	89.6
5	8.25d	7.78d	10.2br	7.30t, 7.42t, 7.83d	1.88s	90.8	86.7
6	7.95d	7.65d	5.52br	7.25t, 7.39t, 7.82d	1.32t, 1.77q	$75.0(^{2}J), 154.5(^{3}J)$	71.8 (² <i>J</i>), 147.5 (³ <i>J</i>)
7	8.32br	7.86br	9.51br	7.31t, 7.44t, 7.80d	1.22t, 1.94q	$87.7(^{2}J), 173.6(^{3}J)$	83.9 (² J), 165.9 (³ J)
8	8.22br	7.41br	9.5br	7.23–7.33m,	1.25t, 1.98q	56.1 (^{2}J) , 147.3 (^{3}J)	50.1 (² <i>J</i>), 140.7 (³ <i>J</i>)
				7.30-7.42m, 7.64d			
9	7.94br	7.64br	5.56br	7.24t, 7.38t, 7.82d	0.89t, 1.27–1.50m,	n.o.	n.o.
					1.65–1.90m		
10	8.34br	7.86br	8.97br	7.30t, 7.43t, 7.81d	0.81t, 1.30ps, 1.55-1.75m,	n.o.	n.o.
					1.90–2.01m		
11	8.17d	7.69d	6.52br	7.26t, 7.30t, 7.77d	7.30t, 7.26-7.36m,		
					7.96–8.01m		
12	8.30br	7.80br	6.0br	7.0–8.0m	7.0-8.0m		
13	7.92br	7.64br	7.06br	7.23t, 7.38t, 7.83d	1.20–1.50m br, 1.50–1.95m br,	n.o.	n.o.
					1.96–2.11m br, 2.35–2.60m		
14	7.86br	7.60d	4.9br	7.21t, 7.36t, 7.83d	1.20–1.55m br, 1.50–1.80m br,	n.o.	n.o.
					1.90–2.10m br, 2.36–2.51m		
15	8.47br	7.83br	4.54br	7.30t br, 7.38t br,	1.28s	131.9	125.6
				7.78d br			
16	9.0br	8.1br	6.5-8.5br	7.3–7.6m, 8.85d	0.9t, 1.3 - 1.6m, 1.8 - 2.1m	n.o.	n.o.
16 [°]	9.05br	7.52br		7.30-7.50m br,	0.99t, 1.51ps, 1.93m, 2.39t	n.o.	n.o.
17	0.11	0.21	10.01	7.000r	72.00		
17	9.1br	8.3Dr	12.3Dr	7.2-8.8m br	/.2-8.8m br		
18 `	9.010 9.065-	7.92d	5.UDr	7.4-7.6m, 7.75d	0.97.	40.2	<i>44</i> 0
19	0.20DF	/./UDF	6 5h-	7.38t, 7.40t, 7.79d	0.0/8	09.2	00.9
20	9.00 F	8.00r	0.301	1.3-1.0m, 1.83d	0.758 Dr, 0.828 Dr, 0.958 Dr	n.o.	n.o.

^a Acetone solution; δ in ppm, J in Hz; s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, ps = pseudosixtet, br = broad. ^b CdCl₃ solution.

^c CD₃OD solution.

2.1.4. Sn-N stretching frequencies

We are not able to assign the ν (Sn-N) vibrations because they are likely to be hidden under some absorptions characteristic of the azole ring system [13].

2.1.5. Skeletal bending modes

In our complexes, no skeletal bending mode assignments are straightforward: $\delta(C-Sn-C)$, $\delta(X-Sn-X)$,

 $\rho(\text{Sn-C}_3)$, $\rho(\text{SnX}_3)$ and $\nu(\text{Sn-Br})$ fall in the region 120–180 cm⁻¹ [19] and are far too close to resolve.

2.1.6. Other data

The perchlorato complexes **19** and **20** were found to be ionic; in fact a single broad absorption at ca. 1100 cm^{-1} and a sharp band at 620 cm^{-1} were observed [20].

Table -	4
---------	---

¹³C NMR data ^a for the ligand and its tin(IV) and organotin(IV) derivatives

Compound	Aromatics	Imidazole	R-Sn	$J(^{119}\text{Sn-C})$	$J(^{117}\text{Sn-C})$
L'	125.8, 127.3, 129.6, 135.9	136.9			
1	125.3, 127.1, 129.2, 134.6	115.8, 136.5, 139.3	1.09	469.1 (¹ <i>J</i>)	$446.4(^{1}J)$
2	125.5, 127.7, 129.4, 132.9	118.4, 137.4	129.2, 129.8, 137.1, 143.7	13.0 $({}^{2}J)$, 69.7 $({}^{3}J)$, 47.3 $({}^{4}J)$	12.9 (² <i>J</i>), 69.5 (³ <i>J</i>), 47.1 (⁴ <i>J</i>)
3	125.4, 127.5, 129.3, 133.6	116.7, 136.7	14.2		
6	125.5, 127.5, 129.3, 135.6	117.1, 137.0	10.0, 19.4	$683.6(^{1}J), 48.8(^{2}J)$	
7	125.6, 128.1, 129.6, 131.9	119.7, 136.0, 137.7	11.0, 33.5	744.6 (^{1}J) , 52.3 (^{2}J)	
9	125.5, 127.7, 129.4, 132.3	117.8, 137.2	13.8, 26.6, 28.1, 33.7	$669.2(^{1}J), 118.6(^{3}J)$	$638.2(^{1}J), 111.6(^{3}J)$
10	125.7, 128.2, 129.6, 131.9	119.2, 136.1, 137.6	13.8, 26.4, 40.1	746.4 (¹ <i>J</i>), 41.9 (² <i>J</i>), 136.0 (³ <i>J</i>)	
11	125.6, 128.4, 129.6, 131.4	119.5, 135.6, 137.7	128.3, 129.3, 136.2	92.3 $({}^{3}J)$, 68.0 $({}^{4}J)$	
15	125.8, 128.6, 129.7	121.7, 138.0	28.0	1196.3 (¹ <i>J</i>)	$1145.7(^{1}J)$

^a Acetone solution.

2.2. NMR data

The ¹H NMR spectra of the donor L' and of its tin(IV) and organotin(IV) complexes 1–20 in acetone solution are reported in Table 3, with the exception of 18 which was recorded in methanol due to poor solubility in the former solvent. The ¹³C NMR spectra (Table 4) were carried out only on the sufficiently soluble derivatives.

The spectra of the triorganotin(IV) complexes 1 and 2 indicate a complete dissociation into the starting reagents. In fact, the Δ (difference in chemical shift for the same type of proton in the free base and in its tin(IV) complex) value is in the range 0.01-0.07 ppm. The tin-proton and tin-carbon coupling constants observed are of the same order of magnitude as those reported for the starting triorganotin acceptors [21].

In the diorganotin(IV) complexes 3-14, upon complexation the signals of the ligand L' are generally displaced downfield. This is likely to be due to a σ -charge donation from the N-donor to the metal center and is evidence of the existence of the complex in solution. The deshielding observed is attenuated at positions remote from the metal (aromatic protons and carbons). For 3-14 the tin-proton and tin-carbon coupling constants are different from those observed for the starting organotin(IV) derivatives [22]; they are of the same order of magnitude as those observed in 1-benzylimidazole organotin derivatives [10] and smaller than those reported in the literature for undissociated *trans*octahedral diorganotin(IV) complexes [23]. This indicates that in acetone the dissociation of 3-14 is only partial and, as previously described for the organotin(IV) derivatives of 1-benzylimidazole [10], much of the complex is still present.

It is worth noting that the Δ value for the tri- and tetrahalidetin(IV) derivatives 15-18 is larger than that for the diorganotin complexes. This is likely to be due to an increase in stability of the adducts and to a stronger bonding interaction between L' and the acceptors with decreasing degree of alkylation of the metal center. This fact is in accordance with the well-known Lewis acid strength of tin(IV) and organotin(IV) acceptors [11].

In most of the ¹H spectra of our tin(IV) compounds, a broad signal appears in the range 5-12 ppm, strongly dependent on the concentration, and assignable to the N-H proton. This further supports the concept that the donor L' coordinates to tin(IV) in the neutral form through the pyridinic nitrogen.

2.3. Diffraction study of $[(L')_2(CH_3)_3 Sn]Cl]$ (1)

A drawing of the [bis(4-phenylimidazole)trimethyltin(IV)]chloride structure, from the diffraction study, is



Fig. 2. The molecular structure of [bis(4-phenylimidazole)trimethyltin(IV)]chloride with atom numbering, as used in the crystallographic work.

Table 5

Interatomic distances (A) with e.s.d.s in parentheses for {	bis(4	4-phenylimidazole)	}trimethy1-tin(IV) chloride
---	-------	--------------------	-------------------	------------

	•			
$\overline{Sn(1)-N(1a)}$	2.351(5)	Sn(1)-N(1b)	2.351(6)	
N(1a)-C(2a)	1.311(9)	N(1b)-C(2b)	1.318(9)	
N(1a)-C(5a)	1.404(8)	N(1b)-C(5b)	1.39(1)	
C(2a)-N(3a)	1.334(8)	C(2b)–N(3b)	1.345(9)	
N(3a)-C(4a)	1.391(8)	N(3b)-C(4b)	1.370(9)	
C(4a)-C(5a)	1.35(1)	C(4b)-C(5b)	1.35(1)	
C(4a)-C(6a)	1.468(9)	C(4b) - C(6b)	1.46(1)	
C(6a)-C(7a)	1.41(1)	C(6b)-C(7b)	1.39(1)	
C(6a) - C(11a)	1.39(1)	C(6b)–C(11b)	1.39(1)	
C(7a)-C(8a)	1.39(1)	C(7b)–C(8b)	1.39(1)	
C(8a)-C(9a)	1.35(1)	C(8b)-C(9b)	1.38(1)	
C(9a) - C(10a)	1.36(1)	C(9b)-C(10b)	1.35(1)	
C(10a) - C(11a)	1.39(1)	C(10b)C(11b)	1.39(1)	
Sn(1)-C(12)	2.131(8)	Sn(1) - C(13)	2.130(7)	
Sn(1) - C(14)	2.125(9)			

presented in Fig. 2 together with the numbering scheme. The bond distances and angles are listed in Tables 5 and 6, respectively. Analysis of the structure shows the tin atom pentacoordinate in a substantially regular trigonal bipyramidal configuration. The three methyls occupy the equatorial positions, and the two 4-phenylimidazole ligands the two axial sites. The chloride group is displaced from the Sn and involved in an H-bond network. It is worth noting that the two donor molecules coordinate the tin atom by means of their N(1) atoms. This is likely to be due to steric hindrance of the phenyl group in the 4-position, which requires a metal/proton exchange between the two nitrogen sites. In this structural study the occurrence of the trigonal bipyramidal geometry agrees well with the observation that in the solid state triorganotin(IV) halides seem to saturate their coordination with a coordination number equal to five

[24]. However, in this case, the unusual 2:1 stochiometry, found for imidazole- [25] and 4-phenylimidazoletriorganotin(IV) halide derivatives, is a rather unexpected result; in fact, for example, even if 4-phenylimidazole and 1-benzylimidazole have the same value of pK_a (6.10 [26]), the latter, which presents a smaller steric hindrance than the former, gives, with trimethyltin(IV) chloride, a 1:1 adduct.

No crystal structure of triorganotin(IV) compounds coordinated to two imidazole-type donors is reported in the literature, so we have compared the structure of $[(L')_2(CH_3)_3Sn]Cl]$ with those of triorganotin derivatives bonded to two N-donor ligands with a trigonal bipyramidal coordination [27–30]. The Sn–N distances in $[(L')_2(CH_3)_3Sn]Cl]$ were found to be very close to the mean value (2.36 Å calculated from six Sn–N bonds) even if, because of the different chemical nature of the

Table 6

Bond angles (deg) with e.s.d.s in parentheses for {bis(4-phenylimidazole)}trimethyl-tin(IV) chloride

$\frac{1}{8n(1)}$ N(1a) C(2a)	121 3(4)	$S_{p}(1)$ N(1b) $C(2b)$	125 8(5)	
$S_{n}(1) = N(1a) - C(2a)$ $S_{n}(1) = N(1a) - C(5a)$	121.3(4) 122.1(4)	$S_{n}(1) = N(10) - C(20)$	125.6(5)	
S(a) = N(a) - C(a)	135.1(4)	S(1) = N(10) = C(50)	120.7(3)	
C(2a) - N(1a) - C(5a)	105.3(5)	C(2b) - N(1b) - C(5b)	105.8(6)	
N(1a)-C(2a)-N(3a)	112.1(6)	N(1b)-C(2b)-N(3b)	110.7(6)	
C(2a) - N(3a) - C(4a)	107.4(5)	C(2b) - N(3b) - C(4b)	108.0(6)	
N(3a) - C(4a) - C(5a)	105.8(6)	N(3b)-C(4b)-C(5b)	105.9(7)	
N(3a) - C(4a) - C(6a)	121.6(6)	N(3b)-C(4b)-C(6b)	123.0(6)	
C(5a) - C(4a) - C(6a)	132.5(6)	C(5b) - C(4b) - C(6b)	130.8(7)	
N(1a)-C(5a)-C(4a)	109.3(6)	N(1b) - C(5b) - C(4b)	109.5(7)	
C(4a) - C(6a) - C(7a)	119.4(6)	C(4b) - C(6b) - C(7b)	119.5(7)	
C(4a) - C(6a) - C(11a)	122.0(6)	C(4b)-C(6b)-C(11b)	122.0(7)	
C(7a) - C(6a) - C(11a)	118.6(6)	C(7b)-C(6b)-C(11b)	118.5(7)	
C(6a) - C(7a) - C(8a)	119.7(8)	C(6b) - C(7b) - C(8b)	120,1(9)	
C(7a) - C(8a) - C(9a)	119.5(8)	C(7b) - C(8b) - C(9b)	120.3(9)	
C(8a) - C(9a) - C(10a)	122.4(7)	C(8b)-C(9b)-C(10b)	119.5(8)	
C(9a) - C(10a) - C(11a)	119.2(9)	C(9b) - C(10b) - C(11b)	121.2(9)	
C(6a) - C(11a) - C(10a)	120.5(8)	C(6b)-C(11b)-C(10b)	120.3(8)	
N(1a) - Sn(1) - C(12)	89.8(3)	N(1b) - Sn(1) - C(12)	87.2(3)	
N(1a) - Sn(1) - C(13)	92.3(3)	N(1b)-Sn(1)-C(13)	91.6(3)	
N(1a) - Sn(1) - C(14)	88.2(3)	N(1b)-Sn(1)-C(14)	91.1(3)	
C(12)-Sn(1)-C(13)	120.8(4)	C(12)-Sn(1)-C(14)	122.3(4)	
C(13) - Sn(1) - C(14)	116.9(4)	N(1a)-Sn(1)-N(1b)	176.0(2)	

ligands, values ranging from 2.472 Å [27] to 2.29 Å [28] are reported.

We have also compared the Sn-N bond distance found in 1 with those reported in the literature for other tin(IV) imidazole adducts [31–36], without taking into account their coordination numbers and chemical nature. Also in these cases the mean value (2.347 \AA) is very close to the one we found, the Sn-N bond lengths ranging from 2.29 to 2.41 Å. The spread of the Sn-N bond lengths in the imidazole-Sn interaction seems to be due to steric effects. It was noted [33], in fact, that the longer bond distances for the $[(ImH)_2R_2SnCl_2]$ (where ImH is a generic imidazole ligand) have been found when the imidazole is substituted in position 2 [33] (our numbering scheme), whereas substitution in positions 3 or 4 seems to produce a lesser effect [31] compared with unsubstituted imidazole adducts [32]. The Sn-N distance is longer than those reported for non-sterically hindered bis(imidazole)-diorganotin complexes. This fact indicates that an increase in the degree of tin acceptor alkylation produces a weaker bonding interaction and so a decrease in stability of the adducts.

The Sn-C distances in **1** are in good agreement with those reported in the literature for trimethyltin(IV) compounds [29].

The geometry of the two imidazole rings is not significantly different, with the exception of the internal angle on C(2) and the bond length between N(3) and

C(4). The differences are, however, less than three times their e.s.d.s.

The torsion angles of the two imidazole rings with the bonded phenyl rings are 6.1° for ring A and 18.0° for ring B. The molecular parameters of the azole ring in 1 agree with those reported in the literature; the conjugation effect of phenyl on the ring geometry seems to be negligible, at least at this level of precision, even if the distance C(4)–C(6) is shorter than the value (1.488 Å) reported from CSD analysis [37] on a sample of 87 C(sp²)–C(arylic) bond lengths.

The most interesting feature of this structure is the position of the Cl atom. In the solid state, the Cl is displaced from the Sn too far (4.564 Å) for any kind of bonding interaction. The Cl and the two N-H are involved in a hydrogen bond network, which connects each molecule of the adduct with another one related by the $(\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z)$ symmetry operation. They thus define a chain extending along the y direction (Fig. 3) which stabilizes the entire crystal packing. The N · · · Cl distances, which are 3.098(6) and 3.147(6) Å for N(3a) and N(3b) respectively, are below the sum of the Van Der Waals radii (3.3 Å) indicated by Hamilton and Ibers [38]. The angle values found for $N(3a) \cdots$ $H(3a) \cdots Cl(1)$ and $N(3b) \cdots H(3b) \cdots Cl(1)$ are 164 and 170°, which are in good agreement with the geometrical requirement for the H-bonds. The angle $N(3a) \cdots Cl(1) \cdots N(3b)$ is 82° .



Fig. 3. Projection of the unit cell.

C. Pettinari et al. / Journal of Organometallic Chemistry 515 (1996) 119-130

3. Experimental details

3.1. General comments

The tin(IV) and organotin(IV) halides were purchased from Alfa (Karlsruhe) and Aldrich (Milwaukee) and used as received. The ligand 4-phenylimidazole (L')was obtained from Aldrich and was crystallized from diethyl ether/petroleum ether (1:2).

The samples for microanalysis were dried in vacuo to constant weight (20°C, ca. 0.1 Torr). Elemental analyses (C, H, N) were performed in-house with a Carlo-Erba model 1106 instrument. IR spectra were recorded from 4000 to 100 cm⁻¹ with a Perkin-Elmer System 2000 FT-IR instrument. ¹H and ¹³C NMR spectra were recorded on a VXR-300 Varian spectrometer operating at room temperature (300 MHz for ¹H, 75 MHZ for ¹³C). Melting points were taken on an IA 8100 electrothermal instrument. The electrical conductance of the solutions was measured with a Crison CDTM 522 conductimeter at room temperature.

3.2. Synthesis of the complexes

3.2.1. [Bis(4-phenylimidazole)trimethyltin(IV)]chloro (1)

To a stirred diethyl ether solution (100 cm³) of (CH₃)₃SnCl (548 mg, 2.8 mmol) at room temperature, a diethyl ether solution (25 cm³) of 4-phenylimidazole (L') (800 mg, 5.5 mmol) was added. A colorless precipitate was formed immediately, which was filtered off after 10 h, washed with diethyl ether and shown to be compound 1. Adducts 3, 4, 6, 7, 9, 11, 13, 15, and 18 were obtained similarly.

3.2.2. [Bis(4-phenylimidazole)triphenyltin(IV)]chloro (2)

To a stirred diethyl ether solution (10 cm^3) of $(C_6H_5)_3$ SnCl (560 mg, 1.45 mmol), a diethyl ether/benzene (1:2) solution (100 cm^3) of 4-phenylimidazole (L') (488 mg, 3.4 mmol) was added under N, stream. The mixture was heated at 40°C and stirred for 1 day. It was then allowed to cool and evaporated under reduced pressure. The colorless residue was crystallized twice from diethyl ether to yield the analytical sample 2. Compound 16 was obtained similarly.

Table 7

Crystal data: data collection and refinement of the structure for {bis(4-nhenylimidazole)}trimethyl tip(IV) chloride

Formula	C ₂₁ H ₂₅ N ₄ ClSn	
Formula weight	487.598	
Space group	C2/c	
<i>a</i> (Å)	19.474(1)	
<i>b</i> (Å)	14.461(1)	
<i>c</i> (Å)	15.989(1)	
β (deg)	98.53(1)	
V_{c} (Å ³)	4508.3(6)	
Z	8	
$D_{\rm c} ({\rm g}{\rm cm}^{-3})$	1.437	
μ (Mo K α) (cm ⁻¹)	104.35	
F(000)	1968	
Radiation (monochromated)	Cu K α ($\lambda = 1.5406$ Å)	
Temperature of data collection (K)	293	
Scan mode	$\omega/2\theta$	
Scan width (deg)	$\Delta \omega = A + B * \tan(\theta), A = 1.365^{\circ}, B = 0.32^{\circ}$	
Scan speed max. (deg min ^{-1})	32	
Background/scan ratio	0.5	
Data collection range (deg)	$5 \le 2\theta \le 124$	
Standards (measured every 97 reflections)	0 - 2 3, $-1 - 3$ 2, 3 1 2	
No. of unique reflections measured	3703	
No. of data with $l \ge 3\sigma(1)$	2578	
Refinement	Full-matrix least-squares on F	
No. of parameters refined	244	
R ^a	0.051	
R _w ^b	0.058	
S ^c (Goodness of fit)	1.21	

^a $R = (\Sigma || F_o || k || F_c ||) / \Sigma || F_o |.$ ^b $R_w = [\Sigma w(|F_o | -k || F_c |)^2 / \Sigma w || F_o |^2]^{1/2}.$

^c $S = [\Sigma w(|F_0| - k|F_c|)^2 / (N_{obs} - N_{par})]^{1/2}$

3.2.3. [Bis(4-phenylimidazole)dimethyldi-iodotin(IV))](5)

A dry THF solution of compound **3** (508 mg, 1.0 mmol) was introduced into a 250 cm³ round-bottomed flask fitted with a condenser. Sodium iodide (600 mg, 4.0 mmol) was then added. The mixture was heated at reflux, under N₂ stream, with stirring for 1 day. It was then allowed to cool and was filtered off. The solvent was removed with a rotary evaporator and the residue was extracted with $CH_2Cl_2(3 \times 15 \text{ cm}^3)$; the organic layer was dried on Na_2SO_4 . It was then filtered and concentrated under reduced pressure. Et₂O (30 cm³) was then added; the solution was left in a freezer for 1 day. A yellow precipitate was formed which was filtered off, washed with Et₂O and crystallized twice from CH_2Cl_2/Et_2O (1:2) to yield the analytical sample **5**. Compounds **8**, **12**, **14**, and **19** were obtained similarly.

3.2.4. [Bis(4-phenylimidazole)dibuthyldibromotin(IV)] (10)

 Bu_2SnBr_2 (166 mg, 0.4 mmol) was added at room temperature to a stirred dichloromethane solution (100 cm³) of the ligand L' (290 mg, 2.0 mmol). After 12 h, the solvent was removed with a rotary evaporator and the residue was crystallized twice from diethyl ether to yield the analytical sample 10.

3.2.5. [Tris(4-phenylimidazole)(phenyltrichlorotin(IV)) \cdot H_2O] (17)

PhSnCl₃ (500 mg, 1.8 mmol) was added at room temperature to a stirred saturated diethyl ether solution (110 cm³) of the ligand L' (145 mg, 1.0 mmol). A colorless precipitate was formed immediately. After 5 min, the suspension was centrifuged, and the residue washed with diethyl ether to yield the analytical sample 17.

3.2.6. [Tris(4-phenylimidazole)bis(dimethyltin(IV)diperchlorate)] (20)

An ethanol solution (100 cm^3) of compound **3** (330 mg, 0.6 mmol) was introduced into a 250 cm³ roundbottomed flask fitted with a condenser. Sodium perchlorate (365 mg, 2.6 mmol) was then added. The mixture was heated at reflux, under N₂ stream, with stirring for 1 day. It was then allowed to cool and was filtered off. The solvent was removed with a rotary evaporator and the residue was extracted with CH₂Cl₂ (3 × 15 cm³); the organic layer was dried on Na₂SO₄. It was then

Table 8

Atomic coordinates and isotropic equivalent thermal parameters (with e.s.d.s in parentheses) for {bis(4-phenylimidazole)}trimethyl-tin(IV) chloride

Atom	<i>x</i>	у	Z	$U_{\rm eq}^{\rm a}$ (Å ²)	-
Sn(1)	0.18991(2)	0.06099(3)	0.27661(3)	0.0463(2)	-
Cl(1)	0.16914(8)	0.2693(1)	-0.1238(1)	0.0471(5)	
N(1a)	0.1273(3)	0.1013(4)	0.1446(4)	0.051(2)	
C(2a)	0.1559(3)	0.1493(5)	0.0898(4)	0.054(2)	
N(3a)	0.1103(3)	0.1724(4)	0.0225(3)	0.050(2)	
C(4a)	0.0463(3)	0.1359(5)	0.0340(4)	0.044(2)	
C(5a)	0.0573(3)	0.0919(5)	0.1089(5)	0.051(2)	
C(6a)	-0.0165(3)	0.1517(4)	-0.0274(4)	0.045(2)	
C(7a)	-0.0812(4)	0.1233(7)	-0.0078(6)	0.071(4)	
C(8a)	-0.1412(4)	0.1398(7)	-0.0649(7)	0.081(4)	
C(9a)	-0.1366(5)	0.1844(7)	-0.1380(6)	0.077(4)	
C(10a)	-0.0751(4)	0.2143(6)	-0.1584(6)	0.071(3)	
C(11a)	-0.0144(4)	0.1974(6)	-0.1033(5)	0.058(3)	
N(1b)	0.2595(3)	0.0246(5)	0.4053(4)	0.057(2)	
C(2b)	0.2638(4)	-0.0561(5)	0.4421(4)	0.051(2)	
N(3b)	0.3197(3)	-0.0604(4)	0.5025(3)	0.049(2)	
C(4b)	0.3526(4)	0.0225(5)	0.5057(4)	0.049(2)	
C(5b)	0.3153(4)	0.0746(5)	0.4456(5)	0.059(3)	
C(6b)	0.4182(4)	0.0405(5)	0.5605(4)	0.049(3)	
C(7b)	0.4412(5)	0.1302(6)	0.5735(6)	0.077(3)	
C(8b)	0.5041(5)	0.1482(8)	0.6245(7)	0.092(4)	
C(9b)	0.5452(5)	0.0772(8)	0.6602(6)	0.084(4)	
C(10b)	0.5228(4)	- 0.0099(8)	0.6477(6)	0.076(3)	
C(11b)	0.4599(4)	- 0.0296(6)	0.5983(6)	0.068(3)	
C(12)	0.2715(4)	0.0090(6)	0.2149(5)	0.067(3)	
C(13)	0.1089(4)	-0.0261(6)	0.3050(5)	0.061(3)	
C(14)	0.1852(5)	0.2001(6)	0.3132(7)	0.079(4)	

^a U_{eq} is defined as the mean of the principal axes of the thermal ellipsoid.

filtered and concentrated under reduced pressure. Et_2O (30 cm³) was then added. A colorless precipitate was formed which was filtered off, washed with diethyl ether, and shown to be compound **20**.

3.3. X-ray structure determination of [Bis(4-phenylimidazole)trimethyltin(IV)]chloro (1)

A summary of the experimental conditions is reported in Table 7. Atomic coordinates and isotropic equivalent thermal parameters are given in Table 8. Crystals of compound 1 were grown from a dichloromethane/diethyl ether solution by slow evaporation. A colorless crystal of approximate dimension $0.38 \times 0.5 \times 0.31 \text{ mm}^3$ was mounted on a Rigaku AFC5 automatic four-circle rotating diffractometer (45 kV, 100 m A). The crystal system was found to be monoclinic, and accurate cell parameters were obtained by least-squares refinement of 40 reflections with $40^\circ \le 2\theta \le 80^\circ$; the space group was found to be C2/c.

A total of 3910 reflections were collected with $0 \le h \le 22$, $0 \le k \le 17$ and $-18 \le l \le 18$; no decay was observed during the data collection. The ψ -scan of three different reflections (-2 4 3, 0 2 3, 0 2 2) gave a minimum and maximum transmission factor equal to 0.3 and 1.0; the data were then corrected for the absorption by using a semi-empirical method [39].

The structure was solved by direct methods using the siR92 program [40], all the non-hydrogen atoms in the asymmetric unit were found at this level.

A first isotropic refinement of all non-hydrogen atoms was performed using the CRYSTALS package [41], giving R = 0.123, and a successive anisotropic refinement of the same atoms lowered the value to R = 0.060. At this stage we were able to locate the hydrogens on the imidazoles by means of a Fourier difference map; all the other hydrogens were imposed by the model with d = 1.06 Å. The structure was then refined, with the hydrogens refined as riding (U_{iso} fixed at 20% greater than the bonded atoms), giving R = 0.055. A final refinement was performed after optimization of the weighting scheme [42–44] using $w^{-1} = P(F_c/F_c^{Max}) \times$ $\{1 - (|F_0 - F_c|/[6 \times |F_0 - F_c|_{est}])^2\}^2$, where $P(F_c/F_c^{Max})$ is a third order optimized truncated Cheybishev polynomial with coefficients $a_1 = 7.56$, $a_2 =$ -2.18, $a_3 = 5.84$, and $|F_0 - F_c|_{est}$ is estimated by using the polynomial to fit $|F_0 - F_c|$ against F_c .

The *R* factor was equal to 0.051 at the end of the last refinement cycle, with a maximum r.m.s. shift equal to 0.01. A difference Fourier gave minimum and maximum peaks equal to -1.41 and $0.31 e \text{ Å}^3$ near the Sn atom. All refinements were carried out taking into account the anomalous scattering contributions but without refining the extinction parameter.

The scattering factors and anomalous contributions were taken from the International Tables for Crystal-

lography [45]. All calculations were carried out on a personal computer; the program PARST [46] was used for some geometrical calculations.

4. Supplementary material available

Tables of anisotropic thermal parameters for non-hydrogen atoms, tables of the least-squares planes, tables of hydrogen atom parameters, as well as tables of structure factors have been deposited at the Cambridge Crystallographic Data Centre.

Acknowledgements

Financial support from the Ministero dell'Università e della Ricerca Scientifica e Tecnologica (MURST) and the Consiglio Nazionale delle Ricerche C.N.R., Rome is acknowledged.

References

- A.J. Crowe and P.J. Smith, *Chem. Ind.*, (1980) 200; A.J. Crowe, P.J. Smith and G. Atassi, *Chem. Biol. Interact.*, 32 (1980) 171.
- M. Gielen (ed.), *Tin-Based Antitumor Drugs*, NATO ASI Ser. H, Cell Biology, Vol. 37, Brussels, 1989.
- [3] G. Ruisi, A. Silvestri, M.T.L. Giudice, R. Barbieri, G. Atassi, F. Huber, K. Gräk and L. Lamartine, J. Inorg. Biochem., 25 (1985) 229.
- [4] A.J. Crowe, P.J. Smith, C.J. Cardin, H.E. Parge and F.E. Smith, Cancer Lett., 24 (1984) 45.
- [5] H. Höpf and P. Köpf Maier, ACS Symp. Ser., (1983) 209.
- [6] V. Narayanan, M. Nasr and K.D. Paull, in M. Gielen (ed.), *Tin-Based Antitumour Drugs*, Springer, Berlin, 1990, p. 201.
- [7] F. Caruso, M. Bol-Schoenmakers and A.H. Penninks, J. Med. Chem., 36 (1993) 1168.
- [8] C.J. Cardin and A. Roy, Inorg. Chim. Acta, 107 (1985) 57.
- [9] C.J. Cardin and A. Roy, Inorg. Chim. Acta, 125 (1986) 63.
- [10] C. Pettinari, F. Marchetti, A. Cingolani and S. Bartolini, *Polyhedron*, in press.
- [11] P.G. Harrison, Compounds of tin: general trends, in P.G. Harrison (ed.), *Chemistry of Tin*, Chapman and Hall, London, 1989, Chapter 2, pp. 9–59.
- [12] N. Ohkaku and K. Nakamoto, *Inorg. Chem.*, 12 (1993) 2440;
 I.R. Beattie and G.P. McQuillan, J. Chem. Soc., (1963) 1519.
- [13] G. Nieuwpoort, J.G. Vos and W.L. Groeneveld, Inorg. Chim. Acta, 29 (1978) 117.
- [14] R. Graziani, U. Casellato, R. Ettorre and G. Plazzogna, J. Chem. Soc., Dalton Trans., (1982) 805; G. Valle, R. Ettorre, V. Peruzzo and G. Plazzogna, J. Organomet. Chem., 326 (1987) 169.
- [15] R.C. Poller, The Chemistry of Organotin Compounds, Logos, London, 1970; W.P. Newman, The Organic Chemistry of Tin, Wiley Interscience, New York, 1970; B.V.K. Ho and J.J. Zuckerman, Inorg. Chem., 12 (1973) 1552; F. Huber, M. Vornefeld, G. Ruisi and R. Barbieri, App. Organomet. Chem., 7 (1993) 243.
- [16] W.F. Edgell and C.H. Ward, J. Mol. Spectrosc., 8 (1962) 343;
 J.K. Sandhu, G. Kaur, J. Holecek and A. Licka, J. Organomet. Chem., 345 (1988) 51.

- [17] J.R. May, W.R. McWhinnie and R.C. Poller, Spectrochim. Acta, 27A (1971) 969; A.L. Smith, Spectrochim. Acta, 24A (1967) 695; M.S. Dance, W.R. McWhinnie and R.C. Poller, J. Chem. Soc., Dalton Trans., (1976) 2349.
- [18] T.S. Basu Baul, D. Dey, D.D. Mishra, W.L. Basaiawmoit and E. Rivarola, J. Organomet. Chem., 447 (1993) 9.
- [19] R.J.H. Clark, A.G. Davies and R.J. Puddephatt, J. Chem. Soc. A, (1968) 1828.
- [20] L.E. Moore, R.B. Gayhart and W.E. Bull, J. Inorg. Nucl. Chem., 26 (1964) 896.
- [21] B. Wrackmeyer, Ann. Rep. NMR Spectrosc., 16 (1985) 73.
- [22] P.G. Harrison, Investigating tin using spectroscopy, in P.G. Harrison (ed.), *Chemistry of Tin*, Chapman and Hall, London, 1989, Chapter 3, pp. 61-115.
- [23] W.D. Honnick, M.C. Hughes, C.D. Schaeffer, Jr. and J.J. Zuckerman, *Inorg. Chem.*, 15 (1976) 1391 and references cited therein; T.P. Lockhart and W.F. Manders, *Inorg. Chem.*, 25 (1986) 892.
- [24] J.T.B.H. Jastrzebski and G. Van Koten, in Advances in Organometallic Chemistry, Vol. 35, 1993.
- [25] C. Pettinari, unpublished results, 1995.
- [26] M.R. Grimmett, Imidazoles and their benzo derivatives: (i) structure, in A.R. Katritzky, C.W. Rees and K.T. Potts (eds.), *Comprehensive Heterocyclic Chemistry*, Vol. 5, Part 4A, Pergamon Press, Oxford, 1984, p. 345.
- [27] W.A. Nugent, R.J. McKinney and R.L. Harlow, Organometallics, 3 (1984) 1315.
- [28] J.P. Charland, E.J. Gabe, L.E. Khoo and F.E. Smith, *Polyhe*dron, 8 (1989) 1897.
- [29] A. Blaschette, I. Hoppel, J. Krahl, E. Wieland, P.G. Jones and A. Sebold, J. Organomet. Chem., 437 (1992) 279.
- [30] G. Van Koten, J.T.B.H. Jastrzebski, J.G. Noltes, A.L. Spek and J.C. Schhone, J. Organomet. Chem., 148 (1978) 233.
- [31] R. Bardi, A. Piazzesi, R. Ettorre and G. Plazzogna, J. Organomet. Chem., 270 (1984) 171.

- [32] E. Garcia Martinez, A. Sanchez Gonzalez, A. Macias, M.V. Castano, J.S. Casa and J. Sordo, J. Organomet. Chem., 385 (1990) 329.
- [33] U. Casellato, R. Graziani and A. Sanchez Gonzalez, Acta Crystallogr., C48 (1992) 2125.
- [34] G. de Sousa, C.A.L. Figueiras, M.Y. Darensbourg and J.H. Reibenspies, *Inorg. Chem.*, 31 (1992) 3044.
- [35] G. Bandoli, A. Dolmella, V. Peruzzo and G. Plazzogna, J. Organomet. Chem., 452 (1993) 47.
- [36] C. Lopez, A. Sanchez Gonzalez, E. Garcia Martinez, J.S. Casas, J. Sordo, R. Graziani and U. Casellato, J. Organomet. Chem., 434 (1992) 261.
- [37] F.H. Allen, O. Kennard, D.G. Watson, L. Brammer, A.G. Orpen and R. Taylor, J. Chem. Soc., Perkin Trans. II, (1987) S1.
- [38] W.C. Hamilton and J.A. Ibers, in W.A. Benjamin (ed.), "Hydrogen Bonding in Solids", New York, 1968.
- [39] A.C.T. North, D.C. Phillips, F.S. Mathews, Acta Crystallogr., A24 (1968) 351.
- [40] A. Altomare, G. Cascarano, C. Giacovazzo and A. Guagliardi, J. Appl. Cryst., 26 (1993) 343.
- [41] D.J. Watkin, J.R. Carruthers and P.W. Bettridge, CRYSTALS User Guide, Chemical Crystallography Laboratory, University of Oxford, Oxford, 1985.
- [42] J.R Carruthers and D.J. Watkin, Acta Crystallogr., A35 (1979) 698.
- [43] D.J. Watkin, Acta Crystallogr., A50 (1994) 411.
- [44] E. Prince and P.T. Boggs, in A.J.C. Wilson (ed.), *International Tables for Crystallography*, Vol. C, Kluwer Academic, Dordrecht, 1992.
- [45] A.J.C. Wilson (ed.), International Tables for Crystallography, Vol. C, Kluwer Academic, Dordrecht, 1992.
- [46] M. Nardelli, Comput. Chem., 7 (1983) 95.