Journal of Organometallic Chemistry, 375 (1989) 173–181
Elsevier Sequoia S.A., Lausanne – Printed in The Netherlands
JOM 20117

Synthesis and structure of pentacoordinate tin(IV) complexes

Ji-tao Wang, Feng-Quan Liu, Yun-wen Zhang,

(Department of Chemistry, Nankai University, Tianjin (China)

Ru-ji Wang, Hong-Gen Wang, and Xin-Kan Yao

Central Laboratory, Nankai University, Tianjin (China)

(Received April 10th, 1989)

Abstract

Ten complexes formulated as Ph_2SnL, where L is $RC_6H_4C(O)N_2CHC_6H_4O$ with R = H, *p*-NO_2, *p*-Cl, *p*-Br, *p*-CH_3, *p*-OCH_3, *o*-NH_2, *m*-NO_2, *m*-OCH_3, and 3,5-dinitro, have been prepared and were characterized by ^1H NMR, IR, and mass spectroscopy and elemental analyses. A single crystal X-ray diffraction study of the complex with R=*o*-NH_2 confirms the molecular structure. The crystals are triclinic, space group $P\bar{1}$ with a 9.608(2), b 11.826(4), c 21.478(6) Å, α 76.17(2), β 76.53(2), γ 88.91(2)°, V 2302.7(4) Å^3, $Z = 4$, D_c 1.52 g/cm^3. The final discrepancy factors are $R = 0.050$ and $R_w = 0.057$ for 3129 observed independent reflections. The tin atom has a distorted trigonal bipyramidal coordination. The two Sn–O bonds of 2.073(6) and 2.116(6) Å, respectively and one Sn–N bond (of 2.143(7) Å) * are shorter than those found in related complexes. A comparison of the IR spectra of the ligands with those of the corresponding complexes, reveals that the disappearance of the bands assigned to carbonyl unambiguously confirms that the ligands coordinate with the tin in the enol form.

Introduction

In a previous paper [1] we described the substitution reactions of dichlorobis[(π-cyclopentadienyl)dicarbonyliron]tin(IV) with *o*-(substituted α-hydroxybenzylidenehydrazonomethyl)phenols. The X-ray and spectroscopic studies showed that in the substitution products the o-(substituted α-hydroxybenzylidenehydrazonomethyl)phenols are tridentate planar ligands and exist in the enol-form. As a continuation of these studies we have now obtained a different type of tin(IV)

---

* The bond distances and angles quoted in the text all refer to the averaged values of two independent molecules.

0022-328X/89/$03.50 © 1989 Elsevier Sequoia S.A.

complex, from the substitution reactions of dichlorodiphenyltin with the same ligands, in order to examine the effects by various substituents attached to the tin atom on the coordination bonds between the tin atom and the ligands. Ten products formulated as Ph_2SnL, where L is $RC_6H_4C(O)N_2CHC_6H_4O$ (R = H, p-NO_2, p-Cl, p-Br, p-CH_3, p-OCH_3, o-NH_2, m-NO_2, m-OCH_3, 3,5-dinitro), have been synthesized and characterized by ^1H NMR, IR, and mass spectroscopy and by elemental analyses. A single crystal X-ray diffraction study (R = o-NH_2) has confirmed the compositions of these complexes and revealed that shorter Sn–N and Sn–O bonds are formed between the central tin atom and the ligand when two phenyl groups replace the two π-cyclopentadienyldicarbonyliron groups in these complexes.

Results and discussion

The analytical data, color, melting points, and the yields of the various Ph_2SnL are listed in Table 1. Triethylamine was used to remove the hydrogen chloride formed in these substitutions. It should be noted that, in order to prepare Ph_2SnL, the ligands and triethylamine must be mixed first, only then can the dichlorodiphenyltin in benzene solution be added dropwise to it. If the ligands and dichlorodiphenyltin are mixed first and triethylamine is then added to the solution, the substitution reactions do not proceed successfully.

A remarkable difference between the IR spectra of the ligands and those of the corresponding complexes is that the stretching vibration bands of carbonyl disappear from the spectra of the complexes. The disappearance of the bands assigned to carbonyls unambiguously confirms that the ligands coordinated with the tin are in the enol form. The characteristic absorptions at 1603–1611 and 1590–1599 cm^{-1} (see Table 2) in the spectra of these complexes indicate the presence of $>C=N-$ and $>C=N-N=C<$ groups [2]. A strong peak at 1073 cm^{-1} is characteristic of the Sn–Ph group [3].

Figure 1 is a perspective drawing of $Ph_2Sn[OC_6H_4CHN_2C(O)C_6H_4NH_2]$, along with the adopted labelling scheme. The final fractional coordinates of nonhydrogen atoms with equivalent isotropic thermal parameters for this complex are listed in Table 3. The crystals of this complex consist of discrete molecules. There are two

Table 1

Physical constants and analytical data of the complexes $Ph_2Sn[OC_6H_4CHN_2C(O)C_6H_4R]$

R	Mol. Wt.	Color	mp °C	Yield %	Analysis (Found (calc.) (%)		
					C	H	N
H	512	pale-yellow	165–167	91	60.92(61.06)	3.98(3.91)	5.60(5.48)
p-NO_2	557	pale-yellow	207–209	90	56.04(56.11)	3.47(3.42)	7.29(7.55)
p-Cl	546	yellow	167–169	90	57.11(57.14)	3.47(3.48)	5.11(5.13)
p-Br	590	pale-yellow	179–181	87	52.96(52.88)	3.17(3.22)	4.72(4.75)
p-CH_3	526	pale-yellow	175–177	92	61.78(61.71)	4.27(4.19)	5.26(5.33)
p-OCH_3	542	pale-yellow	199–201	86	60.05(59.89)	4.03(4.07)	5.11(5.27)
o-NH_2	527	dark-yellow	171–173	83	59.18(59.32)	4.06(3.99)	8.02(7.98)
m-NO_2	557	yellow	234–236	86	56.14(56.11)	3.48(3.42)	7.45(7.55)
m-OCH_3	542	pale-yellow	148–150	90	60.02(59.89)	4.11(4.07)	5.09(5.27)
3,5-dinitro	602	dark-yellow	274–276	85	51.97(51.91)	2.97(3.00)	9.38(9.32)

Table 2

Characteristics IR bands and MS data of the complexes $Ph_2Sn[OC_6H_4CHN_2C(O)C_6H_4R]$

R	IR bands (cm^{-1})					MS data: *m/e* (relative intensity)				
	ν(C=N)	(ν(C=N-N=C)	ν(Sn-Ph)	ν(Sn-O)	ν(Sn←N)	$M^{\cdot+}$	$M^{\cdot+}$ - Ph	$M^{\cdot+}$ - 2Ph	$PhSnOPhCHN^{\cdot+}$	$SnPh^{\cdot+}$
H	1609s	1597s	1073m	572m	478w	512(100)	435(11)	358(5)	316(8)	197(28)
p-NO_2	1611s	1592s	1073m	572m	478w	557(100)	480(15)	403(7)	316(10)	197(41)
p-Cl	1609s	1591m	1073m	580m	480w	546(100)	469(12)	392(7)	316(7)	197(27)
p-Br	1606s	1590m	1073m	564m	480w	590(43)	513(5)	436(3)	316(4)	197(23)
p-CH_3	1605s	-	1073m	570m	480w	526(100)	449(10)	372(3)	316(7)	197(20)
p-OCH_3	1609s	1592m	1073m	572m	478w	542(100)	465(15)	388(5)	316(10)	197(20)
o-NH_2	1603s	1599m	1073m	580m	480w	527(92)	450(4)	373(11)	316(8)	197(30)
m-NO_2	1608s	-	1073m	571m	478w	557(66)	480(15)	403(3)	316(9)	197(21)
m-OCH_3	1609s	1592s	1073m	572m	480w	542(66)	465(9)	388(3)	316(7)	197(14)
3,5-dinitro	1609s	-	1073m	572m	481w	602(100)	525(11)	448(5)	316(5)	197(37)

Table 3

Positional parameters and equivalent isotropic thermal parameters for $Ph_2Sn[NH_2C_6H_4C(O)N_2\text{-}CHC_6H_4O]$

Atom	x	y	z	B_{eq} (Å^2)
Sn(1)	0.7650(1)	0.26635(8)	0.49189(5)	6.03(3)
O(1)	0.948(1)	0.2621(8)	0.4189(5)	8.1(3)
O(2)	0.579(1)	0.2046(7)	0.5672(5)	7.0(3)
C(11)	1.019(2)	0.173(1)	0.3968(6)	6.7(4)
C(12)	1.156(1)	0.204(1)	0.3512(7)	7.0(4)
C(13)	1.230(2)	0.120(1)	0.3235(7)	8.1(5)
C(14)	1.167(2)	0.004(1)	0.3409(8)	8.1(5)
C(15)	1.034(1)	−0.026(1)	0.3872(7)	7.0(4)
C(16)	0.961(1)	0.062(1)	0.4157(6)	6.0(3)
C(17)	0.830(1)	0.022(1)	0.4635(6)	5.7(4)
N(1)	0.749(1)	0.0824(8)	0.4960(5)	5.1(3)
N(2)	0.630(1)	0.0250(9)	0.5413(5)	5.9(3)
C(1)	0.552(1)	0.091(1)	0.5750(6)	6.3(4)
C(21)	0.420(1)	0.047(1)	0.6276(6)	6.7(4)
C(22)	0.384(1)	−0.078(1)	0.6468(6)	6.3(3)
C(23)	0.257(2)	−0.109(1)	0.6976(7)	7.6(4)
C(24)	0.175(2)	−0.032(1)	0.7275(8)	8.6(5)
C(25)	0.211(2)	0.086(2)	0.7078(8)	8.7(5)
C(26)	0.335(2)	0.126(1)	0.6577(7)	7.2(4)
N(22)	0.458(1)	−0.159(1)	0.6201(6)	8.1(4)
C(31)	0.875(2)	0.315(1)	0.5569(7)	6.3(4)
C(32)	0.801(2)	0.306(1)	0.6219(7)	6.6(4)
C(33)	0.881(2)	0.334(1)	0.6653(8)	8.8(5)
C(34)	1.029(2)	0.370(1)	0.6433(8)	9.0(5)
C(35)	1.097(2)	0.375(1)	0.5777(8)	8.4(5)
C(36)	1.026(1)	0.349(1)	0.5301(8)	7.3(4)
C(41)	0.651(1)	0.378(1)	0.4296(7)	5.8(4)
C(42)	0.722(1)	0.422(1)	0.3616(7)	6.7(4)
C(43)	0.641(2)	0.492(1)	0.3198(7)	7.3(4)
C(44)	0.497(2)	0.510(1)	0.3440(7)	7.7(4)
C(45)	0.430(2)	0.463(1)	0.4090(7)	8.2(5)
C(46)	0.508(2)	0.394(1)	0.4527(7)	7.0(4)
Sn(2)	0.22851(9)	0.27645(7)	0.94592(4)	4.57(2)
O(3)	0.152(1)	0.3556(8)	0.8642(4)	6.8(3)
O(4)	0.3266(8)	0.2678(6)	1.0252(4)	4.9(2)
C(51)	0.089(1)	0.458(1)	0.8504(6)	4.9(3)
C(52)	0.005(1)	0.466(1)	0.8015(6)	6.7(4)
C(53)	−0.056(1)	0.574(1)	0.7825(7)	6.9(4)
C(54)	−0.039(2)	0.669(1)	0.8094(7)	7.2(4)
C(55)	0.039(1)	0.654(1)	0.8582(7)	6.1(4)
C(56)	0.103(1)	0.549(1)	0.8785(6)	4.6(3)
C(57)	0.176(1)	0.545(1)	0.9298(6)	5.1(3)
N(3)	0.2279(9)	0.4483(8)	0.9598(4)	4.4(2)
N(4)	0.290(1)	0.4652(8)	1.0105(5)	4.6(3)
C(2)	0.334(1)	0.368(1)	1.0408(5)	4.4(3)
C(61)	0.407(1)	0.365(1)	1.0966(5)	4.2(3)
C(62)	0.416(1)	0.465(1)	1.1205(6)	5.0(3)
C(63)	0.481(1)	0.452(1)	1.1748(7)	6.5(4)
C(64)	0.531(2)	0.345(1)	1.2021(7)	7.2(4)
C(65)	0.524(1)	0.245(1)	1.1772(6)	7.1(4)
C(66)	0.459(1)	0.256(1)	1.1254(6)	5.6(4)
N(62)	0.368(1)	0.5729(9)	1.0950(5)	6.2(3)

Table 3

Atom	x	y	z	B_{eq} (Å^2)
C(71)	0.404(1)	0.201(1)	0.8913(6)	5.1(3)
C(72)	0.503(1)	0.142(1)	0.9236(7)	6.5(4)
C(73)	0.615(2)	0.087(1)	0.8884(8)	8.8(5)
C(74)	0.625(2)	0.089(2)	0.8221(9)	9.1(5)
C(75)	0.525(2)	0.150(1)	0.7903(7)	8.2(5)
C(76)	0.413(1)	0.206(1)	0.8237(6)	6.3(4)
C(81)	0.032(1)	0.183(1)	1.0049(6)	5.5(3)
C(82)	0.038(2)	0.094(1)	1.0596(7)	6.8(4)
C(83)	−0.096(2)	0.037(1)	1.0983(8)	8.4(5)
C(84)	−0.223(2)	0.072(1)	1.0793(8)	8.5(5)
C(85)	−0.223(2)	0.158(1)	1.0246(9)	9.2(5)
C(86)	−0.096(1)	0.218(1)	0.9827(7)	7.5(4)

crystallographically independent molecules in an asymmetric unit. Two sets of bond distances and angles are shown in Tables 4 and 5, respectively. As the configurations of the two independent molecules are essentially identical, and slight differences between the sets of data are regarded as experimental error. As in the case of $[Cp(CO)_2Fe]_2[NH_2C_6H_4C(O)N_2CHC_6H_4O]Sn$, so the *o*-(α-hydroxy-*o*-aminobenzylidenehydrazonomethyl)phenol is a tridentate, planar ligand. The tin atom lies

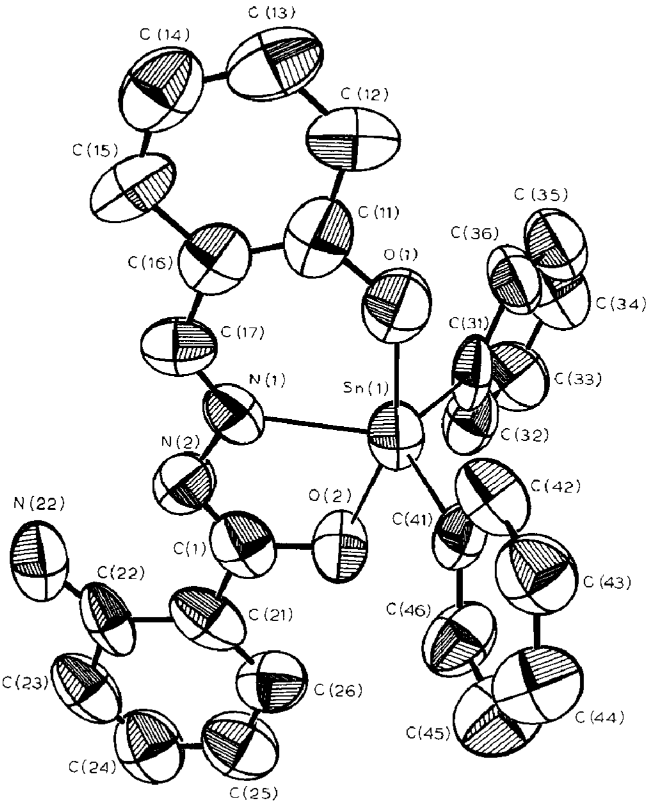

Fig. 1. Structure of $Ph_2Sn[OC_6H_4CHN_2C(O)C_6H_4NH_2]$.

Table 4

Selected bond distances for the two independent molecules (A and B) of $Ph_2Sn[NH_2C_6H_4C(O)$-$N_2CHC_6H_4O]$ (Å)

Molecule A		*Molecule B*	
Sn(1)–O(1)	2.076(6)	Sn(2)–O(3)	2.070(5)
Sn(1)–O(2)	2.119(7)	Sn(2)–O(4)	2.112(5)
Sn(1)–N(1)	2.163(7)	Sn(2)–N(3)	2.123(6)
Sn(1)–C(31)	2.120(9)	Sn(2)–C(71)	2.128(8)
Sn(1)–C(41)	2.122(9)	Sn(2)–C(81)	2.163(8)
O(1)–C(11)	1.36(1)	O(3)–C(51)	1.342(8)
O(2)–C(1)	1.33(1)	O(4)–C(6)	1.312(9)
C(11)–C(12)	1.45(2)	C(51)–C(52)	1.46(2)
C(11)–C(16)	1.37(1)	C(51)–C(56)	1.37(2)
C(12)–C(13)	1.38(1)	C(52)–C(53)	1.41(2)
C(13)–C(14)	1.43(1)	C(53)–C(54)	1.41(1)
C(14)–C(15)	1.42(1)	C(54)–C(55)	1.41(2)
C(15)–C(16)	1.43(1)	C(55)–C(56)	1.39(2)
C(16)–C(17)	1.43(1)	C(56)–C(57)	1.44(2)
C(17)–N(1)	1.25(1)	C(57)–N(3)	1.324(9)
N(1)–N(2)	1.383(9)	N(3)–N(4)	1.409(9)
N(2)–C(1)	1.30(2)	N(4)–C(6)	1.294(9)
C(1)–C(21)	1.50(2)	C(6)–C(61)	1.52(2)
C(21)–C(22)	1.46(1)	C(61)–C(62)	1.41(2)
C(21)–C(26)	1.40(1)	C(61)–C(66)	1.43(1)
C(22)–C(23)	1.42(1)	C(62)–C(63)	1.42(1)
C(22)–N(22)	1.34(1)	C(62)–N(62)	1.38(1)
C(23)–C(24)	1.38(1)	C(63)–C(64)	1.39(2)
C(24)–C(25)	1.38(1)	C(64)–C(65)	1.41(2)
C(25)–C(26)	1.41(1)	C(65)–C(66)	1.38(1)
C(31)–C(32)	1.39(1)	C(71)–C(72)	1.38(2)
C(31)–C(36)	1.46(1)	C(71)–C(76)	1.42(1)
C(32)–C(33)	1.44(1)	C(72)–C(73)	1.41(2)
C(33)–C(34)	1.43(2)	C(73)–C(74)	1.40(1)
C(34)–C(35)	1.39(1)	C(74)–C(75)	1.40(2)
C(35)–C(36)	1.45(2)	C(75)–C(76)	1.40(1)
C(41)–C(42)	1.43(1)	C(81)–C(82)	1.39(2)
C(41)–C(46)	1.38(2)	C(81)–C(86)	1.44(2)
C(42)–C(43)	1.42(1)	C(82)–C(83)	1.44(1)
C(43)–C(44)	1.39(1)	C(83)–C(84)	1.40(1)
C(44)–C(45)	1.38(2)	C(84)–C(85)	1.36(2)
C(45)–C(46)	1.43(1)	C(85)–C(86)	1.42(1)

in this plane and forms a five-membered and a six-membered chelate ring with the ligand. The molecule possesses approximate C_s symmetry with the mirror coinciding with the ligand plane, and the two phenyl groups being mirror images of each other. The tin atom has distorted trigonal bipyramidal coordination in which the two more electronegative oxygen atoms occupy the axial positions. The tin atom is displaced by 0.070 Å from the equatorial plane which consists of the nitrogen and two carbon atoms. The dihedral angles between the equatorial plane and the two phenyl planes are 92.9° and 79.4°, respectively. Some of the Sn–N, Sn–O, and Sn–C bond distances found in this and related complexes are listed in Table 6. It can be seen that the Sn–N bond of 2.143 Å observed in the present study is the shortest of the

Table 5

Selected bond angles for $Ph_2Sn[NH_2C_6H_4C(O)N_2CHC_6H_4O]$ (°)

Molecule A		Molecule B	
O(1)–Sn(1)–O(2)	158.9(2)	O(3)–Sn(1)–O(4)	156.6(2)
O(1)–Sn(1)–N(1)	83.3(3)	O(3)–Sn(2)–N(3)	83.2(2)
O(1)–Sn(1)–C(31)	95.0(4)	O(3)–Sn(2)–C(71)	94.6(4)
O(1)–Sn(1)–C(41)	96.3(4)	O(3)–Sn(2)–C(81)	97.6(4)
O(2)–Sn(1)–N(1)	75.7(3)	O(4)–Sn(2)–N(3)	74.3(2)
O(2)–Sn(1)–C(31)	93.8(3)	O(4)–Sn(2)–C(71)	93.3(3)
O(2)–Sn(1)–C(41)	94.0(4)	O(4)–Sn(2)–C(81)	95.9(3)
N(1)–Sn(1)–C(31)	117.2(3)	N(3)–Sn(2)–C(71)	125.4(2)
N(1)–Sn(1)–C(41)	116.1(3)	N(3)–Sn(2)–C(81)	109.5(2)
C(31)–Sn(1)–C(41)	126.5(3)	C(71)–Sn(2)–C(81)	124.8(3)
Sn(1)–O(1)–C(11)	132.5(6)	Sn(2)–O(3)–C(51)	130.2(5)
Sn(1)–O(2)–C(1)	111.8(6)	Sn(2)–O(4)–C(6)	113.5(5)
O(1)–C(11)–C(12)	115.5(9)	O(3)–C(51)–C(52)	114.0(9)
O(1)–C(11)–C(16)	122(1)	O(3)–C(51)–C(56)	124.2(8)
C(15)–C(16)–C(17)	114.5(9)	C(55)–C(56)–C(57)	115.3(9)
C(16)–C(17)–N(1)	126.1(9)	C(56)–C(57)–N(3)	122.8(7)
C(17)–N(1)–N(2)	116.4(7)	C(57)–N(3)–N(4)	112.3(6)
Sn(1)–N(1)–C(17)	129.7(6)	Sn(2)–N(3)–C(57)	131.2(5)
Sn(1)–N(1)–N(2)	113.9(5)	Sn(2)–N(3)–N(4)	116.5(5)
O(2)–C(1)–C(21)	112(1)	O(4)–C(6)–C(61)	114.6(8)
N(2)–C(1)–C(21)	124(1)	N(4)–C(6)–C(61)	120.4(7)
C(1)–C(21)–C(22)	119(2)	C(6)–C(61)–C(62)	120.7(8)
C(1)–C(21)–C(26)	120(1)	C(6)–C(61)–C(66)	117.1(7)
Sn(1)–C(31)–C(32)	118.2(7)	Sn(2)–C(71)–C(72)	119.0(7)
Sn(1)–C(31)–C(36)	116.3(8)	Sn(2)–C(71)–C(76)	119.6(8)
Sn(1)–C(41)–C(42)	118.4(7)	Sn(2)–C(81)–C(82)	119.1(6)
Sn(1)–C(41)–C(46)	119.9(7)	Sn(2)–C(81)–C(86)	116.0(7)

Table 6

Comparison of coordinated bond lengths on tin in $Ph_2[NH_2C_6H_4C(O)N_2CHC_6H_4O]Sn$ with those in selected organotin(IV) compounds (Å)

Compound	Sn–N	Sn–O	Sn–C	Ref.
Ph_2Sn[3-(2-py)-2-thienyl-C,N]	2.560(2)		2.163(2)	4
			2.179(2)	
Ph_2SnCl_2bipy [a]	2.344(6)		2.151(7)	5
	2.375(6)		2.153(8)	
(p-tolyl)$_2SnCl_2$bipy	2.306(3)		2.159(3)	6
	2.374(3)		2.161(3)	
SnBuCl(dapa) [b]	2.278(5)	2.144(5)	2.138(20)	7
	2.318(9)			
Ph_2Sn(dapa)	2.266(9)	2.207(7)	2.182(12)	7
	2.309(9)	2.187(7)	2.170(12)	
	2.324(9)			
Me_2ClSn(2-pyCOO)	2.505(25)	2.076(29)	2.089(21)	8
		2.375(28)	2.120(24)	
$ClSn(CH_2CH_2CH_2)_3N$	2.372(29)		2.169(8)	9
$[Cp(CO)_2Fe]_2SnL$	2.247(4)	2.149(5)		1
		2.198(4)		
Ph_2SnL	2.163(7)	2.076(6)	2.120(9)	this work
	2.123(6)	2.119(7)	2.122(9)	
		2.070(5)	2.128(8)	
		2.112(5)	2.163(8)	

[a] bipy = 2,2′-bipyridyl. [b] H_2dapa = 2,6-diacetylpyridine bis(2-aminobenzoylhydrazone).

Sn–N bonds listed in Table 6; moreover, compared with the corresponding values found for other complexes, the Sn–O and Sn–C bonds in this complex are also shorter. The short bonds in this type of complex indicate that the coordination bonds of tin are strong, a fact that is supported by MS data obtained by use of an EI source. The molecular ion peaks of ten Ph_2SnL complexes are all over 40%, and most of them are the highest peaks (see Table 2); a phenomenon seldom seen in large organometallic compounds.

In crystals of $Ph_2Sn[OC_6H_4CHN_2C(O)C_6H_4NH_2]$, the distance of 2.717 Å between N(22) and N(2) (the relevant distance in the other molecule is 2.694 Å) suggests the formation of an intramolecular hydrogen bond. Molecular interactions in this crystal are of the normal van der Waals type, and there are no unusual short intermolecular contacts.

Experimental

Synthesis

The chemicals and solvents were dried and purified by standard methods. The ligands and dichlorodiphenyltin were prepared by the reported methods [10,11]. All reactions were carried out under dry argon.

A 100-ml three-necked flask was charged with 0.0025 mol of *o*-(substituted α-hydroxybenzylidenehydrazonomethyl)phenol, 1 ml triethylamine and 50 ml benzene. Dichlorodiphenyltin (0.85 g, 0.0025 mol) in 10 ml benzene was added dropwise into the flask with stirring at room temperature. The solution turned yellow and a white precipitate formed. This solution was stirred for 3 h. The white deposit, which is Et_3N–HCl formed in the reaction, was filtered off, washed with 20 ml benzene, and the filtrate was concentrated to 5 ml. Yellow crystals formed after 7 ml of petroleum ether (30–60 ° C) had been added to the filtrate. The yellow solid was recrystallized from CH_2Cl_2/petroleum ether.

Elemental C, H, N analyses were carried out on Corder model M7-3 automatic equipment. IR of KBr discs were obtained on a Nicolet-FT-IR model 5DX spectrophotometer in the range of 4600–400 cm^{-1}. The mass spectra were determined by a VG 7070E-HF spectrometer (70 eV) by use of an EI source.

X-Ray diffraction analysis

Crystals of the complex ($R = o\text{-}NH_2$) for X-ray study were grown by slow evaporation of solvent from CH_2Cl_2/petroleum ether at room temperature. A yellow prism of approximate dimensions 0.1 × 0.2 × 0.6 mm was mounted on an Enraf–Nonius CAD4 diffractometer equipped with a graphite monochromator and Mo-K_α radiation ($\lambda = 0.71073$ Å). Cell parameters were determined by least-squares refinement of the diffraction geometry for 25 accurately centered reflections. A total of 4248 independent reflections were collected in the range of $2° \leqslant \theta \leqslant 20°$ by ω–2θ scan technique at room temperature, in which 3129 reflections with $I \geqslant 3\sigma(I)$ were considered observed and were used in the subsequent refinements. The corrections for LP and for absorption were based on a series of ψ scans and were applied to the data. The crystals of this complex are triclinic, space group $P\bar{1}$ with a 9.608(2), b 11.826(4), c 21.478(6) Å, α 76.17(2), β 76.53(2), γ 88.91(2)°, V 2302.7(4) Å^3; $Z = 4$, D_c 1.52 g/cm^3.

The structure was solved by direct methods (MULTAN 82). The two Sn atoms were located from an *E*-map. The coordinates of the remaining non-hydrogen atoms were found in subsequent difference Fourier syntheses. The hydrogen atoms were not included in the refinements. The final refinement by full matrix least-squares method with anisotropic thermal parameters for non-hydrogen atoms converged with unweighted and weighted discrepancy factors of 0.050 and 0.057, respectively (unit weights for all observed reflections). The highest peak on the final difference Fourier map had a height of 0.81 e/Å^3.

Acknowledgement

The authors are grateful to the National Science Fundation of China for support of this work.

References

1 F.-Q. Liu, J.-T. Wang, R.-J. Wang, H.-G. Wang, X.-K. Yao, J. Organomet. Chem., 371 (1989) 35.
2 D.K. Rastogi, S.K. Dua. V.B. Rana, S.K. Shni, J. India Chem. Soc. (1978) 1323.
3 M.C. Henry, J.G. Noltes, J. Am. Chem. Soc., 82 (1960) 554.
4 V.G.K. Das, L.K. Mun, C. Wei, T.C.W. Mak, Organometallics, 6 (1987) 10.
5 P.G. Harrison, T.J. King, J.A. Richards, J. Chem. Soc., Dalton Trans., (1974) 1723.
6 V.G.K. Das, C. Wei, Y.C. Keong, T.C.W. Mak, J. Organomet. Chem., 299 (1986) 41.
7 C. Pellizi, G. Pellizi, G. Predieri, J. Organomet. Chem., 263 (1984) 9.
8 I.W. Nowell, J.S. Brooks, G. Beech, R. Hill, J. Organomet. Chem., 244 (1983) 119.
9 K. Jurkschat, A. Tzschach, J. Meunier-Piret, M. van Meerssche, J. Organomet. Chem., 290 (1985) 285.
10 D.K. Rastogi, S.K. Dua, V.B. Rana, S.K. Sahn, J. India Chem. Soc., (1978) 1323.
11 H. Gilman, L.A. Gist, J. Org. Chem., 22 (1957) 368.