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Optically active pentacyclic binuclear diorganotin compounds

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Abstract

Three optically active binuclear diorganotin compounds (**2**–**4**) were prepared from an optically active oxalamide: (1S,2R)-(−)-[*N*-(2-hydroxy-1-methyl-2-phenyl-ethyl)-*N*′-(2-hydroxy-phenyl)-oxalamide (**1**). The new compounds [1-(2′,2′-diorganyl-1′-oxa-3′-aza-2′-stanna-indan-3′-yl)-2-(2″,2″-diorganyl-4″-methyl-5″-phenyl[1,3,2]-oxazastannolidin-3″-yl)-ethane-1,2-dione {organyl = methyl (**2**), *n*-butyl (**3**) or phenyl (**4**)} were characterized by IR, elemental analysis and mass spectrometry. Compounds **3** and **4** were submitted to a detailed NMR study in order to assign their resonances (^{1}H and ^{13}C) and specially the two different ^{119}Sn signals for each compound. The X-ray diffraction analysis of compound **4** showed a planar pentacyclic framework with two penta-coordinated tin atoms with a distorted tbp geometry and 12 intramolecular hydrogen bonds.
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Keywords: Optically active tin compounds; Binuclear tin heterocycles; Tin heteropentacycles

1. Introduction

We have been involved in the spectroscopic study of planar tetracyclic aromatic heterocycles derived from bis-phenolamines [1,2] their importance is based in their rigid structures which give stable compounds, suitable models for NMR and X-ray diffraction studies of hypervalent metallic atoms. The results motivated us to explore the reactions of diphenol-oxalamides with organometallic tin halides which afforded binuclear compounds with delocalized hexacyclic frameworks, being the tin atoms strongly coordinated to the amides oxygen atoms, Scheme 1 [3,4].

One important aspect of the study of organotin compounds is their biocide activity. The understanding of the structural bases of the biological effects needs a careful investigation of the electronic environment, coordination number, geometry and stereochemistry in tin organometallic compounds. It has been shown on tetra- penta- and hexa-coordinated organotin compounds [5–7] that Sn–O, Sn–N or Sn–S bonds make them highly biocides, for example against leukemia P388 in rats [8] or against tumoral cells MCF-7 and WiDr [5].
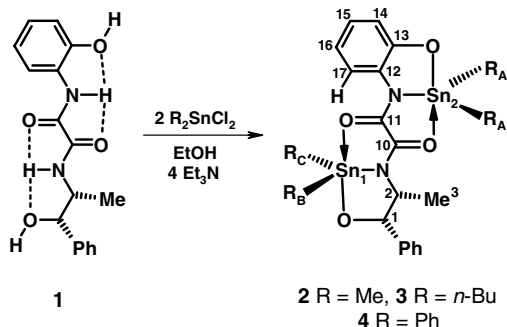
2. Results and discussion

In order to extend our study to other bimetallic heterocycles, we decided to use a related non-symmetrical ligand, compound **1**, to prepare three new polyheterocycles bearing, each one, two non-equivalent diorganyltin groups (**2** R = CH$_3$, **3** R = *n*-C$_4$H$_9$, R = **4** C$_6$H$_5$). The syntheses were performed by reaction of compound **1** and the corresponding organometallic compounds R$_2$SnCl$_2$ in ethanol and in presence of triethylamine, Scheme 2. The two penta-coordinated tin atoms are stereogenic centers and therefore an isomeric mixture should be expected; however, the

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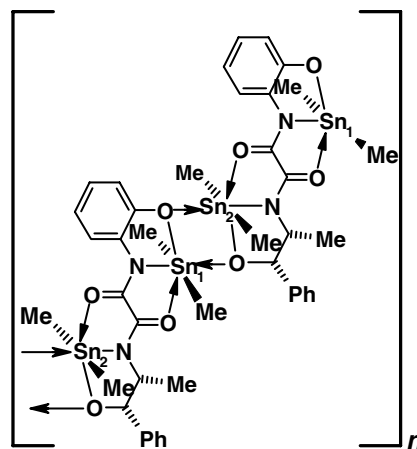*E-mail address:* aflores@cinvestav.mx (A. Flores-Parra).
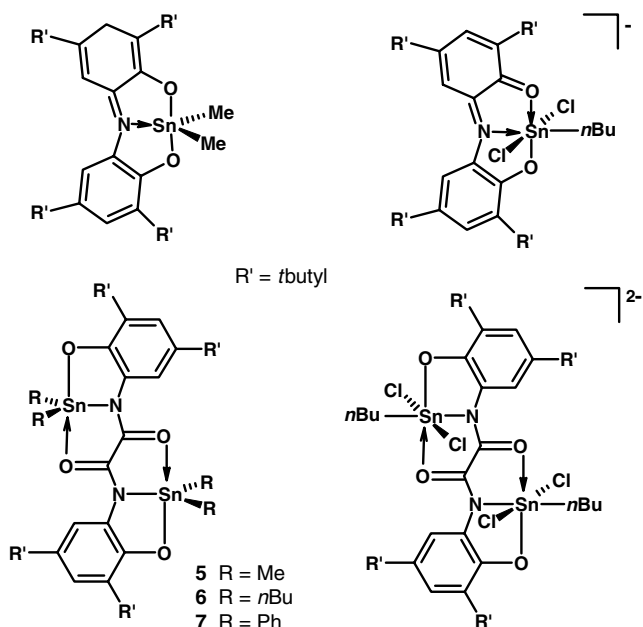
Scheme 1. Some bis-phenolamine[1,2] and bis-(phenol)oxalamide[3,4] tin heterocycles.

Scheme 2. Tin compounds (2–4) derived from the asymmetric oxalamide 1.

Scheme 3. Possible polymeric structure of compound 2.

2.1. NMR analysis

As a consequence of the lack of symmetry in compounds 3 and 4, both tin atoms are not equivalent as it has been shown by ^{119}Sn NMR. The presence of four stereogenic centers (two carbon and two tin atoms) complicated the assignment of the ^{1}H and ^{13}C signals, due to the diasterotopy of the Sn-organyl groups. Owing to the complex spectra, a challenge of this research was the assignment of the ^{1}H, ^{13}C and ^{119}Sn data, for the aromatic groups it was based on the $^nJ(^{13}C, ^{117/119}Sn)$ coupling constants (Scheme 4) and by comparison with reported data for the symmetrical molecules [3,4]. The ^{1}H signals for H-17 in compounds 3 (8.31 ppm) and 4 (8.52 ppm) showed a strong deshielding effect by their short distance to the oxygen atoms of the

reactions gave only one isomer in each case, due to the rigid and planar array of the ligand, which implies that reactions have been stereoselective [9–11].
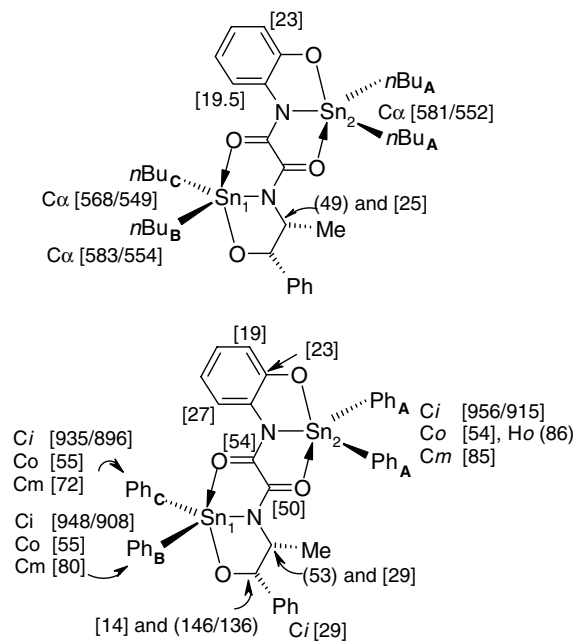
Compound 4 is a yellow-greenish crystalline solid whereas 2 and 3 are white. All compounds were obtained in good yields and they are stable to air and moisture. Compound 2 was not enough soluble in organic solvents in order to record its NMR spectra, this unusual behavior was attributed to the polymeric association shown in Scheme 3. In compounds 2–4, the IR C=O stretching bands were shifted to lower frequencies (2 1602; 3 1599; 4 1619 cm^{-1}) with respect to compound 1 (1670 cm^{-1}) due to their coordination to tin atoms. Compounds 2–3 presented molecular ions at the mass spectra, all of them lose benzaldehyde to give the corresponding fragments, m/z (%): 502(100), 670(23) and 750(40) for 2, 3, and 4, respectively, which indicate that the delocalized fragments of the molecules are stable.

Scheme 4. Tin coupling constants values: $^nJ(^1H, ^{119}Sn)$ between parentheses and $^nJ(^{13}C, ^{119/117}Sn)$ between square parentheses, R_B and R_C could be exchanged.

amide groups (2.48 Å as it was found by X-ray diffraction analysis, vide infra); this effect is an evidence that in solution the phenol fragment is coplanar with the oxalamide group and that all the molecule framework is planar. Once, the hydrogen atoms of compounds **3** and **4** were assigned by COSY experiments, they were correlated with the ^{13}C spectra by HETCOR experiments. The carbonyl carbon atoms C-10 and C-11 are not equivalent (161.4 and 158.3 ppm, respectively), the C=O of the phenolamide (C11) has been assigned by comparison with the symmetric compound **7** (158.0 ppm) [3,4], Scheme 1.

The two ^{119}Sn NMR signals of each compound correspond to penta-coordinated tin atoms [12,13] (**3** Sn_1 −126.2 ppm, Sn_2 −114.8 ppm and **4** Sn_1 −265.2 ppm, Sn_2 −252.7 ppm). The ^{119}Sn chemical shifts and coupling constants $J(^{13}C, ^{119}Sn)$ of **3** and **4** resemble to those of symmetrical compounds **6** and **7** [**6** = −122.5 ppm and **7** = −258.8 ppm] [3] but the information was not enough conclusive to decide the correct assignment, Scheme 1. Compounds **6** and **7** have chemical shifts which are found between those values of the two tin atoms in **3** and **4**.

Only compound **4** clearly presented a coupling between both tin atoms $^4J(^{119}Sn, ^{119}Sn) = 50$ Hz, which is of the same order than those found in the symmetrical compounds between the 119 and 117 tin isotopes [$^4J(^{119}Sn, ^{117}Sn) = 40.8$ (**5**), 41.5 (**6**) and 51.8 Hz (**7**)] [3].

For the unequivocal assignment of both ^{119}Sn signals of compound **4**, it was expected that the Sn1 atom linked to the ethanolamide fragment would present more and larger couplings that those of the Sn2 atom of the phenolamide group and, in consequence, the first one should have a broader signal. Effectively, the ^1H coupled ^{119}Sn spectra showed that the signal at −252.7 ppm is broader ($\Delta v_{1/2} =$ 229 Hz) than the second at −265.2 ($\Delta v_{1/2} = 208$ ppm); however, the latter observation was not enough to sustain the assignment. It was considered that Sn2 heterocycle is planar and very rigid whereas the other is fluxional due to its aliphatic nature and in consequence that the magnetic relaxation at the second tin atom Sn1 may occur more easily. Therefore, a ^{119}Sn variable temperature experiment performed from +60 to −60 °C showed that at low temperature the signal at −252.7 ppm became so broad that was almost lost in the base line, whereas the signal assigned to Sn1 had less changed.

Finally, the unequivocal assignment of the ^{119}Sn resonance has been made by a [^1H, ^{119}Sn] heteronuclear multiple quantum coherence (HMQC) experiment using selective inverse detection which allowed to find a correlation between H-1 (5.2 ppm) and Sn1 (−252.7 ppm) using a coupling constant value of $^3J(^1H, ^{119}Sn) = 146$ Hz. The ^{119}Sn chemical resonances of compound **3** were assigned by comparison of experiments of both compounds **3** and **4**.

2.2. X-ray diffraction analysis

Crystals of compound **4** (orthorhombic system, chiral space group $P2_1$) were suitable for its X-ray diffraction
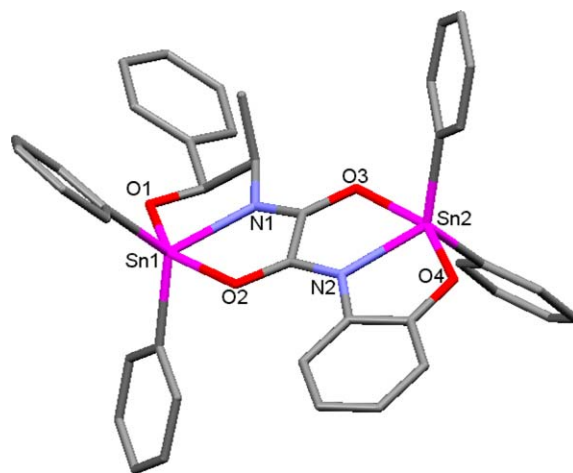

Fig. 1. View of the X-ray diffraction structure of compound **4**, it shows the ring conformation of the ethanolamine fragment, the hydrogen atoms have been eliminated for clarity.

analysis. The structure is pentacyclic with two tin atoms, the phenolamide fragment is almost planar whereas the cyclic aliphatic part of the molecule has an envelop conformation with the benzylic carbon atom being out of the ring plane, the C-phenyl group at C1 is in equatorial and the methyl group in C2 in a pseudoaxial position, as is shown in Fig. 1. The aromatic framework is similar to those found for the symmetrical compounds **5**–**7** [3,4], Scheme 1. Both tin atoms are penta-coordinated, their geometry, due to the ring tension, is a distorted tbp as it is deduced from the angles of the axial oxygen atoms: O4–Sn2–O3 153.13(1)° and O2–Sn1–O1 151.84(1)°, Table 1. The nitrogen and the two Sn-phenyl groups are in equatorial position and their angles are: N2–Sn2–C36 118.45(1)°, N2–Sn2–C30 115.69(1)°, N1–Sn1–C18 118.45(1)° and N1–Sn1–C24 116.55(1)°.

It is quite interesting that the five bonds around the tin atoms are very short and their values very close to those of covalent bonds [for example bonds O3–Sn2 2.207(3) and O2–Sn1 2.292(2) Å are only approximately 10% longer than O4–Sn2 2.057(2) and O1–Sn1 2.037(2) Å] which indicates strong bonds around the tin atom. In order to know the charge distribution and to explain the bond lengths, we have calculated the atomic charges using the program Ms Modeling v3.2.0.0 and the atomic coordinates obtained from the X-ray diffraction structure. The tin atoms have positives charges as well as C10 and C11, the nitrogen and oxygen atoms the negative ones. Coordination of the carbonyl groups to tin atoms creates a positive charge at carbon atoms C10 and C11, the electronic deficiency is solved by attracting electronic density from the neighboring oxygen and nitrogen atoms, Scheme 5. The latter effect could explain the short bond lengths of the C=O [C10–O3 1.268(5), C11–O2 1.256(4) Å] and C=N [C10–N1 1.286(4) and C11–N2 1.311(4) Å] and the long C–C bond of the oxalamide [C10–C11 1.531(4) Å].

Table 1
X-ray diffraction data (selected bond lengths and bond angles) and numbering of compound **4**
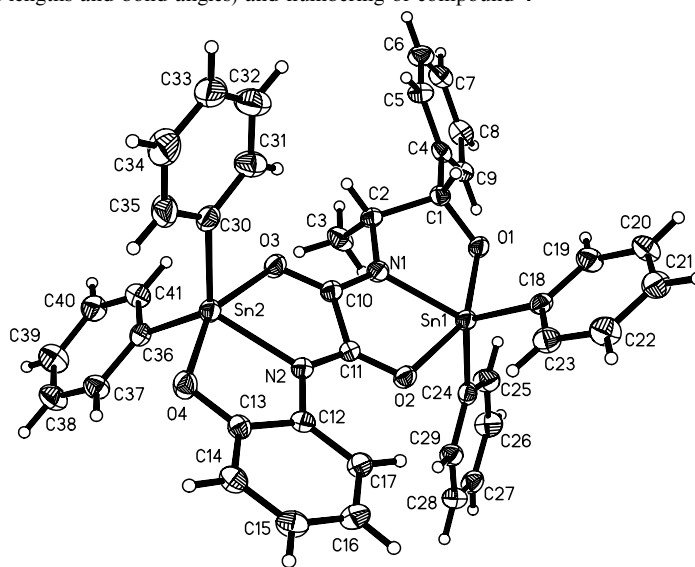
Bond lengths (Å)

Sn2–O4	2.057(2)	Sn2–C36	2.108(4)	Sn2–C30	2.122(4)	Sn2–N2	2.151(3)
Sn2–O3	2.207(3)	Sn1–O1	2.037(2)	Sn1–C24	2.105(4)	Sn1–N1	2.116(3)
Sn1–C18	2.124(4)	Sn1–O2	2.292(2)	O4–C13	1.345(5)	O2–C11	1.256(4)
O3–C10	1.268(5)	O1–C1	1.416(5)	N2–C11	1.311(4)	N2–C12	1.414(4)
N1–C10	1.286(4)	N1–C2	1.484(4)	C10–C11	1.531(4)		

Bond angles (°)

O4–Sn2–C36	99.74(1)	O4–Sn2–C30	101.49(1)	C36–Sn2–C30	124.67(1)
O4–Sn2–O3	153.13(1)	C36–Sn2–N2	118.45(1)	C30–Sn2–O3	91.91(1)
C30–Sn2–N2	115.69(1)	C36–Sn2–O3	91.30(1)	O4–Sn2–N2	78.08(1)
N2–Sn2–O3	75.12(1)	O1–Sn1–C24	100.42(1)	O1–Sn1–N1	78.66(1)
C24–Sn1–N1	116.55(1)	O1–Sn1–C18	101.60(1)	C24–Sn1–C18	123.55(1)
N1–Sn1–C18	118.45(1)	O1–Sn1–O2	151.84(1)	C24–Sn1–O2	93.96(1)
N1–Sn1–O2	73.26(1)	C18–Sn1–O2	90.11(1)		

Twelve intramolecular hydrogen bonds contributes to the rigidity of the framework, each oxygen atom has three hydrogen bonds which give penta-coordinated oxygen atoms, this unusual situation is depicted in Fig. 2. The distances for the hydrogen bonds [14] are O1–H25 2.524, O1–H19 2.714, O1–H9 2.491, O2–H17 2.476, O2–H29 2.661, O2–H23 2.504, O3–H31 2.414, O3–H41 2.572, O3–H2 2.612, O4–H14 2.483, O4–H37 2.732 and O4–H35 3.021.

3. Conclusion

We have reported three optically active new tin compounds, which exhibit a stable tin configuration due to the rigid framework of the ligands. The NMR data was analyzed and almost all their signals were assigned, specially the two different [119]Sn signals for each compound. The X-ray showed some interesting features as the planar pentacyclic framework with two penta-coordinated tin atoms with a distorted tbp geometry and 12 intramolecular hydrogen bonds.

4. Experimental

4.1. Syntheses

4.1.1. General comments

All chemicals were commercial and were used without further purification. Solvents were purified by usual methods. Melting points were obtained in Mel-Temp II apparatus and are uncorrected. IR (KBr, disc) spectra were determined on a Perkin–Elmer 16 FPC spectrometer. Mass spectra in the EI mode were recorded at 20 eV on a Hewlett-Packard HP 5989 spectrometer. NMR spectra were obtained on a Jeol GSX-270, Bruker 300 Advance or Jeol Eclipse 400 MHz spectrometers. Optical rotation angle values were obtained in Perkin–Elmer 241 polarimeter. Elemental analyses were performed at Eager 300 at Cinvestav, Mexico. Compound **1** was prepared following the reported procedure [15,16].

4.2. Synthesis of compounds 2–4

A solution of 0.2 g (1 mmol) of **1** in ethanol (10 mL) and Et$_3$N (0.37 mL, 2.7 mmol) was mixed with a solution of
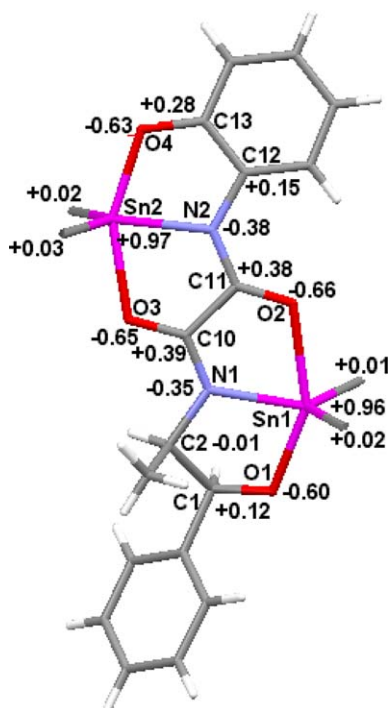
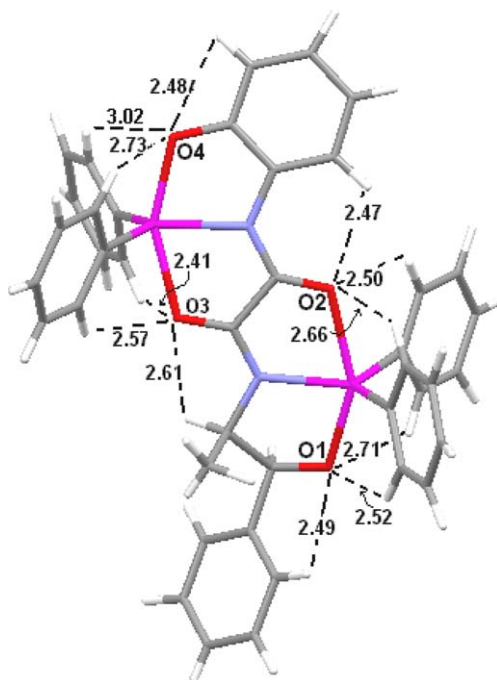Scheme 5. Atomic charges for the framework of compound **4**.

Fig. 2. Intramolecular hydrogen bonds (Å) with the four oxygen atoms.

SnR$_2$Cl$_2$ (1.82 mmol) in ethanol (5 mL). After some minutes a crystalline precipitate was formed which was filtered and washed with *n*-hexane.

4.3. Analytical data

4.3.1. 1-(2′,2′-Dimethyl-1′-oxa-3′-aza-2′-stanna-indan-3′-yl)-2-(2″,2″-dimethyl-4″-methyl-5″-phenyl-[1,3,2]oxazastannolidin-3″-yl)-ethane-1,2-dione (2)

White powder, 71% (250 mg), m.p. 212–214 °C. MS (EI, 20 eV), *m/z* (%): 608 [M]$^+$ (10), 502 (100). IR (KBr), *v* (cm^{-1}): 1601.8, 1476, 1284, 992, 781. Anal. Calc. for C$_{21}$H$_{26}$N$_2$O$_4$Sn$_2$: C, 41.49; H, 4.31; N, 4.60. Found: C, 41.70; H, 4.34; N, 4.57%.

4.3.2. 1-(2′,2′-Dibutyl-1′-oxa-3′-aza-2′-stanna-indan-3′-yl)-2-(2″,2″-dibutyl-4″-methy-5″-phenyl-[1,3,2]oxazastannolidin-3″-yl)-ethane-1,2-dione (3)

White powder, 98%. $[\alpha]_{578\,Hg}^{25} = +25°$ (*c* = 0.5, AcOEt), m.p. 253 °C. ^{1}H NMR (400 MHz, CDCl$_3$), assignments for Bu$_B$ or Bu$_C$ groups could be interchanged. δ [*n*BuSn groups: 0.91 (t, 6H, C*H$_3$* in *n*Bu$_A$Sn$_2$), 0.84 (t, 3H, C*H$_3$* in *n*Bu$_C$Sn$_1$), 0.95 (t, 3H, C*H$_3$* in *n*Bu$_B$Sn$_1$), 1.27–1.71 (m, 24H, 12[C*H$_2$*] in *n*Bu$_2$Sn)], [ethanolamide group: 0.72 (d, ^3J = 6.3 Hz, H-11), 4.04 (dq, ^3J = 4.3 and 6.4 Hz, 1H, H-2), 4.92 (d, ^3J = 4.1 Hz, 1H, H-1), 7.22 (t, ^3J = 6.95 Hz, 1H, H*p*), 7.33 (t, ^3J = 7.69 Hz, 2H, H*m*), 7.42 (d, ^3J = 6.95 Hz, 2H, H*o*)], [phenoxyamide group: 6.67 (t, ^3J = 6.59 Hz, 1H, H-16), 6.84 (d, ^3J = 6.59 Hz, 1H, H-14), 7.03 (td, ^3J = 6.23 and ^4J = 1.54 Hz, 1H, H-15), 8.31 (dd, ^3J = 6.59 and ^4J = 1.55 Hz, 1H, H-17)]. ^{13}C NMR (270 MHz, CDCl$_3$), δ: [*n*BuSn groups: 13.73 (2C*H$_3$*, *n*Bu$_A$Sn$_2$ and *n*Bu$_B$Sn$_1$), 13.78 (C*H$_3$*, *n*Bu$_C$Sn$_1$), 20.46 (6C*H$_2$*-α, *n*Bu$_A$Sn$_2$), 21.18 (C*H$_2$*-α, *n*Bu$_B$Sn$_1$), 21.21 (C*H$_2$*-α, *n*Bu$_C$Sn$_1$), 26.72–27.69 (8[C*H$_2$*], *n*Bu$_2$Sn)], [ethanolamide group: 13.00 (C*H$_3$*-3), 54.18 (C2), 77.62 (C1), 126.19 (C*o*), 126.82 (C*p*), 128.08 (C*m*), 143.57 (d, C*i*), 162.18 (C10)], [phenoxyamide group: 116.22 (C16), 116.69 (C14), 120.96 (C17, d), 128.3 (C15), 128.89 (C12), 157.58 (C13), 159.3 (C11)]. ^{119}Sn NMR (400 MHz, CDCl$_3$), δ: −126.2 (Sn$_1$), −114.8 (Sn$_2$). MS (EI, 20 eV), *m/z* (%): 776 [M]$^+$ (3), 670 (40), 557 (100). IR (KBr), *v* (cm^{-1}): 2961.6, 2926.6, 1599.18, 1472.3, 1280.5, 991.2. Anal. Calc. for C$_{33}$H$_{50}$N$_2$O$_4$Sn$_2$: C, 51.06; H, 6.49; N, 3.6. Found: C, 50.91; H, 6.24; N, 3.59%.

4.3.3. 1-(2′,2′-Diphenyl-1′-oxa-3′-aza-2′-stanna-indan-3′-yl)-2-(2″,2″-diphenyl-4″-methyl-5″-phenyl-[1,3,2]oxazastannolidin-3″-yl)-ethane-1,2-dione (4)

The pale greenish yellow crystalline powder was recrystallyzed from AcOEt: CH$_2$Cl$_2$ (8:2), 90%. $[\alpha]_{578\,Hg}^{25} = -4.1°$ (*c* = 0.5, AcOEt), m.p. 211 °C. ^{1}H NMR (300 MHz, CDCl$_3$), assignments for Ph$_B$ or Ph$_C$ groups could be exchanged. δ: [phenoxyamide group: 6.87 (t, ^3J = 6.65 Hz, 1H, H-16), 7.16 (d, ^3J = 6.65 Hz, 1H, H-14), 7.20 (td, ^3J = 6.23 and ^4J = 1.54 Hz, 1H, H-15), 8.52 (dd, ^3J = 6.65 and ^4J = 1.55 Hz, 1H, H-17)], [ethanolamide group: 0.85 (d, ^3J = 6.44 Hz, 3H, H-3), 4.35 (dq, ^3J = 4.1 and 6.2 Hz, 1H, H-2), 5.16 (d, ^3J = 4 Hz, H-1), 7.31 (t, ^3J = 7.45 Hz, 1H, H*p*), 7.41 (t, ^3J = 7.45 Hz, 2H, H*m*), 7.62 (d, ^3J = 7.45 Hz, 2H, H*o*)]; [PhSn groups: 7.37 (m, 12H, 8H*m*, 4H*p*), 8.03 (dd, ^3J = 7.52, 8H, 4H, H*o$_A$*), 7.89 (dd,

$^3J = 7.5, 2H, Ho_B$), 7.80 (dd, $^3J = 7.5, 2H, Ho_C$)]. ^{13}C NMR (300 MHz, CDCl$_3$), δ: [ethanolamide group: 13.72 (CH_3-3), 54.56 (CH-2), 77.55 (CH-1), 126.28 (Co), 127.24 (Cp), 128.8 (Cm), 143.26 (d, Ci), 161.38 (d, C10)], [phenoxyamide group: 117.32 (C16), 117.35 (C14), 121.15 (C17), 127.5 (C15), 129.35 (C12), 157.35 (d, C13)], 158.35 (d, C11)] [PhSn groups: 138.06 (d, Ci_B), 137.72 (2Ci_A), 137.58 (d, Ci_C), 136.56 (d, 4Co_A), 136.47 (d, 2Co_B), 136.56 (d, 2Co_C), 129.24 (d, 2Cm_C), 129.27 (d, 2Cm_B), 129.24 (d, 4Cm_A), 131.1 (2Cp_A), 131.0 (Cp_B), 130.8 (Cp_C)]. ^{119}Sn NMR (400 MHz, CDCl$_3$), δ: −265.2 (Sn$_1$), −252.7 (Sn$_2$). MS (EI, 20 eV), m/z (%): 855 [M]$^+$ (2), 750 (40), 478 (100). IR (KBr), v (cm^{-1}): 3049.4, 1618.8, 1470.2, 1430.9, 1364.4, 1282.3, 1077.1. Anal. Calc. for C$_{41}$H$_{34}$N$_2$O$_4$Sn$_2$: C, 56.33; H, 6.49; N, 3.2. Found: C, 56.16; H, 4.09; N, 3.18%.

4.4. X-ray structural analysis of 4

C$_{41}$H$_{34}$N$_2$O$_4$Sn$_2$, green-yellow, which crystallizes in the monoclinic space group $P2_1$ with the lattice parameters: $a = 11.7063$ (8) pm, $b = 12.9654$ (9) pm, $c = 12.4214$ (8) pm, $\alpha = 90.00°$, $\beta = 93.2480(10)°$, $\gamma = 90.00°$, $V = 1882.3(2)$ Å^3, $Z = 2$, $\mu = 1.3697$ mm^{-1}, $F(000) = 852$; diffractometer: Siemens P4 instrument equipped with a CCD area detector and a low temperature device LTP2 (Mo Kα with $\lambda = 0.71073$ Å); 11010 reflections collected at 198 K in the range $3.48° \leqslant 2\theta \leqslant 58.26°$, 5808 reflections independent, 5568 assigned to be observed [$F > 4\sigma(F)$], solution by direct method; full-matrix least-squares refinement (SHELXL) against F^2 with 437 parameters converged at R_1:w_2-values of 0.0212:0.0511.

Acknowledgment

Appendix A. Supplementary information

Crystallographic data (excluding structure factors) for compound **4** have been deposited with the Cambridge Crystallographic Data as Supplementary Publications Nos. CCDC-289015. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: (internat.) + 44 1223 336 033; e-mail: deposit@ccdc.cam.ac.uk]. Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jorganchem.2005.11.072.

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