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The Synthesis and Biological Activity of two Organostannoxane Ladder-like Complexes Derived from Schiff Bases

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Abstract. Two complexes containing $(n\text{Bu})_2\text{Sn}_4\text{O}_4\text{L}_4$ (L = salicylaminoaryl alcohols) were synthesized and characterized by elemental analysis and ^{1}H, ^{13}C, and ^{119}Sn NMR as well as IR spectroscopy. The crystal structure of complex **2.2** was determined by X-ray crystallography. It consists of three, four-member Sn_2O_2 rings in a ladder-like, structural arrangement with four tin centers, bridged by four, three-coordinate μ_3-oxygen atoms. The dimeric distannoxane have two different, pentacoordinate tin atoms, where the Schiff base acts as monodentate ligand. The complexes were subjected to a thiobarbituric acid, reactive substance (TBARS) assay, which displayed a higher antioxidant activity than the α-tocopherol and Butylated hydroxy toluene (BHT), used as positive controls.

Introduction

The organotin complexes are interesting, due to their structural versatility and diverse applications [1]. Several synthetic strategies, based on self-assembly, have been developed for the construction of structures, including macrocycles, networks, cages, and clusters [2, 3]. The organooxotin clusters are attractive because of the diversity of arrangements that they adopt, such as ladder, O-caped, cube, butterfly, drum, one, two and three-dimensional structures, (1D, 2D, and 3D) [4–14]. The distannoxanes play an important role as catalysts in transesterification reactions in neutral reaction conditions [11, 15, 16–20], as well as in the formation of gels in aromatic solvents [21]. Several organic ligands have been used to obtain complexes with fascinating topologies, such as monoaryl phosphates used in the synthesis of organotin clusters and polymer [22], triazolacetic acid derivatives with multiplebidentate coordination properties used to generate 40-membered macrocycles. The latter contain two distannoxane ladders [23], the trifluoromethanesulfonates that give rise to ladder-like, tin(IV) dinuclear complexes [24] and benzoic acid derivatives, which form tetraorganostanooxanes with antibacterial and cytotoxic activity. [25] Although several studies have been devoted to this type of chemistry, only a few examples of organooxotin clusters containing Schiff base ligands are described in the literature. For example, Schiff bases containing triazole have different coordination environments and supramolecular structures in non covalent interactions [26–28]. Pyruvic acid hydrazone ligands form monomeric or polymeric complexes, whose nuclearity and stereochemistry are dependent upon the nature of the starting acceptor and the reaction conditions [29].

The aim of the present contribution was to prepare and characterize distannoxane-type structures from tridentate Schiff base ligands, and evaluate the antioxidant properties of their complexes.

Results

Infrared Spectroscopy

The salicylaminoaryl alcohols L1 and L2 used were prepared from salicylaldehyde and 4-amino or 3-aminophenol, according to known methods [30]. The Schiff-base ligands L1 and L2 were transformed into the distannoxanes **2.1** and **2.2** by using a 1:1 stoichiometric ratio of the proper ligand and dibutyl tin oxide in methanol, as outlined in Figure 1.

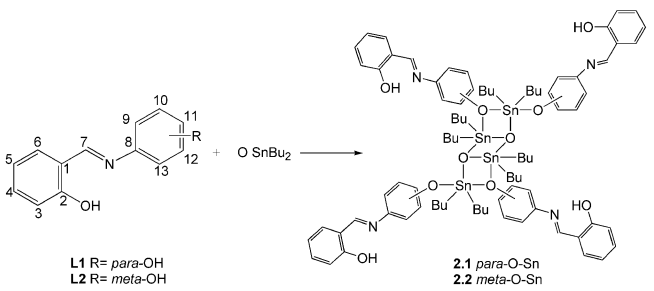

Figure 1. Stoichoimetric ratio of the proper ligand and dibutyl tin oxide in methanol.

IR spectroscopic analysis of complexes **2.1** and **2.2** shows characteristic absorption bands in the region for stretching and deformation of the phenoxy group $\nu_{\text{O–H}}$ = 3400 and 3379 cm^{-1}, indicating that only one phenoxy group is bonded to the tin

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metal atom. The spectrum also shows the Sn–O vibration band at 522 and 549 cm^{-1}, proving that the metal ligand bond is through this site. Additionally, a strong band in the region of imine group, $\nu_{N=C}$ = 1616 and 1582 cm^{-1}, was observed with values similar to those of the Schiff base ligands. This observation of the stretching and deformation bands in the phenoxy group indicates that the imine fragment does not participate in the coordinative bond.

NMR Spectroscopy

The evidence of the formation of the pentacoordinate species was provided by ^{119}Sn NMR spectrocsopy, which shows two different resonances for the two equivalent metal atoms at –173.4, –173.7 ppm for **2.1** and at –173.4, and –174.0 ppm for **2.2**. These two were assigned to the exo and endocyclic tin atoms of the corresponding tetrabutyldistannoxane with values of the chemical shifts similar to those reported for other tetraorganodistannoxanes [29]. The ^{1}H NMR spectrum showed multiple signals in the methylene region, attributed to the butyl group of the tin oxide fragment. A couple of triplets at 0.97 and 1.01 ppm for **2.1** and 0.86 and 0.91 ppm for **2.2** were assigned to the terminal methyl groups. The ^{1}H NMR pattern of the aromatic and the iminic protons is similar to that of the free, Schiff-base ligand, confirming that the nitrogen atom is not connected with the metal atom, as deduced from the IR spectroscopic data.

The ^{13}C NMR spectra of **2.1** and **2.2** are not substantially different from the free ligand and show two sets of signals for the *n*-butyl groups, agreeing with the presence of two different tin sites. The carbon atom C-12 of complex **2.2** is shifted to lower frequencies ($\Delta\delta$ = 8 ppm) with respect to the free ligand, probably induced by the O–Sn bond. Conversely, the carbon atom C-11 of complex **2.1** is slightly shifted to higher frequencies.

Crystal Structure

Complex **2.2** was crystallized from hexane. The ORTEP view is shown in Figure 2. Selected bond lengths and angles are listed in Table 1. The title compound exhibits a ladder-like structure, shaped by three planar, four-member Sn_2O_2 rings. The structure is a centrosymmetric dimer with the exocyclic tin atoms, Sn(2) and Sn(2A) and the endocyclic tin atoms, Sn(1) and Sn(1A), connected by two oxygen atoms. The Schiff base ligands are coordinated to the exocyclic tin atoms in a monodentate mode. The tin atoms adopt a distorted, pentacoordinate, trigonal, bipyramidal arrangement, in which the axial positions are occupied by the oxygen atoms from the O–Sn–O moiety, forming angles of 148.8(1)° and 143.2(1)° for Sn(2) and Sn(1), respectively. The equatorial plane contains the carbon atoms of the butyl groups, attached to the metal atom, and the triple-bridged oxygen atom, forming the following angles: C(39)–Sn(2)–C(35) 130.4(2)°, O(5)–Sn(2)–C(35) 116.9 (1)°, O(5)–Sn(2)–C(39) 110.7(2)° and C(27)–Sn(1)–C(31) 131.4(1)°, C(27)–Sn(1)–O(5) 111.5(1)°, and C(31)–Sn(1)–O(5) 115.9(1)°.

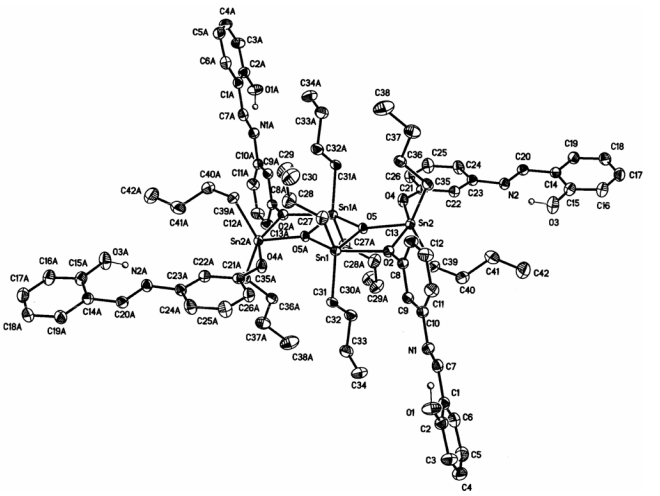

Figure 2. Perspective view of the molecular structure of compound **2.2** ORTEP (Thermal ellipsoids at 30 % of probability level).

Table 1. Bond lengths /Å and angles /° for compound **2.2**.

Bond lengths			
Sn(1)–O(2)	2.245(2)	Sn(2)–O(2)	2.299(2)
Sn(1)–O(5)	2.044(2)	Sn(2)–O(4)	2.089(2)
Sn(1)–O(5A)	2.101(2)	Sn(2)–O(5)	2.002(2)
Sn(1A)-O(5)	2.101(2)	Sn(2)–C(35)	2.120(3)
Sn(1)–C(27)	2.128(3)	Sn(2)–C(39)	2.139(7)
Sn(1)–C(31)	2.125(2)		
Bond angles			
O(2)–Sn(1)–O(5)	70.4 (1)	O(2)–Sn(2)–O(4)	148.8(1)
O(2)–Sn(1)–O(5A)	143.2 (1)	O(2)–Sn(2)–O(5)	70.0(1)
O(5)–Sn(1)–O(5A)	72.8(1)	O(4)–Sn(2)–O(5)	78.9(1)
C(27)–Sn(1)–C(31)	131.4(1)	C(35)–Sn(2)–C(39)	130.4(2)
C(27)–Sn(1)–O(2)	91.5(2)	C(35)–Sn(2)–O(2)	93.9(1)
C(27)–Sn(1)–O(5)	111.5(1)	C(35)–Sn(2)–O(4)	98.6(1)
C(27)–Sn(1)–O(5A)	102.5 (11)	C(35)–Sn(2)–O(5)	116.9(1)
C(31)–Sn(1)–O(2)	94.5(1)	C(39)–Sn(2)–O(2)	90.3(3)
C(31)–Sn(1)–O(5)	115.91(1)	C(39)–Sn(2)–O(4)	103.2(3)
C(31)–Sn(1)–O(5A)	100.8 (1)	C(39)–Sn(2)–O(5)	110.7(2)
Sn(1A)–O(5)–Sn(2)	133.6(1)		

The arrangement distortions could be associated with the presence of the intramolecular bond Sn–O Sn(1)–O(5A) 2.101(2) Å forcing the C–Sn–C angle to open and resulting in the deviation from the ideal value of 120°. The exocyclic tin atoms form two Sn–O bonds with the oxygen atoms. The bond length of Sn(2)–O(5) 2.002(2) Å is slightly shorter than that of Sn(2)–O(2) 2.299(2) Å and Sn(1)–O(5) 2.245(2) Å. However, all of the values are in agreement with those described for ladder-like structures [23–25] and are considerably lower than the sum of their van der Waals radii for tin and oxygen atoms of 3.68 Å. [31]

Antioxidant Activity

The antioxidant response of compounds **2.1** and **2.2** was tested for the inhibition of the formation of thiobarbituric acid-

Table 2. Anti-oxidant activity IC50 values for compounds **2.1** and **2.2** in Inhibition of Lipids Peroxidations TBARS[a)].

Complex	Concentration /μM	TBARS /μmol/mg prot	Inhibition /%	IC_{50} /μM
2.1	0	8.85 ± 0.66	–	6.11 ± 0.32
	2.75	6.27 ± 0.56**	29.37 ± 1.64	
	4.91	5.04 ± 0.45**	43.29 ± 1.11	
	8.73	3.63 ± 0.44**	59.39 ± 1.92	
	15.53	2.16 ± 0.40**	76.18 ± 2.58	
	27.61	0.87 ± 0.16**	90.42 ± 1.03	
2.2	0	7.62 ± 0.33	–	18.99 ± 0.55
	4.91	5.95 ± 0.33**	18.86 ± 3.26	
	8.73	5.43 ± 0.27**	28.50 ± 0.67	
	15.53	4.23 ± 0.19**	43.52 ± 2.35	
	27.61	3.09 ± 0.09**	59.91 ± 0.88	
	49.11	1.78 ± 0.07**	78.11 ± 1.55	
butylated hydroxy toluene BHT	0	8.84 ± 0.59	–	12.86 ± 0.64
	7.50	7.65 ± 0.42	8.16 ± 1.78	
	10	5.86 ± 0.78*	23.98 ± 6.41	
	13.34	3.90 ± 0.71*	54.99 ± 7.25	
	17.78	1.09 ± 0.21*	82.15 ± 6.98	
	23.71	0.79 ± 0.18*	90.27 ± 2.51	
quercetine	0	9.52 ± 0.19	–	4.11 ± 0.26
	1	8.33 ± 0.33	11.70 ± 2.14	
	1.78	7.64 ± 0.47*	19.29 ± 3.32	
	3.16	6.21 ± 0.24*	34.16 ± 3.05	
	5.62	3.20 ± 0.55*	66.05 ± 5.62	
	10	1.27 ± 0.29*	86.61 ± 2.92	
α-tocopherol	0	9.48 ± 0.79	–	569.09 ± 24.54
	100	8.43 ± 0.45	11.25 ± 2.71	
	177.83	8.01 ± 0.35	15.65 ± 2.07	
	316.23	7.49 ± 0.16*	21.39 ± 1.20	
	562.34	5.09 ± 0.37*	46.36 ± 3.38	
	1000	1.66 ± 0.44*	82.28 ± 4.80	

a) Each value represents the mean of 3–4 observations. The value $p \leq 0.05$ (*) and $p \leq 0.01$ (**) were considered as significant difference with respect to the standard.

reactive substances (TBARS). The results are summarized in Table 2. The α-tocopherol, quercetine, and butylated hydroxy toluene (BHT) were used as positive controls. The complexes showed that the percentage of inhibition for the lipid peroxidation depends upon the concentration. Complex **2.1** showed higher lipid peroxidation inhibition activity than the references BHT and α-tocopherol. However, both complexes **2.1** and **2.2** are less active than the quercetine. It is clear that the position of the phenol in the compound **2.1** decreases the IC_{50} value by three times, compared with complex **2.2**. Regarding the structure/activity relationship of phenolic antioxidants, *Zhang* and co-workers have previously reported that species without electron-donating substituents at the *para* or *ortho* positions of the hydroxyl group exhibit poor properties as antioxidants [32]. This leads us to hypothesize that the activity of **2.1** and **2.2** could be the result of the electron donating effect exerted on the hydroxyl group. These results are in complete agreement with those described for heptacoordinate tin, Schiff-base derivatives [33].

Conclusions

This contribution reveals that the salicilaldenaminoaryl alcohol derivatives react with dibutyltin oxides yielding organostannoxane, ladder-type complexes. The resulting ligand is connected with the tin metal atom by the oxygen atoms of one phenol group, leaving eight coordination sites available for connection with other metal atoms. These complexes displayed antioxidant activity superior to the α-tocopherol. The complex **2.1** exhibits the best inhibitory effect increase in activity because of the position of the phenol in the used ligand.

Experimental Section

3-Aminophenol, 4-aminophenol, salicylaldehyde, and dibutyltin oxide were purchased from the Aldrich Chemical Co. The ^{1}H-, ^{13}C- and ^{119}Sn-NMR spectra were recorded with a JEOL Eclipse +300. Chemical shifts (ppm) are relative to $(CH_3)_4Si$, and coupling constants are quoted in Hz. Melting points were measured with a Fisher Johns apparatus and are uncorrected. The elemental analyses were determined with an Exeter Analytical CE-440. The IR spectra were recorded with a Bruker Tensor 27. The X-ray crystallographic study of **2.2** was done with a Bruker Smart Apex CCD diffractometer with a $\lambda = 0.71073$ Å (Mo-K_α) graphite monochromator, at $T = 173$ K. The structure was solved by direct methods. All non-hydrogen atoms were refined anisotropically, using full-matrix, least square techniques. All hydrogen atoms were placed in idealized positions based on their hybridization with thermal parameters fixed at 1.2 times (for –CH) and 1.5 times (for $–CH_3$) the value of the attached atom. The butyl groups formed by the carbons C-27 to C-30 and C-39 to C-42 exhibited disorder in the two positions. Structure solutions and refinements were performed using SHELXTL v 6.10.

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Crystallographic data for complex **2.2**: $C_{84}H_{112}N_4O_{10}Sn_4$ M = 1812.54, triclinic, space group $P\bar{1}$, a = 11.318(1), b = 11.531(1), c = 16.684(2) Å, α = 89.713(2), β = 73.978(2), γ = 78.072(2)°, V = 2044.6(4) Å^3, Z = 1, ρ_{calc} = 1.472 g·cm^{-3}, T = 173(2) K, F(000) = 924, Crystal size 0.148 × 0.25 × 0.142 mm, color orange, reflections collected, 28313, Independent reflections 7513 R(int) = 0.0443, No. of variables 546, R_1 = 0.0268, wR_2 = 0.0594.

Crystallographic data in this paper have been deposited with the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB21EZ, UK. Copies of the data can be obtained on quoting the depository numbers CCDC-756277 (**2.2**) (Fax: +44-1223-336-033; E-Mail: deposit@ccdc.cam.ac.uk).

The assay for the antioxidant activity was determined by using the Inhibition of TBARS formation in a rat brain homogenate, as has been described previously [33].

Complex 2.1: Dibutyltin oxide (0.2338 g, 0.93 mmol) was added to a solution of the Schiff-base L1 (0.200 g, 0.93 mmol) in methanol (40 mL). The reaction mixture was heated under reflux for ten hours. Afterwards, the solvent was evaporated under reduced pressure, yielding yellow oil. The yellow oil was crystallized in methanol and provided 0.4391g (25.8 %) of yellow crystals; m.p. 125–127 °C. **^{1}H NMR** (300 MHz, $CDCl_3$): δ = 0.97 [t, J = 7.4 Hz, 6 H, CH_3-$(CH_2)_3$-Sn], 1.01 [t, J = 7.4 Hz, 6 H, CH_3-$(CH_2)_3$-Sn], 1.32–1.94 [m, CH_3-$(CH_2)_3$-Sn,12 H], 6.76 (d, J = 7.4 Hz, 1 H, H-9), 6.95 (t, J = 7.4 Hz, 1 H, H-5), 7.06 (d, J = 7.8 Hz, 2 H, H-3), 7.29 (d, J = 7.4 Hz, 2 H, H-10), 7.34 (d, J = 7.7 Hz, 1 H, H-6), 7.37 (t, J = 7.7 Hz, 2 H, H-4), 8.65 (s, 1 H, H-7). **^{13}C NMR** (300 MHz, $CDCl_3$): δ = 13.7, 13.8 (C_δ), 23.5, 25.2 (C_α), 27.1, 27.3 (C_β), 27.5, 27.6 (C_γ), 117.2 (C-3), 119.0 (C-5), 119.5 (C-10), 119.7 (C-1), 122.7 (C-9), 131.8 (C-6), 132.4 (C-4), 138.9 (C-8), 158.7 (C-7), 158.4(C-11), 161.1 (C-2). **^{119}Sn NMR** (112 MHz, $CDCl_3$): δ = –173.4, –173.7. **IR** (KBr): ν(OH) 3400, ν(C=N) 1619.7 cm^{-1}. $C_{84}H_{112}N_4O_{10}Sn_4$: C 55.74 (calcd. 55.60); H 6.19 (calcd. 6.16); N 3.24 (calcd. 3.08) %.

Complex 2.2: Dibutyltin oxide (0.2338g, 0.93 mmol) was added to a solution of the Schiff-base L2 (0.200 g, 0.93 mmol) and methanol (40 mL). The reaction mixture was heated under reflux for ten hours. Afterwards, the solvent was evaporated under reduced pressure, yielding yellow oil. The yellow oil was crystallized in hexane and provided 0.4285 g (25.2 %) of yellow crystals; m.p. 11 0 °C. **^{1}H NMR** (300 MHz, $CDCl_3$): δ = 0.86 [t, J = 7.4 Hz, 6 H, CH_3-$(CH_2)_3$-Sn], 0.91 [t, J = 7.4 Hz, 6 H, CH_3-$(CH_2)_3$-Sn], 1.30–1.83 [m, 24 H, CH_3-$(CH_2)_3$-Sn], 6.53–6.6.57 (m, 2 H, H-11, H-13,), 6.71 (d, J = 6.9 Hz, 1 H, H-9) 6.93 (t, J = 7.7, 1 H, H-5), 7.00 (d, J = 8.7 Hz, 1 H, H-3), 7.21 (t, J = 7.7, 2 H, H-10), 7.33 (t, J = 7.4, 2 H, H-4, H-6), 8.61 (s, 2 H, 1 H, C=N). **^{13}C NMR** (75 MHz, $CDCl_3$) : δ = 13.6, 13.7 (C_δ), 23.5, 25.1 (C_α), 27.1, 27.2 (C_β), 27.4, 27.5 (C_γ), 110.7 (C-9), 112.2 (C-13), 117.3 (C-3), 117.7 (C-11), 119.0 (C-5), 119.3 (C-1), 130.2 (C-10), 132.7 (C-4), 133.0 (C-6), 149.9 (C-12), 161.2 (C-2), 161.5 (C-8), 162.1 (C-7). **^{119}Sn NMR** (112 MHz, $CDCl_3$): δ = –173.7, –174.0. **IR** (KBr): ν(OH) 3379, ν (C=N) 1617 cm^{-1}. $C_{84}H_{112}N_4O_{10}Sn_4$: C 55.82, (calcd. 55.60); H 6.24 (6.16); N 3.08 (3.23) %.

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