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Synthesis, Characterization, and Antibacterial Evaluation of Dinuclear Organotin(IV) Complexes Derived from Oxalyldihydrazone-Based Ligands †

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Abstract

In this study, three novel oxalyldihydrazone-based ligands were synthesized through the condensation of oxalyldihydrazide with three different aldehydes: 5-bromo-2-hydroxybenzaldehyde, 2-hydroxynaphthaldehyde, and 2-hydroxy-3-methoxybenzaldehyde. These ligands were subsequently reacted with two equivalents of dimethyltin(IV) dichloride, resulting in the formation of symmetrical dinuclear organotin(IV) complexes in a 1:2 metal-to-ligand molar ratio. The synthesized ligands and their corresponding complexes were fully characterized using FT-IR spectroscopy, ^{1}H NMR, ^{119}Sn NMR, and elemental analysis. The spectral data confirmed successful coordination of the tin centers with donor atoms of the ligands, consistent with the proposed dinuclear structures. The antibacterial activities of the ligands and complexes were evaluated against both Gram-positive bacteria (*Staphylococcus aureus*, *Bacillus subtilis*) and Gram-negative bacteria (*Escherichia coli*, *Pseudomonas aeruginosa*). Notably, the organotin(IV) complexes displayed significantly enhanced antibacterial activity compared to their free ligands, especially against **P. aeruginosa*, a clinically important multidrug-resistant pathogen. This enhanced activity is attributed to the chelation effect, which increases the lipophilicity of the complexes, thus improving their ability to penetrate bacterial cell membranes. Furthermore, the proposed mechanism involves hydrolysis of the organotin complexes in biological media, allowing interaction with cellular enzymes and disruption of bacterial metabolic pathways. The findings suggest that these novel organotin(IV) complexes are promising candidates for further development as potent antibacterial agents.

Keywords: dinuclear complex; Dihydrazone; antibacterial activity

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1. Introduction

Hydrazone-based ligands and their metal complexes are an important group of coordination compounds because they contain several donor atoms and show a wide range of biological and industrial uses. Among them, acyl- and aroyl-dihydrazones are especially interesting due to their flexibility in binding metals and their ability to form mono- or multinuclear complexes with different geometries. These compounds are known for various biological effects, such as antibacterial, antifungal, and anticancer activities, and

are also useful in catalysis, pigment formation, analytical chemistry, and material science [1–5]. Bis(acyl/aroyl)dihydrazones, in particular, are attractive because they include two tridentate binding sites connected by a flexible linker. This structure allows them to coordinate metals through phenolic, azomethine, amide, and enolic groups. Although hydrazone coordination chemistry has been widely studied, dinuclear organotin(IV) complexes of these ligands have not yet been fully explored [6–8]. Organotin(IV) compounds are well known for their chemical flexibility, structural variety, and strong biological activities. The geometry around the tin atom and its bonding envi-ronment have a major effect on their reactivity and biological behavior [9,10]. Previous studies have shown that the structure and substituents of dihydrazone ligands strongly influence the physical and biological properties of organotin compounds. Based on these findings, the present work describes the synthesis and detailed characterization of new dimethyltin(IV) complexes containing oxalyldihydrazone ligands. The effect of ligand structure on the geometry and antibacterial activity of the products was investigated to better understand how structural changes influence the biological be-havior of binuclear organotin(IV) systems.

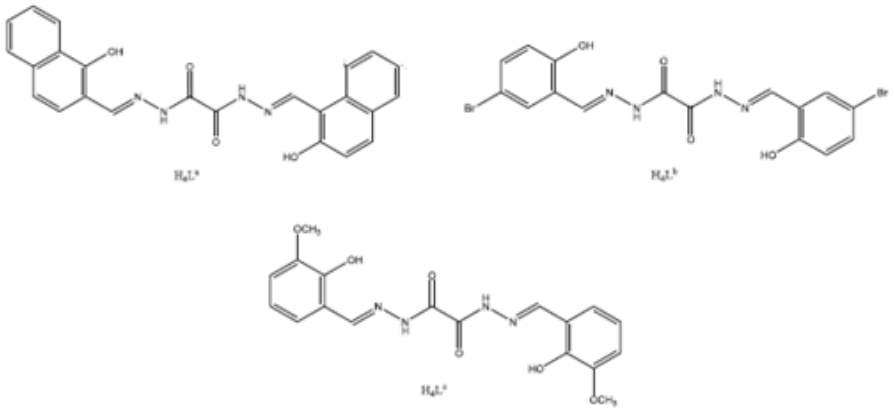

Figure 1. Structure of dihydrazone ligands.

2. Experimental

2.1. Materials and Methods

All reagents and solvents were of analytical grade and used as supplied. Most of the chemicals were purchased from Merck, while dimethyltin dichloride was obtained from Acros Organics. No additional purification was carried out prior to use. Infrared spectra were recorded on a BOMEM MB-102 FT-IR spectrometer using KBr pellets. Proton and carbon NMR spectra were obtained on Bruker Avance Ultrashield instruments, employing tetramethylsilane (TMS) and tetramethyltin ($SnMe_4$) as internal standards for calibration.

2.2. Synthesis of Bis(2-hydroxy-1-naphthaldehyde)oxalyldihydrazone (**H_4L^a**)

An ethanolic solution containing oxalyldihydrazide (0.098 g, 1 mmol) and 2-hydroxynaphthaldehyde (0.430 g, 2.5 mmol) was heated under reflux for approximately 7 h. Upon completion, the reaction mixture produced a yellow solid, which was separated by filtration, washed several times with hot ethanol, and then air-dried to obtain the purified ligand. ^{1}H NMR (DMSO-d_6, δ/ppm): 7.20 (d, 2H, H_1, ^{3}JHH = 8.7 Hz), 7.40 (m, 2H, H_4, ^{3}JHH = 7.6 Hz), 7.58 (m, 2H, H_5, ^{3}JHH = 7.7 Hz), 7.87 (m, 4H, $H_{2,3}$), 8.20 (d, 2H, H_6, ^{3}JHH = 8.6 Hz), 9.76 (s, 2H, HC=N), 11.75 (s, 2H, NH), 12.65 (s, 2H, OH). IR (KBr, cm^{-1}): ν(NH/OH) 3100–3200 (br), ν(C=O) 1659, ν(C=N) 1615. Yield: 0.310 g (82%); m.p. > 300 °C.

2.3. Synthesis of Bis(5-bromo-2-hydroxybenzaldehyde) Oxalyldihydrazone (**H_4L^b**)

A mixture of oxalyldihydrazide (0.12 g, 1 mmol) and 5-bromo-2-hydroxybenzaldehyde (0.57 g, 2.5 mmol) in ethanol was refluxed for 5 h. Upon completion of the reaction, a milky solid formed, which was filtered, washed several times with hot ethanol, and dried in air. ^{1}H NMR (400 MHz, DMSO-d_6, δ/ppm): 6.92 (d, 2H, H_1, ^{3}JHH = 8.5 Hz), 7.45 (dd, 2H, H_2, ^{3}JHH = 8.5 Hz, ^{4}JHH = 2.5 Hz), 7.77 (d, 2H, H_3, ^{4}JHH = 2.5 Hz), 8.79 (s, 2H, HC=N), 11.14 (s, 2H, NH), 12.82 (s, 2H, OH). IR (KBr, cm^{-1}): ν(OH) 3255, ν(NH) 3218, ν(C=O) 1708, ν(C=N) 1615. Yield: 0.35 g (90%); m.p. 247 °C.

2.4. Synthesis of Bis(2-hydroxy-3-methoxybenzaldehyde) Oxalyldihydrazone (**H_4L^c**)

H_4L^c was synthesized as described for H_4L^b, using oxalyldihydrazide (0.12 g, 1 mmol) and 2-hydroxy-3-methoxybenzaldehyde (0.19 g, 1.25 mmol) in ethanol. The mixture was refluxed for 5 h to afford a yellow solid, which was filtered, washed several times with ethanol, and dried in air. ^{1}H NMR (400 MHz, DMSO-d_6, δ/ppm): 3.70 (s, 6H, OCH_3), 6.80 (t, 2H, H_2, ^{3}JHH = 7.9 Hz), 7.05 (dd, 2H, H_1, ^{3}JHH = 8.0 Hz, ^{4}JHH = 2.2 Hz), 7.15 (dd, 2H, H_3, ^{3}JHH = 8.2 Hz, ^{4}JHH = 1.2 Hz), 8.77 (s, 2H, HC=N), 10.85 (s, 2H, NH), 12.68 (s, 2H, OH). IR (KBr, cm^{-1}): ν(O–H) 3452, ν(N–H) 3223, ν(C=O) 1708, ν(C=N) 1613. Yield: 0.13 g (72%); m.p. > 300 °C.

2.5. Synthesis of $(Me_2Sn)_2L^a$ (**1**)

A suspension of H_4L^a (0.106 g, 0.25 mmol) was prepared in 10 mL of ethanol and magnetically stirred for 30 min after the addition of triethylamine (1 mmol). Subsequently, an ethanolic solution (10 mL) of Me_2SnCl_2 (0.137 g, 0.62 mmol) was added gradually to the mixture. The reaction was maintained under reflux for approximately 5 h. After cooling to room temperature, the resulting white product was isolated by filtration, repeatedly rinsed with ethanol, and dried in air. Yield: 0.08 g (44%); m.p. > 300 °C. Anal. Cal. for $C_{28}H_{26}N_4O_4Sn_2$: C, 59.54; H, 3.51; N, 5.70%. Found: C, 59.16; H, 3.49; N, 5.31%. IR (KBr, cm^{-1}): 1610, ν (C=N); 580, ν (Sn–O); 450, ν (Sn–N). ^{1}H NMR (DMSO-d_6, δ ppm): 0.65 [s, 12H, $SnMe_2$, ^{2}J(^{119}Sn–^{1}H) = 86.0 Hz], 6.88 (d, 2H, H_1, ^{3}JHH = 9.1 Hz), 7.28 (t, 2H, H_4, ^{3}JHH = 7.6 Hz), 7.46 (t, 2H, H_5, ^{3}JHH = 7.7 Hz), 7.83–7.87 (m, 4H, $H_{2,3}$), 8.16 (d, 2H, H_6, ^{3}JHH = 8.7 Hz), 9.45 [s, 2H, HC=N, ^{3}J(^{119}Sn–^{1}H) = 40.4 Hz]. ^{119}Sn NMR (DMSO-d_6, δ ppm): −209.

2.6. Synthesis of $(Me_2Sn)_2L^b$ (**2**)

An ethanolic solution of H_4L^b (0.051 g, 0.10 mmol) was prepared in 20 mL of ethanol, followed by the addition of triethylamine (0.4 mmol) under continuous stirring. After maintaining the mixture under agitation for 30 min, a separately prepared solution of Me_2SnCl_2 (0.055 g, 0.25 mmol) in ethanol (10 mL) was added dropwise to the reaction flask. The combined mixture was then heated under reflux for approximately 7 h. Upon cooling, a yellow solid formed, which was collected by filtration, repeatedly washed with ethanol, and dried in open air (Figure 2). Yield: 0.042 g (48%); m.p. 243 °C. Anal. Cal. for $C_{20}H_{20}N_4O_4Sn_2Br_2$: C, 30.36; H, 2.76; N, 6.44%. Found: C, 30.00; H, 2.76; N, 6.61%. IR (KBr, cm^{-1}): 1602, ν (C=N); 639, ν (Sn–C); 581, ν (Sn–O); 486, ν (Sn–N). ^{1}H NMR (300 MHz, $CDCl_3$, δ/ppm): 0.78 [s, 12H, $SnMe_2$, ^{2}J(^{119}Sn–^{1}H) = 78.8 Hz, ^{2}J(^{117}Sn–^{1}H) = 75.3 Hz], 6.65 (d, 2H, H_2, ^{3}JHH = 8.8 Hz), 7.25 (d, 2H, H_5, ^{3}JHH = 2.7 Hz), 7.37 (dd, 2H, H_3, ^{3}JHH = 8.9 Hz, ^{4}JHH = 2.5 Hz), 8.50 [s, 2H, HC=N, ^{3}J(^{119}Sn–^{1}H) = 45.5 Hz]. ^{13}C NMR (62 MHz, $CDCl_3$, δ/ppm): 2.38 (C_{10}, C_{11}), 108.36 (C_4), 118.22 (C_6), 123.92 (C_2), 135.66 (C_5), 137.85 (C_3), 159.94 (C_7), 165.45 (C_1), 175.30 (C_8). ^{119}Sn NMR ($CDCl_3$, δ/ppm): –116**.**

2.7. Synthesis of $(Me_2Sn)_2L^c$ (**3**)

A homogeneous solution of H_4L^c (0.041 g, 0.10 mmol) in 20 mL of ethanol was first obtained under magnetic stirring. Then, triethylamine (0.4 mmol) was slowly added to facilitate the deprotonation of the ligand. After continuous stirring for approximately 30 min, an ethanolic solution of Me_2SnCl_2 (0.050 g, 0.25 mmol) was added dropwise to the reaction mixture. The combined solution was heated under reflux for 4 h, leading to the gradual formation of a yellow precipitate. The resulting solid was separated by filtration, rinsed several times with ethanol, and dried in air (Figure 2). Yield: 0.10 g (56%); m.p. 155 °C. Anal. Cal. for $C_{22}H_{28}N_4O_6Sn_2$: C, 40.59; H, 4.79; N, 7.89%. Found: C, 40.21; H, 4.31; N, 8.19%. IR (KBr, cm^{-1}): ν(C=N) 1609, ν(Sn–O) 562, ν(Sn–N) 460. ^1H NMR (300 MHz, $CDCl_3$, δ ppm): 0.84 [s, 12H, $SnMe_2$, $^2J(^{119}Sn–^1H)$ = 77.3 Hz, $^2J(^{117}Sn–^1H)$ = 75.6 Hz], 3.80 (s, 6H, OCH_3), 6.67 (t, 2H, H_8, ^3JHH = 7.8 Hz), 6.75 (dd, 2H, H_7, ^3JHH = 7.7 Hz, ^4JHH = 1.3 Hz), 6.94 (dd, 2H, H_9, ^3JHH = 7.7 Hz, ^4JHH = 1.27 Hz), 8.62 [s, 2H, HC=N, $^3J(^{119}Sn–^1H)$ = 46.5 Hz]. ^{13}C NMR (62 MHz, DMSO-d_6, δ ppm): 4.62 (C_{11}, C_{12}), 53.91 (C_{10}), 113.88 (C_4), 114.90 (C_8), 117.93 (C_7), 125.18 (C_9), 150.32 (C_3), 158.33 (C_5), 162.21 (C_6), 172.11 (C_2). ^{119}Sn NMR (111 MHz, $CDCl_3$): δ = −151 (Figure 3).

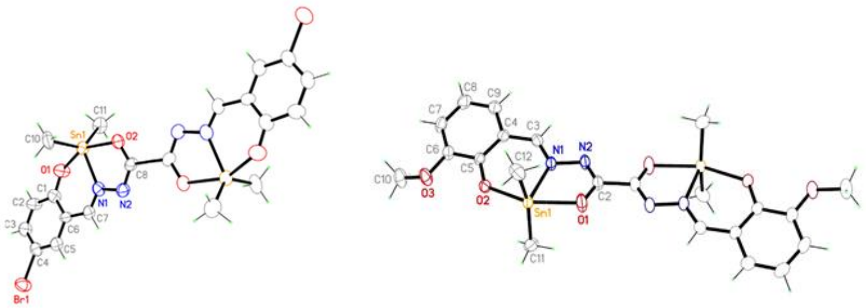

Figure 2. Perspective view of **2**, **3**.

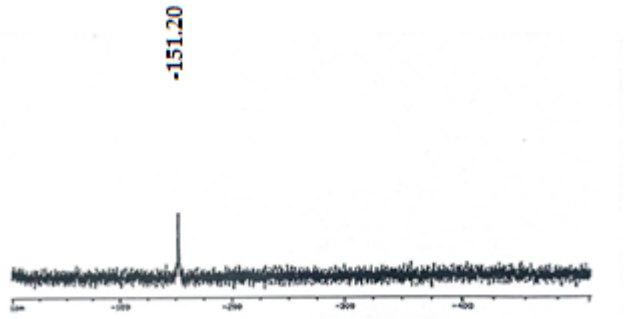

Figure 3. ^{119}SnNMR spectrum of **3**.

3. Results and Discussion

The ligands H_4L^a, H_4L^b, and H_4L^c were obtained through the condensation of oxalyl dihydrazide with 2-hydroxynaphthaldehyde, 5-bromo-2-hydroxybenzaldehyde, and 2-hydroxy-3-methoxybenzaldehyde, respectively. Each ligand contains four dissociable hydrogen atoms and behaves as a hexadentate chelating system composed of two tridentate segments joined by a flexible spacer. Reaction of these bis-acylhydrazone ligands with dimethyltin(IV) dichloride in the presence of triethylamine, using a molar ratio of 1:2:4 (ligand:base:organotin reagent), afforded the dinuclear complexes (**1–3**). A slight excess of Me_2SnCl_2 was used to ensure complete coordination. The resulting products were characterized by elemental analysis, IR, and NMR (^1H and ^{119}Sn) spectroscopy, which confirmed the formation of the expected molecular structures.

3.1. Antibacterial Tests

The antibacterial potential of the synthesized Schiff base ligands and their diorganotin(IV) derivatives was investigated in comparison with standard antibiotics, Nalidixic acid and Vancomycin. Tests were carried out against Bacillus subtilis and Staphylococcus aureus (Gram-positive), as well as Pseudomonas aeruginosa (Gram-negative), known for its multidrug resistance. The organotin(IV) complexes exhibited notably higher growth inhibition than the uncoordinated ligands, indicating that metal coordination enhances biological performance. This improvement can be attributed to increased electron delocalization within the metal–ligand framework, which enhances the complexes' lipophilicity and promotes easier diffusion through bacterial lipid membranes. Remarkably, complexes **1–3** showed significant activity even against Pseudomonas aeruginosa, which is typically resistant to conventional antibiotics. The differences in antibacterial response among the examined compounds could arise from variations in cell wall structure and permeability. The antibacterial tests were carried out using the disc diffusion assay, where DMSO solutions of each sample (10, 20, and 40 mg/mL) were applied on paper discs placed over freshly cultured bacterial plates.

Table 1.

Antibacterial activity data of ligands and their complexes.

Compound	Conc. (mg/mL)	Inhibition zone (mm)			
		E. coli	*P. aeruginosa*	*S. aureus*	*B. subtilis*
H_4L^a	10	10	10	12	12
	20	11	12	14	13
	40	12	13	16	15
H_4L^b	10	11	n.a.	13	9
	20	12	n.a.	15	11
	40	13	n.a.	16	13
H_4L^c	10	10	11	13	11
	20	11	12	14	12
	40	12	13	16	11
1	10	16	13	15	15
	20	17	14	18	19
	40	22	15	23	23
2	10	n.a.	12	19	13
	20	12	13	20	15
	40	13	14	22	16
3	10	10	n.a.	14	14
	20	12	n.a.	16	15
	40	13	n.a.	17	17
Vancomycin		13	n.a.	17	23
Nalidixic acid		24	n.a.	12	22

n.a. = no activity.

3.2. Spectroscopic Studies

The IR spectra of the prepared complexes showed that the ν(C=N) stretching bands shifted to lower frequencies compared with those of the free ligands, which confirms the coordination of the imine nitrogen atoms to the tin(IV) centers. In addition, new absorption bands appeared in the 400–600 cm^{-1} region, assigned to Sn–O and Sn–N stretching vibrations, supporting the involvement of both oxygen and nitrogen atoms in coordination. In the ^{1}H and ^{13}C NMR spectra of all complexes, only one set of signals was observed, indicating that both parts of the molecules are magnetically equivalent in solution. On the other hand, the spectra of the uncoordinated ligands showed several overlapping peaks, probably due to the presence of different tautomeric or conformational forms in solution. The disappearance of –OH proton peaks confirmed that the ligands were completely deprotonated and coordinated through oxygen atoms. The integration values of the proton signals were consistent with a 2:1 ligand-to-metal ratio. The imine protons also showed fine coupling with ^{119}Sn nuclei, confirming coordination through the azomethine nitrogen atoms. A single peak observed in the $^{119}Sn\{^{1}H\}$ NMR spectra of the complexes suggested the presence of one type of tin environment. The ^{119}Sn chemical shifts moved to lower fields compared with that of Me_2SnCl_2 (+137 ppm), which indicates an increase in the coordination number of tin after complex formation.

4. Conclusions

The spectroscopic analyzes clearly demonstrate that the bis-acylhydrazone ligands act in their completely deprotonated tetrabasic forms, coordinating through the imine nitrogen and phenolic/enolic oxygen atoms to the diorganotin(IV) centers. The obtained complexes are dinuclear, and each tin center exhibits a distorted trigonal bipyramidal geometry, as supported by the ^{119}Sn NMR results. The antimicrobial assays revealed that all synthesized complexes show enhanced inhibition compared to their uncoordinated ligands against both Gram-positive (*B. subtilis*, *S. aureus*) and Gram-negative (*E. coli*, *P. aeruginosa*) strains. Notably, in several cases, their activity surpassed that of the standard antibiotics, highlighting the potential of these dimethyltin(IV) complexes as effective antibacterial candidates.

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