

SYNTHESIS, CHARACTERIZATION, AND ANTIOXIDANT ACTIVITY OF NOVEL DIORGANOTIN(IV) COMPLEXES DERIVED FROM IBUPROFEN-5-AMINOSALICYLIC ACID LIGAND

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(Received June 21, 2024; Revised September 24, 2024; Accepted September 25, 2024)

ABSTRACT. This study focuses on the preparations of a new ligand prepared by the condensation reaction between two substances ibuprofen and 5-aminosalicylic acid, and complexes of diorganotin(IV) salts were prepared using this produced ligand to yield the appropriate complexes. The generated compounds were identified using FTIR and elemental analysis, in addition (tin, proton and carbon) magnetic resonance (^{119}Sn , ^1H , and ^{13}C , NMR). Octahedral geometry was suggested for the synthesized complexes based on the observations of the spectra. Two techniques were used to examine the antioxidant activity of the compounds: DPPH and CUPRAC. Because the tin element was present, the resultant complexes exhibited a higher rate of inhibition than the ligand. Di-methyltin-di-IAS also showed a greater effect as antioxidants than the other compounds.

KEY WORDS: Diorganotin(IV), Antioxidant activity, Ligand (IAS), DPPH method, CUPRAC method

INTRODUCTION

Compounds having organotin(IV) or simply organotins, are those organometallic compounds that include a minimum of one direct single bond C covalent bond. They have the general formula of $\text{R}_n\text{SnX}_{4-n}$; when $n = 1-4$, R is the organic part, and X can be any group that is anionic such as Cl^- , OH^- , etc. Based on the organic constituent quantity (n) linked to the Sn atom, they are classified into four different kinds of organotin(IV) compounds: tetra, tri, di, and mono, represented by the symbols RSnX_3 , R_2SnX_2 , R_3SnX , and R_4Sn , respectively [1, 2].

It has been found that organic tin(IV) compounds possess a variety of biological functions [3-6]. The complexes containing organic tin(IV) carboxyl radical have garnered a lot of interest due to their high biological activity. In comparison to other organic tin(IV) complexes which have different gaps [7-17]. Tin may form a wide range of stable complexes with a number organic ligands that contain electron-rich atoms, such as heteroatoms (like nitrogen, oxygen, sulfur, etc) [18]. The number and kind of substituents (aryl or alkyl moieties, for example) bonded to tin have a significant effect on the diversity and activity of these compounds [18, 19]. In current state, medical chemistry's fundamental objective is to meet the need for novel, more potent metal-based anti-cancer medications that either directly destroy cancer cells or stop them from growing without having a negative overall impact [20].

organotin(IV) complexes have shown encouraging activity, anticancer activity in vitro maybe even more effective than cisplatin when it comes to human tumor cell lines [21].

In particular, diorganotin complexes have appropriate biological activity and provide simple structural modification [22, 23]. Therefore, to enhance the therapeutic potential of the resulting organotin complexes, diorganotin compounds may contain ligand molecules with a variety of physiological roles to assist control their biological activity [24].

Variations in the aryl and alkyl alternatives of organotin(IV) have a significant impact on these complexes' biological activities employ organotin chemicals, such as antioxidants, in biological activities based on their response [25-29].

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The formation of novel carboxylates of organotins with varied structural characteristics will help in the research and creation of a wide range of medical and industrial uses, including the development of anti-tuberculosis, anticancer, antioxidant, plastic stabilizer, and polymer catalysts [30, 31].

Antioxidant is a substance that inhibits other molecules from oxidation where free radicals can be produced. Such radicals are electrons and hydrogen. Electrons or hydrogen are transferred from substances to an oxidizing agent during the chemical process of oxidation. In turn, Chain reactions can be initiated by these radicals, when the chain reactions happens in a cell, it can cause damage to cells or even kill them when they occur inside them. Antioxidants eliminate free radical intermediates, which prevents these chain reactions and prevent further oxidative reactions [32].

Techniques that use diphenylpicrylhydrazyl (DPPH) and CUPRAC were employed to evaluate the antioxidant activity of different diorganotin(IV)-IAS complexes. The purpose of this work is to study the antioxidant activity of diorganotin(IV) compounds and compare them with the ligand (IAS) and observe whether there is an improvement or decrease in the antioxidant activity of the complexes and the ligand.

EXPERIMENTAL

Materials

Every substance and solvent used were used without extra purification. Methanol, Ibuprofen, 5-aminosalicylic acid, dibutyltin dichloride, diphenyltin dichloride, dimethyltin dichloride, were all obtained from Sigma-Aldrich located in Schnelldorf, Germany.

Ligand synthesis

The ligand was prepared by reacting a suitable amount of substance Ibuprofen (4.1 g, 20 mmol) with (3.06 g, 20 mmol) substance 5-aminosalicylic acid (by converting the carboxylic group in ibuprofen to acyl group by SOCl_2 first then reaction with 5-aminosalicylic acid). Each substance was dissolved in 30 mL of methanol, and after that the two substances were mixed together in a condensing flask for 4 h. The precipitate was obtained by filtering, drying, and recrystallizing the resultant solution.

Synthesis of di-organotin(IV) complexes

The molar ratios for synthesizing the complexes (metal-ligand) are (1:2), respectively, where an appropriate amount was used of Ph_2SnCl_2 (0.688 g, 2 mmol), Bu_2SnCl_2 (0.607 g, 2 mmol) and Me_2SnCl_2 (0.439 g, 2 mmol) were added to the IAS solution after being dissolved in 30 mL of methanol that had been stirred (1.366 g, 4 mmol), in 30 mL methanol. For 5 h, this mixture was refluxed. To form the complexes precipitates, the resulting mixture was filtered, dried, and then crystallized again [19, 30].

Antioxidant activity tests

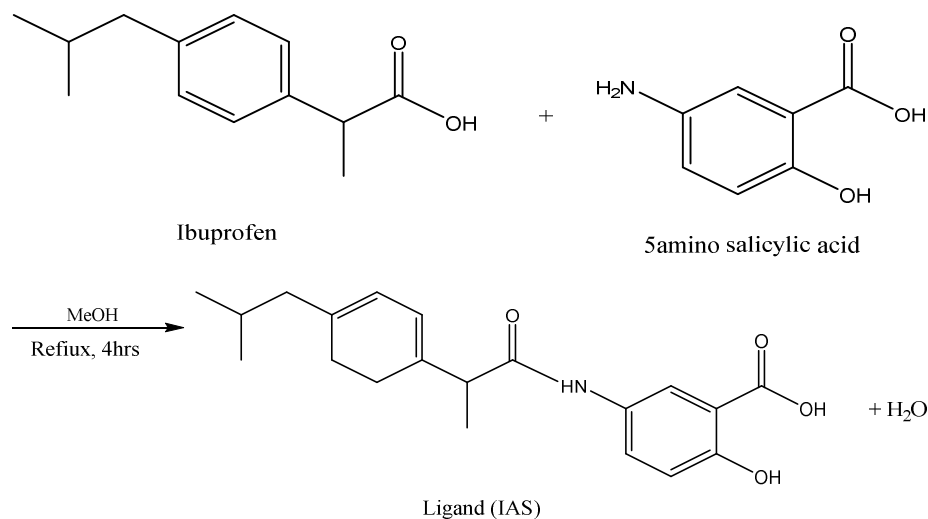
DPPH method. As previously mentioned, the 1,1-diphenyl-2-picrylhydrazine (DPPH) method was used to evaluate antioxidant activity [33, 34]. Methanol was used to dissolve the compounds at different concentrations: 2, 4, 8, 16, and 32 mM. Each tested solution mixed with a solution containing 0.1 mM of DPPH in methanol, which was added and well mixed. The mixed solution was removed after 30 min. The mixture's absorbance was measured at 517 nm using a UV-Vis spectrophotometer. The antioxidant activity was determined by calculating the proportion of inhibition against DPPH. The percentage inhibition was found by applying equation (1) [35]:

$$I\% = \left[\frac{\text{Control absorbance} - \text{Sample absorbance}}{\text{Control absorbance}} \right] \times 100 \quad (1)$$

CUPRAC method. The CUPRAC antioxidant activity test was conducted in accordance with other people's methods [35, 36].

$$\text{Total antioxidants levels} = \left[\frac{A_{\text{test}}}{A_{\text{STD}}} \right] \times \text{Conc of STD} \left(\frac{\text{mmol}}{\text{L}} \right) \quad (2)$$

RESULTS AND DISCUSSION

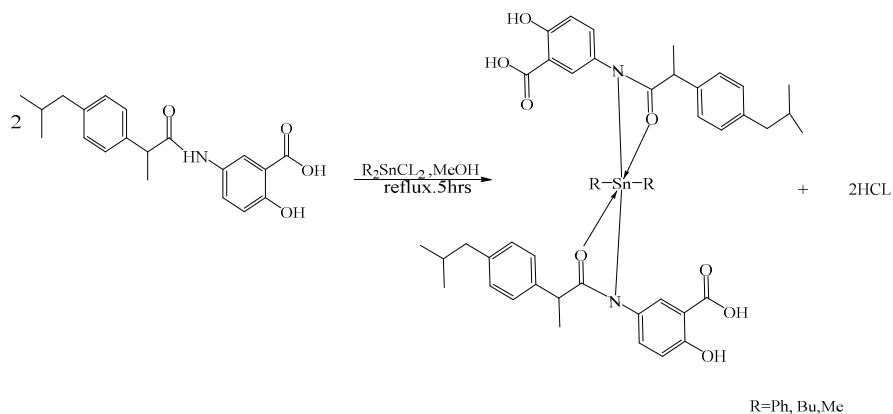


Scheme 1. Synthesis of Ibuprofen 5-amino salicylic acid (IAS).

Following an IAS reaction (4 mol equivalents) with Bu₂SnCl₂, Ph₂SnCl₂, and Me₂SnCl₂ (2 mol equivalents), conforming IAS di organotin(IV) complexes were produced with yield percentages of 82, 93, and 88.2% in a solvent of methanol with reflux for around 5 h (Scheme 2).

Table 1. The physical properties and elemental analysis of ligand and its di-organotin(IV) complexes.

Compound	Color	Yield (%)	m.p. (°C)	Elemental analysis % calculated (found)		
				C%	H%	N%
Ligand (IAS)	Gray	-	245-247	67.36 (66.79)	6.79 (6.66)	4.10 (3.99)
Ph ₂ Sn(IAS ₂)	Pale gray	93	269-271	60.49 (58.85)	5.71 (5.19)	2.94 (2.65)
Bu ₂ Sn(IAS ₂)	Black	82	246-248	57.10 (56.55)	6.84 (6.18)	3.07 (2.75)
Me ₂ Sn(IAS ₂)	Brown	88	240-242	54.81 (53.71)	6.08 (5.31)	3.38 (2.97)



Scheme 2. Synthesis of di-organotin(IV) complexes.

FTIR spectra

Displays the significant vibration numbers in the FTIR spectrum of ligand and its complexes. Complexes **1–3** possess pronounced peaks in the 544–546, 482–483 and 424–434 cm^{-1} in the FTIR spectra, which correspond to the vibrations of the Sn–C, Sn–O, and Sn–N groups, respectively [37]. Additionally, carbonyl group vibrations absorb much at (1650–1711 cm^{-1}). Also that the NH group disappeared in the complexes spectra.

Table 2. IR spectral data of ligand (IAS) and its diorganotin(IV) complexes.

Compounds	C=O	C-N	Sn-C	Sn-O	Sn-N
Ligand (IAS)	1719	1233	--	--	--
Ph ₂ Sn (IAS) ₂	1618	1119	544	482	424
Bu ₂ Sn (IAS) ₂	1715	1017	546	482	434
Me ₂ Sn (IAS) ₂	1653	1234	542	482	424

¹H-NMR spectra of di-organotin(IV) complexes

¹H-NMR spectrum of ligand (IAS) shows many signals related to the benzene ring at 7.09–6.77, NH group at 10.8, C-CH₃ at 1.34 and 0.84 ppm. Due to complexation, all of these signals were changed as can be seen in Table 3. To determine the synthesized compounds' geometrical shape, ¹¹⁹Sn-NMR analysis was conducted, when it rises to greater than 200, the complex was predicted to have an octahedral shape with six coordinations site [13].

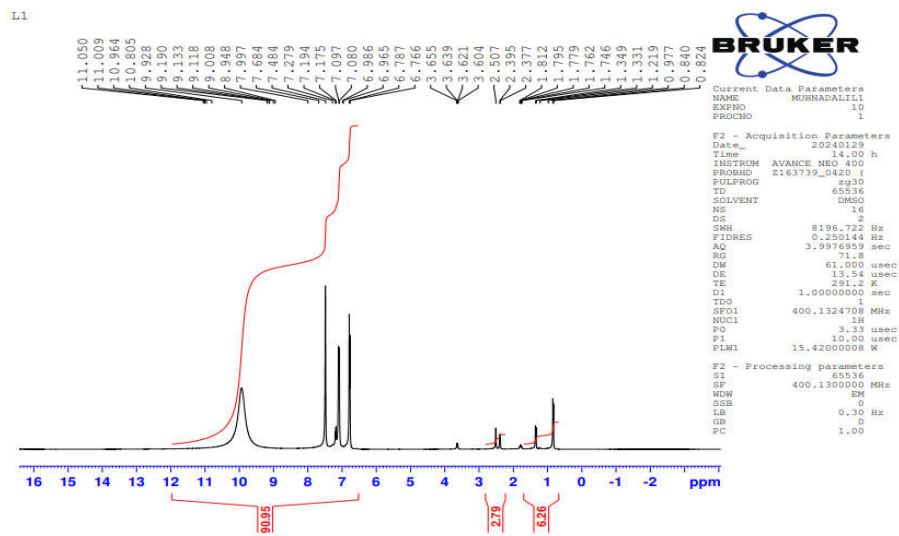


Figure 1. The ¹H-NMR spectra of ligand (IAS).

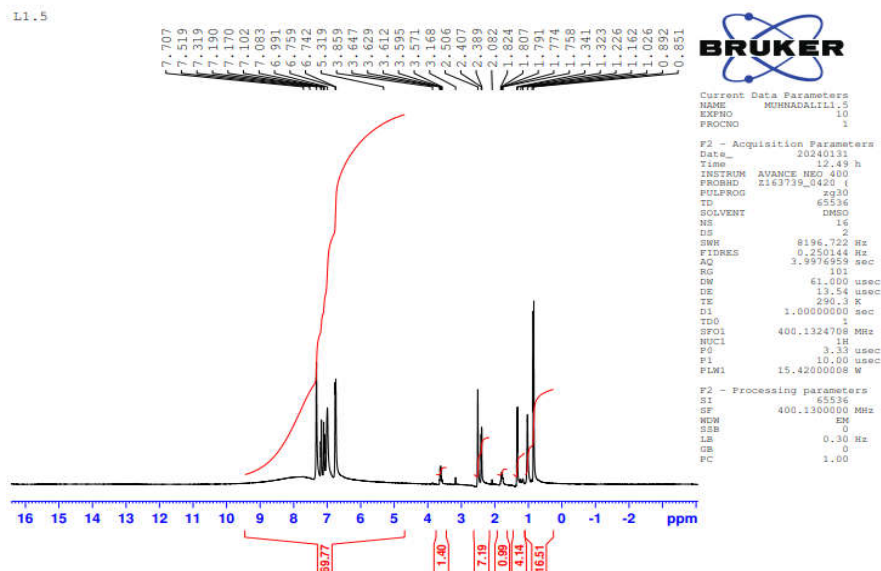
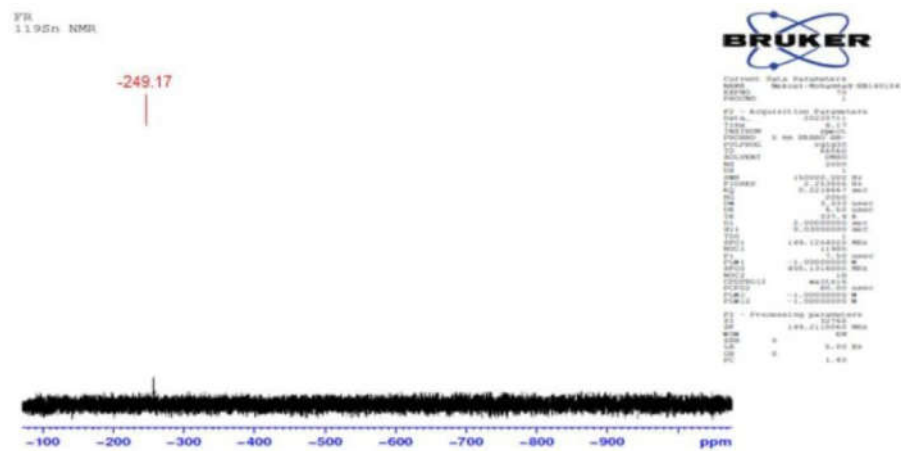


Figure 2. The ¹H-NMR spectra of complex (Me₂SnI₂).

Figure 3. ^{119}Sn -NMR spectrum of complex (Me_2SnI_2).Table 3. ^1H -NMR spectra of Ibuprofen 5 amino salicylic acid (IAS) and its complexes.

Compounds	^1H -NMR
Ligand (IAS)	10.8 (s, 1H, NH), 7.68–6.78 (m, 7H, Ar), 3.66 (s, 1H, CH), 2.39 (d, 2H, CH_2), 1.81 (s, 1H, CH), 1.34 (s, 3H, Me), 0.84 (s, 6H, 2Me).
$\text{Ph}_2\text{Sn}(\text{IAS})_2$	7.75 (s, 10H, Ar), 7.17 (m, 8H, Ar), 6.82 (d, 4H, Ar), 2.94 (s, 2H, 2CH), 41 (s, 4H, 2 CH_2), 1.81 (m, 2H, 2CH), 1.32 (s, 6H, 2Me), 0.87 (s, 12H, 4Me).
$\text{Bu}_2\text{Sn}(\text{IAS})_2$	7.29 (s, 2H, 2CH), 7.17 (d, 8H, Ar), 6.88 (s, 4H, 4CH), 3.56 (m, 2H, 2CH), 2.40 (d, 4H, 2 CH_2), 1.78 (m, 2H, 2CH), 1.32 (d, 12H, 2 $\text{CH}_2\text{CH}_2\text{CH}_2$), 0.88 (s, 18H, 6Me).
$\text{Me}_2\text{Sn}(\text{IAS})_2$	7.3 (s, 2H, 2CH), 7.17 (m, 8H, Ar), 6.9 (s, 4H, 4CH), 3.8 (s, 2H, 2CH), 2.4 (d, 4H, 2 CH_2), 1.82 (s, 2H, 2CH), 1.32 (s, 6H, 2Me), 0.89 (d, 18H, 6Me).

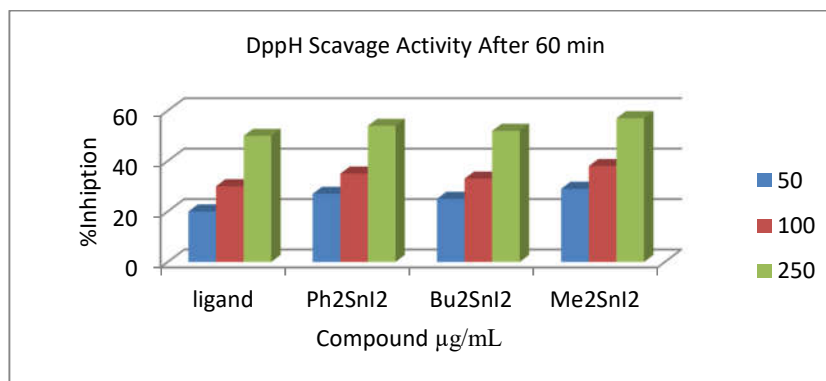


Figure 4. DPPH scavenging activity of ligand (IAS) and its diorganotin(IV) complexes.

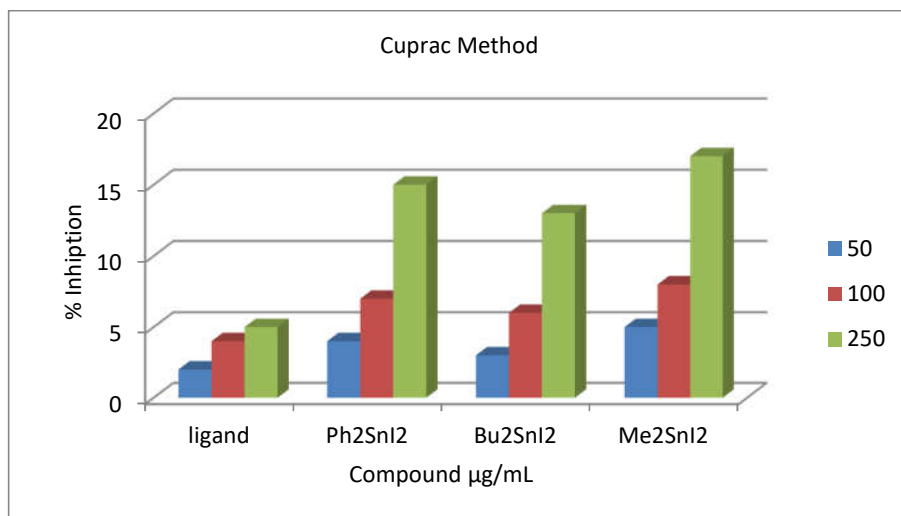


Figure 5. CUPRAC method activity of ligand (IAS) and its diorganotin(IV) complexes.

Antioxidant activity

The four synthesized compounds (ligand and its complexes) were examined in various amounts in the investigation of antioxidant activity using the two methods (DPPH, CUPRAC). According to the literature, the amount of metal moiety will increase the antioxidant activity of metallic complexes with ligands [38-40]. Because the metallic component increased the ligand's ability to donate protons as in Figures 4 and 5 [41]. The complexes prepared from ligand (IAS) had a significant level of antioxidant activity, according to the results compared to ligand alone, and this is because to the tin element's presence, which increased the antioxidant activity [41-45].

CONCLUSION

Two well-known methods were employed to evaluate the ligand's and its three novel tin(IV) complexes' antioxidant activities after they had been synthesized and characterized: DPPH and CUPRAC. Compared to ligand (IAS), the organotin(IV) complexes have more antioxidant activity. The complexes prepared from ligand (IAS) had a significant level of antioxidant activity, according to the results compared to ligand alone.

ACKNOWLEDGMENTS

The authors are appreciative of Babylon University's fantastic assistance.

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