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Enhanced Antioxidant Activity of Di- and Triorganotin Complexes Derived from Mefenamic Acid

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Abstract

Organotin **(IV) compounds have gained significant interest in both the chemical and pharmaceutical industry. This research** concentrates on synthesis of di and tri-organotin complexes with mefenamic acid and characterized with deferent techniques such as elemental analysis, FTIR, proton and carbon NMR. The anti-oxidant properties of the complexes were assessed using the 1,1-diphenyl-2-picrylhydrazyl, DPPH and CUPRAC Method . The complexes' scavenging activities were assessed and compared to those of the free drug. **The synthesized complexes gives higher antioxidant activity than free drug, also, complex 3** of di methyltin-mefenamic was the highest as compared with the other complexes.

Key words: **Antioxidant activities; Organotin(IV); Carboxylates; DPPH; Antitumor; Triorganotin(IV); Diorganotin** (IV).

1. Introduction

In the chemical and pharmaceutical industries, organotin (IV) compounds have gotten a lot of interest. Tin (IV) generates stable complexes with distinct structural, physical, and chemical properties that are utilized as a thermostat and catalyst in organic synthesis, as well as in the production of pharmaceuticals as biologically active compounds and in other fields. One of the most important types of chemicals is organotin carboxylates [1]. Organotin carboxylates are important in industry, the environment, and agriculture, in addition to their theoretical and structural interests [2-4].

Organotin(IV) compounds, particularly those produced from carboxylate ligands, have been extensively researched as bactericides [5-9], antitumoral [10-12], anti-inflammatory [13,14], and antifungal agents [15], wood preservatives and catalysts [16-18], and pesticides to find the best performance based on the ligand attached to the organometallic fragment and the origin.Organotin (IV) compounds' biological activities are determined by the length of the alkyl chain and the structure of the chemical, therefore the longer the alkyl chain, the less hazardous it is. [20,19] Triorganotin(IV) compounds, which have three Sn-C bonds, exhibit the maximum cytotoxicity [21-23], and compounds with aryl groups are less toxic than those without aryl groups [24, 25].Mineral complexes containing active medicines, such as ligands, are a burgeoning area of research in inorganic and medicinal chemistry, and have gotten a lot of attention as a way to produce new medications [26,27]. In the recent decade, new drivers have received a mixture of mineral complexes and non-steroidal anti-inflammatory medicines (NSAIDs). First, because they are based on pure coordination chemistry, NSAIDs are an extremely versatile coastline that can provide a wide range of bonding conditions depending on the metal and environment. Once the coordination compound has separated within the target tissues, the synergistic action of the mineral residues can boost the potency of the multiple

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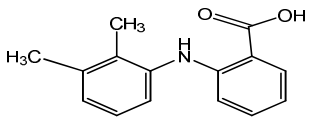
therapeutic agent created by combining two or more distinct types in the same compound. Some complexes have more pharmacological or biological activity than the drug, or they're intriguing from a strictly chemical standpoint [28-31].The carboxyl group in most NSAIDs, including aspirin, can coordinate metal ions. Non-steroidal anti-inflammatory medications, or NSAIDs, including N-phenyl anthranilic acid derivatives, such as mefenamic acid, tofenamic acid, and diclofenac sodium, are widely utilized in inflammatory and traumatic disorders in both rheumatic and non-rheumatic disorders.

Mefenamic acid isone of the analgesic drug that has been widely used in themarket (Scheme 1). This drug inhibits the enzyme cyclooxygenase (COX) 1 and 2.



Scheme 1. Chemical Structure of Mefenamic acid

We try to modify the structure of mefenamic acid to obtain greater activity through molecular approaches.The antioxidant activity of Sn(IV) compounds was investigated in this study and compared it to mefenamic acid and organic substituents of organotin (IV) compounds formed from mefenamic acid to see if there was an increase in antioxidant activity.

2. Experimental

General

KBr discs were used to record FTIR spectra (400–4000 cm^{-1}) using an FTIR 8300 Shimadzu spectrophotometer (Tokyo, Japan).

The elemental analyses were carried out using an EM-017mth instrument.Melting points were determined using an MPD Mitamura Riken Kogyo apparatus (Tokushima, Japan). On a Bruker DRX300NMRspectrometer, ^{1}H (300 MHz) and ^{13}C (125 MHz) NMR spectra were acquired (Zurich, Switzerland).

*Synthesis of di- organotin(IV) Complexes **1**–**3***

A boiling solution of di butyl, di phenyl, or di methyltin salt (one mmol) was slowly added to a refluxed solution of mefenamic acid (0.482 g, two mmol) in methanol (15 mL) and the combination was refluxed about 8 hours with constant stirring. After cooling, the solid precipitate was recovered and recrystallized to yield diorganotin(IV) **1** or **2** or **3**.

*Synthesis of Triorganotin(IV) Complex **4***

A boiling solution of tri phenyltin chloride (one mmol) in methanol (10 mL) was slowly added to a solution of (0.241 g, one mmol) mefenamic acid in methanol (10 mL), the combination was refluxed about 8 hours. After cooling, the solid crystals was recovered and re-crystallized, yielding triorganotin (IV) **4**.

DPPH free radical scavenging activity

An ethanol compound solution was added (0.1 mm) to a DPPH-ethanol solution in an equivalent volume. The chemicals' solution had a concentration of 0.1 mM. A control solution of ethanol was also employed. For a time dependency analysis of DPPH radical scavenging activity, absorbance was measured at 517 nm at room temperature after 20 and 60 minutes [32]. The practice was dubbed "radical scavenging" by the participants. Using the following equation, the DPPH radical scavenging activity of the compounds was calculated as a percentage reduction in the absorbance values of the initial DPPH solution:

$$I(\%) = (A_{blank} - A_{sample} / A_{blank}) \times 100 \quad ...(1)$$

Where A blank denotes the absorbance of the control sample (which contains all reagents except the testing chemical) and A sample denotes the absorbance of the experimental sample (which contains all reagents).

CUPRAC Method

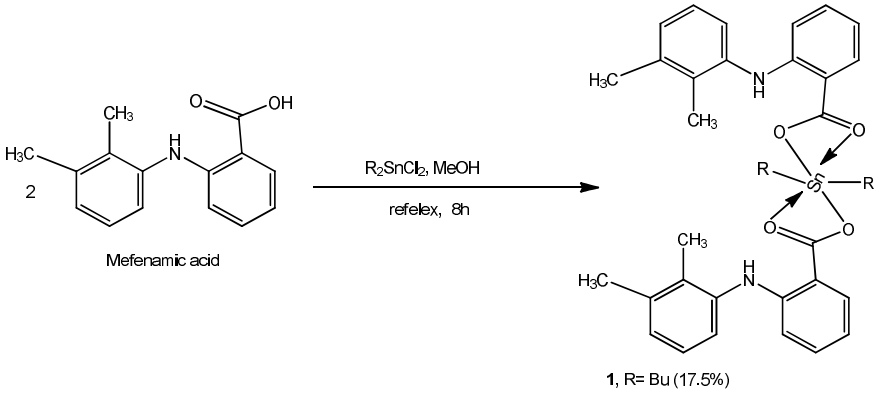
0.01M Copper(II) chloride solution was made by dissolving 0.4262g of copper(II) chloride in 250mL of water. 19.27 g of Ammonium acetate (NH_4Ac) was dissolved in 250 mL water to make a buffer solution (pH =7). By dissolving 0.039 g Neocuproine (Nc) 2,9-dimethyl-1,10-phenanthroline in 96 percent EtOH and diluting to 25 mL with ethanol, a 0.0075M Neocuproine (Nc)2,9-dimethyl-1,10-phenanthroline solution was generated [33].

Total antioxidants levals =(A_{test} / A_{STD})× Conc. of STD (mmole/L) ...(2)

3. Results and Discussion

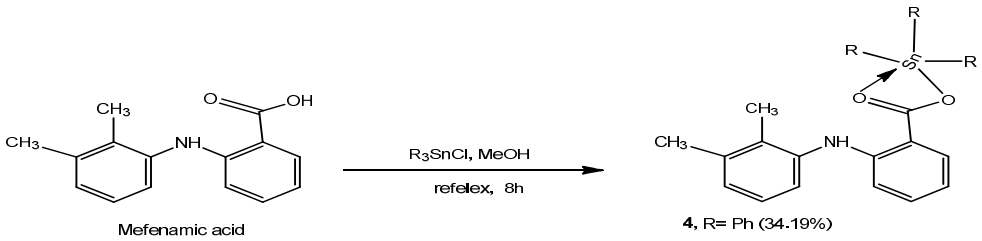
Synthesis of Organotin(IV) Complexes 1–4

Mefenamic acid reaction (2 mole equivalents) with Bu_2SnCl_2, Ph_2SnCl_2, or Me_2SnCl_2 in (methanol solvent) with reflux about 8 hours produced the coforming bis(mefenamic) di-organotin (IV) complexes **1–3** with yield percentage of 17.5% , 27.08% and 28.3% yield, respectively [2,4] (Scheme 2).



Scheme 2. Synthesis of Organotin (IV) Complexes

At the same method, the reaction of an equimolar mixture of mefenamic acid and triphenyl(IV) chloride in methanol under reflux for 8 hours gave the corresponding (mefenamic acid) triorganotin(IV) complex **4** with 34.19% yield, (Scheme3).



Scheme 3. Synthesis of Triorganotin (IV) Complex **4**

The Elemental Analysis and Physical properties of Organotin(IV) Complexes **1–4**, are shown in Table 1.

Table 1 The Elemental Analysis and Physical properties of Organotin(IV) Complexes **1–4**

Sn (IV)Complex	R	color	Yield	Melting point	Calcd. (Found); %		
					C	H	N
L	-	White	-	230-231	74.67(73.83)	6.27(6.07)	5.81(6.21)
1	Bu	White	17.5	118-120	63.97(62.88)	6.5(6.23)	3.93(3.57)
2	Ph	Light green	27.08	134-136	66.95(65.69)	5.08(6.15)	3.72(3.1)
3	Me	White	28.30	213-215	61.07(62.14)	5.45(6.35)	4.45(5.02)
4	Ph	White	34.19	123-125	67.14(66.85)	4.95(5.32)	2.37(3.11)

Table 2 shows the important FTIR spectrum vibrations numbers of compounds **1–4**. Complexes **1–4** have prominent peaks in the 526–536 cm^{-1} and 445–447 cm^{-1} FTIR spectra, which correspond to Sn–C and Sn–O group vibrations, respectively [34]. Also they have high absorption at (1685–1697 cm^{-1}) that correlates to carbonyl group vibrations.

Table 2. FTIR Spectral Data of Complexes **1–4**.

Sn (IV)Complex	FTIR (υ, cm^{-1})				
	N-H	C=O	C=C	Sn-C	Sn-O
1	3323	1653	1456	524	449
2	3311	1739	1456	520	430
3	3309	1660	1450	520	445
4	3313	1651	1452	522	449

*^{1}H-NMR Spectroscopy of Organotin(IV) Complexes **1–4***

NMR spectroscopy was used to confirm the structures of organotin (IV) complexes **1–4.**The NMR shows all of the expected signals for all of the predicted chemical shifts (Table 3). However, inside the aromatic area, multiple signals are visible in the ^{13}C-NMR spectra of **1–4**. (Table 4).

Table 3. Complexes **1–4** ^{1}H-NMR Spectral Data (ppm, DMSO-d6)**1–4.**

Sn(IV) Complex	^{1}H-NMR
L	13.005(s, 1H, OH), 9.48(s, H, NH), 6.65-7.89(m, 6H, Ar), 2.501(s, 3H, CH_3), 2.08(s, 3H, CH_3).
1	9.47(s, H, NH), 6.67-7.97(m, 6H, Ar), 2.50(s, 3H, CH_3), 2.04(s, 3H, CH_3), 1.86(s, 2H, CH_2), 1.46-1.70(m, 5H, CH_2)0.8-0.87(m, 2H, CH_3).
2	9.45(s, H, NH), 7.86-7.89(m, 12H, Ph), 6.65-7.35(m, 6H, Ar), 2.50(s, 3H, CH_3), 2.11(s, 3H, CH_3).
3	9.44(s, H, NH), 6.67-7.88(m, 6H, Ar), 2.49(s, 3H, CH_3), 2.08(s, 3H, CH_3), 1.01(s, 3H, Me).
4	9.46(s, H, NH), 7.83-7.89(m, 18H, Ph), 6.66-7.48(m, 6H, Ar), 2.50(s, 3H, CH_3), 2.09(s, 3H, CH_3).

^{13}C-*NMR Spectroscopy of Organotin(IV) Complexes **1–4***

Table 4. ^{13}C-NMR Spectral data (ppm, DMSO-d6) of Complexes **1–4**.

Sn(IV) Complex	C^{13}-NMR
L	172.70(C=O), 149.217(C-NH), 138.315(C-NH), 134.31(C-CH_3), 132.59, 126.83, 126.45, 122.60, 116.67, 113.52, 20.65, 14.09.
1	170.71(C=O), 149.21(C-NH), 138.81(C-NH), 138.31(C-CH_3), 134.59, 132.18, 131.66, 126.83, 126.45, 122.6, 116.67, 113.52, 111.75, 33(C-Sn), 27(CH_2-Bu), 24(CH_2-Bu), 13(CH_3-Bu), 14.09 and 20.65 CH_3.
2	171.21(C=O), 149.62(C-NH), 138.83(C-NH), 138.34(C-CH_3), 134.65, 132.20, 131.70, 126.88, 126.48, 122.66, 116.72, 113.55, 111.73, 14.11 and 20.67 CH_3.
3	171.24(C=O), 149.64(C-NH), 138.33(C-NH), 138.34(C-CH_3), 134.64, 132.21, 131.70, 126.87, 126.48, 122.65, 116.72, 113.55, 111.73, 14.11 and 20.67 CH_3.
4	170.21(C=O), 149.62(C-NH), 136.29(C-NH), 136.24(C-CH_3), 129.53, 129.48, 129.12, 128.90, 128.85, 128.80, 128.78, 128.57, 111.71, 14.14 and 20.54 CH .

Antioxidant activities

There have been reports in the literature of metallic complexes with antioxidant activity in the ligand, and it is believed that the metal moiety will boost that activity [35-37]. Because the ligand's proton donor capacity was improved by the addition of the metallic moiety, the ligand's antioxidant activity was increased as in Figures 1 and 2.

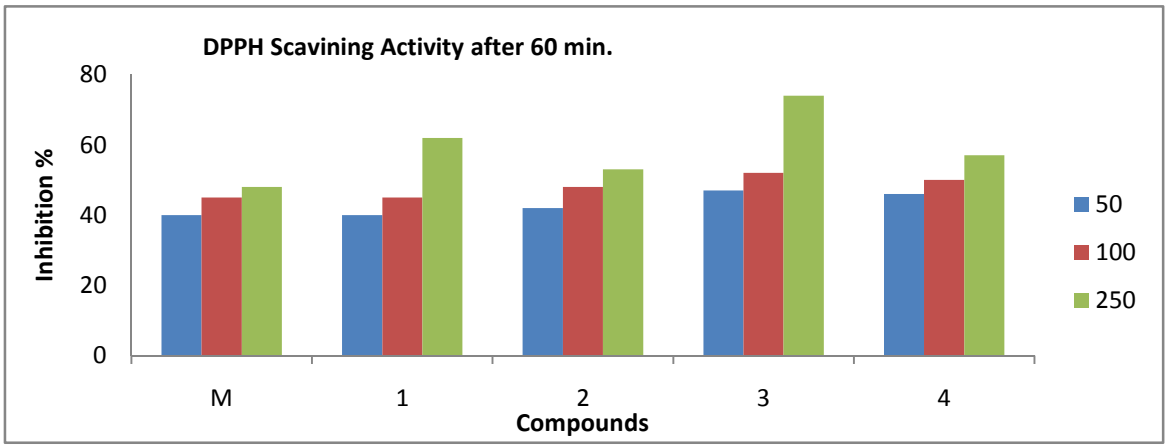


Figure 1. DPPH scavenging activity of Mefenamic acid (M) and its complexes were analyzed at 50-250 µg/mL DMSO solutions at T = 1 hour

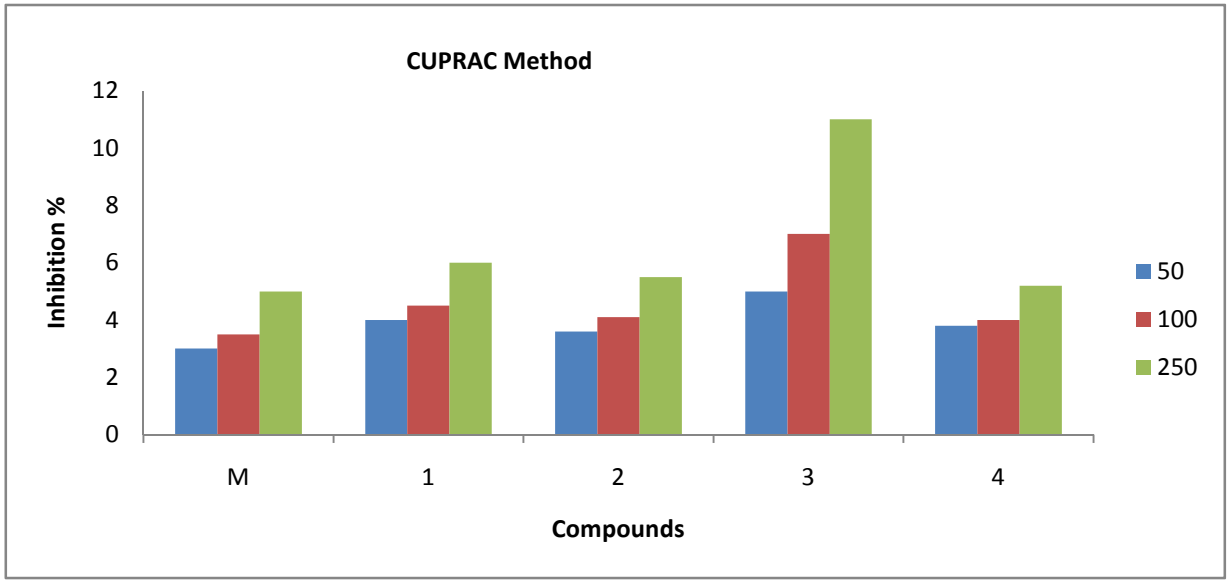


Figure 2. CUPRAC Method activity of Mefenamic acid (M) and its complexes at 50-250 µg/mL DMSO solutions at T = 1hour.

All complexes have higher scavenging activity than the pure drug (mefenamic acid) [38,39], but complex 3 (dimethyltin–mef) has the highest; this is due to the symmetric complex's stability, as well as the fact that this complex contains greater tin content than the others, resulting in increased antioxidant ability[40].

The application of both of the aforesaid procedures (CUPRAC and DPPH) took place at two periods: 20 and 60 minutes. The results were the same for both times, indicating that time had no bearing on the outcomes.

4. Conclusions

The reaction with mefenamic acid produced four novel organotin(IV) complexes. The synthesized complexes' structures were approved. The antioxidant activity of mefenamic and the complexes produced from it was calculated using the DPPH and Cupric techniques. The most active derivative was Complex 3

5. Conflicts of interest

The authors declare no conflict of interest.

6. Formatting of funding sources

The authors declare that they didn't receive any type of fund.

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