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SYNTHESIS, STRUCTURAL ELUCIDATION, AND ANTIOXIDANT EVALUATION OF DI- AND TRIORGANOTIN (IV) COMPLEXES WITH MODIFIED 4-AMINOANTIPYRINE LIGAND

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A novel ligand, 2-[(1,5-di methyl -3- oxo -2- phenyl pyrazolidin -4-yl) amino] acetic acid (DPA), was synthesized by reacting of 4-aminoantipyrine with chloro acetic acid. This prepared ligand was (DPA), then used to prepare triphenyltin(IV), dibutyltin(IV), and dimethyltin(IV) complexes through condensation reactions with the corresponding organotin(IV) chloride salts (Ph_3SnCl, Bu_2SnCl_2, and Me_2SnCl_2) respectively. The synthesized complexes were methodically characterized by using infrared spectroscopy (IR), multinuclear magnetic resonance spectroscopy (^1H and ^{119}Sn NMR), and elemental analysis, confirming their structural integrity. The antioxidant ability of DPA and its organotin(IV) complexes was evaluated via two established methods: DPPH (2, 2- diphenyl-1- picrylhydrazyl) radical scavenging and CUPRAC (cupric ion reducing antioxidant capacity) assays. Results showed that both triorganotin(IV) and diorganotin(IV) complexes exhibited significantly higher inhibition percentages compared to the free DPA ligand, underscoring the critical role of the Sn(IV) centers in increasing antioxidant ability. Notably, the triphenyltin(IV) carboxylate complex demonstrated superior performance in both assays, attributed to its distinct electronic and steric properties arising from the phenyl substituents.

Keywords: Chloro acetic acid, condensation, carboxylate complex, organotin substituents, antioxidant ability, coordination geometry, lipophilicity.

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INTRODUCTION

Organotin(IV) complexes (OTS) have garnered large attention in medicinal and materials chemistry due to their diverse structural architectures and biological activities, including anticancer, antimicrobial, and antioxidant properties [1, 2]. The biological efficacy of these complexes is highly influenced by the ligand framework and the organometallic moiety, which modulate electronic and steric environments [3, 4]. Ligands derived from 4-aminoantipyrine, a pyrazolone derivative with inherent pharmacological activity, are particularly promising due to their versatile coordination modes and ability to enhance metal-centered reactivity [5, 6].

Modification of 4-aminoantipyrine to generate functionalized ligands, such as carboxylate or Schiff base derivatives, has been widely explored to optimize metal-ligand synergism [7, 8]. For instance, chloroacetic acid-functionalized ligands enable the formation of stable organotin(IV) complexes via condensation reactions, leveraging the carboxylate group's chelating ability [9, 10]. Structural characterization of such complexes is typically achieved through spectroscopic techniques like IR, which identifies coordination modes [11], and multinuclear NMR ($^{1}H/^{119}Sn$), which provides insights into tin coordination geometry [12, 13].

Antioxidant evaluation of organotin(IV) complexes is critical, as oxidative stress underpins numerous pathologies, including inflammation and neurodegeneration [14]. The DPPH and CUPRAC assays are widely used to quantify radical scavenging and metal-reducing capacities, respectively [15, 16]. Studies indicate that tin(IV) centers enhance antioxidant activity by stabilizing radical intermediates or facilitating electron transfer [17, 18]. For example, triorganotin(IV) complexes often outperform their diorganotin counterparts due to increased lipophilicity and tailored steric effects [19, 20].

In this study, we synthesized a novel 4-aminoantipyrine-derived ligand, 2-[(1,5-di methyl -3- oxo -2- phenyl pyrazolidin-4-yl) amino] acetic acid (DPA), and its triphenyltin(IV), dibutyltin(IV), and dimethyltin(IV) complexes. The complexes were characterized spectroscopically, and their antioxidant activities were systematically compared. Our findings align with prior reports on the superior bioactivity of triorganotin species while highlighting the role of phenyl substituents in enhancing radical scavenging efficacy, as observed in related systems.

EXPERIMENTAL PART

MATERIALS AND METHODS

General

Chemicals, reagents, and solvents from Merck were used (Gillingham, UK). A Shimadzu FTIR 8300 spectrophotometer (Kyoto, Japan) was used to record FT-IR spectra in the 400–4000 cm-1 spectral area using the KBr disc method. Elemental analyses were carried out using the Vario EL III apparatus (Analysensysteme GmbH, Hanau, Germany). The melting points were recorded using a Mitamura Riken Kogyo MPD melting point device (Tokushima, Japan). 1H- (300 MHz) spectra in DMSO-d6 were recorded using a Bruker DRX300 NMR spectrometer (Bruker, Zurich, Switzerland). A Q-Panel tester (Homestead, Florida) was used for UV radiation studies.

Preparation of Ligand

The ligand was prepared by reacting a suitable amount of substance 4-Aminoantipyrine (0.203 g, 1 mmol) with (0.094 g, 1 mmol) substance of chloro acetic acid in methanolic solution stirred about 30 min. at room temp. The yellow precipitate of ligand (DPA) was filtered and dried

Preparation of Triphenyl Tin(IV) Complex 1

The prepared ligand (DPA) in methanolic solution (1 mmol, 0.261 g) was stirred with (1 mmol, 0.4 g) NaOH about 30 minute at room temperature. A (1 mmol, 0.385 g) from triphenyl tin chloride (Ph_3SnCl) was dissolved in 30 ml hot methanol then added to DPA ligand, left to reflux about 4 h with continuously stirred [21- 23]. The yellow precipitate was left to evaporate under vacuum, washed with diethyl ether and dried.

Preparation of di-Butyl or Methyltin(IV) Complexes 2-3

30 ml methanolic solution of prepared ligand (DPA) (2 mmol, 0.522 g) was mixed with NaOH (2 mmol, 0.8 g) for 30 min at room temperature. (1 mmol, 0.303 g or 0.22 g) from dibutyl or dimethyltin dichloride (Bu_2SnCl_2 or Me_2SnCl_2) was dissolved in 20 ml of hot methanol then added to (DPA) mixture, left to reflux for 4 h with continuously stirred [24- 27]. The precipitate was left to evaporate under vacuum, washed with diethyl ether and dried.

Antioxidant Activity Tests

DPPH technique

Antioxidant activity was measured using the 1,1-di phenyl-2-picrylhydrazine (DPPH)

method, as explained by researchers [28, 29]. The compound was dissolved in MeOH at special concentrations from (2 - 32 M). A solution of (0.1 mM DPPH in MeOH) can be added to the test miture and mixed with careful. After thirty min, the mixture was waste. the mixture's absorbance was measured at a UV-vis spectrophotometer at (λ_{max} = 517 nm). The inhibition percentage was measured by using equation (1);

$$Inhibition\ Percentage = (Control\ Absorbance - Sample\ Absorbance) \times Control\ Absorbance)^{-1} \times 100 \quad (1)$$

CUPRAC Method

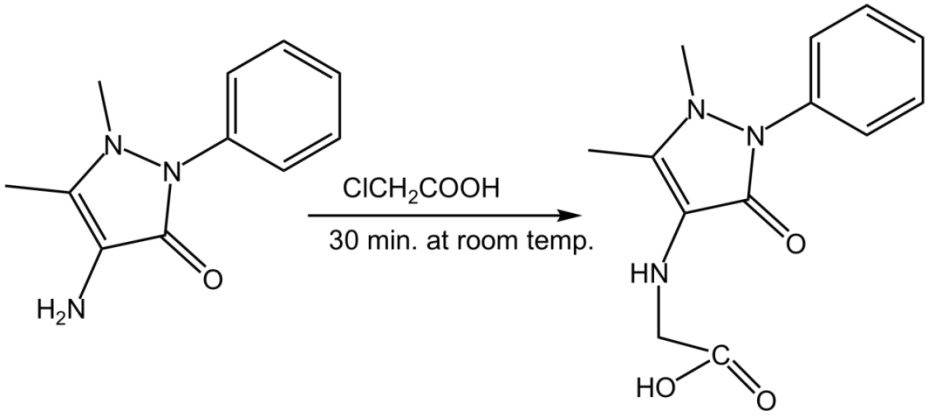
CUPRAC method was used as a second teqnique to test antioxidant activity , according to method used by others [30].

$$Total\ antioidants\ levals\ = \left[\frac{A\ test}{A\ STD}\right] \times Conce.of\ STD\ \left(\frac{mmole}{L}\right) \quad (2)$$

RESULTS AND DISCUSSION

Preparation of ligand DPA

The synthesis of the preparation of new ligand DPA was expresed in Scheme 1 by stirring 4-aminoantipyrine with chloroacetic acid for 30 min and at room temperture.



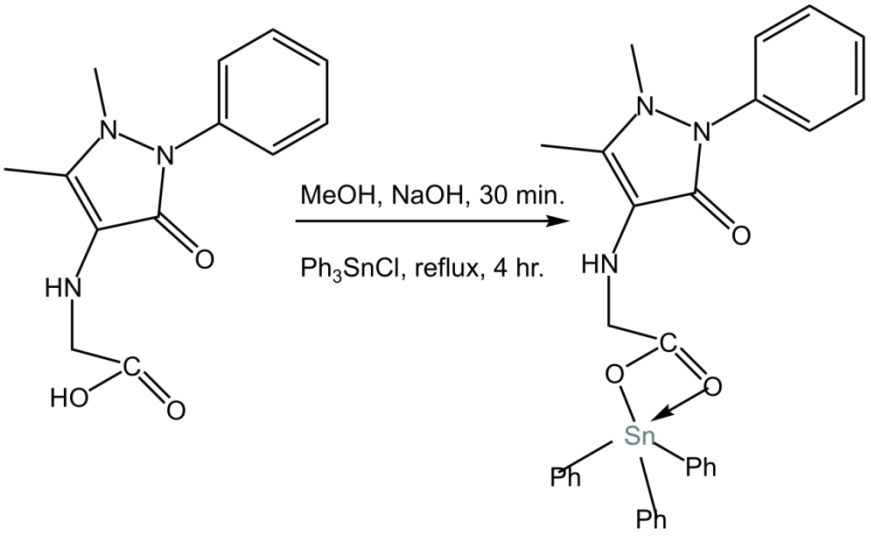
Scheme 1. Shows Synthesis of New Ligand DPA.

Synthesis of DPA- Organotin(IV) Complexes 1–3

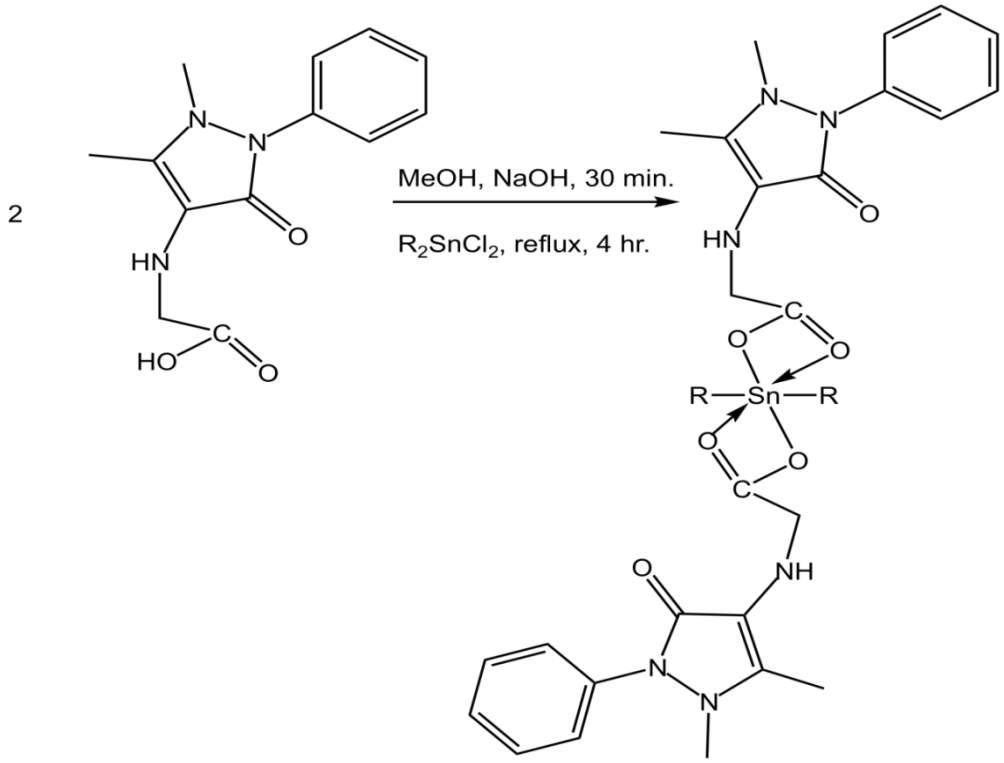
The novel complexes of triphenyl, di-butyl or dimethyltin were achieved via refluxing reaction of tri and di-organotin chloride in methanolic solutions with ligand DPA, to give a large yield percent of complexes (Scheme 2 and 3).

The resulting compounds were characterized via using various techniques for example FT-IR, NMR (^{1}H and Sn^{119}) spectroscopy methods, in addition to the elemental analyses. Tables 1, 2 and 3 review the results of every method.

FT-IR spectral data of DPA distinguish by many bands such as 3433-3329 cm^{-1} absorption of that related to amine group. Carboxyl group appeared as broad band at 2500-3100 cm^{-1}.The CH stretching frequency absorb at 3000-2900 cm^{-1} . Because of complexation, these bands were shifted. At 516-520cm^{-1} and 445-440 cm^{-1} Sn-O band Sn-N band were appeared respectively. complexes.



Scheme 2. Synthesis of Ph_3Sn(DPA) Complex 1.



Scheme 3. Synthesis of Bu_2SnCl_2 and Me_2SnCl_2 Complexes(R=Bu or Me).

Table 1. Physical Analysis Data of DPA with its Complexes.

Comp.	Color	Yield percent	Melting point(°C)	Elemental analysis % Calculated (Found)		
				C	H	N
Ligand- DPA	Pale-Yellow	93.2	120-122	50.57(51.06)	6.13(6.71)	32.18(32.93)
Ph_3SnL	Off white	91.5	230-232	63.85(64.15)	11.55(12.21)	15.41(15.43)
Bu_2SnL_2	Orange	93.6	220-221	28.67(28.92)	4.65(5.22)	10.56(10.97)
$Me_2\ SnL_2$	Grey	90.3	212-214	24.76(25.13)	3.81(4.21)	13.33(14.01)

Table 2. FT-IR Spectral data of DPA and its

Comp.	NH	C=O	C=C	M-O	M-N
DPA	3430-3326	1652	1590	-	-
Ph_3- DPA	3406-3323	1643	1572	520	440
Bu_2 $(DPA)_2$	3407-3328	1647	1591	516	445
Me_2 $(DPA)_2$	3405-3324	1645	1589	518	442

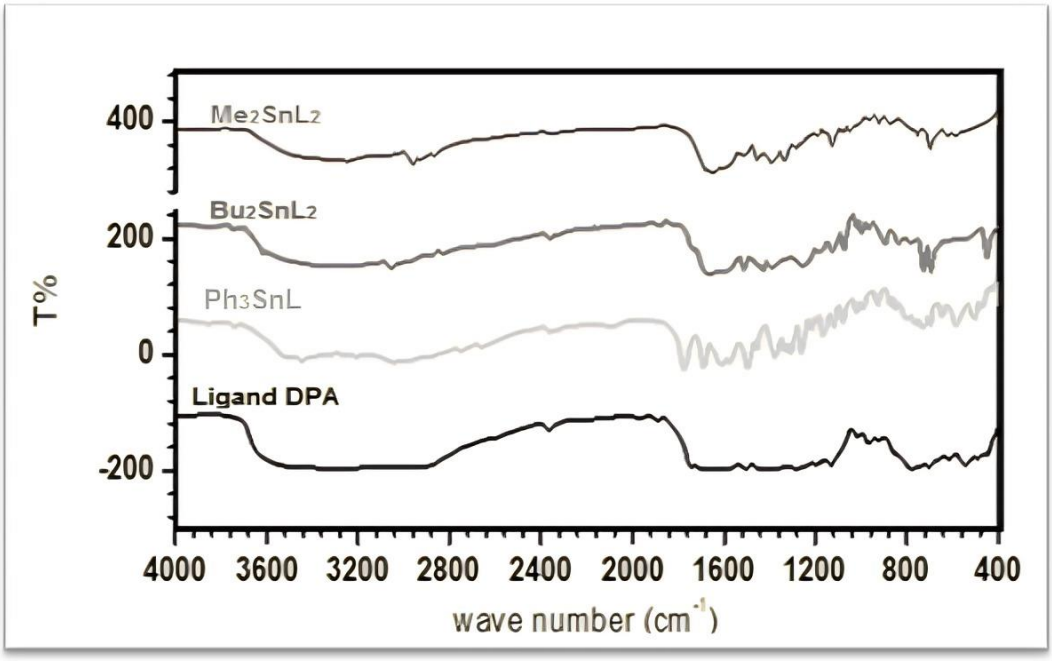


Fig. 1. FT-IR Spectra of DPA and its complexes.

^{1}H-NMR Spectra of DPA shows altered signals for the prepared complexes. If the value of the related to benzene ring at 7.21-7.47. Additionally ^{119}Sn-NMR <200 this mean 5 coordination at 9.45 for NH-group, 2.69-2.78 related to N- complexes, when this value increases > 200, hence methyl, 2.09-2.27 for C- methyl. As a result of six coordination complex was proposed, with octa complexation, these signals were shifted. Table 3 hedral shape [31].
expressed ^{1}H-NMR values. In order to discover
geometrical shape, the ^{119}Sn-NMR were studied

Table 3. ^{1}H-NMR Spectra of DPA and complexes.

Compound	^{1}H-NMR	^{119}Sn-NMR
DPA	13.05 (s, COOH), 9.45 (s, NH), 7.21-7.97(m, 5proton, Ar-proton), 2.70-2.78(s, 3proton, N- methyl), 2.09-2.27(s, 3 proton, C- methyl)	---
Ph_3 DPA	9.55 (s, NH), 7.26-7.50 (m, 5 proton, Ar. proton), 7.55-7.78(m, 5 proton, phenyl group), 2.82(s, 3 proton, N- methyl), 2.1(s, 3 proton, C- methyl)	-174
Bu_2 $(DPA)_2$	8.08(s, 1proton, NH), 7.23-7.45(m, 5 proton, Ar. proton), 2.69-2.78(s, 3 proton, N- methyl), 2.09-2.64(s, 3 proton, C- methyl), 0.83-1.59m, m, 18 proton, $2C_4H_9$).	-249
Me_2 $(DPA)_2$	9.5(s, proton, NH), 6.21-7.95(m, 5 proton, Ar. proton), 2.37-2.96(s, 3 proton, N- methyl), 2.04-2.24(s, 3 proton, C- methyl), 0.80-1.23(s, 6 proton, $2CH_3$)	-234

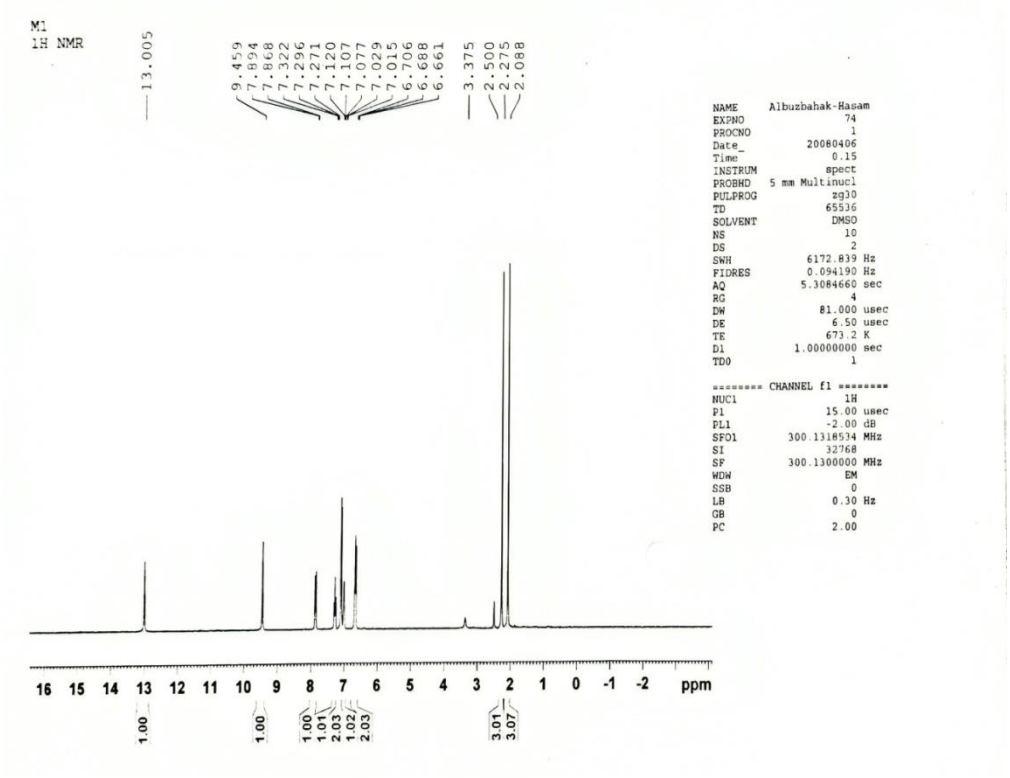


Fig. 2. ^{1}H-NMR of DPA.

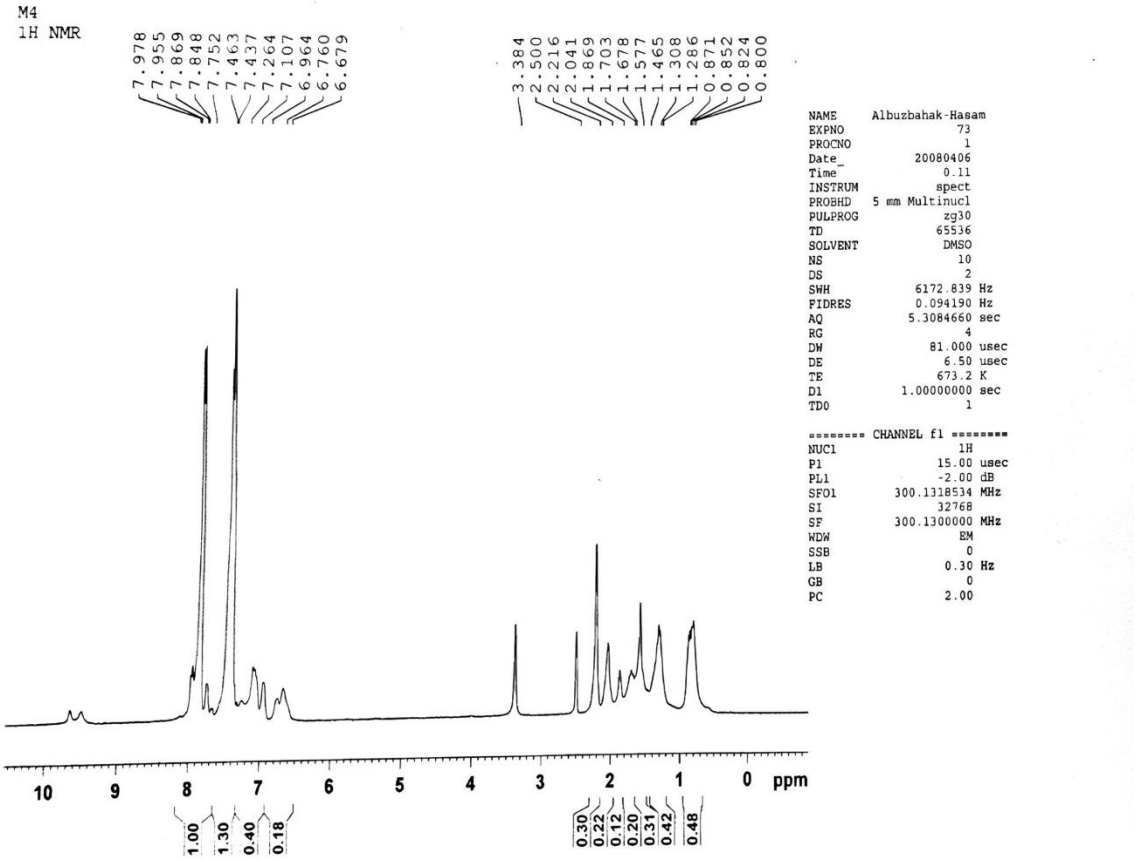


Fig. 3. ^{1}H-NMR of Bu_2Sn-DPA.

Singlet signal are present at(-174, -249 and -234)ppm region of the ^{119}Sn NMR-spectra of Ph_3, Bu_2 and Me_2-complexes respectively. The complexes tin atom coordination number has increased [32]. The complex of Ph_3SnL has trigonal bipyramidal geometry, while Bu_2SnCl_2 complexes geometry determine the chemical shift, and Me_2SnCl_2 have octahedral geometry [33]. and these changes are in line with the theory that

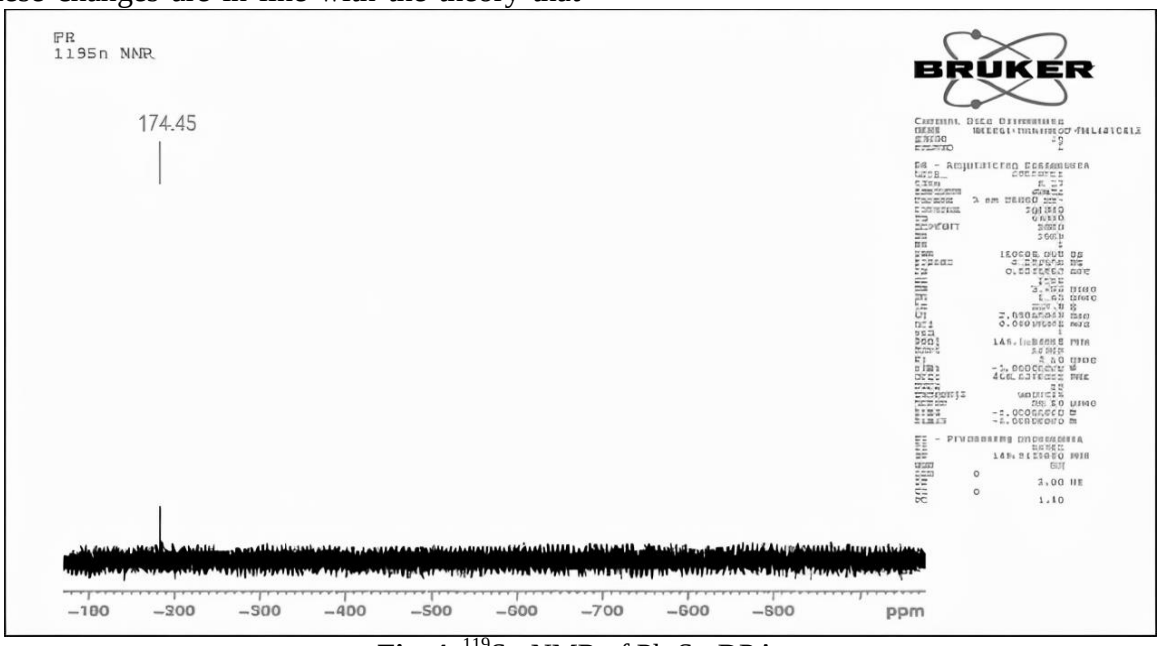


Fig. 4. ^{119}Sn-NMR of Ph_3Sn-DPA.

Antioxidant Activity

Two methods were applied to study the 3 antioxidant activity assay. The % inhibition can be measured when the absorbance for each measurement has been achieved. As shown in Figurs. 5 and 6.

Complex 1 (Triphenyltin-DPA) exhibited higher activity than the other synthesized prepared complexes in different amounts for the complexes; this possibly because that the complex has 3 groups of phenol also due to higher content of aromaticity as compared with di butyl or dimethyltin complexes.

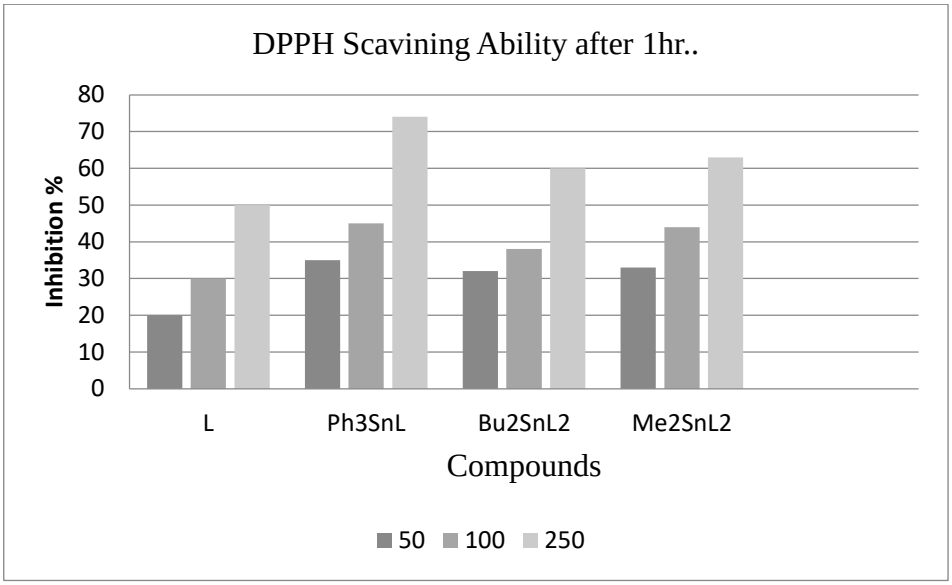


Fig. 5. DPPH scavenging Ability of DPA and complexes.

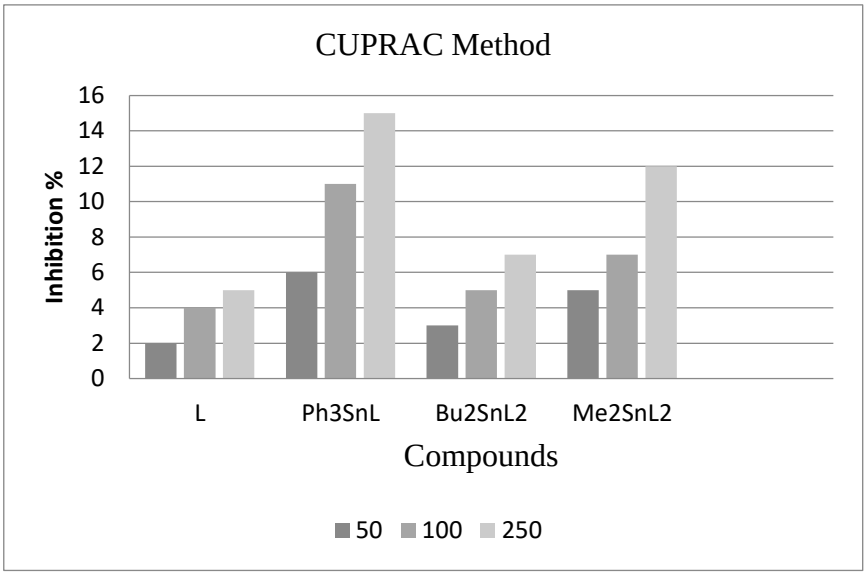


Fig. 6. CUPRAC Method for DPA and complexes.

Conclusions

The DPA reaction as a new ligand with di and tri-organotin chloride produced three corresponding organotin (IV) complexes with high yield percentage. DPA and its organotin(IV)complexes antioxidant activity was regulated by the use of DPPH and CUPRAC techniques. The prepared complexes showed larger antioxidant activity than that of the DPA using two approaches. This is may be related to metal Sn content besides existence of different organic groups.

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Conflicts of interest

The authors have no conflicts of interest regarding this investigation.

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MODİFİKASİYA OLUNMUŞ 4-AMİNOANTİPİRİN LİQANDI İLƏ Dİ- VƏ TRİORQANOQALAY(IV) KOMPLEKSLƏRİNİN SİNTEZİ, QURULUŞUNUN MÜƏYYƏN EDİLMƏSİ VƏ ANTİOKSİDANT AKTİVLİYİNİN QİYMƏTLƏNDİRİLMƏSİ

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Yeni liqand olan 2-[(1,5-dimetil-3-okso-2-fenilpirazolidin-4-il)amino] sirkə turşusu (DPA) 4-aminoantipirinin xlorsirkə turşusu ilə reaksiyası nəticəsində sintez edilmişdir. Alınmış DPA liqandı müvafiq orqanoqalay(IV) xlorid duzları (Ph_3SnCl, Bu_2SnCl_2 və Me_2SnCl_2) ilə kondensləşmə reaksiyaları vasitəsilə uyğun olaraq trifenilqalay(IV), dibutilqalay(IV) və dimetilqalay(IV) komplekslərinin alınması üçün istifadə olunmuşdur. Sintez edilmiş komplekslər infraqırmızı spektroskopiya (İQ), çoxnüvəli nüvə maqnit rezonansı spektroskopiyası (^1H və ^{119}Sn NMR) və element analizi üsulları ilə sistemli şəkildə xarakterizə edilmiş və onların quruluşu təsdiq olunmuşdur. DPA-nın və onun orqanoqalay(IV) komplekslərinin antioksidant qabiliyyəti iki standart üsulla qiymətləndirilmişdir: DPPH (2,2-difenil-1-pikrilhidrazil) radikalının neytrallaşdırılması və CUPRAC (mis(II) ionunu reduksiya edən antioksidant tutum) üsulu. Nəticələr göstərmişdir ki, həm triorqanoqalay(IV), həm də diorqanoqalay(IV) kompleksləri sərbəst DPA liqandı ilə müqayisədə xeyli yüksək inhibisiya faizləri nümayiş etdirir. Bu, antioksidant qabiliyyətinin artırılmasında Sn(IV) mərkəzlərinin həlledici rolunu vurğulayır. Xüsusilə trifenilqalay(IV) karboksilat kompleksi hər iki üsulda ən yüksək nəticəni göstərmişdir. Bu, fenil əvəzedicilərinin yaratdığı spesifik elektron və sterik xassələrlə izah olunur

Açar sözlər: xlorsirkə turşusu, kondensləşmə, karboksilat kompleksi, orqanoqalay əvəzediciləri, antioksidant qabiliyyəti, koordinasiya həndəsəsi, lipofillik.