

## Research Article

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# Synthesis, characterization and electroluminescence properties of a new mixed-ligand diorganotin(IV) complex

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**Abstract:** A new mixed-ligand diorganotin(IV) complex,  $[\mu\text{-}(4\text{-dpe})\{\text{Me}_2\text{Sn}(\text{cup})_2\}]$  (**1**), was synthesized by reacting dimethyltin(IV) dichloride with 1,2-di(4-pyridyl)ethylene (4-dpe) and ammonium N-Nitroso-N-phenylhydroxylamine (cup). The prepared complex was fully characterized by PXRD,  $^1\text{H}$ ,  $^{13}\text{C}$  and  $^{119}\text{Sn}$  NMR, IR, and UV spectra and elemental analysis (CHN). The structural analysis of complex **1** by X-ray diffraction showed that this compound consists of centrosymmetric binuclear units that crystallize in the monoclinic system with the space group of  $P2_{1/c}$ . Thermal behavior of this complex was interrogated by thermogravimetric and differential thermal analysis (TGA and DTA) under air atmosphere. The study of thermal behavior and luminescence properties of prepared diorganotin(IV) complex exhibited that this complex can be used as a emitting layer in the preparation of optical devices. The prepared coordination compound was used in two different concentrations in the manufacture of two light-emitting diodes (OLEDs).

**Keywords:** diorganotin(IV) complex; single crystal XRD; optical properties; OLED

## 1 Introduction

Recently, the synthesis and design of luminescence coordination compounds has attracted the attention of several research groups due to the widespread use of them as a precursor in the preparation of optical devices. In this regard, extensive efforts have been made and the results have shown that the proper choice of ligand and central metal has a consequential effect on the optical properties and molecular structure of luminescence coordination compounds (Li et al., 2018; Najafi et al., 2013; Puttock et al., 2018; Utochnikova et al., 2017). The nature of metal ion and the structure of coordinated ligands are two important factors in determining the physical and chemical properties of metal complexes (Fazaeli et al., 2012; Hashemzadeh et al., 2017; Najafi et al., 2017; Najafi et al., 2018). The interactions of some of pyridyl-based ligands such as 2,2-bipyridin and 1,10-phenanthroline with particular symmetry and stereochemistry with organotin(IV) acceptors with the Lewis acid character have led to the production of metal complexes with fascinating and unique molecular structure. Despite the numerous reports that exist about the crystal structure of organotin complexes and their biological properties (Bhatra et al., 2016; Casas et al., 2011; Choudhary et al., 2018; Diop et al., 2016;), little consideration has been paid to the synthesis of mixed-ligand organotin(IV) compounds and their optical properties (Álvarez-Boo et al., 2003; Arnold and Blok, 2004; Devi and Pachwania, 2018; Hodaie et al., 2017; Molter et al., 2012; Momeni et al., 2013). The studies show that the preparation of mixed-ligand coordination compounds by increasing the auxiliary ligands to the structure of metal complexes can be a suitable method for tuning their physical and chemical properties (Amani et al., 2007; Yin et al., 2015). Usually, mixed-ligand metal complexes perform better than single-ligand complexes in many fields, such as biological activities and optical devices (Sadeek et al., 2016; Saha et al., 2007). Therefore,

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the increase of auxiliary ligands to the structure of metal complexes and the synthesis of novel luminescent mixed-ligand metal complexes can provide an opportunity to use of them as a precursor in the preparation of optical devices.

So far, many transitional metal complexes of cupferron (Cup,  $[\text{PhN}(\text{O})\text{NO}]\text{NH}_4$ ) as an analytical reagent in quantitative determination of some of metal ions have been reported and the structure of some of them has been characterized. However, this ligand is less used in the synthesis of main metal complexes (Hui et al., 2013; Wang et al., 2002; Wang et al., 2006).

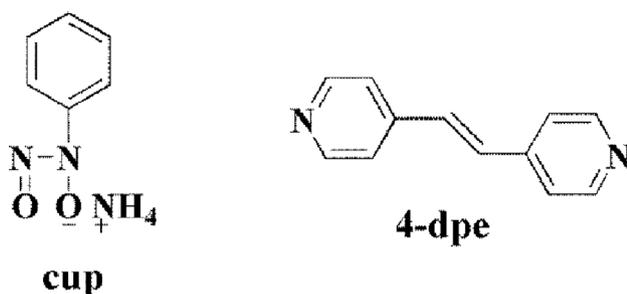
Our previous efforts to synthesize mixed-ligand Pb(II) complexes in the presence of Cup as a primary ligand and 4,4'-bipyridine or 4-bpdb as ancillary ligands led to the formation of two new coordination polymers of  $[\text{Pb}_4(4,4'\text{-bipy})_2(\text{NO}_3)_8(\text{Cup})_2]_n$  and  $[[\text{Pb}(4\text{-bpdb})(\text{NO}_3)(\text{Cup})]_2]_n$ , respectively. Investigation of the results showed that the auxiliary ligands had an important effect on the structure and absorption and fluorescence spectra of Pb(II) complexes and prepared compounds were utilized as precursors for the preparation of nano-metal oxides (Amini et al., 2015).

Following our interest in the synthesis of mixed-ligand metal complexes, a new mixed-ligand diorganotin(IV) complex of cupferron ligand as a primary ligand and 1,2-Di(4-pyridyl)ethylene ligand, a well-known N,N'-donor ligand in optical studies, as an ancillary ligand (Scheme 1) was prepared. The investigation of the optical properties and thermal behavior showed that this complex can be used as a precursor in the preparation of organic light emitting diodes.

## 2 Results and discussion

### 2.1 Synthesis

A new binuclear tin(IV) complex has been synthesized from the reaction of 1,2-di(4-pyridyl)ethylene (4-dpe),

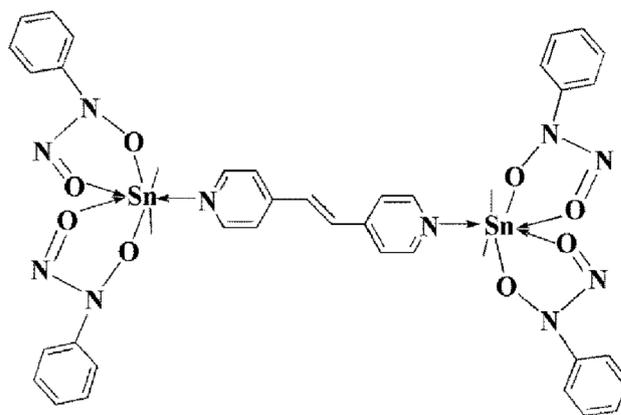


**Scheme 1:** The structures of cupferron (cup) and 1,2-Di(4-pyridyl)ethylene (4-dpe) ligands.

Cupferron (cup) and dimethyltin(IV) dichloride. All general characterization of this complex were carried out with its crystal samples and the results revealed that there is an excellent concurrence between the components of this compound and the outcomes of structural analysis. The molecular structure of this compound was also investigated by X-ray diffraction (Scheme 2).

Relatively weak bands in the range of 3000-3050 and 2930-3000  $\text{cm}^{-1}$  are related to the stretching vibrations of aromatic and aliphatic C-H bonds, respectively. The position of the bands of N-N and N-O previously reported for cupferron ligand shows a significant shift in comparison to those of prepared complex because of coordination of ONNO unit to tin atom. The appearance of the new Sn-O band in the IR spectra of compound **1** at about 400  $\text{cm}^{-1}$  in the IR spectra of prepared compound in comparison with that of free cupferron ligand can be related to the coordination of this ligand to the diorganotin species in bidentate chelating mode. Significant information can be obtained with regard to the wave number of this new bond about the coordination mode of ONNO group of cup ligand to tin atom. The coordination of cupferron ligand to the diorganotin species in the bridging coordination mode shows a band at about 388  $\text{cm}^{-1}$ , while, the  $\nu(\text{Sn}-\text{O})$  band for ordinary bidentate chelating mode is centered in the range of 395-408  $\text{cm}^{-1}$  (Baul et al., 2007). Furthermore, the attendance of a single band at about 560  $\text{cm}^{-1}$  in the infrared spectra of **1** exhibits that the C-Sn-C moiety has a relatively linear configuration. It is completely consistent with the results of structural analysis (Najafi et al., 2014).

The  $^1\text{H}$ ,  $^{13}\text{C}$ , and  $^{119}\text{Sn}$  NMR spectra of prepared complex have been recorded to conclude its precise structural properties in the solution. Coordination of ligands to diorganotin species has led to a shift in their signal in the



**Scheme 2:** Line drawing structure of tin complex.

$^1\text{H}$  NMR spectra of compound **1** to lower fields. In the  $^1\text{H}$  spectra of complex **1**, the methyl groups bound to the tin atom exhibit a singlet signal at 0.69 ppm, indicating the attendance of one type tin atom in its crystal structure. In accordance with the results of the IR spectrum and structural analysis, the  $^2J^{17/119}\text{Sn-H}$  coupling value of compound **1** shows a linear configuration of the C–Sn–C skeleton and a heptacoordinated geometry for tin atom in its structure in solution. In the  $^{13}\text{C}$  NMR spectra of prepared compound, the formation of complexes by coordination of pyridyl and cupferron ligands to diorganotin species and electron transfer from them to tin atom resulted in a frail shift between the signals of ligands and complex **1**.

The value of chemical shift of  $^{119}\text{Sn}$  NMR can be utilized to predict the coordination number of central tin atom in the structure of tin complexes. The  $^{119}\text{Sn}$  NMR spectra of complex **1** exhibit only one resonance at -225.9 ppm. This value of chemical shift is in the anticipated range for seven-coordinate Sn for this compound (Najafi et al., 2014).

## 2.2 Crystal structure of complex 1

Crystallographic data and refinement details for the complex **1** are listed in Table 1. The results show that this compound in solid-state structure consists of centrosymmetric binuclear  $[\text{Me}_2\text{Sn}(\text{N}_2\text{PhO}_2)_2]_2$  dpe units (Figure 1). The unit cell of this complex is exhibited in Figure 2. The asymmetric unit of this complex with the monoclinic system and space group of  $P2_1/c$  has one tin(IV) atom, a half 4-dpe ligand, two cupferronato anions and two methyl groups. The sum of the bond angles of O(1)–Sn(1)–O(2), 68.57(7)°; O(2)–Sn(1)–O(4), 75.34(7)°; O(4)–Sn(1)–O(3), 67.62(7)°, O(3)–Sn(1)–N(5) 73.64(8)° and O(1)–Sn(1)–N(5), 74.87(8)° is 360.04°. These results show that the tin(IV) centres in the structure of complex **1** have a pentagonal bipyramidal environment. The  $\text{O}_4\text{N}$  donor set of the cupferronato and the bipy ligands are in the equatorial plane, and two *trans* carbon atoms from the methyl groups bound to the tin atom are in axial positions. The coordination of cupferronato anions to tin atom in a bidentate chelating mode led to formation of two  $\text{SnO}_2\text{N}_2$  units as five-membered chelate rings (A and B). The O–Sn(1)–O bite angle of rings A and B is 68.57(7)° and 67.62(7)°, respectively (Figure 1). The Sn–O bond lengths which are in the range of 2.231(2)–2.310(2) Å (Table 2) in five-membered chelate rings A and B are relatively different ( $\Delta = 0.056$  Å and  $\Delta = 0.066$  Å), respectively. These bond lengths are relatively shorter than the same bond length in the structure of dimethylbis(N-nitroso-

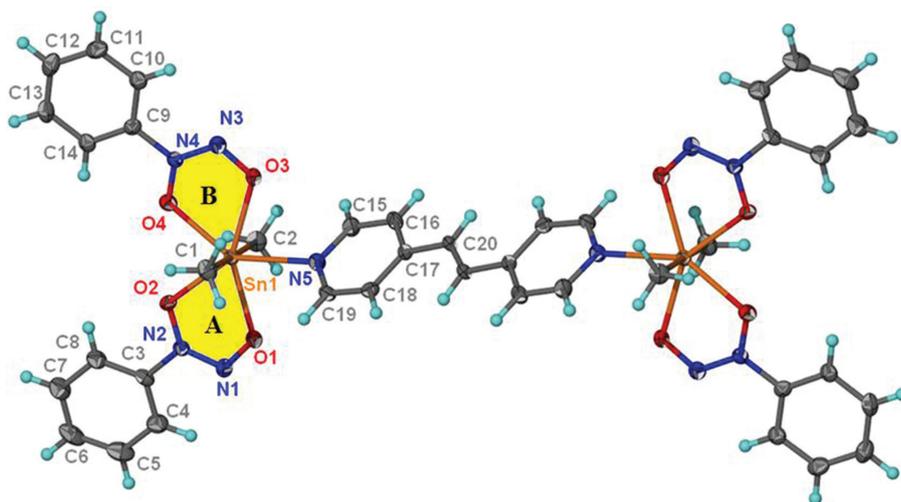
**Table 1:** Crystal data, data collection and structure refinement details of complex **1**.

Chemical formula	$\text{Sn}_2(\text{CH}_3)_4(\text{C}_{12}\text{H}_{10}\text{N}_2)(\text{C}_6\text{H}_5\text{N}_2\text{O}_2)_4$
$M_r$	1028.22
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	100
$a, b, c$ (Å)	12.1762 (5), 15.1477 (6), 11.2520 (4)
$\beta$ (°)	93.466 (4)
$V$ (Å <sup>3</sup> )	2071.54 (14)
$Z$	2
Radiation type	Mo $K\alpha$
$\mu$ (mm <sup>-1</sup> )	1.27
Crystal size (mm)	0.40 × 0.30 × 0.20
Data collection	
Diffractometer	Agilent Technologies SuperNova Dual diffractometer with Atlas detector
Absorption correction	Gaussian <i>CrysAlis PRO</i> (Agilent, 2014)
$T_{\min}, T_{\max}$	0.717, 0.794
No. of measured, independent and observed [ $I > 2\sigma(I)$ ] reflections	10820, 4782, 3917
$R_{\text{int}}$	0.030
$(\sin \theta/\lambda)_{\text{max}}$ (Å <sup>-1</sup> )	0.651
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.032, 0.083, 1.01
No. of reflections	4782
No. of parameters	271
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å <sup>-3</sup> )	1.00, -0.67

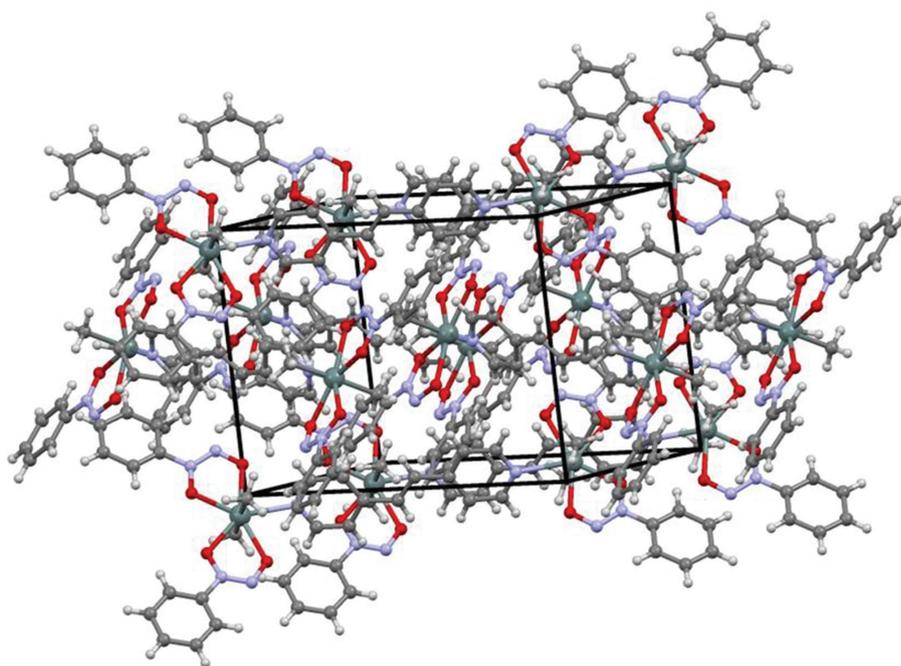
Computer programs: *CrysAlis PRO* (Agilent, 2014), *SHELXS97* (Sheldrick, 2008), *SHELXL97* (Sheldrick, 2008), *X-SEED* (Barbour, 2001), *pubCIF* (Westrip, 2010).

N-phenyl-hydroxylaminato)-tin(IV) complex with the triconnective bridging ligand and adjacent to those reported already for other diorganotin(IV) cupferronato complexes with the chelating ligand (Deak et al., 2000; Deak et al., 2001). The distance of Sn1...Sn1 separated by 4-dpe ligand is 14.076 Å.

It should be noted that the Sn–C, Sn–N bond distances are within the ranges discovered in  $[\text{R}_2\text{SnCl}_2(\text{bpy})]$  derivatives (Deak et al., 2001). The point in the structure of the complexes is that the non-covalent interactions and weak intra- and intermolecular interactions have a important influence on the molecular assemblies and crystal packing of coordination compounds. In the crystal packing of this complex, as well as the structure of many complexes, the existence of hydrogen bonding (Table 3) and face-to-face  $\pi$ – $\pi$  interactions (Figure 3) has led to the



**Figure 1:** A perspective view of the molecular structure of tin complex drawn with 50% probability displacement ellipsoids.



**Figure 2:** Crystal packing in 1.

connection of dimer units to each other and the expansion of the crystal structure to a three-dimensional network.

### 2.3 Thermal behavior and XRD pattern of prepared complex

The acceptable match of the simulated XRD pattern of complex 1 with its experimental XRD pattern indicates their structural similarity and high purity of complex 1 (Figure 4).

Thermal behavior of this complex was interrogated by thermogravimetric and differential thermal analysis (TGA and DTA) under air atmosphere (Figure 5). The results show that this complex is stable up to ca. 320°C. The endothermic peak at 150°C in the DTA curve is related to the melting of the complex. The existence of a severe weight loss and an extreme exothermic peak in TGA and DTA curves of prepared complex in the temperature range of 300 to 450°C is related to the removal of organic groups from the complex structure. The residual component above 500°C

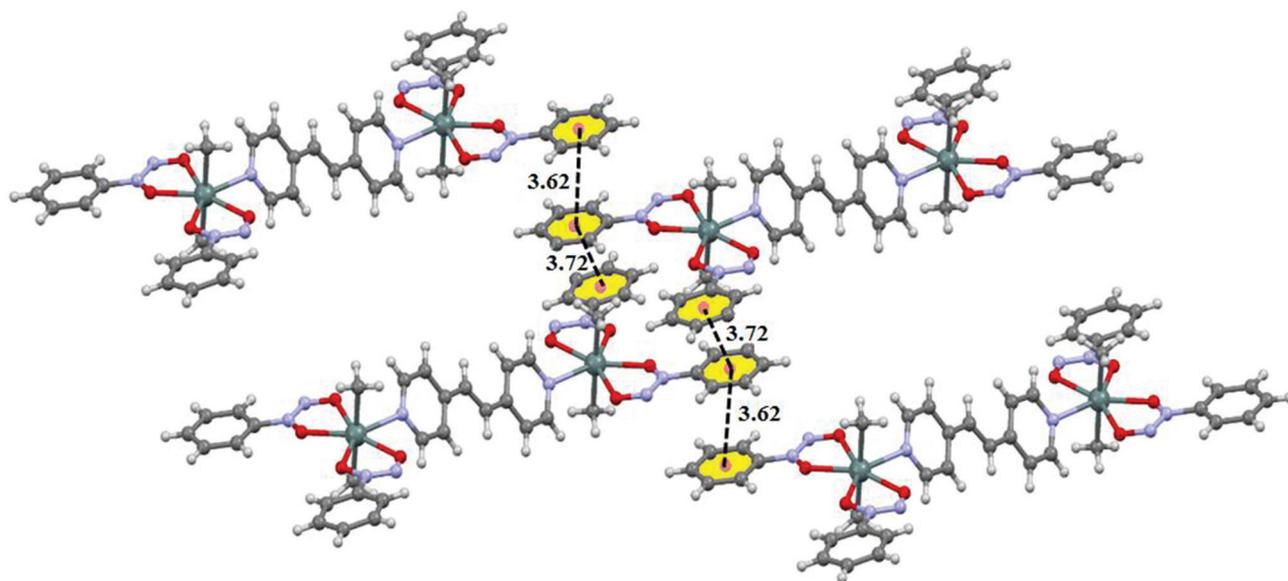
**Table 2:** Selected bond lengths (Å) and angles (°) for (1).

Bond lengths (Å)			
Sn1—C1	2.116(3)	Sn1—O2	2.231(2)
Sn1—C2	22.123(3)	Sn1—O4	2.244(2)
Sn1—O1	2.287(2)	Sn1—O3	2.310(2)
Sn1—N5	2.432(2)		
Bond angles (°)			
C2—Sn1—C1	177.76(12)	O2—Sn1—O4	75.34(7)
C1—Sn1—O2	91.10(10)	C2—Sn1—O4	90.34(10)
C1—Sn1—O4	89.15(11)	C1—Sn1—O1	91.98(10)
C2—Sn1—O2	90.88(10)	O4—Sn1—O1	143.91(7)
C2—Sn1—O1	89.71(10)	O2—Sn1—O1	68.57(7)
C1—Sn1—O3	88.75(10)	C2—Sn1—O3	89.04(10)
O2—Sn1—O3	142.96(7)	O4—Sn1—O3	67.62(7)
O1—Sn1—O3	148.46(7)	C1—Sn1—N5	88.15(11)
C2—Sn1—N5	90.88(11)	O2—Sn1—N5	143.39(7)
O4—Sn1—N5	141.21(8)	O1—Sn1—N5	74.87(8)
O3—Sn1—N5	73.64(8)		

**Table 3:** Significant intermolecular interactions (interatomic distance (Å)) and bond angles.

D-H...A	D-H	H...A	D...A	D-H...A
C(8)–H(8)..O(2)	0.95	2.37	2.708(4)	100
C(14)–H(14)..O(41)	0.95	2.39	2.719(3)	100

is clearly related to the creation of tin oxide particles. The high thermal stability of this complex is one of its most important advantages in order to use it in the production of the optical devices.

**Figure 3:** A perspective view of  $\pi$ - $\pi$  interactions of tin complex.

## 2.4 Optical properties of compound 1

Recently, the synthesis of complexes and their use in the manufacture of optical devices has been very much considered. In this work, a new tin complex of cupferron ligand was prepared and 1,2-Di(4-pyridyl)ethylene was added as an ancillary ligand in the structure of it to improve its optical properties. The solid-state absorption spectra of complex **1** and bare ligands, Figure 6, show that, the coordination of ligands to the diorganotin species has led to a red shift in the absorption spectrum of prepared complex relative to that of ligands because of the formation of the rigid conjugated systems (Amini et al., 2017). It is noteworthy that the principal source of bands in the absorption spectra of complex **1** and bare ligands is related to the  $\pi \rightarrow \pi^*$  transitions of aromatic rings of ligands.

The fluorescence spectra of complex **1** in the solid state and MeOH is shown in Figure 7. The photoluminescence spectra of complex in methanol and solid state have the similar appearance with a slight shift, which can be related to the presence of weak intermolecular interactions. Interestingly, luminescence spectrum of complex in MeOH shows a narrower FWHM than that of solid-state. In the emission spectra of complex **1**, the existence of the emission bands can be attributed to  $\pi^* \rightarrow \pi$  transitions of the aromatic rings of ligands.

Measuring absolute quantum yield of prepared complex in solid state, 0.79 according to the methodology used by Moreno, (Moreno, 2012), suggests that this complex can be utilized in the manufacture of optical devices such as organic light emitting diodes.

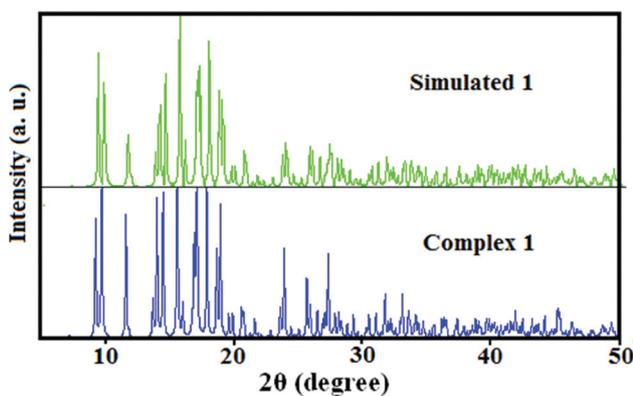


Figure 4: XRD patterns of complex 1.

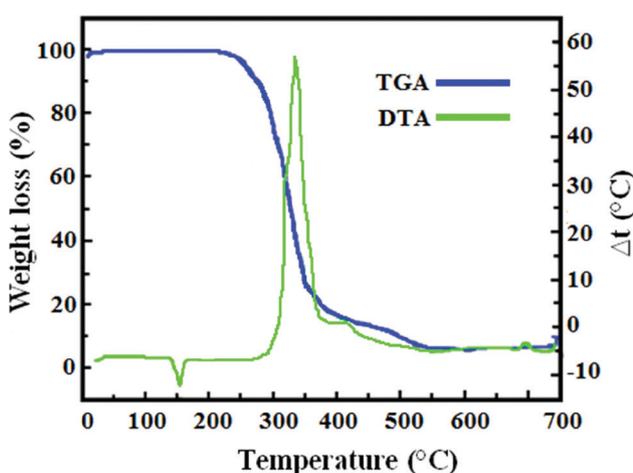


Figure 5: TGA diagram of complex 1.

## 2.5 Electroluminescence characteristic

Two OLED devices with a general configuration of ITO/PEDOT:PSS(90nm)/PVK:PBD:complex(80nm)/Al(200nm) (Figure 8) with two distinctive concentrations of complex 1 were constructed and their electroluminescence properties were investigated. The effect of dopant concentration on the electroluminescence properties of diodes were also investigated. It should be noted that in this work PBD, PEDOT:PSS and PVK molecules as electron transport layer, hole injection layer and hole transport layer were used in the structure of prepared diodes.

The combination of electrons and cavities injected from the cathode and anode leads to the light emission and the formation of excitons. Adding the PBD into the PVK molecules with a ratio of 40 to 100 is the best solution to increase the efficiency of the diodes and reduce the formation of the excimers (Chang et al., 2006; Yang et al., 2004). The electroluminescence spectrum of PVK:PBD device without complex 1 in the structure of it shows a

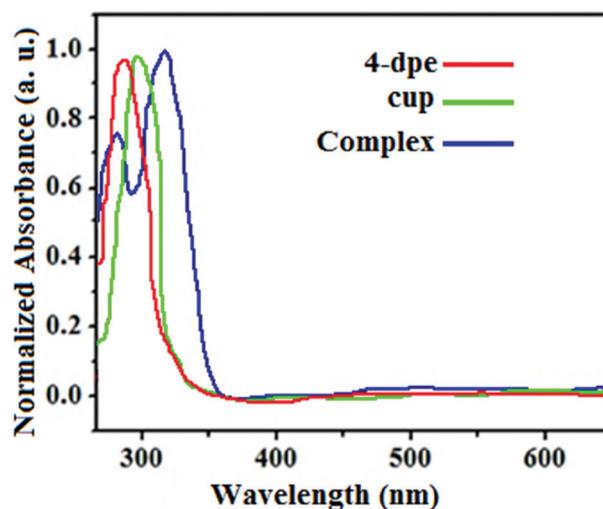
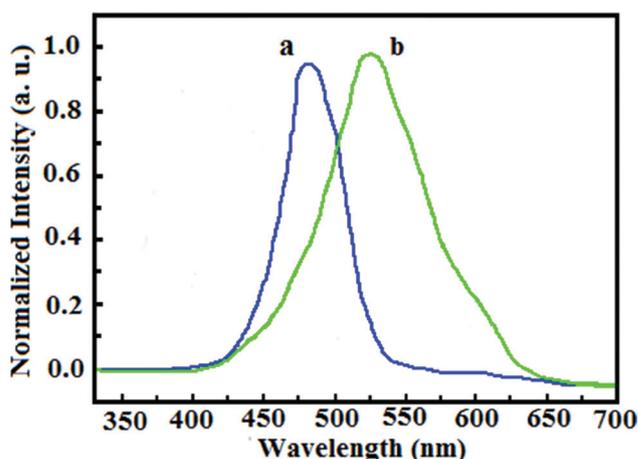


Figure 6: Absorption spectra of the bare ligands and tin complex in the solid state.

Figure 7: Fluorescence spectra of tin complex in solid state (b) and methanol (ca.  $1 \times 10^{-5}$  M) (a).

relatively broad peak at about 450 nm, while the EL spectra of diodes with 10% (diode 1) and 30% (w/w) (diode 2) demonstrate a band at about 550 nm (Figure 9). This long red shift reveals that tin complex work as the emission layer in the structure of complex based devices. Figure 9 shows that the complex concentration has a meaningful influence on the electroluminescence properties of diodes. One of the main reasons for reducing the efficiency of the diodes can be due to the high concentration of the complex in their structure.

Figure 10 presents the current density versus the voltage of diodes. As can be seen, decreasing the complex concentration in the structure of the diodes leads to an increase in their current density. The diode 2 due to the

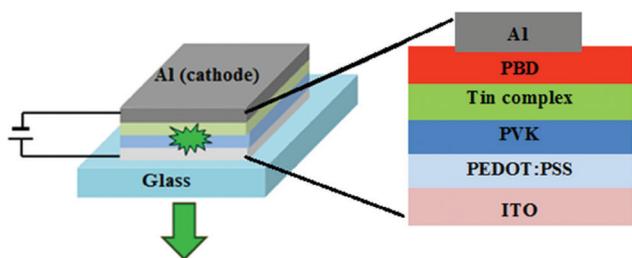


Figure 8: Layer arrangement of the OLED device.

high surface roughness and the high electron injection barrier relative to the diode 1 requires a higher operation voltage that reduces the diode's efficiency.

Figure 11 reveals the luminescence-voltage characteristics of devices 1 and 2. The complex concentration is impressive on the turn-on voltage. The device 1, which contains less concentration of the complex, has a higher brightness.

### 3 Conclusions

In conclusion a novel mixed-ligand organotin(IV) luminescence coordination complex has been successfully synthesized and characterized by spectroscopic methods. The X-ray diffraction was also used to determine its crystal structure. The photoluminescence properties and thermal behavior of this complex was investigated and the results revealed that this complex can be used as an emitting layer material in the manufacture of optical devices such as organic light emitting diodes. Finally, the production of two diodes using the prepared complex and examination of their electrical properties showed that tin complexes with favorable optical properties can be used to make optical devices. The results showed that the complex concentration has a significant effect on the electroluminescence properties of diodes.

## Experimental

### Materials and physical techniques

All commercially available reagents were purchased from Merck and used without additional purification. The reported standard methods were used to dry the solvents (Perrin et al., 1980). C, H, and N microanalyses and thermal analyses (TGA) of compounds have been appraised by Thermo Finnigan Flash-1112EA microanalyzer and Bahr STA-503 instrument, respectively. For the recording of

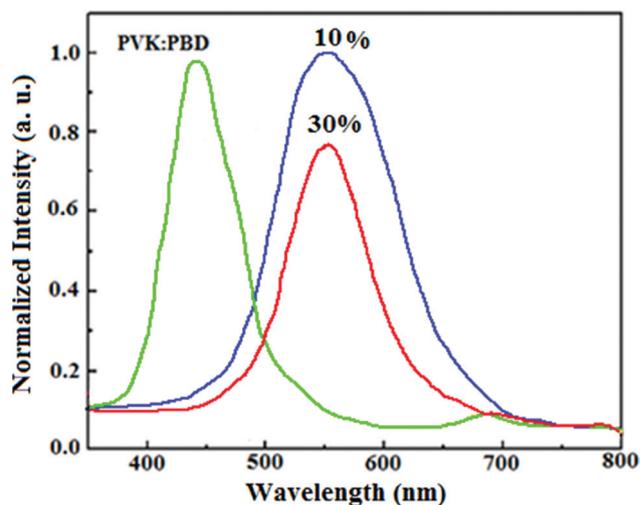


Figure 9: Electroluminescence spectra of prepared OLED devices.

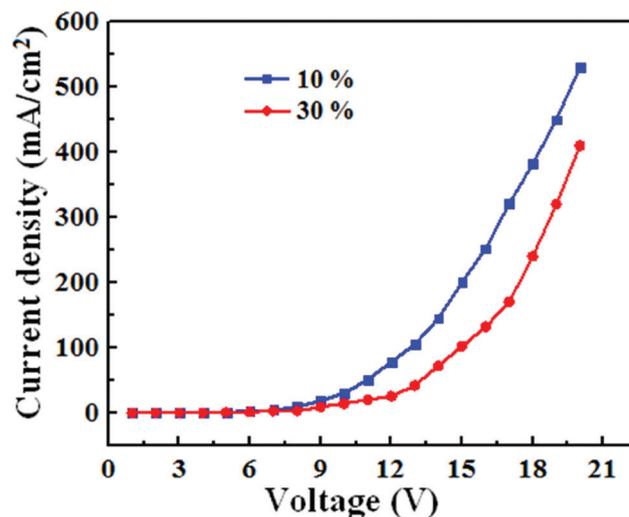
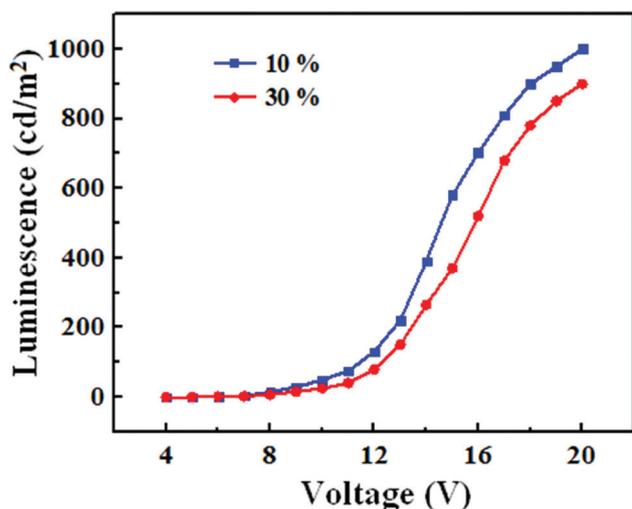


Figure 10: Current density ( $J$ )-voltage ( $V$ ) characteristics of prepared OLED device.

infrared spectra, the Bomem MB-series FT-IR instrument has been utilized. NMR spectra were inscribed on a Bruker AVANCE 300 operating at 300.3 MHz. The luminescence and absorption spectra of compounds were obtained on USB2000 and Shimadzu 2,100 spectrometers, respectively.

Crystallographic measurements were acquired by utilizing a Bruker SMART APEX diffractometer supplied with a graphite monochromated Mo-K $\alpha$  radiation at 100 K. Accurate unit cell parameters and orientation matrices were obtained from least-squares refinements. The structure solved by direct method was refined by full-matrix least-squares procedure on F<sup>2</sup>. The SHELXL97 crystallographic software package was used for all refinements.



**Figure 11:** Luminance–current density characteristics of prepared OLED device.

For the preparation of complex **1**, a solution of dimethyltin(IV) dichloride (1 mmol) was added to a solution of 1,2-di(4-pyridyl)ethylene. Stirring this solution for 1 h led to the formation of a white precipitate. Increasing ammonium N-Nitroso-N-phenylhydroxylamine (1 mmol) to the resulting solution resulted in the formation of  $\text{NH}_4\text{Cl}$  and a yellow solution. After stirring for an hour, the  $\text{NH}_4\text{Cl}$  was filtered off and the yellow solution was left to evaporate slowly at room temperature. After two week, yellow crystals of the complex were formed (yield 82%, m.p: 150-152°C). Anal. Calc. for  $\text{C}_{40}\text{H}_{42}\text{N}_{10}\text{O}_8\text{Sn}_2$ : C 46.72, H 4.12, N 13.62%. Found: C 46.93, H 4.21, N 13.63%. IR ( $\text{cm}^{-1}$ ): 3030, 2985, 1462, 1342, 1221, 1054, 528, 468, 405.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ ):  $\delta$  = 0.69 (6H, s, Sn- $\text{CH}_3$ ,  $^2J^{117/119}\text{Sn-H}$  = 89.4 Hz), 6.43, 6.55, 7.15, 7.44, 8.68 (9H,  $\text{C}_5\text{H}_5\text{N}$  and  $\text{C}_6\text{H}_5$ ), 7.18 (CH).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ ):  $\delta$  8.8 (Sn $\text{CH}_3$ ,  $^1J^{117/119}\text{Sn-}^{13}\text{C}$  = 750.5Hz), 115.3, 120.8, 123.7, 127.5, 135.9, 143.9, 151.1 (Ar-C).  $^{119}\text{Sn}$  NMR ( $\text{CDCl}_3$ ):  $\delta$  -225.9 ppm.

**Supplementary material:** CCDC 1849720 embraces the crystallographic data for complex **1** informed in the article. Copies of the data can be acquired from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk.

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