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Note

# Synthesis, crystal structures and *in vitro* antibacterial activity of two novel organotin(IV) complexes

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ABSTRACT

Two triorganotin(IV) carboxylates $[n\mathrm{Bu_3SnOL}]_n$ (**KK1**) and $[\mathrm{Ph_3SnOL}]_n$ (**KK2**) have been prepared by the reactions of (*E*)-3-(4-(diphenylamino)phenyl)acrylic acid (**HL**) with $n(\mathrm{Bu_3Sn})_2\mathrm{O}$ and $\mathrm{Ph_3Sn(OH)}$, respectively. Complexes **KK1** and **KK2** have been structurally characterized by IR, elemental analysis and X-ray crystallography, confirming that both complexes possess infinite 1D chain structures. It's exciting to discover that **KK1** and **KK2** exhibit strong solid-state luminescence emission while the **HL** almost quenches. Furthermore, both complexes were assayed for *in vitro* antibacterial activity against two Gram-positive bacterial strains (*Bacillus subtilis* ATCC 6633 and *Staphylococcus aureus* ATCC 6538) and two Gram-negative bacterial strains (*Pseudomonas aeruginosa* ATCC 13525 and *Escherichia coli* ATCC 35218) by MTT method. Complex **KK2** exhibited powerful antibacterial activities against *S. aureus* with MIC value of 0.78 μg/mL, which was superior to the positive controls penicillin G. On the basis of the biological results, structure–activity relationships were discussed.



## 1. Introduction

The increasing interest in organotin(IV) carboxylates is attributed to their important biological properties and their considerable structural diversity. In general, organotin(IV) carboxylates show significant *in vitro* antifungal, antibacterial, antiviral, wood preservatives, pesticides and antitumor activities [1–8] which is essentially related to the number and nature of the organic groups attached to the central Sn atoms, however, the role of carboxylate ligand cannot be ignored. Usually, triorganotin(IV) compounds display a higher biological activity than their di-and monoorganotin(IV) analogues [9,10]. Literature divulges that little is known about the mode/mechanism of action of such complexes and more information is required. As an outcome of several attempts it has been assumed that the organic ligand facilitates the transportation of the complexes across the cell membrane and the groups attached to the central Sn atoms may enhance the binding to proteins [11]. In addition to their biological activities, the organotin(IV) carboxylates also present an interesting structural diversity. This is supported through the observation of monomeric, dimeric, tetrameric, oligomeric ladder, cyclic and drums structures [12–16]. It is generally believed that a combination of steric and electronic factors determine the specific structure adapted by a particular organotin carboxylate [17]. Furthermore, it has been reported that the size of the carboxylic acids used and the stoichiometry of the reactants play an important role in the formation of solid-state frameworks [12]. For better understanding of the structural diversity and the structure–activity relationships of such complexes, we have synthesized a series of carboxylic acids to react with some organotin(IV) precursors. Herein, we report the synthesis, characterization, structural study of two new organotin(IV) carboxylates with interesting infinite 1D chain structures. Both complexes and the ligand were tested for *in vitro* antibacterial activity against two Gram-positive bacterial strains (*Bacillus subtilis* ATCC 6633 and *Staphylococcus aureus* ATCC 6538) and two Gram-negative bacterial strains (*Pseudomonas aeruginosa* ATCC 13525 and *Escherichia coli* ATCC 35218).

## 2. Results and discussion

### 2.1. Structural studies

In the IR spectra of complexes **KK1** and **KK2**, the absence of broad $\upsilon$(O–H) absorption of **HL** in the range 2500-3000 $\mathrm{cm^{-1}}$ indicates a deprotonation of the ligand during coordination to the

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tin atom. The strong absorption appearing at 625 and 448 $cm^{-1}$ in the respective IR spectrum of complexes **KK1** and **KK2**, which is absent in the free ligand, is assigned to the Sn–O stretching mode of vibration. Also strong absorption appearing at 1566 and

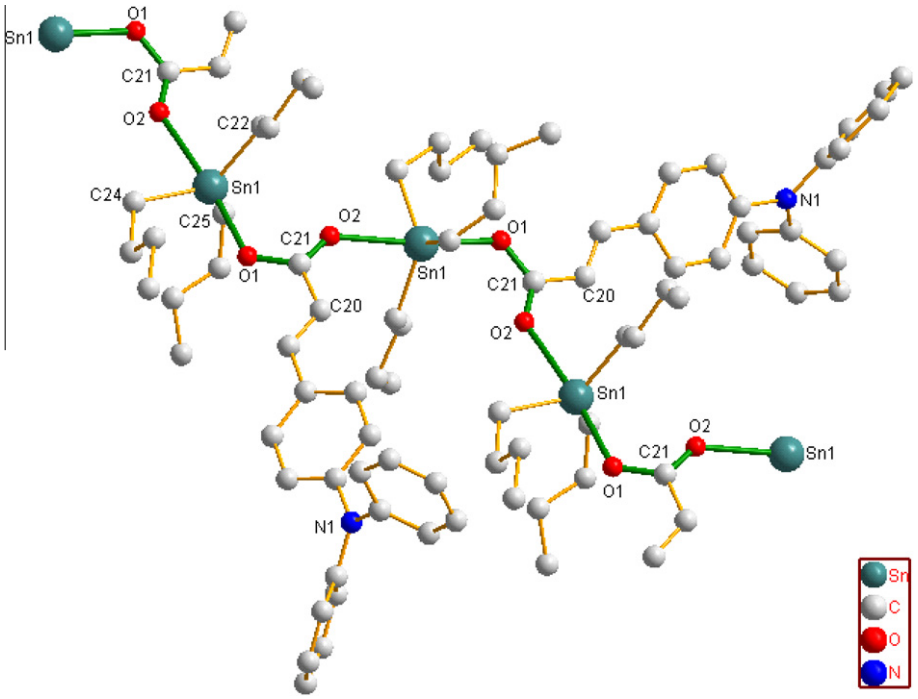



**Fig. 1.** Coordination environment of Sn in **KK1** (All hydrogen atoms have been omitted for clarity).

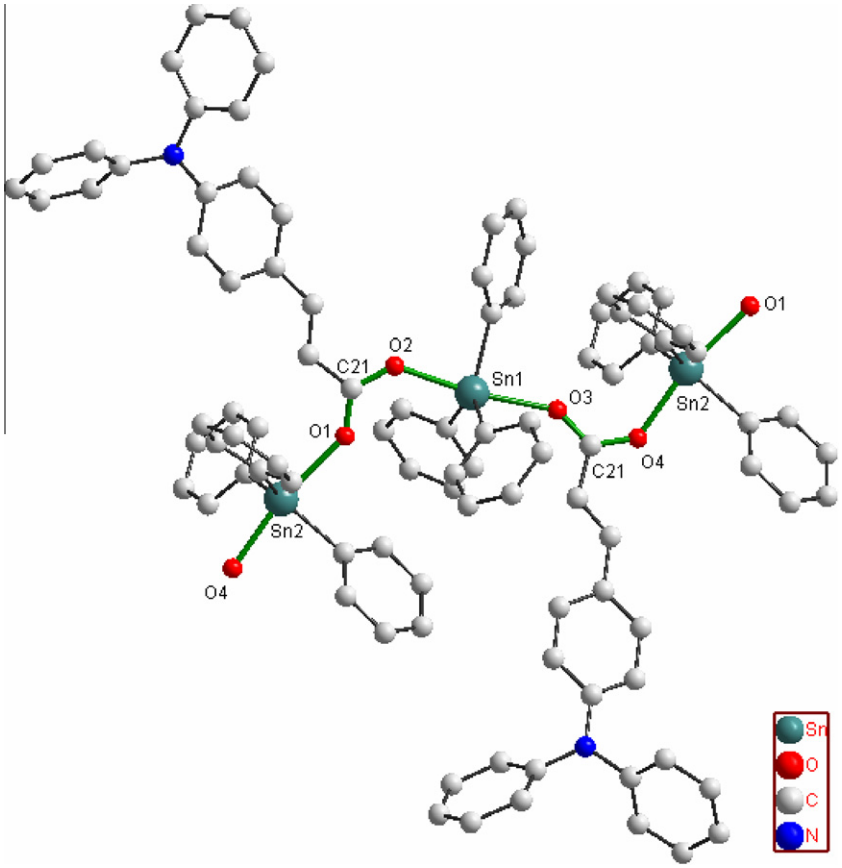



**Fig. 2.** Coordination environment of Sn in **KK2** (All hydrogen atoms have been omitted for clarity).

**Table 1**
Selected bond lengths (Å) and angles (°) for complexes **KK1** and **KK2**.

| | | | |
|---|---|---|---|
| 1. $[n\mathrm{Bu_3SnOL}]_n$ (**KK1**) | | | |
| *Bond lengths* | | | |
| Sn(1)–O(1) | 2.1689(117) | Sn(1)–O(2) | 2.4137(86) |
| C(21)–O(1) | 1.3006(116) | C(21)–O(2) | 1.2187(161) |
| *Bond angles* | | | |
| O(2)–Sn(1)–O(1) | 172.851(354) | Sn(1)–O(1)–C(21) | 121.888(859) |
| O(1)–C(21)–O(2) | 122.669(1199) | C(21)–O(2)–Sn(1) | 136.795(811) |
| $[\mathrm{Ph_3SnOL}]_n$ (**KK2**) | | | |
| *Bond lengths* | | | |
| Sn(2)–O(4) | 2.1812(24) | Sn(2)–O(1) | 2.3083(24) |
| C(21)–O(1) | 1.2552(40) | C(21)–O(2) | 1.2661(41) |
| Sn(1)–O(2) | 2.1855(27) | Sn(1)–O(3) | 2.2963(26) |
| C(21)–O(3) | 1.2526(51) | C(21)–O(4) | 1.2690(44) |
| *Bond angles* | | | |
| O(4)–Sn(2)–O(1) | 169.634(89) | Sn(2)–O(1)–C(21) | 140.159(229) |
| O(1)–C(21)–O(2) | 122.674(323) | C(21)–O(2)–Sn(1) | 134.084(223) |
| O(2)–Sn(1)–O(3) | 172.177(89) | Sn(1)–O(3)–C(21) | 141.049(230) |
| O(3)–C(21)–O(4) | 122.043(323) | C(21)–O(4)–Sn(2) | 133.613(222) |

1599 $cm^{-1}$ in the respective IR spectrum of complexes **KK1** and **KK2** is assigned the asymmetric vibration of the COO moiety. As for complexes **KK1** and **KK2**, the $^{119}$Sn NMR spectra exhibit a single resonance at $\delta$ −146.5 and −121.4 ppm, respectively, indicating that they both have only one type of penta-coordinate tin atoms [18].

The molecular structures of complexes **KK1** and **KK2** with the atom numbering scheme are depicted in Figs. 1 and 2, respectively. Selected bond lengths and angles with their estimated standard deviations are listed in Table 1. Both the complexes **KK1** and **KK2** possess infinite 1D zigzag chain structures with a five-coordinated tin center, which are generated by the bidentate bridging carboxylic acid ligands and the Sn center. Thus, the coordination geometry about the Sn atom is best described as distorted trigonal bipyramidal with two O atoms and three C atoms, which exhibits *trans*-$R_3SnO_2$ geometry. The two O atoms occupy the apical positions [axial angles: O(2)–Sn(1)–O(1) = 172.851(354)° for complex **KK1**; O(2)–Sn(1)–O(3) = 172.177(89)°, O(4)–Sn(2)–O(1) = 169.634(89)°, for complex **KK2**], while the carbon atoms are in the equatorial plane. The Sn, 3C system is basically planar [Sn(1)C(22)C(24)C(25), the C–Sn–C angles ranging from 118.437(592) to 120.931(563)°, for complex **KK1**; Sn(1)C(66)C(72)C(73), the C–Sn–C angles ranging from 112.410(146) to 125.468(182)°, Sn(2)C(48)C(54)C(60), the C–Sn–C angles ranging from 117.819(158) to 123.718(183)°, for complex **KK2**]. The O atoms of the ligand bridges the Sn atoms and gives rise to different Sn–O bond lengths [Sn(1)–O(1) = 2.1689 (117), Sn(1)–O(2) = 2.4137(86), for complex **KK1**; Sn(1)–O(2) = 2.1855 (27), Sn(1)–O(3) = 2.2963(26), Sn(2)–O(1) = 2.3083(24), Sn(2)–O(4) = 2.1812(24) Å, for complex **KK2**]. These Sn–O bond lengths are a little longer than the Sn–O covalent bond lengths [2.038–2.115 Å]. The Sn–C bond lengths are consistent with those reported in other organotin(IV) complexes. Layers of the two complexes are depicted in Figs. 3 and 4, respectively.

### 2.2. Solid-state luminescence emission

The solid-state luminescence spectra measured by F-4500 FL Spectrophotometer was shown in Fig. 5. It's exciting to discover that **KK1** and **KK2** exhibit strong solid-state fluorescence emission while the **HL** almost quenches. The luminescence photograph of the three compounds under 365 nm irradiation was shown in Fig. 6. **KK1** shows blue color and **KK2** shows blue/green color. The reason for this huge variation is that most coordination process is the process in which the addition of metal cores to the organic ligands always breaks down the original π–π interactions between them, which usually produce the fluorescence quench and the red-shifted fluorescence of organic intra-molecular charge transfer of π–π*. Proved by the X-ray structures, both the **KK1** and **KK2** avoid π–π interactions either by *n*-butyl or angled phenyl groups.

### 2.3. Antibacterial activity

Data on the antibacterial activity of the complexes **KK1** and **KK2**, together with those of the starting materials [HL, $n(\mathrm{Bu_3Sn})_2\mathrm{O}$ and $\mathrm{Ph_3Sn(OH)}$], against *B. subtilis*, *S. aureus* (Gram positive), *P. aeruginosa* and *E. coli* (Gram-negative) bacterial, are presented in Table 2. From the MIC values, it is apparent that the organotin(IV) complexes were more toxic towards Gram-positive strains than Gram-negative strains. The reason may be the difference in the structures of the cell walls [19]. The walls of the Gram-negative cells are more complex than those of Gram-positive cells. Lipopolysaccharides form an outer lipid membrane and contribute to the complex antigenic specificity of Gram-negative cells. It is suggested that the anti-microbial activity of the complexes is due to either by killing the microbes or inhibiting their multiplication by blocking their active sites [20]. Since the molecular structures of the two complexes are quite similar, the only difference being the groups attaching to the central tin atoms (butyl in complex **KK1** versus phenyl in complex **KK2**), the slightly better antibacterial activity of complex **KK2** can be attribute to the presence of phenyl groups, which facilitate binding to biological molecules by π–π interactions.

The enhanced bactericidal activity of the ligand on complexation with organotin(IV) precursors may be explained by chelation theory, according to which chelation reduces the polarity of the central metal atom because of partial sharing of its positive charge with the donor groups and possible π-electron delocalization within the whole chelate ring [21,22]. This chelation increases the lipophilic nature of the central atom, which favours the permeation of the complexes through the lipid layer of the cell membrane. Compounds inhibit the growth of bacterial to greater extent as concentration increased.

## 3. Experimental

### 3.1. Materials and methods

The reagents employed in the present study were purchased from commercial sources and used without further purification. The ligand (*E*)-3-(4-(diphenylamino)phenyl)acrylic acid was prepared by the literature method [23,24], see Scheme 1. Carbon, hydrogen and nitrogen assays were carried out with a CHN–O-Rapid instrument and were within ±0.4% of the theoretical values. IR spectra were record on a Nicolet 470 FT-IR spectrophotometer using KBr discs in the range 4000–400 $cm^{-1}$. $^1$H and $^{13}$C NMR spectra were recorded on a Bruker AV 400 spectrometer with TMS as internal standard. $^{119}$Sn NMR spectra (proton-decoupled) were recorded on a Bruker AV 400 spectrometer operating at 150 MHz; resonances are referenced to tetramethyltin (external standard, $^{119}$Sn). The solid-state luminescence spectra measured by F-4500 FL Spectrophotometer. The PMT Voltage is 700 V and the Scan speed is 240 nm/min. The EX and EM Slit are 1.0 nm and 5.0 nm, respectively.

### 3.2. Synthesis of (E)-3-(4-(diphenylamino)phenyl)acrylic acid (*HL*)

4-(Diphenylamino)benzaldehyde (2.73 g, 10 mmol) and malonic acid (2.16 g, 30 mmol) were added to 60 mL pyridine and stirred at 90 °C for 2 h in the presence of 1 mL of piperidine, followed by adjustment of pH 1 with dilute HCl solution. A yellow crude

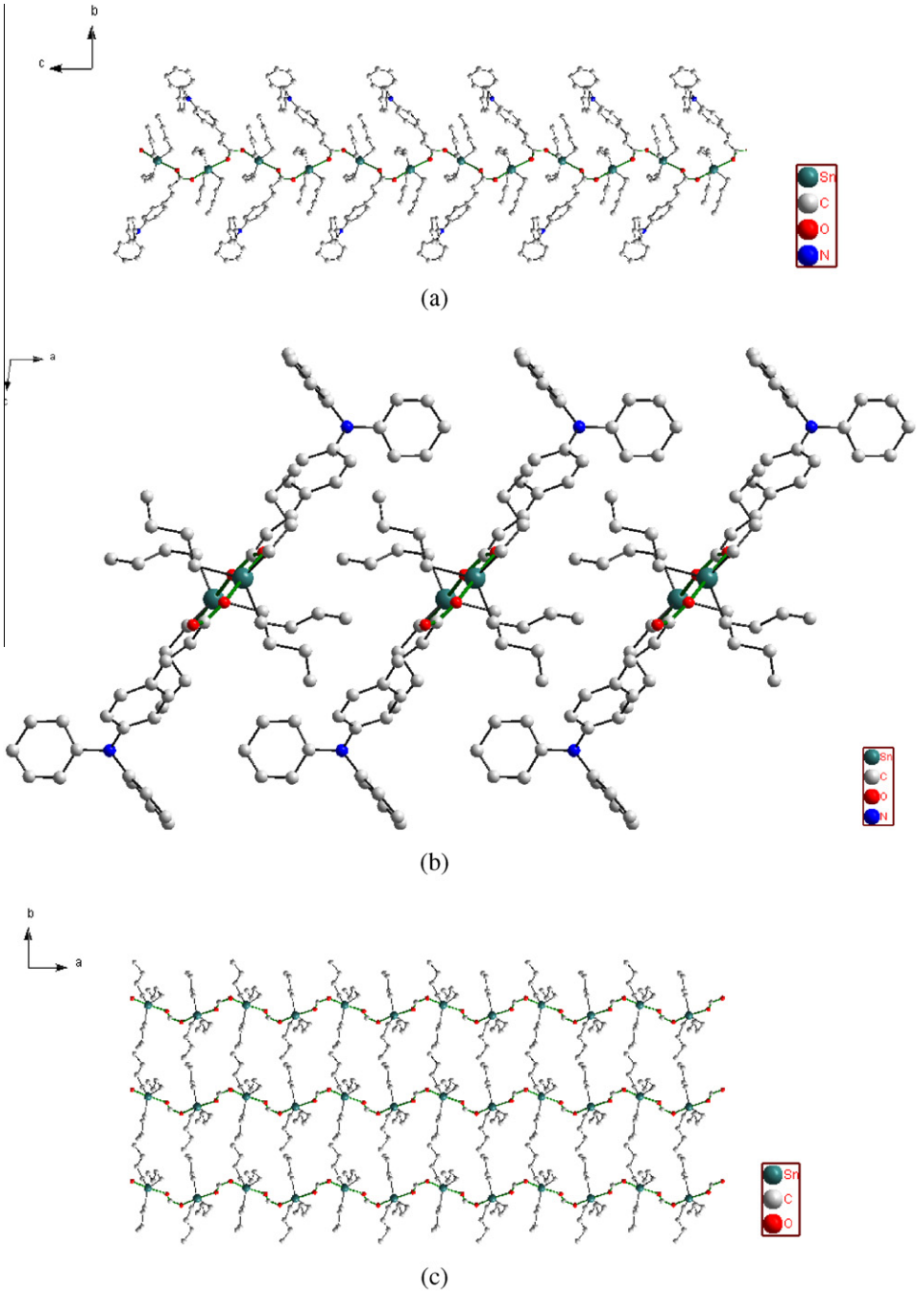



**Fig. 3.** (a) View of the layers of **KK1** along the *a*-axis. (b) View of the layers of **KK1** along the *b*-axis. (c) View of the layers of **KK1** along the *c*-axis (Benzyl groups are omitted for clarity). All the H atoms are omitted for clarity.

product was obtained and recrystallized with ethanol and dried in vacuum. Yield: 86%. M.p. = 137 °C. $^1$H NMR (400 MHz, $CDCl_3$) $\delta$ (ppm): 6.32 (d, $J$ = 16.0 Hz, 1H), 7.02–7.43 (*Ar*H, 14H), 7.75 (d, $J$ = 16.0 Hz, 1H). *Anal.* Calc. for $C_{21}H_{17}NO_2$ ($Mr$ = 315.4): C, 79.98; H, 5.43; N, 4.44. Found: C, 79.77; H, 5.45; N, 4.43%. ESI-MS: $m/z$ 316.4 ($[M+H]^+$).

### 3.3. Synthesis of the complexes ***KK1*** and ***KK2***

#### 3.3.1. $[nBu_3SnOL]_n$ (***KK1***)

A solution of HL (0.630 g, 2 mmol) in benzene (15 mL) was added to a solution of $n(Bu_3Sn)_2O$ (0.596 g, 1 mmol) in benzene (15 mL). The reaction mixture was refluxed for 12 h. The solvent was removed under vacuum and the residue was dissolved in $CH_2Cl_2$ (10 mL) and filtered; methanol (5 mL) was added, and the solution was left to crystallize at room temperature. Colorless crystals of **KK1** were formed from this solution after 5 d. Yield: 83%. M.p. = 217 °C. $^1$H NMR (400 MHz, $CDCl_3$) $\delta$ (ppm): 7.56 (d, 1H, $J$ = 16.4 Hz), 7.38 (d, 2H, $J$ = 8.0 Hz), 7.30 (d, 4H, $J$ = 7.6 Hz), 7.15–7.09 (m, 6H), 7.01 (d, 2H, $J$ = 8 Hz), 6.36 (d, 1H, $J$ = 16.0 Hz), 1.30–1.70 (m, 18H), 0.94 (t, 9H, $J$ = 7.2 Hz). $^{13}$C NMR ($CDCl_3$) $\delta$ (ppm): 13.7, 16.5, 27.1, 27.9, 76.7, 77.0, 77.3, 117.4, 122.1, 123.7, 125.2, 128.9, 129.4, 143.5, 147.1. $^{119}$Sn NMR ($CDCl_3$): $\delta$ = −146.5 ppm. *Anal.* Calc. for $C_{33}H_{43}NO_2Sn$ ($Mr$ = 604.37): C, 65.58; H, 7.17; N, 2.32. Found: C, 65.77; H, 7.16; N, 2.31%. IR (KBr): 3048 (w), 2954 (s), 2823 (s), 2854 (m), 1641 (s), 1602 (m), 1545 (s), 1514 (s),

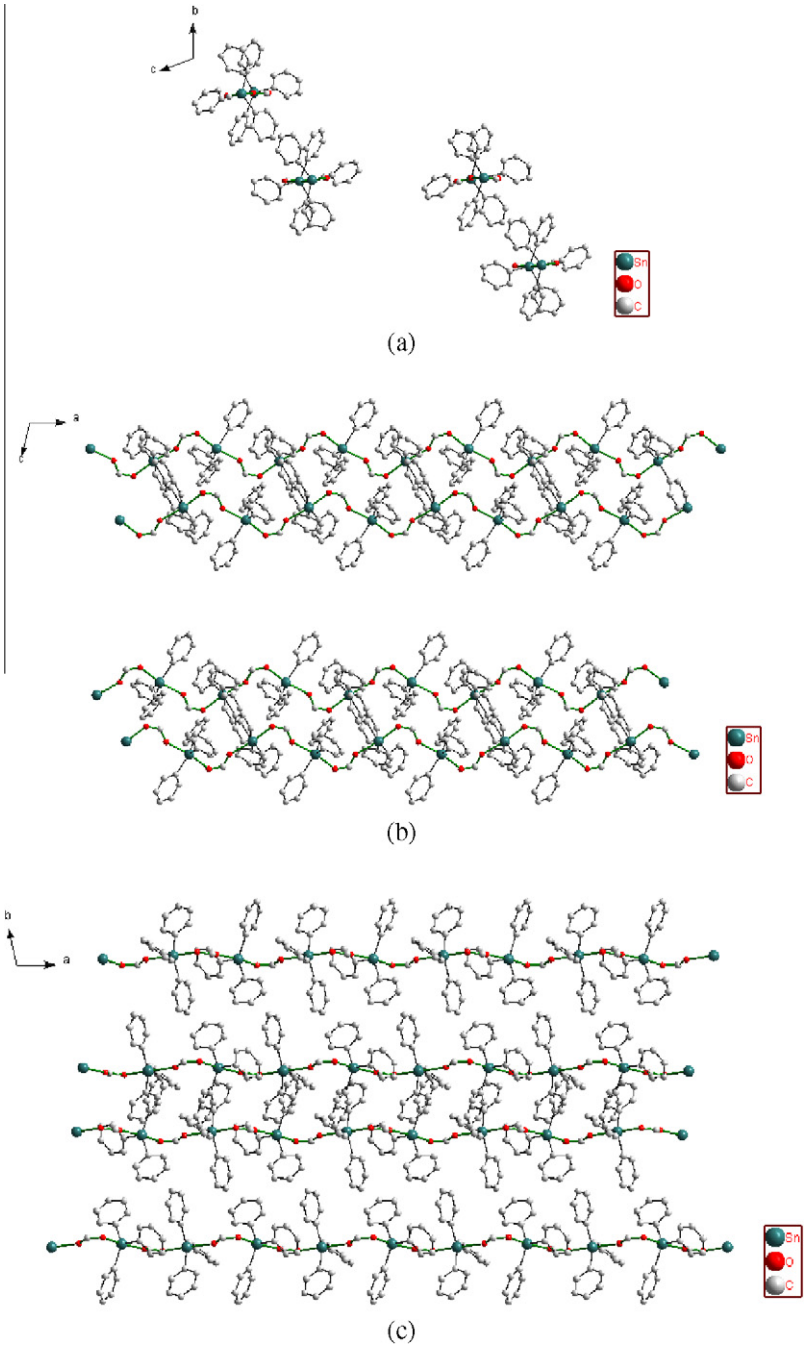



**Fig. 4.** (a) View of the layers of **KK2** along the *a*-axis. (b) View of the layers of **KK2** along the *b*-axis. (c) View of the layers of **KK2** along the *c*-axis. Benzyl groups except for the carbon atoms bonded to tin atoms and all the H atoms are omitted for clarity.

1452 (s), 1393 (s), 1364 (s), 1231 (m), 1169 (w), 983 (w), 832 (w), 744 (s), 723 (m), 670 (w), 621 (w), 509 (w) $cm^{-1}$.

### 3.3.2. $[Ph_3SnOL]_n$ (**KK2**)

A solution of HL (0.315 g, 1 mmol) in benzene (15 mL) was added to a solution of $Ph_3Sn(OH)$ (0.368 g, 1 mmol) in benzene (15 mL). The reaction mixture was refluxed for 12 h. The solvent was removed under vacuum and the residue was dissolved in $CH_2Cl_2$ (10 mL) and filtered; methanol (5 mL) was added, and the solution was left to crystallize at room temperature. Yellow crystals of **KK2** were formed from this solution after 7 d. Yield: 71%. M.p. = 164 °C. $^1H$ NMR (400 MHz, $CDCl_3$) $\delta$ (ppm): 7.87 (d, 1H, $J$ = 6.0 Hz), 7.79–7.80 (m, 5H), 7.68–7.72 (m, 2H), 7.47–7.49 (m, 9H), 7.38 (d, 2H, $J$ = 8.4 Hz), 7.32 (d, 3H, $J$ = 7.6 Hz), 7.08–7.15 (m, 6H), 7.01 (d, 2H, $J$ = 8.4 Hz), 6.43 (d, 1H, $J$ = 15.6 Hz). $^{13}C$ NMR ($CDCl_3$) $\delta$ (ppm): 76.7, 77.0, 77.3, 123.9, 125.4, 128.3, 128.8, 129.0, 129.2, 129.5, 130.0, 135.7, 136.1, 136.9, 137.2, 138.7. *Anal.* Calc. for $C_{39}H_{31}NO_2Sn$ ($Mr$ = 664.34): C, 70.50; H, 4.70; N, 2.11. Found: C, 70.37; H, 4.72; N, 2.10%. $^{119}Sn$ NMR ($CDCl_3$): $\delta$ = −121.4 ppm. IR (KBr): 3053 (w), 1641 (m), 1599 (s), 1515 (s), 1451 (s), 1427 (m), 1358 (s), 1336 (s), 1226 (s), 1167 (w), 1075 (w), 990 (w), 831 (w), 751 (s), 728 (s), 698 (s), 625 (w), 448 (m) $cm^{-1}$.

### 3.4. X-ray crystallography

The crystallographic data for **KK1** and **KK2** were collected on a Bruker Smart 1000 CCD area detector diffractometer. Equipped

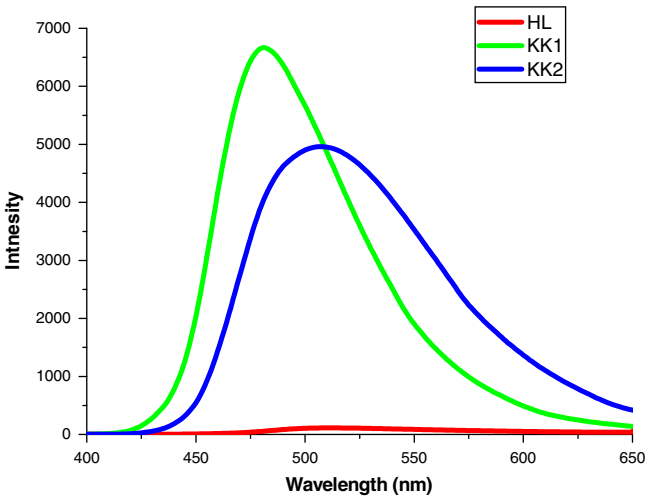



**Fig. 5.** Solid-state luminescence spectra of **HL**, **KK1** and **KK2**.

with Mo Kα ($\lambda$ = 0.71073 Å) radiation using ω-scan mode. Empirical absorption correction was applied to the data. The structures were solved by direct methods and refined by full-matrix least-squares methods on $F$ [2]. All non-hydrogen atoms were located from the trial structure and then refined anisotropically. All hydrogen atoms were generated in idealized positions. All calculations were performed with SHELXL-97 programs. Other relevant parameters of the crystal structure are listed in Table 3.

### 3.5. Antibacterial activity

The antibacterial activities of the synthesized complexes were tested against *B. subtilis* ATCC 6633, *E. coli* ATCC 35218, *P. aeruginosa* ATCC 13525 and *S. aureus* ATCC 6538 using MH medium (Muellere-Hinton medium: casein hydrolysate 17.5 g, soluble starch 1.5 g and beef extract 1000 mL) by MTT method. The MICs of the tested compounds were determined by a colorimetric method using the dye 3-(4,5-dimethyl-2-triazyl)-2,5-diphenyl-2*H*-tetrazolium bromide (MTT) [25]. This yellow tetrazolium salt is cleaved by dehydrogenases inside mitochondria or in other cellular locations possessing dehydrogenase activity to form its purple formazan derivative [26,27], which can be measured spectrophotometrically at 550 nm. MTT is cleaved by all living, metabolically active microorganisms impendent of proliferation and irrespective of unicellular or multicellular growth and thus is a measure of metabolic activity.

A stock solution of the synthesized compound (50 μg/mL) in DMSO was prepared and graded quantities of the tested compounds were incorporated in specified quantity of sterilized liquid medium (MH medium for antibacterial activity). A specified quantity of the medium containing the compound was poured into microtitration plates. Suspension of the microorganism was prepared to contain approximately $10^5$ cfu/mL and applied to microtitration plates with serially diluted compounds in DMSO to be tested and incubated at 37 °C for 24 h. After the MICs were visually determined on each of the microtitration plates, 50 mL of PBS (phosphate buffered saline 0.01 mol/L, pH 7.4, $Na_2HPO_4{\cdot}12H_2O$ (2.9 g), $KH_2PO_4$ (0.2 g), NaCl (8.0 g), KCl (0.2 g), distilled water (1000 mL) containing 2 mg of MTT/mL was added to each well. Incubation was continued at room temperature for 4–5 h. The content of each well was removed, and 100 mL of isopropanol containing 5% 1 mol/L HCl was added to extract the dye. After 12 h of incubation at room temperature, the optical density (OD) was measured with a microplate reader at 550 nm. The OD of the blank, which consisted of an uninoculated plate incubated together with the inoculated plates, was subtracted from the ODs of the inoculated plates. The percentage of MTT conversion to its formazan derivative for each well was calculated by comparing the OD at 550 nm ($OD_{550}$) of the wells with that of the drug-free control based on the following equation: ($A_{550}$ of wells that contained the drug/$A_{550}$ of the drug-free well) × 100%. Growth inhibition then was assessed by visual observation of the wells containing MTT and compared with the MTT-free wells.

The observed MICs are presented in Table 2. The experiment has been done in triplicate and the results were averaged.

**Table 2**
Crystallographic data and structure refinements for complexes **KK1** and **KK2**.

| Compound | **KK1** | **KK2** |
|---|---|---|
| Formula | $C_{33}H_{43}NO_2Sn$ | $C_{78}H_{62}N_2O_4Sn_2$ |
| Formula weight | 604.37 | 1328.68 |
| Crystal system | monoclinic | Triclinic |
| Space group | $P2_1$ | $P\bar{1}$ |
| Crystal size ($mm^3$) | 0.30 × 0.20 × 0.12 | 0.21 × 0.11 × 0.08 |
| $a$ (Å) | 9.796(5) | 11.077(5) |
| $b$ (Å) | 10.430(5) | 15.362(5) |
| $c$ (Å) | 15.889(5) | 22.079(5) |
| $\alpha$ (°) | 90.000(5) | 108.526(5) |
| $\beta$ (°) | 97.604(5) | 100.061(5) |
| $\gamma$ (°) | 90.000(5) | 98.304(5) |
| $V$ ($Å^3$) | 1609.1(12) | 3426(2) |
| $Z$ | 2 | 2 |
| $D_{calc}$ ($g/cm^{-3}$) | 1.247 | 1.288 |
| $\mu$ ($mm^{-1}$) | 0.820 | 0.778 |
| $F$(0 0 0) | 628 | 1352 |
| $\theta$ range (°) | 1.29–25.00 | 1.43–27.49 |
| Reflections collected | 8604 | 15 567 |
| Reflections unique | 5020 | 10 511 |
| Parameters | 337 | 775 |
| Goodness-of-fit (GOF)on $F^2$ | 0.945 | 0.844 |
| $R_1$, $wR_2$ [$I > 2\sigma(I)$] | 0.0622, 0.1489 | 0.0381, 0.1084 |
| $R_1$, $wR_2$ [all data] | 0.1033, 0.1783 | 0.0709, 0.1345 |

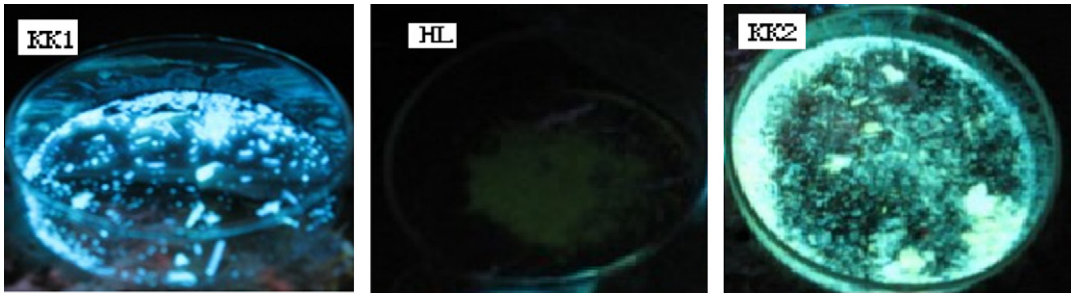
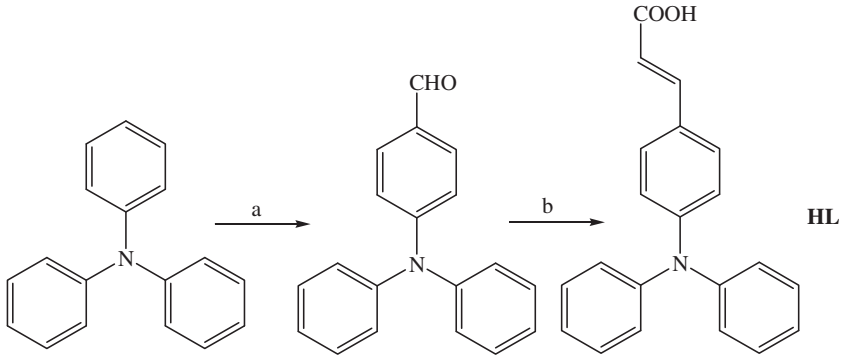



**Fig. 6.** Luminescence photograph of **HL**, **KK1** and **KK2** under 365 nm irradiation.




**Scheme 1.** (a) DMF/$POCl_3$, 0 °C and (b) malonic acid/pyridine/piperidine, 90 °C.

**Table 3**
MICs of the synthesized compounds.

| Compound | Minimum inhibitory concentrations (μg/mL) | | | |
|---|---|---|---|---|
| | *B. subtilis* | *S. aureus* | *P. aeruginosa* | *E. coli* |
| **KK1** | 6.25 | 1.562 | 25 | 50 |
| **KK2** | 3.125 | 0.78 | >50 | >50 |
| **HL** | >50 | >50 | >50 | >50 |
| ***n*(Bu$_3$Sn)$_2$O** | 12.5 | 25 | 50 | >50 |
| **Ph$_3$Sn(OH)** | 25 | 50 | 50 | >50 |
| Kanamycin B | / | / | 3.125 | 3.125 |
| Penicillin G | 1.562 | 1.562 | / | / |

## 4. Conclusion

Two organotin(IV) complexes had been obtained in the crystalline state, and the structures of them were reported here. The structures of the complexes **KK1** and **KK2** reveal that the ligand acts as a linker to connect metal centers to give rise to 1D infinite chain. It's exciting to discover that both complexes exhibit strong solid-state luminescence emission while the HL almost quenches. The *in vitro* antibacterial activities of the synthesized complexes had been studied by testing them in four bacterial strains which shows their inhibitory effect. Complex **KK2** exhibited considerable antibacterial activity against *S. aureus* with MIC value of 0.78 μg/mL, which was superior to the positive controls penicillin G.

## Acknowledgements

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## Appendix A. Supplementary material

CCDC 743473 and 743474 contain the supplementary crystallographic data for complexes **KK1** and **KK2**, respectively. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.ica.2010.09.004.

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