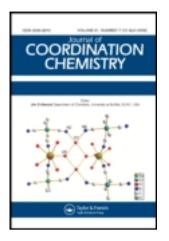
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# Synthesis, characterization and antibacterial activity of indium(III) complexes with adamantane-ring containing Schiff bases

# XU-DONG JIN\*†, YUE-HONG JIN‡, ZHI-YUAN ZOU§, ZHI-GANG CUI†, HAI-BO WANG†, PING-LI KANG†, CHUN-HUA GE† and KAI LI†

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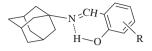
Indium(III) chloride tetrahydrate and Schiff-base ligands derived from adamantaneamine and 3-/4-methoxysalicylaldehyde gave two complexes,  $C_{22}H_{32}Cl_3InN_2O_3$  (1) and  $C_{36}H_{44}C_{13}InN_2O_4$  (2), respectively. The complexes were characterized by IR, <sup>1</sup>H NMR, elemental analysis, molar conductance, thermal analysis, and single-crystal X-ray diffraction. Complex 1 crystallizes in the monoclinic system,  $P_{1/n}$  space group with the asymmetric unit consisting of one indium(III), one *N*-(3-methoxysalicylidene)-aminoadamantane (L<sup>1</sup>), three chlorides and one *N*,*N*-dimethylformamide molecule. The indium is six-coordinate with reversed triangular-prism geometry via three oxygens and three chlorides. Complex 2 crystallizes in the triclinic system,  $P_{\overline{1}}$  space group; the asymmetric unit consists of one indium(III), two *N*-(4methoxysalicylidene)-aminoadamantane (L<sup>2</sup>), and three chlorides. The indium is five-coordinate with distorted trigonal-bipyramidal geometry via two oxygens and three chlorides. Antibacterial activities of the complexes have been investigated against *Escherichia coli* and *Staphylococcus aureus*.

Keywords: Indium(III) complexes; Synthesis; Antibacterial activity; Adamantane; Schiff bases

# 1. Introduction

A relatively small number of theoretical and practical routes for Schiff base indium complexes [1–5] have been reported compared with other metal complexes. Schiff base indium complexes exhibit advantages in antibacterial and anti-cancer activities than other metal complexes or than the free Schiff bases [6–8]. Adamantaneamine has an obvious effect on controlling exuviating of the influenza virus, and also restrains virus going into host cells playing an effective role of preventing influenza  $A_2$ . In addition it can alleviate Parkinson symptoms [9]. We designed and synthesized a series of complexes to demonstrate biological activity by introducing liposoluble adamantane rings as well as integrating of the biochemical characteristics of Schiff base indium complexes. In this work, a six-coordinate complex (1) and a five-coordinate complex (2)

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 $L^1$ : R= 3-OCH<sub>3</sub>; L<sup>2</sup>: R= 4-OCH<sub>3</sub> Figure 1. Structures of L<sup>1</sup> and L<sup>2</sup>.

were synthesized and the structures were characterized by IR, <sup>1</sup>H NMR, elemental analysis, molar conductance, thermal analysis, and single-crystal X-ray diffraction. The antibacterial activities of complexes against two bacteria *Escherichia coli* and *Staphylococcus aureus* were simultaneously investigated.

#### 2. Experimental

# 2.1. Materials and methods

All chemicals and solvents were of analytical grade and used as received. Elemental analysis was carried out on a Perkin Elmer Flash EA 1112. Chemical shifts ( $\delta$ ) for <sup>1</sup>H NMR spectra were recorded at 300 MHz on a Varian Mercury-Vx300 spectrometer in CDCl<sub>3</sub> containing TMS as an internal standard. Infrared spectra (IR) were scanned from 4000 to 400 cm<sup>-1</sup> with KBr pellets on a Nicolet NEXUS FT-IR 5700 spectrophotometer. Thermal analysis was carried out on a METTLER TOLEDO TGA/SDTA851e. Melting points were measured on a WRS-1B micro melting point apparatus and are uncorrected. The molar conductance of the complexes in DMF ( $1.0 \times 10^{-3} \text{ mol L}^{-1}$ ) was measured on a DDS-11A conductometer.

# 2.2. Synthesis of ligands

 $L^1$  and  $L^2$  (figure 1) were prepared by condensation of adamantaneamine and 3- or 4-methoxysalicylaldehyde in anhydrous alcohol.

Adamantaneamine 3.03 g (0.02 mo1) in 20 mL anhydrous alcohol was added to a solution of 3- or 4-methoxysalicylaldehyde 3.04 g (0.02 mo1) in 60 mL anhydrous alcohol. The mixture was refluxed for 2 h and cooled to room temperature. A colored crystal was filtered and evaporated under reduced pressure.

L<sup>1</sup>: 4.45 g, yield 78%. Brown needles. m.p. 108.5 ~ 110.0°C. Lit. [10]. 109°C. IR (KBr): 3433(w), 3009(w), 2912(s), 2849(s), 1629(s), 1468(s), 1453(m), 1420(w), 1376(w), 1367(w), 1344(w), 1327(w), 1306(w), 1276(m), 1249(s), 1171(w), 1119(m), 1090(s), 986(w), 966(m), 940(w), 932(w), 966(w), 843(w), 814(w), 787(w), 755(w), 713(w), and 544(w). <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz):  $\delta$  15.19 (*s*, 1H, Ar–OH); 8.24 (*s*, 1H, CH=N); 6.84 (*d*, <sup>3</sup>*J*=7.5, 1H, Ar–H); 6.83 (*d*, <sup>3</sup>*J*=8.1, 1H, Ar–H); 6.68 (*t*-like, <sup>3</sup>*J*=7.8/8.1, 1H, Ar–H); 3.88 (*s*, 3H, Ar–OCH<sub>3</sub>); 2.17 (*s*, 3H, CH, adamantane ring); 1.85 (*s*, 6H, CH<sub>2</sub>, adamantane ring); 1.79–1.71 (*m*, 6H, CH<sub>2</sub>, adamantane ring). Anal. Calcd for C<sub>18</sub>H<sub>23</sub>NO<sub>2</sub> (285.38): C, 75.76; H, 8.12; N, 4.91. Found: C, 75.74; H, 8.15; N, 4.88.

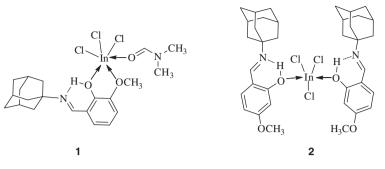


Figure 2. Proposed structures of 1 and 2.

L<sup>2</sup>: 3.71 g, yield 65%. Yellow needles. m.p. 106.7 ~ 108.7°C. IR (KBr): 3434(w), 2907(s), 2848(m), 1625(s), 1531(w), 1452(w), 1342(w), 1309(w), 1227(s), 1210(w), 1187(w), 1169(w), 1116(m), 1088(w), 1032(w), 967(w), 939(w), 922(w), 831(m), 814(w), 787(w), 645(w), 585(w). <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz):  $\delta$  11.51 (*s*, 1H, Ar–OH); 8.00 (*s*, 1H, CH=N); 7.01 (*d*, <sup>3</sup>*J*=9.6, 1H, Ar–H); 6.29 (*s*, 1H, Ar–H); 6.27 (*d*, <sup>3</sup>*J*=8.7, 1H, Ar–H); 3.79 (*s*, 3H, Ar–OCH<sub>3</sub>); 2.19 (*s*, 3H, CH, adamantane ring); 1.89 (*s*, 6H, CH<sub>2</sub>, adamantane ring); 1.79–1.71 (*m*, 6H, CH<sub>2</sub>, adamantane ring). Anal. Calcd for C<sub>18</sub>H<sub>23</sub>NO<sub>2</sub> (285.38): C, 75.76; H, 8.12; N, 4.91. Found: C, 75.77; H, 8.10; N, 4.85.

# 2.3. Synthesis of the complexes

Complexes 1 and 2 (figure 2) were prepared in a similar procedure with indium(III) chloride tetrahydrate and the corresponding Schiff base in appropriate solvent.

**2.3.1.** Synthesis of 1. Indium(III) chloride tetrahydrate (0.29 g, 1.0 mmol) in 20 mL anhydrous alcohol was added dropwise to a hot solution of  $L^1$  (0.29 g, 1.0 mmol) in 20 mL anhydrous alcohol. Thereafter the mixture was refluxed for 1.5 h and then kept at room temperature overnight; powdery precipitates were crystallized in ethyl acetate/N,N-dimethylformamide 10:1 (v/v) to give 1 (0.22 g, 38%) as yellow plates.

Complex 1: m.p.:  $126.0 \sim 126.2^{\circ}$ C. IR (KBr): 3431(m), 2918(s), 2853(m), 1647(s), 1610(m), 1560(w), 1499(s), 1455(w), 1309(m), 1226(s), 1087(w), 957(w), 851(w), 781(w), 745(m), 721(w), 547(w), 500(w), 448(w). <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz):  $\delta$  13.26 (*s*, 1H, Ar–OH); 8.19 (*d*, <sup>3</sup>*J* = 14.7, 1H, CH=N); 7.09 (*d*, <sup>3</sup>*J* = 7.8, 1H, Ar–H); 7.03 (*d*, <sup>3</sup>*J* = 6.6, 1H, Ar–H); 6.80 (*t*-like, <sup>3</sup>*J* = 7.8/8.1, 1H, Ar–H); 4.05 (*s*, 3H, Ar–OCH<sub>3</sub>); 2.30 (*s*, 3H, CH, adamantane ring); 2.06 (*s*, 6H, CH<sub>2</sub>, adamantane ring); 1.83-1.71 (*m*, 6H, CH<sub>2</sub>, adamantane ring). Anal. Calcd for  $C_{22}H_{32}Cl_{3}InN_2O_3$  (593.68): C, 44.51; H, 5.43; N, 4.72. Found: C, 44.32; H, 5.55; N, 4.66.

**2.3.2.** Synthesis of 2. Indium(III) chloride tetrahydrate (0.29 g, 1.0 mmol) in 20 mL anhydrous alcohol was added dropwise to a hot solution of L<sup>2</sup> (0.59 g, 2.0 mmol) in 20 mL anhydrous alcohol. The color of the mixture changed from yellow to white during 2h reflux. The reactant was kept at room temperature overnight and red

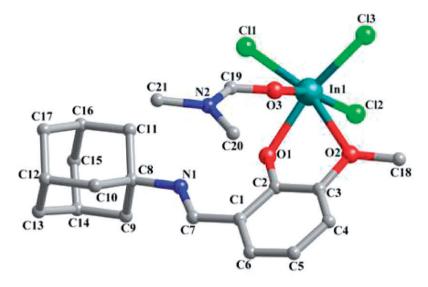


Figure 3. X-ray structure of 1. Hydrogens and ethanol are omitted for clarity.

powdery precipitate was filtered off; recrystallization from ethanol afforded 2 (0.43 g, 55%) as white plates.

Complex **2**: m.p.:  $269.5 \sim 270.7^{\circ}$ C. IR (KBr): 3436(m), 2908(s), 2854(m), 1647(s), 1611(m), 1540(m), 1481(m), 1458(w), 1366(w), 1303(m), 1230(s), 1216(s), 1176(w), 1122(w), 1083(w), 1026(w), 975(w), 840(w), 785(w), 608(w), 538(w), 502(w), 448(w). <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz):  $\delta$  12.67 (*s*, 1H, Ar–OH); 7.78 (*d*, <sup>3</sup>*J* = 13.8, 1H, CH=N); 7.36 (*d*, <sup>3</sup>*J* = 8.4, 1H, Ar–H); 6.29 (*d*, <sup>3</sup>*J* = 8.4, 1H, Ar–H); 6.97 (*s*, 1H, Ar–H); 3.86 (*s*, 3H, Ar–OCH<sub>3</sub>); 2.22 (*s*, 3H, CH, adamantane ring); 2.05 (*s*, 6H, CH<sub>2</sub>, adamantane ring); 1.72 (*s*, 6H, CH<sub>2</sub>, adamantane ring). Anal. Calcd for C<sub>36</sub>H<sub>44</sub>Cl<sub>3</sub>InN<sub>2</sub>O<sub>4</sub> (789.92): C, 54.74; H, 5.61; N, 3.55. Found: C, 55.12; H, 5.78; N, 3.47.

#### 2.4. X-ray crystallography

The crystallographic data collections for 1 and 2 were conducted on a Bruker Smart Apex II CCD with graphite monochromated Mo-K $\alpha$  radiation ( $\lambda = 0.71073$  Å) at 296(2) K using the  $\omega$ -scan technique. The data were integrated using the SAINT program, which also corrected the intensities for Lorentz and polarization effects [11]. An empirical absorption correction was applied using SADABS [12]. The structures were solved by direct methods using SHELXS-97 and all non-hydrogen atoms were refined anisotropically on  $F^2$  by full-matrix least-squares using the SHELXL-97 crystallographic software package [13]. The hydrogens were generated geometrically. All calculations were performed on a personal computer with the SHELXL-97 crystallographic software package. X-ray structures are shown in figures 3 and 4. The details of the crystal parameters, data collection and refinement for 1 and 2 are summarized in table 1. Selected bond lengths and angles with their estimated standard deviations are given in table 2.

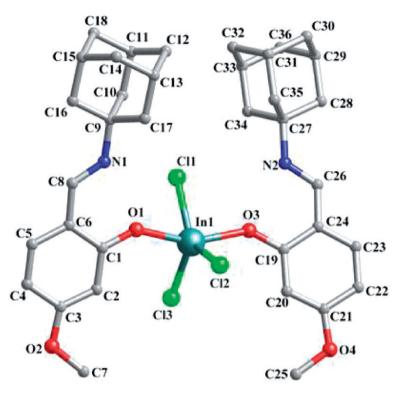


Figure 4. X-ray structure of 2. Hydrogens are omitted for clarity.

#### 3. Results and discussion

#### 3.1. Elemental analysis and molar conductance

The C, H and N contents are in agreement with the formulas of  $C_{22}H_{32}Cl_3InN_2O_3$  for 1 and  $C_{36}H_{44}Cl_3InN_2O_4$  for 2. The molar conductance values ( $\Lambda_M$ ) are 5.13 and 6.88 S cm<sup>2</sup> mol<sup>-1</sup> for 1 and 2, indicating non-electrolytes [14].

#### 3.2. IR spectra

IR data for 1 and 2 are given in table 3. Broad and intense absorptions at  $3431 \text{ cm}^{-1}$  for 1 and  $3436 \text{ cm}^{-1}$  for 2 can be identified as OH stretching vibration, indicating that phenolic hydroxyl was not deprotonated when the complexes were formed, and oxygens of phenolic hydroxyl participate in coordination. The strongest absorption at  $1647 \text{ cm}^{-1}$  for 1 and 2 is characteristic of the C=N. The strong absorptions at  $1226 \text{ cm}^{-1}$  for 1 and  $1230 \text{ cm}^{-1}$  for 2 are C–O stretch of phenolic hydroxyls. The absorption at  $500 \text{ cm}^{-1}$  for 1,  $502 \text{ cm}^{-1}$  for 2, are attributed to In–O vibration, indicating that oxygens of the Schiff bases are coordinated to In.

Empirical formula	$C_{22}H_{32}Cl_{3}InN_{2}O_{3}(1)$	$C_{36}H_{44}Cl_{13}InN_2O_4$ (2)
Formula weight $(g mol^{-1})$	593.68	789.92
Crystal size (mm <sup>3</sup> )	$0.22 \times 0.21 \times 0.20$	$0.20 \times 0.16 \times 0.10$
Crystal system	Monoclinic	Triclinic
Space group	$P2_{1}/n$	$P\bar{1}$
Unit cell dimensions (Å, °)	1) 1	
a	9.5789(6)	10.072(2)
b	23.2272(15)	13.589(3)
С	12.5084(9)	14.027(3)
α	90.00(2)	72.68(3)
β	101.0100(10)	81.42(3)
γ	90.00(2)	76.20(3)
$V(Å^3), Z$	2731.8(3), 4	1773.6(6), 2
$\theta$ range for data collection (°)	2.41-25.01	1.53-25.02
Limiting indices	$-11 \le h \le 11$ ,	$-10 \le h \le 11$ ,
-	$-15 \le k \le 27$ ,	$-16 \le k \le 16$ ,
	$-14 \le l \le 14$	$-13 \le l \le 16$
$\rho (\text{g cm}^{-3})$	1.463	1.479
$\mu (mm^{-1})$	1.185	0.933
Reflections collected/unique	13,644/4790 [R(int) = 0.0332]	12,935/6203 [R(int) = 0.0299]
Data/restraints/parameters	4790/32/393	6203/0/418
Goodness-of-fit	1.059	1.062
F(000)	1224	812
$T(\mathbf{K})$	296(2)	296(2)
$R_1^{a}/wR_2^{b}(I > 2\sigma(I))$	0.0328/0.0912	0.0361/0.0980
$R_1/wR_2$ (all data)	0.0353/0.0928	0.0423/0.1032

Table 1. Crystallographic data for 1 and 2.

<sup>a</sup> $R_1 = \Sigma ||F_o| - |F_c|| / |F_o|;$  <sup>b</sup> $wR_2 = [\Sigma w (F_o^2 - F_c^2)^2 / \Sigma w (F_o^2)^2]^{1/2}$ .

# 3.3. <sup>1</sup>H NMR analysis

<sup>1</sup>H NMR data for **1** and **2** in CDCl<sub>3</sub> are given in table 4. Singlets at 13.26 for **1** and 12.67 for **2** are assigned to the phenolic hydroxyl proton because of intramolecular hydrogen bond formation of  $-CH=N\cdots$ HO- between imino nitrogen and phenolic hydroxyl hydrogen. A sharp singlet at 8.19 for **1** and 7.78 for **2** is assigned to the -CH=N proton. A couple of peaks at 7.09–6.80 for **1** and 7.36–6.97 for **2** are assigned to Ar–H. An intense singlet at 4.05 for **1** and 3.86 for **2** was assigned to OMe. The CH and CH<sub>2</sub> from adamantane were identified in the range of 2.30–1.71.

## 3.4. Crystal structure of 1 and 2

The single-crystal X-ray analysis reveals 1 crystallizes in the monoclinic system,  $P2_1/n$  space group. As shown in figure 3, the asymmetric unit consists of one indium(III), one L<sup>1</sup>, three chlorides and one DMF. The indium is six-coordinate with reversed triangular-prism geometry via three oxygens (O1, O2, O3) as a bottom plane and three chlorides as another plane. The dihedral angle of two approximately parallel planes is  $4.700(73)^{\circ}$ . O1 and O2 are from bidentate ligand L<sup>1</sup> and O3 is from coordinated DMF. The In–O bond lengths are 2.162(2), 2.425(3) and 2.273(2) Å and In–Cl are 2.3960(11), 2.4317(9) and 2.4134(10) Å. The longest bond length between indium and oxygen is In1–O2 due to the effect of methyl. For a similar reason the longest bond length between indium and chloride is In1–Cl2.

1			
In(1)–O(1)	2.162(2)	In(1)-O(3)	2.273(2)
In(1)-Cl(1)	2.3960(11)	In(1)-Cl(3)	2.4134(10)
In(1)-O(2)	2.425(3)	In(1)-Cl(2)	2.4317(9)
N(1)-C(7)	1.289(5)	N(1)-C(8)	1.496(4)
N(2)-C(19)	1.304(5)	N(2)-C(20)	1.460(6)
O(1) - C(2)	1.317(4)	O(2) - C(3)	1.390(4)
O(2)-C(18)	1.415(5)	O(3)-C(19)	1.254(4)
O(1)–In(1)–O(3)	77.94(10)	O(1) - In(1) - Cl(1)	92.27(7)
O(3)-In(1)-Cl(1)	91.97(8)	O(1)-In(1)-Cl(3)	157.39(7)
O(3)-In(1)-Cl(3)	88.07(7)	Cl(1)-In(1)-Cl(3)	106.01(4)
O(1)-In(1)-O(2)	69.60(9)	O(3) - In(1) - O(2)	77.56(10)
Cl(1)–In(1)–O(2)	160.48(7)	Cl(3)-In(1)-O(2)	90.23(7)
O(1)-In(1)-Cl(2)	94.16(7)	O(3) - In(1) - Cl(2)	163.56(8)
Cl(1)-In(1)-Cl(2)	102.83(4)	Cl(3)-In(1)-Cl(2)	94.58(4)
O(2)-In(1)-Cl(2)	86.20(7)	C(2)-O(1)-In(1)	117.9(2)
C(3) - O(2) - In(1)	111.2(2)	C(18)-O(2)-In(1)	129.1(3)
C(19)–O(3)–In(1)	122.8(2)	C(7)–N(1)–C(8)	127.0(3)
2			
In(1)-O(3)	2.161(2)	In(1)-O(1)	2.166(2)
In(1)-Cl(3)	2.3827(11)	In(1)-Cl(2)	2.3849(10)
In(1)-Cl(1)	2.3941(13)	O(1) - C(1)	1.307(4)
O(2) - C(3)	1.362(4)	O(2) - C(7)	1.432(4)
N(1) - C(8)	1.296(4)	N(1) - C(9)	1.483(4)
N(2) - C(26)	1.299(4)	N(2) - C(27)	1.486(4)
Cl(2)-In(1)-Cl(1)	134.54(6)	Cl(3)-In(1)-Cl(1)	115.50(6)
O(3) - In(1) - O(1)	157.63(9)	O(3) - In(1) - Cl(3)	99.23(7)
O(1) - In(1) - Cl(3)	101.45(7)	O(3) - In(1) - Cl(2)	92.37(7)
O(1) - In(1) - Cl(2)	88.55(8)	Cl(3)-In(1)-Cl(2)	109.95(5)
O(3) - In(1) - Cl(1)	81.73(7)	O(1) - In(1) - Cl(1)	81.75(7)
O(1) - C(1) - C(2)	123.2(3)	C(1) - O(1) - In(1)	136.8(2)
C(19) - O(3) - In(1)	136.7(2)	C(8) - N(1) - C(9)	127.4(3)
C(26) - N(2) - C(27)	126.1(3)	O(1) - C(1) - C(6)	118.7(3)
	× /		× /

Table 2. Selected bond lengths (Å) and angles (°) in 1 and 2.

Table 3. Main IR data for 1 and 2 (cm<sup>-1</sup>).

Complex	γо–н	γсн	γсн₂	$\gamma_{C=N}$	γс−о	γм−о
1 2	3431(m)	2918(s)	2853(m)	1647(s)	1226(s)	500(w)
	3436(m)	2908(s)	2854(m)	1647(s)	1230(s)	502(w)

s, strong; m, medium; w, weak.

Complex 2 crystallizes in the triclinic system,  $P_{1}$  space group. As shown in figure 4, the asymmetric unit consists of one indium(III), two L<sup>2</sup> and three chlorides. The indium is five-coordinate with distorted trigonal-bipyramidal geometry via two oxygens and three chlorides. The equatorial coordination sites of In1 are occupied by three chlorides (Cl1, Cl2, Cl3) with bond lengths of 2.3941(13), 2.3849(10) and 2.3827(11)Å for In1–Cl1, In1–Cl2 and In1–Cl3, respectively. Axial positions of In1 are occupied by two oxygens (O1, O3) from two monodentate L<sup>2</sup> with bond lengths of 2.166(2) and 2.161(2)Å for In1–O1 and In1–O3. The distance between two adamantane rings from

Compound	Ar–OH	CH=N	Ar–H	$\operatorname{OCH}_3$	Adamantane ring
1 <sup>a</sup>		$\substack{8.19\\(d, {}^{3}J=14.7, 1\mathrm{H})}$	7.09 ( $d$ , ${}^{3}J$ = 7.8, 1H); 7.03 ( $d$ , ${}^{3}J$ = 6.6, 1H); 6.80 ( <i>t</i> -like, ${}^{3}J$ = 7.8/8.1, 1H)		2.30 ( <i>s</i> , 3H, CH); 2.06 ( <i>s</i> , 6H, CH <sub>2</sub> ); 1.83-1.71 ( <i>m</i> , 6H, CH <sub>2</sub> )
2	12.67 (s, 1H)	7.78 ( $d$ , ${}^{3}J = 13.8$ , 1H)	7.36	3.86 (s, 3H)	2.22 (s, 3H, CH); 2.05 (s, 6H, CH <sub>2</sub> ); 1.72 (s, 6H, CH <sub>2</sub> )

Table 4. <sup>1</sup>HNMR data for **1** and **2** ( $\delta$  in ppm, J in Hz).

<sup>a</sup>8.05(s, 1H, CHO of DMF); 2.97(s, 3H, CH<sub>3</sub> of DMF); 2.87(s, 3H, CH<sub>3</sub> of DMF).

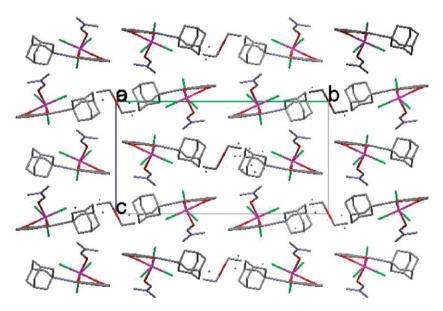


Figure 5. The packing diagram of 1 viewed along the *a*-axis.

two ligands is 4.492 Å for C32–C11, indicating *cis*-coordination of two ligands to indium. Dihedral angle of two phenyl planes is  $16.004(3)^{\circ}$ .

The molecular packing in the unit cell demonstrates no significant intermolecular hydrogen bonding interactions or  $\pi$ - $\pi$  stacking for 1 and 2; molecules are arranged by weak van der Waals interactions to form network structures (figures 5 and 6). Ethanol was found in the crystal of 1.

#### 3.5. Thermal analysis

Thermal gravimetric analysis was carried out with a heating rate of  $20^{\circ}$ C min<sup>-1</sup> in argon from  $25 \sim 900^{\circ}$ C. The TG-DTG curves of **1** and **2** are provided in "Supplementary material". A difference of thermal decomposition was observed with four stages for **1** 

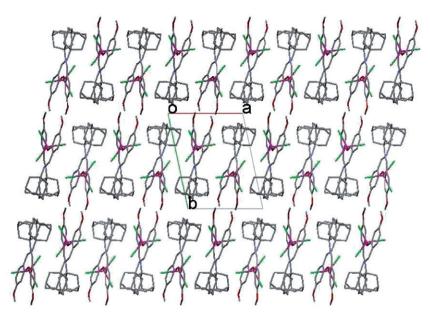


Figure 6. The packing diagram of **2** viewed along the *c*-axis.

and three stages for **2**. The starting decomposition and weight losses were approximately  $120^{\circ}$ C for **1** and  $250^{\circ}$ C for **2**. When the complexes were heated above  $550^{\circ}$ C, the residues were deduced as oxides.

#### 3.6. Antibacterial activity

Complexes 1 and 2 were tested *in vitro* to assess their growth inhibitory activity against *E. coli* and *S. aureus* by Kirby methods [15, 16]; the corresponding Schiff bases were also investigated. The compounds were prepared with four concentrations of  $1.0 \times 10^{-1}$ ,  $1.0 \times 10^{-2}$ ,  $1.0 \times 10^{-3}$ ,  $1.0 \times 10^{-4}$  mol L<sup>-1</sup> in DMF. The diameters of inhibition zone were measured after 48 h and the results are presented in table 5.

Both complexes exhibit antibacterial activities against *E. coli* and *S. aureus* compared with the corresponding ligands; the antibacterial abilities of the complexes were concentration-dependent. Complex **2** was more active against *E. coli* than *S. aureus* in concentrated solution, while **1** displayed almost the same activities against both bacteria at  $1.0 \times 10^{-1}$  and  $1.0 \times 10^{-2}$ . In contrast with complexes, L<sup>2</sup> exhibited relatively poor antibacterial activity against *Escherichia coli* at any concentrations.

### 4. Conclusions

Single-crystal X-ray diffraction revealed that 1 consists of one indium(III), one Schiff-base ligand, three chlorides and one DMF. The indium is six-coordinate with reversed triangular-prism. Complex 2 consists of one indium(III), two Schiff-base

		Escherichia coli	hia coli			Staphylococ	Staphylococcus aureus	
Concentration $(mol L^{-1})$ bacteria name	$1.0 \times 10^{-1}$	$1.0 \times 10^{-2}$ $1.0 \times 10^{-3}$ $1.0 \times 10^{-4}$	$1.0 \times 10^{-3}$	$1.0 \times 10^{-4}$	$1.0  imes 10^{-1}$	$1.0 \times 10^{-2}$	$1.0 \times 10^{-2}$ $1.0 \times 10^{-3}$ $1.0 \times 10^{-4}$	$1.0  imes 10^{-4}$
L <sup>1</sup>	$7.0 \pm 1.0$	$7.0 \pm 1.0$	$7.0 \pm 1.0$	6.0	$7.0 \pm 1.0$	$6.5 \pm 0.5$	$6.75 \pm 0.25$	$6.5 \pm 0.5$
1	$23.0 \pm 1.0$	$15.0 \pm 1.0$	$9.5 \pm 0.5$	$7.0 \pm 1.0$	$22.0 \pm 1.0$	$14.0 \pm 1.0$	$7.0 \pm 1.0$	$7.0 \pm 1.0$
$L^2$	$6.5\pm0.5$	6.0	6.0	6.0	$7.0 \pm 0.5$	$7.0 \pm 1.0$	$6.5\pm1.0$	$6.5\pm0.5$
2	$19.0 \pm 1.0$	$13.0 \pm 1.0$	$9.0 \pm 1.0$	6.0	$10.0 \pm 1.0$	$8.0\pm1.0$	$6.75 \pm 0.25$	$6.75 \pm 0.25$
Filter paper diameter is 6 mm.								

Table 5. Inhibition of ligands and complexes against bacteria growth (inhibition zone/mm).

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ligands and three chlorides. The indium is five-coordinate with distorted trigonal-bipyramidal geometry.

Antibacterial activity showed both complexes exhibited activities against *E. coli* and *S. aureus*. Antibacterial activities of both complexes were better than that of the corresponding ligand.

# Supplementary material

Supplementary X-ray crystallographic data for 1 (CCDC 761183) and 2 (CCDC 797037) can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or 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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