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Synthesis, Characterization, and Antibacterial Activity of Two Zinc(II) Complexes with Schiff Bases from Halogenated Salicylaldehyde and Amantadine[1]

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Abstract—By a condensation reaction of halogenated salicylaldehyde and amantadine, two new Schiff base ligands (HL^1 and HL^2) were synthesized, respectively. A followed mixture of the ligands and zinc(II) chloride in the presence of NaOH in an alcoholic medium brought out two novel complexes (ZnL^1_2) (**I**) and (ZnL^2_2) (**II**). These two complexes were characterized by the means of melting point, elemental analysis, IR, UV-Vis, ^{1}H NMR, molar conductance and single-crystal X-ray diffraction analysis. X-ray diffraction analysis reveals that **I** crystallizes in monoclinic system, $P2_1/c$ space group, a = 9.7812(5), b = 25.6198(12), c = 27.7381(18) Å, β = 105.881(4), $F(000)$ = 1416, R_1 = 0.0731, wR_2 = 0.1147; **II** crystallizes in orthorhombic system, *Pbca* space group, a = 11.1717(10), b = 20.5888(15), c = 27.7381(18) Å, $F(000)$ = 2976, R_1 = 0.1341, wR_2 = 0.1410. Both in **I** and **II**, the central zinc(II) atom is four-coordinated via two nitrogen atoms and two oxygen atoms from the corresponding Schiff base ligands, forming a distorted tetrahedral geometry.

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INTRODUCTION

Schiff-bases are a large class of organic compounds with imino (–HC= N–) group and simple structures [1–3]. Transition metal complexes of Schiff bases have been amongst the most widely studied coordination compounds in the past few years, since they are found to be of importance as biochemical, analytical and antimicrobial reagents [4, 5]. Zinc is a vital element in the life and an important enzyme active site [6, 7]. The Zn(II) complexes of Schiff bases are also biologically active and they exhibit enhanced activities as compared to the free Schiff bases [8]. Tricyclo [3.3.1.1(3,7)] decane-1-amine (amantadine) is an antivirotic drug that has been used to treat influenza and Parkinson disease [9–11]. Salicylaldehyde derivatives, with one or more halo-atoms in the aromatic ring, showed variety of biological activities such as antibacterial and antifungal activities [12, 13]. In view of these points above, in this paper, two zinc(II) complexes with Schiff bases from halogenated salicylaldehyde and amantadine, which are bis(2-(1-adamantyliminomethyl)-4-chlorophenolato-N,O)-zinc(II) (**I**) and bis(2-(1-adamantyliminomethyl)-4-bromophenolato-N,O)-zinc(II) (**II**), were designed and synthesized in the presence of NaOH. Their structures were characterized by the means of IR, UV-VIS, ^{1}H NMR, elemental analysis and molar conductance. More over, the coordination behaviors of the ligands towards zinc(II) ion were also investigated and their absolute structures were determined by a single-crystal X-ray diffraction analysis. The antibacterial activities of complexes against two bacteria *Escherichia coli* and *Bacillus subtilis* were simultaneously investigated.

EXPERIMENTAL

Materials and methods. All chemicals and solvents were of analytical grade and used as received. Elemental analysis was carried out on Perkin Elmer Flash EA 1112. Chemical shifts (δ) for ^{1}H NMR spectra were recorded at 300 MHz on a Varian Mercury-Vx300 spectrometer in $CDCl_3$ solvent containing TMS as an internal standard. IR spectra were scanned in the range 4000 to 400 cm^{-1} with KBr pellets on a Nicolet NEXUS FT-IR 5700 spectrophotometer. UV-Vis spectrum was measured on a Perkin Elmer Lambda 25 spectrophotometer. Melting points were measured on a WRS-1B micro melting point apparatus which were uncorrected. The molar conductance of the complexes in DMF (1.0×10^{-3} mol L^{-1}) at room temperature was measured on a DDS-11A conductormeter.

Synthesis of ligands. Two Schiff base ligands, 2-((*E*)-(-adamantan-1-ylimino)methyl)-4-chlorophenol (HL^1) and 2-((*E*)-(-adamantan-1-ylimino)methyl)-4-bromophenol (HL^2), were prepared analogously to the literatures [14–17]. The synthetic route in this work was shown below:

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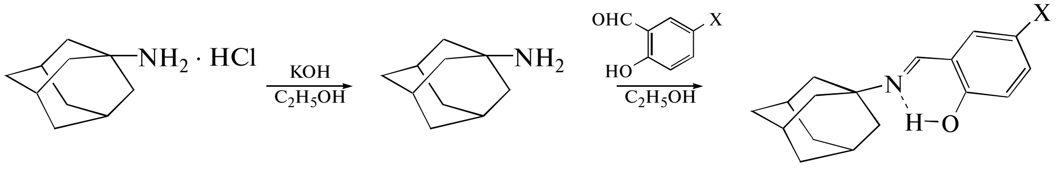


HL^1: X = Cl; HL^2: X = Br

Amantadine hydrochloride (0.375 g, 2.0 mmol) and KOH (0.112 g, 2.0 mmol) in 50 mL anhydrous alcohol were stirred for 1 h. The produced white precipitates (KCl) were filtered out and the transparent liquid was added dropwise to aldehyde (2.0 mmol) in 30 mL anhydrous alcohol under constant stirring. The resulting solution was refluxed for ~1 h, concentrated to about 20 mL through reduced pressure distillation and then stood at room temperature. A yellow solid appeared after 2–3 days with the solvent evaporation. The solid was filtered off and washed with anhydrous alcohol three times and air-dried.

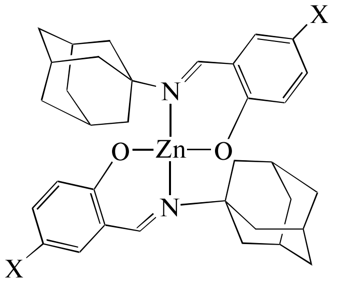
Syntheses of complexes I and II were carried out in a similar procedure with NaOH, the corresponding Schiff base ligand and zinc(II) chloride in appropriate solvents, respectively. 20 mL ethanolic NaOH (0.080 g, 2.0 mmol) was added to a solution of ligand (2.0 mmol) in 20 mL anhydrous ethanol. After the solution was stirred for ten minutes, zinc(II) chloride (0.136 g, 1.0 mmol) in 20 mL anhydrous ethanol was added and the mixture was refluxed for another 2 h at 60°C. Then the solution was kept at room temperature overnight and yellowish complex precipitates were filtered off and dried. The yields were 58% (**I**) and 61% (**II**).

For $C_{34}H_{38}N_2O_2Cl_2Zn \cdot CH_3OH$ (**I**)
anal. calcd., %: C, 63.51; H, 5.96; N, 4.36.
Found, %: C, 63.30; H, 6.00; N, 4.21.

For $C_{34}H_{38}N_2O_2Br_2Zn$ (**II**)
anal. calcd., %: C, 55.80; H, 5.23; N, 3.83.
Found, %: C, 55.75; H, 5.21; N, 4.05.

The molar conductance values (λ_M) are 1.95 and 2.03 S cm^2 mol^{-1} for **I** and **II**. It was concluded from the results that complexes were non-electrolytic in nature [18].

Proposed structures of complexes are the following:



I: X = Cl; **II**: X = Br

X-ray structure determination. The crystals of complexes suitable for X-ray analysis were obtained after about one week with a solvent CH_2Cl_2/MeOH (1 : 1 v/v) slow evaporation. The crystallographic data collections for complexes **I** and **II** were conducted on a Bruker Smart Apex II CCD with graphite monochromated MoK_α radiation (λ = 0.71073 Å) at 296(2) K using the ω-scan technique. The data were integrated by using the SAINT program, which also did the intensities corrected for Lorentz and polarization effect [19]. An empirical absorption correction was applied using the SADABS program [20]. The structures were solved by direct methods using the program SHELXS-97 and all non-hydrogen atoms were refined anisotropically on F^2 by the full-matrix least-squares technique using the SHELXL-97 crystallographic software package [21]. The hydrogen atoms were generated geometrically. All calculations were performed on a personal computer with the SHELXL-97 crystallographic software package. The details of the crystal parameters, data collection and refinement for **I** and **II** are summarized in Table 1. Selected bond lengths and angles with their estimated standard deviations are given in Table 2. X-ray structures of two complexes are shown in Fig. 1; they were visualized by Diamond [22].

Supplementary material for structures **I** and **II** has been deposited with the Cambridge Crystallographic Data Centre (no. 893297 (**I**), 893298 (**II**); deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk).

RESULTS AND DISCUSSION

In IR spectra of ligands, broad and intensity absorptions at 3443 and 3463 cm^{-1} for HL^1 and HL^2 can be identified as OH stretching vibration. Free OH stretching vibration is generally observed at 3500–3600 cm^{-1}; the

Table 1. Crystal data and structure refinement information for compounds **I** and **II**

Parameter	Value	
	I	**II**
Formula weight	674.98	731.85
Crystal size, mm	0.43 × 0.34 × 0.28	0.41 × 0.31 × 0.23
Crystal system	Monoclinic	Orthorhombic
Space group	$P2_1/c$	*Pbca*
a, Å	9.7812(5)	11.1717(10)
b, Å	25.6198(12)	20.5888(15)
c, Å	13.5526(6)	27.7381(18)
β, deg	105.881(4)	90
V, Å^3	3266.6(3)	6380.1(8)
Z	4	8
ρ_{calcd}, g cm^{-3}	1.372	1.524
F(000)	1416	2976
μ, mm^{-1}	0.953	3.308
θ Range, deg	2.56–28.13	2.46–25.00
Index ranges	$-11 \le h \le 8$, $-30 \le k \le 22$, $-11 \le l \le 16$	$-13 \le h \le 8$, $-24 \le k \le 15$, $-32 \le l \le 21$
Reflections collected/unique	12102/5746	14936/5619
R_{int}	0.0250	0.0625
Data/restraints/parameters	5746/0/472	5619/0/452
GOOF	1.028	1.033
R_1/wR_2 ($I > 2\sigma(I)$)*	0.0458/0.0982	0.0585/0.1077
R_1/wR_2 (all data)	0.0731/0.1147	0.1341/0.1410
Absorption correction	Empirical	
Refinement method	Full-matrix least-squares on F^2	
$\Delta\rho_{max}/\Delta\rho_{min}$, e Å^{-3}	0.586/–0.324	0.922/–0.594

* $R_1 = \Sigma||F_o| - |F_c||/|F_o|$; $wR_2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^2)^2]^{1/2}$.

observed lower value is due to intramolecular hydrogen bonding. This band is absent in **I** and **II**, illustrating deprotonation of phenolic OH group occurs prior to the coordination. A band assigned to C=N stretching vibration 1629 cm^{-1} in HL^1 and 1626 cm^{-1} in HL^2 moves to lower wavenumber 1612 cm^{-1} in **I** and 1611 cm^{-1} in **II**, implying a coordination of the Schiff bases through azomethine nitrogen to Zn(II) in **I** and **II**. The spectra of HL^1 and HL^2 displays similar strong bands exactly at 1279 cm^{-1}, which are fairly certain to C–O stretching vibration; these bands occur in **I** and **II** at lower frequency 1162 cm^{-1}. In the low frequency regions, the presence of absorptions at 500 and 495 cm^{-1} for **I** and **II** reveals that there exists Zn–O vibration, indicating that oxygen of the Schiff bases is coordinated to Zn(II).

The main UV-Vis data for two complexes are showed in Table 3. These two complexes exhibited similar pattern in the ultraviolet spectra but significant changes in the visible spectra compared to their ligands. There exist obvious absorption bands at 382 nm in the complexes, which are not found in the ligands. This change indicates that these zinc(II) chloride interact strongly with the Schiff base ligands. The zinc(II) ion is coordinated by oxygen atom and nitrogen atom. The maximal absorption bands at about 230 nm for ligands and complexes are due to the benzenoid conjugate structure.

^{1}H NMR data for complexes in CDCl$_3$ was given in Table 4, in which all data for ligands are also provided for comparison. Single peaks at 14.51 and 14.55 ppm for HL^1 and HL^2 are assigned to the phenolic hydroxyl protons. Because of the intramolecular hydrogen bond

Table 2. Selected bond length (Å) and angles (deg) in **I** and **II**

Bond	*d*, Å	Bond	*d*, Å
		I	
Zn(1)–O(2)	1.909(2)	Zn(1)–O(1)	1.933(2)
Zn(1)–N(2)	2.009(3)	Zn(1)–N(1)	2.011(3)
C(13)–C(28)	4.483(2)	C(12)–C(16)	1.512(6)
N(1)–C(7)	1.284(4)	N(1)–C(8)	1.499(4)
N(2)–C(24)	1.285(4)	N(2)–C(25)	1.493(4)
		II	
N(1)–Zn(1)	1.993(5)	O(1)–Zn(1)	1.919(4)
N(2)–Zn(1)	1.998(5)	O(2)–Zn(1)	1.915(4)
C(9)–C(26)	4.422(6)	C(12)–C(17)	1.530(4)
N(1)–C(7)	1.285(6)	N(1)–C(8)	1.481(9)
N(2)–C(24)	1.280(7)	N(2)–C(25)	1.493(7)
Angle	ω, deg	Angle	ω, deg
		I	
O(2)Zn(1)O(1)	111.97(11)	O(2)Zn(1)N(2)	97.07(11)
O(1)Zn(1)N(2)	110.16(11)	O(1)Zn(1)N(1)	97.17(10)
N(2)Zn(1)N(1)	124.57(11)	O(2)Zn(1)N(1)	116.35(10)
C(7)N(1)C(8)	117.8(3)	C(7)N(1)Zn(1)	118.7(2)
C(8)N(1)Zn(1)	123.52(19)	C(24)N(2)C(25)	118.4(3)
C(24)N(2)Zn(1)	120.0(2)	C(25)N(2)Zn(1)	121.6(2)
C(1)O(1)Zn(1)	124.9(2)	C(18)O(2)Zn(1)	125.3(2)
		II	
C(7)N(1)C(8)	119.8(6)	C(24)N(2)Zn(1)	118.9(4)
C(7)N(1)Zn(1)	118.9(4)	C(25)N(2)Zn(1)	121.5(4)
C(8)N(1)Zn(1)	121.2(5)	C(1)O(1)Zn(1)	123.8(4)
C(24)N(2)C(25)	119.1(5)	C(18)O(2)Zn(1)	125.5(4)
O(2)Zn(1)O(1)	112.2(2)	O(2)Zn(1)N(2)	97.80(19)
O(2)Zn(1)N(1)	111.9(2)	O(1)Zn(1)N(1)	98.45(19)
O(1)Zn(1)N(2)	108.26(19)	N(1)Zn(1)N(2)	128.5(2)

formation of CH = N···HO, they are observed in lower field. While in complexes **I** and **II**, the phenolic hydroxyl protons of HL^1 and HL^2 are eliminated as the formation of complexes, resulting in a disappearance of the single peak to the phenolic hydroxyl proton as well as the intramolecular hydrogen bond. In HL^1 and HL^2 signals appear in 8.24 and 8.23 ppm can be certainly attributed to HC=N proton [23]. However, in the spectra of complexes, these signals are observed in lower field regions of 8.17 ppm. Multiplet peaks at 6.73–7.36 ppm can be assigned to aromatic protons in ligands and complexes. The CH and CH_2 groups from adamantane are identified in the range of 1.52–2.19 ppm.

The crystallographic analysis reveals that in **I** each asymmetric unit has one zinc(II) mononuclear complex and one solvent methanol molecule. The central zinc(II) atom in **I** lies on a twofold rotation axis and is bonded to the oxygen and nitrogen donor atoms of the

Table 3. Main UV-Vis data for ligands and complexes (nm)*

Compound	λ_{max}	λ_{min}
HL^1	228(1.614), 254(0.597), 328(0.239)	244(0.496), 290(0.032)
I	230(1.432), 248(0.950), 330(0.162), 382(0.171)	244(0.918), 292(0.046), 352(0.118)
HL^2	230(1.460), 252(0.522), 328(0.199)	248(0.494), 290(0.036)
II	230(1.474), 248(0.973), 330(0.177), 382(0.213)	244(0.948), 294(0.055), 348(0.134)

* The values in brackets being absorption intensities.

two bidentate ligand molecules in a *trans*-arrangement. The geometry around Zn(II) in **I** is intermediate between square planar and tetrahedral, where the dihedral angle between the two coordination planes defined by O(2)Zn(1)N(2) and O(1)Zn(1)N(1) is 86.55° [24]. Bond angles also show that the coordination geometry about the zinc atom in **I** is distorted tetrahedral (τ_4 = [360° – (α + β)]/141° = 0.84) [25] with N(1)Zn(1)N(2) (α) and O(2)Zn(1)N(1) (β) angles of 124.57(11)° and 116.35(10)°, respectively, which is expected for a typical Schiff base ligand (containing a short C=N bond distance of 1.283 Å) coordinated to a metal centre, where the imine form is predominant. The distinctly shortened Zn(1)–O(1) and elongated Zn(1)–N(1) bonds in **I** are 1.933 and 2.011 Å, respectively (the normal bond length of them are about 1.950 and 1.970 Å), so the coordination geometry around the Zn^{2+} ion reflects Jahn–Teller effect [26]. The shortest distance between two adamantane carbons from two ligands is 4.483 Å for C(13)–C(28) in **I**, indicating *trans*-coordination of two ligands to zinc (Fig. 1a) The distances of two neighboring zinc atoms along the *x* axis are 8.612 Å in **I**. The bidentate coordination mode of **I** refrains itself from forming intramolecular hydrogen bonds because of deprotonated ligands reason, but there also exists two six-membered rings through a zinc ion, two oxygen atoms and two nitrogen atoms [27]. In addition to complex **I**, one methanol molecule is distinctly found in crystal, which is involved in an intermolecular O–H···O hydrogen bond in **I** (Fig. 2a). Geometric parameters of O(3)–H(3*A*)···O(1) hydrogen bond in **I** are the following: D–H 0.84; H···A 1.949; D···A 2.766(4) Å; angle DHA 164.18°.

The crystallographic analysis reveals that in **II** each asymmetric unit has only one independent zinc(II) mononuclear complex. The central zinc(II) atom in **I** lies on a twofold rotation axis and is bonded to the oxygen and nitrogen donor atoms of the two bidentate ligand molecules in a *trans*-arrangement. The geometry around Zn(II) in **II** is intermediate between square planar and tetrahedral, where the dihedral angle between the two coordination planes defined by O(2)Zn(1)N(2) and O(1)Zn(1)N(1) is 83.09°. Bond angles also show that the coordination geometry about the zinc atom in **II** is distorted tetrahedral (τ_4= [360° – (α + β)]/141° = 0.85) with

Table 4. ^1H NMR data for ligands and complexes (δ in ppm, *J* in Hz)

Compound	Ar–OH	CH=N	Ar–H	Adamantane ring–H
HL^1	14.51 (s., 1H)	8.24 (s., 1H)	7.27–7.15 (m., 2H); 6.87 (d., ^3J = 9.6, 1H)	2.19 (s., 3H, CH); 1.83 (s., 6H, CH_2); 1.79–1.67 (m., 6H, CH_2)
I		8.17 (s., 1H)	7.19 (d.d., ^3J = 9.0, ^4J = 2.7, 1H); 7.08 (d., ^4J = 2.7, 1H); 6.78 (d., ^3J = 9.0, 1H)	2.09 (s., 3H, CH); 1.94 (d., ^3J = 11.7, 3H, CH_2); 1.68 (t-like, ^3J = 11.4/13.8, 6H, CH_2); 1.52 (d., ^3J = 12.3, 3H, CH_2)
HL^2	14.55 (s., 1H)	8.23 (s., 1H)	7.36 (s., 1H); 7.34 (d.d., ^3J = 8.7, ^4J = 2.4, 1H, Ar–H); 6.83 (d.d., ^3J = 8.1, ^4J = 1.2, 1H, Ar–H)	2.19 (s., 3H, CH); 1.83–1.67 (m., 12H, CH_2)
II		8.17 (s., 1H)	7.30 (d.d., ^3J = 9.3, ^4J = 2.7, 1H); 7.22 (d., ^4J = 2.7, 1H); 6.73 (d., ^3J = 9.3, 1H)	2.10 (s., 3H, CH); 1.93 (d., ^3J = 11.7, 3H, CH_2); 1.68 (t-like, ^3J = 11.1/12.6, 6H, CH_2); 1.52 (d., ^3J = 12.3, 3H, CH_2)

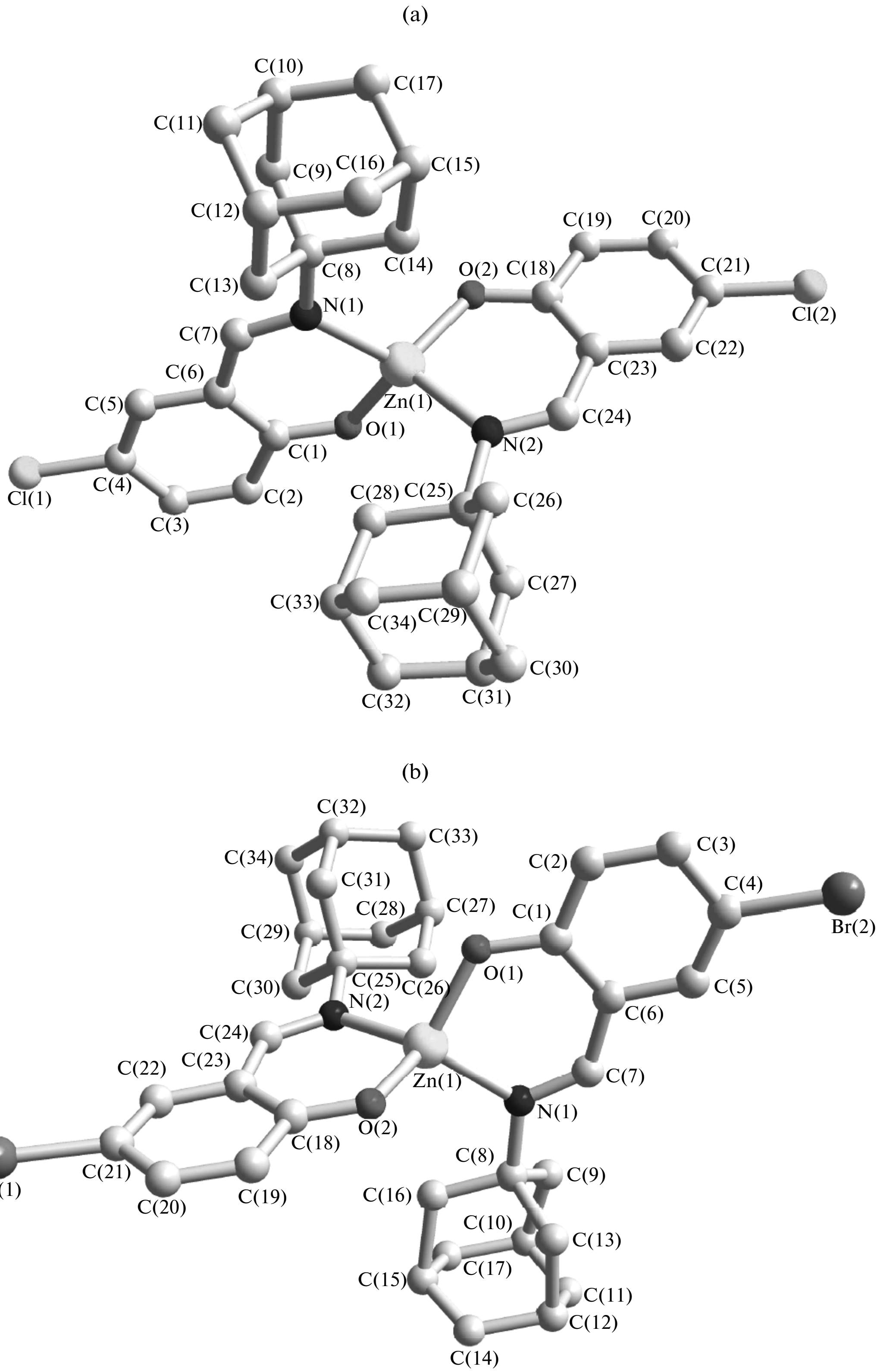


Fig. 1. X-ray structure of **I** (a) and **II** (b). Hydrogen atoms and methanol are omitted for clarity.

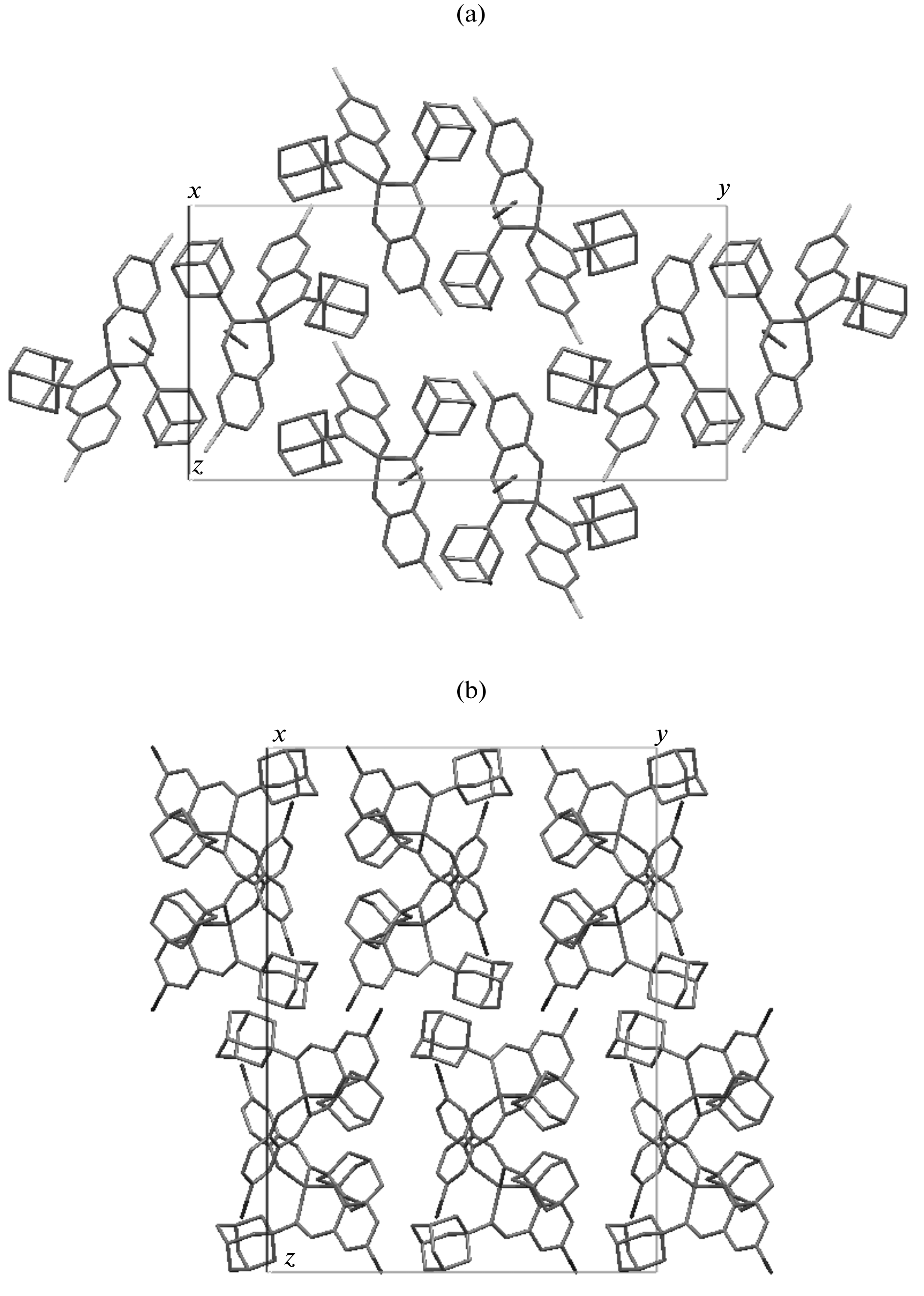


Fig. 2. The packing diagram of **I** (a) and **II** (b) viewing along the *z* axis for I and along the *x* axis for **II**.

N(1)Zn(1)N(2) (α) and O(2)Zn(1)O(1) (β) angles of 128.5(2)° and 112.2(2)°, respectively, which is expected for a typical Schiff base ligand (containing a short C=N bond distance of 1.286 Å) coordinated to a metal centre, where the imine form is predominant. The distinctly shortened Zn(1)–O(1) and elongated Zn(1)–N(1) bonds in **II** are 1.919 and 1.993 Å, respectively, so the coordination geometry around the Zn^{2+} ion reflects Jahn–Teller effect. The shortest distance between two adamantane carbons from two ligands is 4.422 Å for C(9)–C(26) in **II**, indicating *trans*-coordination of two ligands to zinc (Fig. 1b). The distances of two neighboring zinc atoms along the *x* axis are 7.371 Å in **II**. The bidentate coordination mode of complexes **II** does not involve intramolecular hydrogen bonds because the ligands have been deprotonated, but there also exists two six-membered rings through a zinc ion, two oxygen atoms and two nitrogen atoms. The complex **II** is found neither to possess intermolecular hydrogen bonding nor π–π interaction existence in stacking. The complex molecule is regularly arranged by weak vander Waals forces to construct a net

Table 5. Inhibitory of compounds against bacteria growth (inhibition zone, mm*)

Bacteria	*Escherichia coli*					*Bacillus subtilis*				
Concentration (mol L^{-1})	1.0×10^{-1}	1.0×10^{-2}	1.0×10^{-3}	1.0×10^{-4}	1.0×10^{-5}	1.0×10^{-1}	1.0×10^{-2}	1.0×10^{-3}	1.0×10^{-4}	1.0×10^{-5}
HL^1	6.0	6.0	6.0	6.0	6.0	14.0	7.0	6.0	6.0	6.0
I	12.0	8.0	8.0	8.0	7.0	13.0	12.0	10.0	9.0	8.0
HL^2	7.0	7.0	7.0	6.0	6.0	6.0	6.0	6.0	6.0	6.0
II	23.0	10.0	9.0	8.0	6.0	14.0	12.0	9.0	7.0	6.0

* Filter paper diameter being 6.0 mm.

structure containing adamantine cages (Fig. 2b). There are no any solvent molecules in crystal of **II**.

Two Schiff bases ligands and their zinc complexes were studied against a gram positive and a gram negative bacterium by inhibition zone method [28]. The compounds were prepared with five concentrations of 1.0×10^{-1}, 1.0×10^{-2}, 1.0×10^{-3}, 1.0×10^{-4}, 1.0×10^{-5} mol L^{-1} in DMF. The diameters of inhibition zone were measured after 48 h and the results are presented in Table 5.

A significant inhibitory activity much higher than that of the corresponding ligands was displayed by both the metal complexes against *Escherichia coli* and *Bacillus subtilis*. Antibacterial ability of complexes was in a concentration-dependent and with reduction of concentration, the antibacterial ability of complexes decreased significantly. It is worth to note that complex **I** and **II** showed the maximum antibacterial effect against *Escherichia coli* and *Bacillus subtilis* in concentrated solution of 1.0×10^{-1} mol L^{-1}. In contrast with **I**, complex **II** demonstrated inferior antibacterial activity under same testing conditions against *Escherichia coli*. Especially, HL^1 was not found to have antibacterial activity against *Escherichia coli* and HL^2 was not found to have antibacterial activity against *Bacillus subtilis* at above mentioned concentrations.

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