Scientific paper

Syntheses, Characterization and Crystal Structures of Schiff Base Zinc(II) Complexes with Antibacterial Activity

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Abstract

Two polynuclear zinc(II) complexes, $[Zn_2(L^1)_2(OH_2)_2]$ (**1**) and $[ZnL^2(\mu_{1,1}\text{-}N_3)]_n$ (**2**), where L^1 is the dianionic form of *N,N'*-bis(3,5-difluoro-2-hydroxybenzylidene)-1,3-diaminopropane, and L^2 is the monoanionic form of 2-[(2-dimethylaminoethylimino)methyl]-4,6-difluorophenol, have been prepared and structurally characterized by elemental analyses, IR and UV-Vis spectroscopy, as well as single-crystal X-ray diffraction. In complex **1**, the Zn atom is in octahedral coordination, with the donor atoms of the Schiff base ligands L^1 and one water O atom. In complex **2**, the Zn atom is in trigonal-bipyramidal coordination, with the three donor atoms of the Schiff base ligand L^2 and two azido N atoms. The complexes have strong antibacterial activity against *B. subtilis* and *S. aureus*.

Keywords: Schiff base; zinc complex; hydrogen bonds; crystal structure; antibacterial activity

1. Introduction

The rational design and preparation of new coordination compounds have attracted remarkable attention in coordination chemistry.[1] Azide anion is an interesting ligand in the self-assembly of new structures of complexes. A variety of azido complexes with discrete or one-, two-, and three-dimensional polymeric structures have been reported.[2] Schiff bases derived from salicylaldehyde and its derivatives usually possess two or more donor atoms, which can chelate to transition metal atoms, to form a variety of complexes.[3] Metal complexes with Schiff base ligands have attracted much attention in the fields of magnetic, catalytic, as well as biological materials.[4] Zinc complexes with Schiff base ligands are reported to have interesting antibacterial activities.[5] As a continuation of the work on Schiff base complexes, and to explore new and effective antibacterial materials, we report here the synthesis, characterization, and self-assembly of two new zinc(II) complexes, $[Zn_2(L^1)_2(OH_2)_2]$ (**1**) and $[ZnL^2(\mu_{1,1}\text{-}N_3)]_n$ (**2**), where L^1 is the dianionic form of *N,N'*-bis(3,5-difluoro-2-hydroxybenzylidene)-1,3-diaminopropane, and L^2 is the monoanionic form of 2-[(2-dimethylaminoethylimino)methyl]-4,6-difluorophenol. The antibacterial activities of the complexes were investigated.

2. Experimental

2. 1. Materials and Physical Measurements

3,5-Difluorosalicylaldehyde, propane-1,3-diamine and *N,N*-dimethylethane-1,2-diamine were purchased from Aldrich. All other reagents and solvents used in the synthesis were procured commercially and used without subsequent purification. The Schiff bases were synthesized according to the literature method.[6] Microanalyses (C,H,N) were performed using a Perkin-Elmer 2400 elemental analyzer. Infrared spectra were measured on KBr

disks with a Hitachi I-5040 FT-IR spectrophotometer. Electronic spectra were measured with a Lambda 35 spectrophotometer. Single crystal X-ray data were collected on a Bruker SMART APEX II diffractometer.

Caution! Azide complexes of metal ions are potentially explosive. Only a small amount of material should be prepared, and they should be handled with caution.

2. 2. Synthesis of Complex 1

A methanol solution (10 mL) of zinc(II) bromide (0.10 mmol, 22.5 mg) was added to the methanol solution (10 mL) of H_2L^1 (0.10 mmol, 35.4 mg) and NaN_3 (0.10 mmol, 6.5 mg). The reaction mixture was magnetic stirred for 1 h at ambient temperature to give colorless solution. Single-crystals suitable for X-ray diffraction were obtained from the filtrate by slow evaporation in a refrigerator. Yield: 205 mg, 47%. λ_{max} (nm) [ε_{max} (L mol^{-1} cm^{-1})] in methanol: 270 (16,550), 365 (12,070). IR data (KBr, cm^{-1}): 3454 (OH), 1638 (CH=N), 1461, 1362, 1259, 1155, 952, 853, 532. Anal. calcd. for $C_{34}H_{28}F_8N_4O_6Zn_2$ (%): C, 46.86; H, 3.24; N, 6.43. Found (%): C, 46.67; H, 3.33; N, 6.54.

2. 3. Synthesis of Complex 2

A methanol solution (10 mL) of zinc(II) bromide (0.10 mmol, 22.5 mg) was added to the methanol solution (10 mL) of HL^2 (0.10 mmol, 22.8 mg) and NaN_3 (0.10 mmol, 6.5 mg). The reaction mixture was magnetic stirred for 1 h at ambient temperature to give colorless solution. Single-crystals suitable for X-ray diffraction were obtained from the filtrate by slow evaporation in a refrigerator. Yield: 173 mg, 52%. λ_{max} (nm) [ε_{max} (L mol^{-1} cm^{-1})] in methanol: 270 (17,380), 377 (11,025). IR data (KBr, cm^{-1}): 2052 (N_3), 1642 (CH=N), 1556, 1469, 1353, 1289, 1254, 1123, 989, 820, 781, 753, 578. Anal. calcd. for $C_{11}H_{13}F_2N_5OZn$ (%): C, 39.48; H, 3.92; N, 20.93. Found (%): C, 39.27; H, 4.03; N, 20.72.

2. 4. X-ray Crystallography

Data collection for the complexes was performed with a Bruker Apex II CCD diffractometer at 298 K using Mo Kα (λ = 0.71073 Å) radiation. The structures were solved by direct methods with SHELXS-97 and refined by full-matrix least squares (SHELXL-97) on F^2.[7] All non-hydrogen atoms were refined anisotropically. The water H atoms of complex **1** were located from an electronic density map and refined isotropically, with O–H and H···H distances restrained to 0.85(1) and 1.37(2) Å, respectively. The remaining hydrogen atoms were placed geometrically and refined with a riding model, with isotropic displacement coefficients U(H) = 1.2 U(C) or 1.5 U(C_{methyl}). Crystallographic data for the complexes are summarized in Table 1. Selected bond lengths and angles are listed in Table 2.

Table 1. Crystallographic data for the complexes

	1	**2**
Empirical formula	$C_{34}H_{28}F_8N_4O_6Zn_2$	$C_{11}H_{13}F_2N_5OZn$
Formula weight	871.34	334.63
Crystal system	Triclinic	Monoclinic
Space group	*P*–1	$P2_1/c$
a /Å	6.9736(8)	20.034(2)
b /Å	10.8352(12)	10.1610(17)
c /Å	11.3007(13)	6.7339(12)
α /°	75.958(2)	90
β /°	89.667(2)	92.477(2)
γ /°	79.652(2)	90
V /Å^3	814.26(16)	1369.5(4)
Z	1	4
D_{calc} /g cm^{-3}	1.777	1.623
Crystal size /mm	0.33 × 0.30 × 0.28	0.16 × 0.15 × 0.15
μ(Mo Kα) /mm^{-1}	1.574	1.819
F(000)	440	680
Number of reflections	4324	4580
Unique reflections	3004	1946
Observed reflections ($I > 2\sigma(I)$)	2527	1055
Parameters	250	183
Restraints	3	0
R_{int}	0.0125	0.1137
Goodness of fit on F^2	1.047	0.971
R_1, wR_2 ($I > 2\sigma(I)$)	0.0345, 0.0739	0.0863, 0.1668
R_1, wR_2 (all data)	0.0455, 0.0792	0.1553, 0.2087

Table 2. Selected bond lengths (Å) and angles (°) for the complexes with estimated standard deviations (e.s.d.s) in parentheses

1			
Zn1–O1	2.0519(18)	Zn1–O2	2.0073(17)
Zn1–N1	2.091(2)	Zn1–N2	2.126(2)
Zn1–O3	2.201(2)	Zn1–O1A	2.560(2)
O2-Zn1-O1	89.14(7)	O2–Zn1–N1	176.20(8)
O1–Zn1–N1	87.05(8)	O2–Zn1–N2	88.87(8)
O1–Zn1–N2	170.55(9)	N1–Zn1–N2	94.91(8)
O2–Zn1–O3	90.32(8)	O1–Zn1–O3	96.92(9)
N1–Zn1–O3	90.00(9)	N2–Zn1–O3	92.33(8)
N1–Zn1–O1A	86.31(9)	N2–Zn1–O1A	88.72(9)
O1–Zn1–O1A	82.17(9)	O2–Zn1–O1A	93.31(9)
O3–Zn1–O1A	176.24(9)		
2			
Zn1–O1	2.024(8)	Zn1–N1	2.055(8)
Zn1–N2	2.280(9)	Zn1–N3	2.053(8)
Zn1–N3B	2.097(8)		
O1–Zn1–N3	94.2(4)	O1–Zn1–N1	88.7(3)
N3–Zn1–N1	127.6(3)	O1–Zn1–N3B	92.5(3)
N3–Zn1–N3B	108.5(3)	N1–Zn1–N3B	123.6(3)
O1–Zn1–N2	169.8(3)	N3–Zn1–N2	93.1(4)
N1–Zn1–N2	81.2(3)	N3A–Zn1–N2	91.8(3)

Symmetry codes: A: –*x*, 1 – *y*, 1 – *z*; B: *x*, ½ – *y*, ½ + *z*.

2. 5. Antibacterial Activity

The antibacterial activities were tested against *B. subtilis* ATCC 6633, *E. coli* ATCC 35218, *P. putida* TS 1138 and *S. aureus* ATCC 25923 using MH medium (Mueller–Hinton medium: casein hydrolysate 17.5 g, soluble starch 1.5 g, beef extract 1000 mL). The MICs (minimum inhibitory concentrations) of the test compounds were determined by a colorimetric method using the dye MTT [3-(4,5-dimethylthiazol-2-yl)-2,5-diphenyl tetrazolium bromide]. A stock solution of the synthesized compound (50 μg mL^{-1}) in DMSO was prepared and quantities of the test compounds were incorporated in specified quantity of sterilized liquid MH medium. A specified quantity of the medium containing the compound was poured into microtitration plates. A suspension of the microorganism was prepared to contain approximately 10^5 cfu mL^{-1} and applied to micro-titration 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 micro-titration plates, 50 μL of PBS (phosphate buffered saline 0.01 mol L^{-1}, pH 7.4: Na_2HPO_4 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 per mL^{-1} was added to each well. Incubation was continued at room temperature for 4–5 h. The content of each well was removed and 100 μL of isopropanol containing 5% HCl (1 mol L^{-1}) 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.

3. Results and Discussion

3. 1. Synthesis

The complexes were prepared by reaction of equimolar quantities of the Schiff base ligands with zinc bromide and sodium azide in methanol. It is interesting that the azide anion did not coordinate to the Zn atom in complex **1**, and the bromide anion did not coordinate to the Zn atom in complex **2**. Crystals of the complexes are stable in air, and soluble in methanol, ethanol, DMF and DMSO, insoluble in water. The molar conductance of the complexes is 27 Ω^{-1} cm^2 mol^{-1} for **1** and 19 Ω^{-1} cm^2 mol^{-1} for **2**, indicating that the complexes are non-electrolytes.

3. 2. Description of the Structure of Complex 1

Complex **1** is a phenolate oxygen bridged dinuclear zinc(II) compound, with the Zn···Zn distance of 3.492(2) Å (Figure 1). The molecule of the complex possesses crystallographic inversion center symmetry, with the inversion center located at the midpoint of the two Zn atoms. The Zn atom is in octahedral coordination, with the phenolate oxygen and imino nitrogen of one Schiff base ligand defining the equatorial plane, and with the phenolate oxygen of the other Schiff base ligand and one water O atom occupying the axial positions. The Zn–O and Zn–N bond lengths in the equatorial plane involving donor atoms from the Schiff base ligand are Zn1–O1 2.0519(18) Å, Zn1–O2 2.0073(17) Å, Zn1–N1 2.091(2) Å, and Zn1–N2 2.126(2) Å. The Zn–O bond lengths in the axial positions are Zn1–O3 2.201(2) Å and Zn1–O1A 2.560(2) Å, which are much longer than those in the equatorial plane. The Zn–O and Zn–N bond lengths in the complex are comparable to those reported for Schiff

Table 3. Hydrogen bond distances (Å) and bond angles (°) for complex **1**

D–H···*A*	*d*(*D*–H)	*d*(H···*A*)	*d*(*D*···*A*)	Angle (*D*–H···*A*)
O3–H3A···F1$^{\#}$	0.85(1)	2.27(2)	3.057(3)	155(3)
O3–H3A···O1$^{\#}$	0.85(1)	2.44(3)	3.099(3)	135(3)
O3–H3B···O2$^{\#}$	0.85(1)	1.96(2)	2.746(3)	155(3)
O3–H3B···F3$^{\#}$	0.85(1)	2.52(3)	3.119(3)	128(3)

Symmetry code for #: 1 – *x*, 1 – *y*, 1 – *z*.

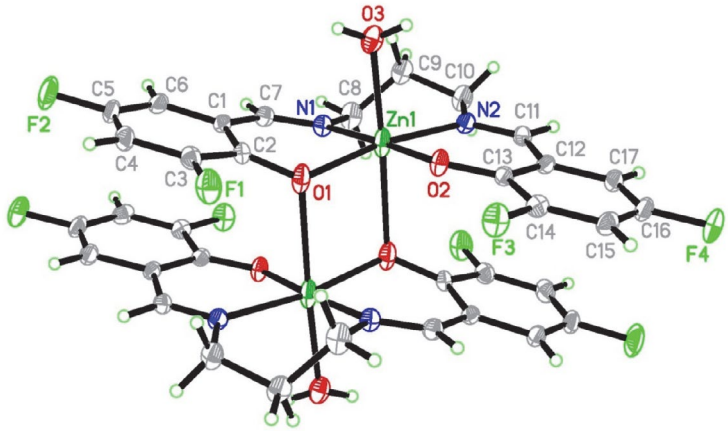

Figure 1. Molecular structure of complex **1**, with 30% thermal probability. Unlabeled atoms are at the symmetry position –*x*, 1 – *y*, 1 – *z*.

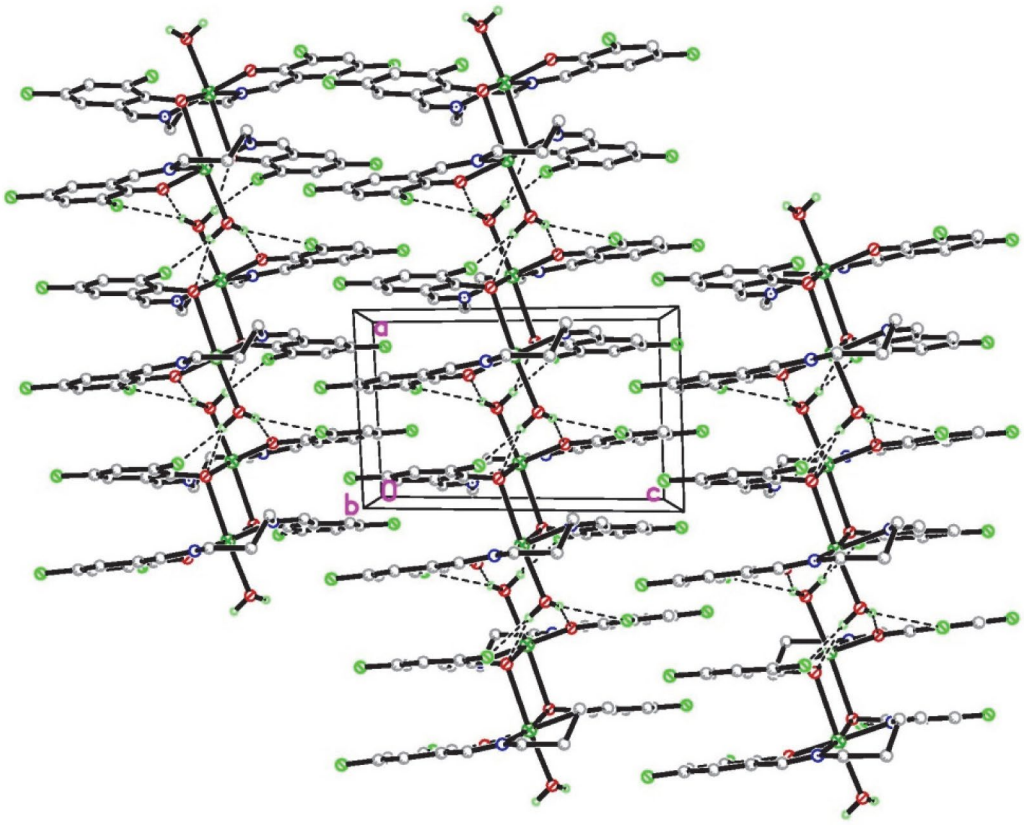

Figure 2. The hydrogen linked structure of complex **1**, viewed along the axis-*b* direction. Hydrogen bonds are shown as dashed lines.

base zinc(II) complexes.[8] As is apparent from Table 2, there are only slight deviation from an ideal octahedral geometry, with all *cis* angles within 7 ° of 90 °, with the exception of the chelating O1–Zn1–O1A angle of 82.17(9)°. The four atoms in the equatorial plane, O1, O2, N1 and N2, are approximately in a plane, with mean deviation of 0.082(3) Å, and with the Zn atom being 0.086(2) Å from the plane in the direction of O3.

In the crystal structure of the complex, the water ligands participate in the hydrogen bonds with the phenolate oxygen and fluorine groups of the Schiff base ligands. The molecules are linked through O–H···O and O–H···F hydrogen bonds (Table 3), to form chains running along the *a* axis (Figure 2).

3. 3. Description of the Structure of Complex 2

Complex **2** is an end-on azido-bridged polynuclear zinc(II) compound, with the Zn···Zn distance of 3.533(2) Å (Figure 3). The Zn atom is in a trigonal bipyramidal coordination, with the imino nitrogen of the Schiff base ligand and two azido nitrogen defining the basal plane, and with the phenolate oxygen and amino nitrogen occupying the axial positions. The Zn–N bond lengths in the basal plane involving donor atoms from the Schiff base and azide ligands are Zn1–N1 2.055(8) Å, Zn1–N3 2.053(8) Å, and Zn1–N3B 2.097(8) Å. The Zn–O and Zn–N bond lengths in the axial positions are Zn1–O1 2.024(8) Å and Zn1–N2 2.280(9) Å. The Zn–O and Zn–N bond lengths in the complex are similar to those of complex 1, and also comparable to those reported for Schiff base zinc(II) com-

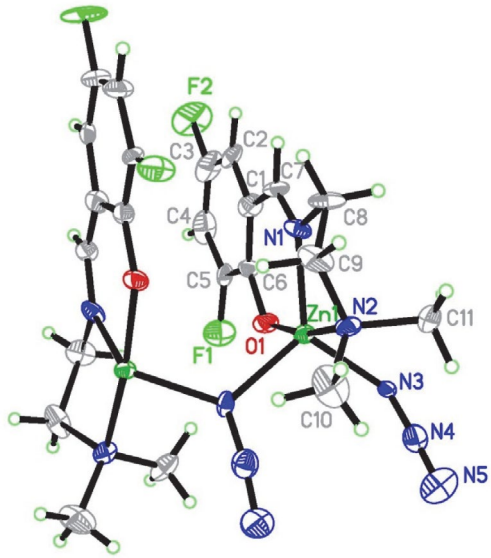

Figure 3. Molecular structure of complex **2**, with 30% thermal probability. Unlabeled atoms are at the symmetry position *x*, ½ – *y*, ½ + *z*.

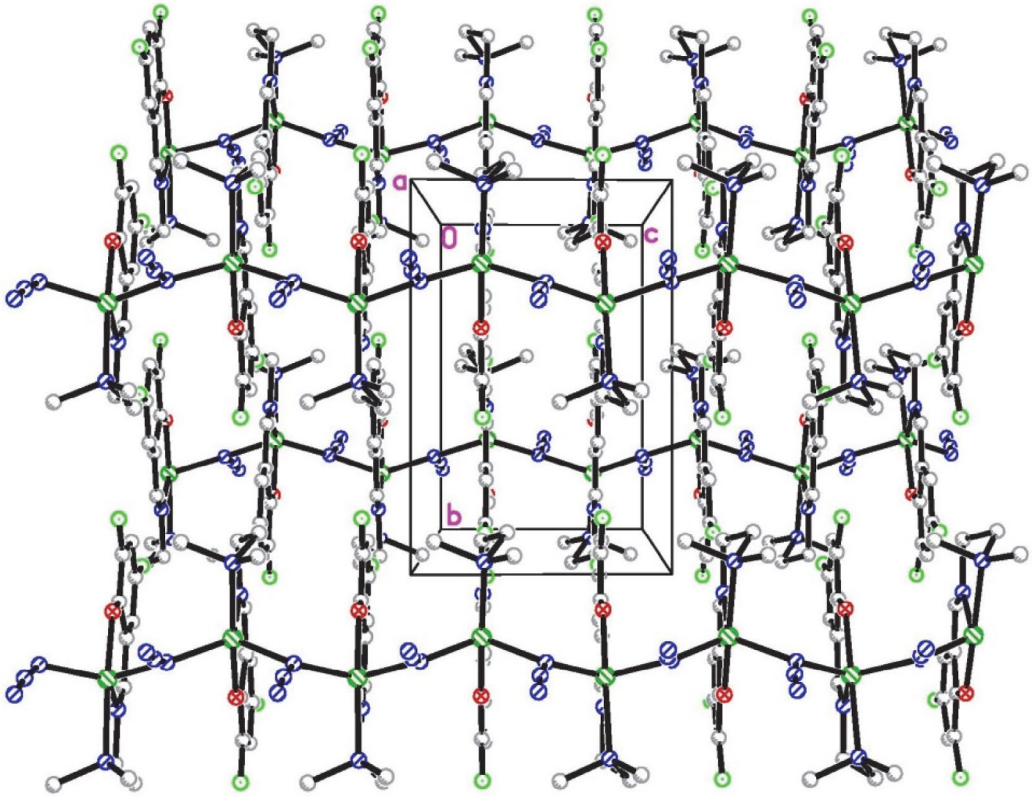

Figure 4. The end-on azido bridged polynuclear structure of complex **2**, viewed along the axis-*a* direction.

plexes.[9] As is apparent from Table 2, there are only slight deviation from an ideal trigonal bipyramidal geometry, with the angles at the basal plane within 12° of 120°. The three atoms in the basal plane, N1, N3 and N3B, are coplar, and with the Zn atom being 0.060(2) Å from the plane in the direction of O1. The [ZnL^2] moieties are linked through end-on azido bridges, to form chains running along the *c* axis (Figure 4).

The question arises as to whether the coordination polyhedron around the five-coordinated zinc atom can be described as a distorted square pyramid or a distorted trigonal bipyramid. Further information can be obtained by determining the structural index τ which represents the relative amount of trigonality (square pyramid, τ = 0; trigonal bipyramid, τ = 1); $\tau = (\beta - \alpha)/60^{o}$, α and β being the two largest angles around the central atom.[10] The values of τ is 0.70. The coordination geometry of the zinc atom in this complex is therefore approximately described as a trigonal bipyramid.

3. 4. Spectral Characterization

In the infrared spectra of the complexes, the bands corresponding to the azomethine (CH=N) groups are observed at 1638 cm^{-1} for **1** and 1642 cm^{-1} for **2**.[11] The typical absorptions for the azide ligand in 2 is located at 2052 cm^{-1}.[12] The appearance of the band at 1353 cm^{-1} indicates the asymmetric nature of the azide groups in complex **2**. The weak and broad band centered at 3512 cm^{-1} for **1** is ascribes to the O–H vibrations of the water ligands. The weak bands in the region of 420–570 cm^{-1} for the complexes can be assigned to ν(Zn–N) and ν(Zn–O). The electronic spectra of the complexes were recorded using the methanol as solvent. The absorptions appear in the range 270–380 nm are most likely due to the π→π* transitions and ligand-to-metal charge transfer.

3. 5. Antibacterial Activity

The complexes were screened for antibacterial activity against *B. subtilis* ATCC 6633, *E. coli* ATCC 35218, *P. putida* TS 1138 and *S. aureus* ATCC 25923 by the MTT method. The MIC values of the complexes against these bacteria are presented in Table 4. The antibiotic Penicillin was included as a reference. In general, the two zinc complexes have effective activities against the bacteria *B. subtilis* and *S. aureus*, medium activity against *E. coli*, and weak activity agaist *P. putida*. It is interesting that for *B. subtilis*, complex **1** has the most activity with MIC value of 0.39 μg mL^{-1}, and for *S. aureus*, complexes **1** and **2** have effective activity with MIC values of 0.39 and 0.78 μg mL^{-1}, respectively. So, the complexes showed a wide range of bactericidal activities against the bacteria, more potent than, or similar with, commercial antibiotic Penicillin.

Table 4. MIC (μg mL^{-1}) values of the antibacterial activity of the complexes

Compound	*B. subtilis*	*E. coli*	*P. putida*	*S. aureus*
1	0.39	12.5	25.0	0.39
2	1.56	25.0	50.0	0.78
Penicillin	0.78	>100	12.5	3.13

4. Conclusion

A new phenolate oxygen bridged dinuclear zinc(II) complex and a new end-on azido-bridged polynuclear zinc(II) complex have been prepared and structurally characterized in this article. The dianionic Schiff base ligand in the phenolate oxygen bridged complex coordinates to the Zn atom through the phenolate oxygen and imino nitrogen. The monoanionic Schiff base ligand in the end-on azido bridged complex coordinates to the Zn atom through the phenolate oxygen, imino nitrogen and amino nitrogen. During the self-assembly of the complexes, the bis-Schiff base zinc complex choose the neutral water molecule as co-ligand, while the mono-Schiff base zinc complex choose the azide as co-ligand. The complexes have strong antibacterial activity against *B. subtilis* and *S. aureus*.

5. Supplementary Data

Crystallographic data for the analysis have been deposited with the Cambridge Crystallographic Data Centre, CCDC Nos. 1848822 (**1**) and 1848823 (**2**). Copies of this information may be obtained free of charge from CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: +44 1223 336 033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

6. Acknowledgments

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7. References

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Povzetek

Sintetizirali smo dva cinkova(II) kompleksa, $[Zn_2(L^1)_2(OH_2)_2]$ (**1**) in $[ZnL^2(\mu_{1,1}\text{-}N_3)]_n$ (**2**), kjer je L^1 dianionska oblika *N,N'*-bis(3,5-difluoro-2-hidroksibenziliden)-1,3-diaminopropana, in L^2 monoanionska oblika 2-[(2-dimetilaminoetilimino)metil]-4,6-difluorofenola, ter ju okarakterizirali z elementno analizo, IR in UV-Vis spektroskopijo ter rentgensko difrakcijo. V kompleksu **1** je Zn atom oktaedrično koordiniran z donorskimi atomi liganda Schiffove baze L^1 in enim kisikovim atomom molekule vode. V kompleksu **2** je Zn atom trikotno-bipiramidalno koordiniran s tremi donorskimi atomi Schiffove baze L^2 in dveh atomov dušika z azidnih ionov. Kompleksa izkazujeta močno protibakterijsko aktivnost proti *B. subtilis* in *S. aureus*.