DOI: 10.17344/acsi.2022.7815 *Acta Chim. Slov.* **2023,** *70,* 148–154

Scientific paper

Trinuclear Zinc(II) Complexes Derived from *N,N'*-Bis-(5-bromosalicylidene)-1,2-cyclohexanediamine: Syntheses, Crystal Structures and Antimicrobial Activity

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Received: 09-18-2022

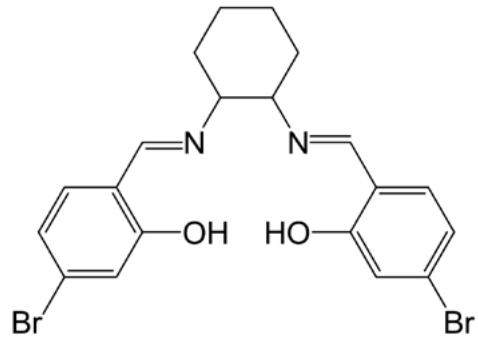
Abstract

Two new trinuclear zinc(II) complexes, $[Zn_3I_2L_2(H_2O)_2]$ (**1**) and $[Zn_3(CH_3OH)(DMF)L_2(NCS)_2]$ (**2**), where L is the dianionic form of *N,N'*-bis(5-bromosalicylidene)-1,2-cyclohexanediamine (H_2L), have been synthesized and characterized by elemental analyses, IR and UV spectra. Structures of the complexes were further confirmed by single crystal X-ray diffraction. Both complexes are trinuclear zinc compounds. Both compounds are solvated, with water ligand for **1** and methanol ligand for **2**. The outer two Zn atoms are in square pyramidal coordination, while the inner one is in octahedral coordination. The effect of the complexes on the antimicrobial activity against *Staphylococcus aureus*, *Escherichia coli* and *Candida albicans* were evaluated, and gave interesting results.

Keywords: Zinc complex, Trinuclear complex, Schiff base, Crystal structure, Antimicrobial activity

1. Introduction

Schiff bases have received increasing interest in the synthesis and structural studies because of their bioactivity and coordination properties.[1] Schiff bases have been reported to be active on fungal, cancer, convulsant, oxidant and diuretic activities.[2] The complexes of Schiff bases with various metal atoms have attracted remarkable interest due to their extensive biological activity like antibacterial, antifungal and antitumor.[3] Moreover, the metal complexes have usually better biological activities than the free Schiff bases. It has been reported that the Salen type Schiff base complexes have excellent antimicrobial activities.[4] Among the metal atoms, zinc is an important biological element, and its complexes with Schiff base ligands have interesting antimicrobial potential.[5] Recent research indicated that the halide and pseudohalide groups can severely increase the antimicrobial activities.[6] Our research group has reported some metal complexes with effective antimicrobial activities.[7] In pursuit of new and efficient antimicrobial agents, in the present work, two new trinuclear zinc(II) complexes, $[Zn_3I_2L_2(H_2O)_2]$ (**1**) and $[Zn_3(CH_3OH)(DMF)L_2(NCS)_2]$ (**2**), where L is the dianionic form of *N,N'*-bis(5-bromosalicylidene)-1,2-cyclohexanediamine (H_2L) (Scheme 1), are reported. To our knowledge, no complexes with L have been reported so far.



Scheme 1. The Schiff base H_2L

2. Experimental

2. 1. Material and Methods

4-Bromosalicylaldehyde and 1,2-diaminocyclohexane were purchased from Fluka. Other reagents and solvents were analytical grade and used without further puri-

fication. The Schiff base was prepared according to the literature method.[8] Elemental (C, H, and N) analyses were made on a PerkinElmer Model 240B automatic analyser. Zinc analysis was carried out by EDTA titration. Infrared (IR) spectra were recorded on an IR-408 Shimadzu 568 spectrophotometer. UV-Vis spectra were recorded on a Lambda 900 spectrometer. X-ray diffraction was carried out on a Bruker SMART 1000 CCD area diffractometer.

2. 2. Synthesis of $[Zn_3I_2L_2(H_2O)_2]$ (1)

H_2L (0.48 g, 1.0 mmol) was dissolved in 50 mL hot methanol, then, a methanolic solution (20 mL) of ZnI_2 (0.64 g, 2.0 mmol) was added dropwise at reflux. The mixture was stirred for 1 h to give a colorless solution. Colorless block-shaped single crystals suitable for X-ray diffraction were formed by slow evaporation of the solution in air for several days. Yield: 0.26 g (36%). Analysis Calcd. (%) for $C_{40}H_{40}Br_4I_2N_4O_6Zn_3$: C 33.31, H 2.80, N 3.88, Zn 13.60. Found (%): C 33.12, H 2.93, N 3.75, Zn 13.87. IR data (KBr, cm^{-1}): 1636 (CH=N). UV in methanol (λ, ε): 227 nm, 2.32×10^3 L mol^{-1} cm^{-1}; 245 nm, 2.41×10^3 L mol^{-1} cm^{-1}; 265 nm, 1.38×10^3 L mol^{-1} cm^{-1}; 350 nm, 5.25×10^2 L mol^{-1} cm^{-1}.

2. 3. Synthesis of $[Zn_3(CH_3OH)(DMF)L_2(NCS)_2]$ (2)

H_2L (0.48 g, 1.0 mmol) was dissolved in 50 mL hot methanol, then, a methanolic solution (20 mL) of $Zn(NO_3)_2{\cdot}6H_2O$ (0.60 g, 2.0 mmol) and ammonium thiocyanate (0.15 g, 2.0 mmol) was added dropwise at reflux. DMF (10 mL) was added to the mixture to dissolve the precipitate. The mixture was stirred for 1 h to give a colorless solution. Colorless block-shaped single crystals suitable for X-ray diffraction were formed by slow evaporation of the solution in air for several days. Yield: 0.31 g (45%). Analysis Calcd. (%) for $C_{46}H_{47}Br_4N_7O_6S_2Zn_3$: C 40.22, H 3.45, N 7.14, Zn 14.28. Found (%): C 40.51, H 3.35, N 7.23, Zn 14.50. IR data (KBr, cm^{-1}): 2080 (NCS), 1638 (CH=N). UV in methanol (λ, ε): 230 nm, 2.46×10^3 L mol^{-1} cm^{-1}; 245 nm, 2.53×10^3 L mol^{-1} cm^{-1}; 262 nm, 1.16×10^3 L mol^{-1} cm^{-1}; 353 nm, 4.61×10^2 L mol^{-1} cm^{-1}.

2. 4. X-Ray Diffraction

Data were collected from selected crystals mounted on glass fibers. The data were collected with MoK_α radia-

Table 1. Crystallographic data and experimental details for the complexes

	1	**2**
Molecular formula	$C_{40}H_{40}Br_4I_2N_4O_6Zn_3$	$C_{46}H_{47}Br_4N_7O_6S_2Zn_3$
Formula weight	1442.31	1373.77
Crystal size, mm	0.19×0.15×0.15	0.25×0.23×0.22
Radiation (λ, Å)	MoK_α (0.71073)	MoK_α (0.71073)
Crystal system	Monoclinic	Monoclinic
Space group	*C*2/*c*	$P2_1/c$
Unit cell dimensions:		
a, Å	25.7336(13)	16.7986(14)
b, Å	14.6897(12)	15.1552(13)
c, Å	15.8224(12)	20.5748(15)
α, °	90	90
β, °	112.643(1)	99.539(1)
γ, °	90	90
V, Å^3	5520.1(7)	5165.6(7)
Z	4	4
ρ_{calcd}, g cm^{-3}	1.735	1.766
F(000)	2768	2728
T_{min}, T_{max}	0.430, 0.501	0.392, 0.430
Absorption coefficient, mm^{-1}	5.348	4.610
θ Range for data collection, °	1.63–25.50	1.23–25.50
Reflections collected	13966	27194
Independent reflections (R_{int})	5023 (0.0656)	9620 (0.0636)
Reflections with $I > 2\sigma(I)$	2362	5155
Data/parameters	5023/273	9620/619
Restraints	3	43
Goodness-of-fit on F^2	0.943	1.020
Final *R* indices ($I > 2\sigma(I)$)	$R_1 = 0.0696$	$R_1 = 0.0626$
	$wR_2 = 0.1674$	$wR_2 = 0.1506$
R indices (all data)	$R_1 = 0.1542$	$R_1 = 0.1335$
	$wR_2 = 0.2153$	$wR_2 = 0.1892$
Largest diff. peak and hole, *e*/A^3	1.578, –1.043	1.107, –0.882

tion (0.71073 Å) at 298(2) K with a Bruker SMART 1000 CCD area diffractometer. The data for the complexes were processed with SAINT[9] and corrected for absorption using SADABS.[10] Multi-scan absorption corrections were applied with ψ-scans.[11] Structures of the two complexes were solved by direct method using SHELXS-97 and refined by full-matrix least-squares techniques on F^2 using anisotropic displacement parameters.[12] The water H atoms in complex **1** and the methanol H atom in complex **2** were located form difference Fourier maps and refined with O–H distances of 0.85(1) Å. All other hydrogen atoms were placed at the calculated positions. Idealized H atoms were refined with isotropic displacement parameters set to 1.2 (1.5 for methyl groups) times the equivalent isotropic *U* values of the parent carbon atoms. The crystallographic data for the complexes are listed Table 1.

Supplementary material has been deposited with the Cambridge Crystallographic Data Centre (nos. 2207640 (1), 2207645 (2)); deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk).

2. 5. Antimicrobial Assay

Qualitative determination of antimicrobial activity was done using the disk diffusion method.[13] The antibacterial activity was tested against *B. subtilis*, *E. coli*, *P. fluorescence* and *S. aureus* using MH medium (Mueller–Hinton medium). 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-diphenyltetrazolium 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 micro-titration 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 containing 2 mg of MTT per millilitre 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 hydrochloric acid was added to extract the dye. After 12 h of incubation at room temperature, the optical density (OD) was measured with a micro-plate reader at 550 nm.

3. Results and Discussion

3. 1. Chemistry

The Schiff base was facile prepared by the reaction of equimolar quantities of 4-bromosalicylaldehyde and 1,2-diaminocyclohexane in methanol. The two zinc complexes are stable at room temperature in the solid state and soluble in common organic solvents, such as methanol, ethanol, chloroform, and acetonitrile. The results of the elemental analyses are in accord with the composition suggested for the complexes.

3.2. IR and Electronic Spectra

The IR spectra of the complexes were analyzed and compared with those of their free Schiff base. The intense absorption band at 1645 cm^{-1} in the spectrum of the Schiff base can be assigned to the C=N stretching. In the zinc complexes, these bands are shifted to 1636 cm^{-1} for **1** and 1638 cm^{-1} for **2** upon complexation, which can be attributed to the coordination of the imine nitrogen to the metal centre.[14] The typical intense absorption at 2080 cm^{-1} in the spectrum of complex **2** is assigned to the vibration of the NCS ligand.[15]

UV–Vis spectra of the zinc complexes were recorded in methanol solution. The charge transfer bands at 220–270 nm can be assigned to $\pi\rightarrow\pi^*$ and $n\rightarrow\pi^*$ transitions of the Schiff base ligands. The bands at 350–355 nm can be assigned to the metal to ligand charge transfer (MLCT) transition.[16]

3. 4. Crystal Structure Description of Complex 1

Selected bond lengths and angles are listed in Table 2. The complex is a phenolato-bridged trinuclear zinc(II) compound (Figure 1), with Zn···Zn distance of 3.179(2) Å. The crystal of the complex possesses crystallographic two-fold rotation axis symmetry. The Zn1 atom is five-coordinated in a square pyramidal coordination, as evidenced by the structural index τ of 0.22.[17] The basal plane of the square pyramidal coordination is defined by two phenolate O (O1, O2) and two imine N (N1, N2) atoms of the Schiff base ligand, and the apical position is occupied by I1 atom. The Zn1 atom deviates from the least-squares plane defined by the four basal donor atoms by 0.671(2) Å. The Schiff base acts as a tetradentate ligand, and forms one five- and two six-membered chelate rings with the Zn1 atom. The Zn2 atom is six-coordinated in an octahedral coordination. The equatorial plane of the octahedral coordination is defined by three phenolate O (O1, O2, O1A) atoms from two Schiff base ligands, and one O (O3) atom of a water ligand, and the axial positions are occupied by one phenolate O (O2A) atom of a Schiff base ligand, and one O (O3A) atom of the other water ligand. The dihedral angle between the two benzene rings C1-C6 and C15-C20 is 10.3(5)°. The bond distances subtended at the Zn atoms are comparable to those observed in similar zinc(II) complexes with Schiff bases.[18] There present C–H···I hydrogen bonds (C6–H6 = 0.93 Å, H6···I1 = 3.129(3) Å, C6–H6···I1 = 165.0(3)°) in the crystal structure (Figure 2).

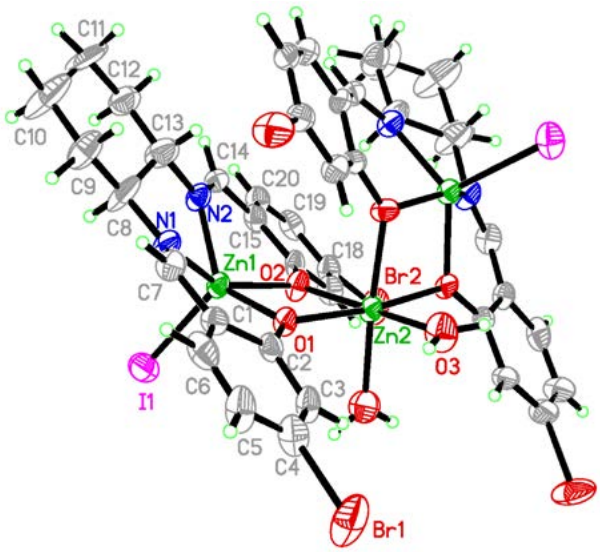


Figure 1. Molecular structure of complex **1**. Unlabeled atoms are at the symmetry position 1 – *x*, – *y*, – *z*.

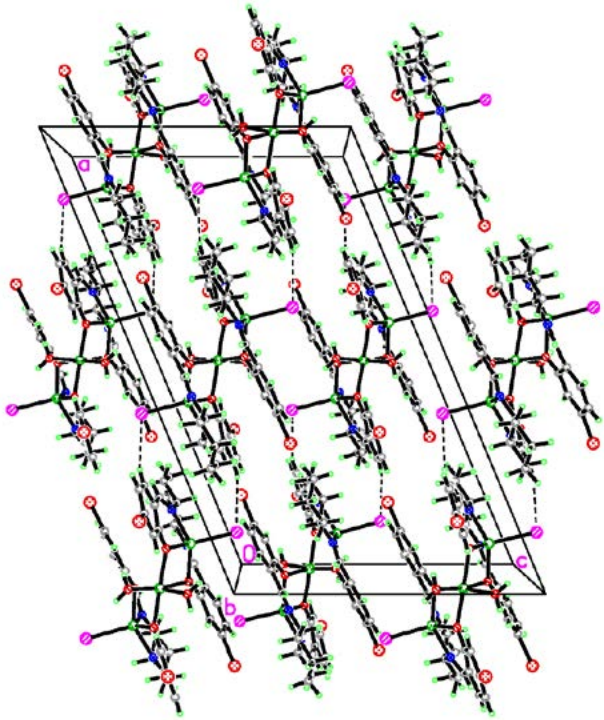


Figure 2. Molecular packing structure of complex **1**, viewed along the *b* axis. Hydrogen bonds are shown as dashed lines.

3. 5. Crystal Structure Description of Complex 2

Selected bond lengths and angles are listed in Table 2. The complex is a phenolato-bridged trinuclear zinc(II) compound (Figure 3), with Zn···Zn distances of 3.170(2) and 3.202(2) Å. The Zn1 and Zn3 atoms are five-coordinated in square pyramidal coordination, as evidenced by the structural index τ of 0.27 for Zn1 and 0.19 for Zn3.[17] The basal planes of the square pyramidal coordination are defined by two phenolate O (O1 and O2 for Zn1, O3 and O4 for Zn3) and two imine N (N1 and N2 for Zn1, N3 and N4 for Zn3) atoms of the Schiff base ligands, and the apical positions are occupied by N5 atom for Zn1 and N6 atom for Zn3. The Zn1 and Zn3 atoms deviate from the least-squares planes defined by the four basal donor atoms by 0.640(2) and 0.646(2) Å, respectively. The Schiff base acts as a tetradentate ligand, and forms one five- and two six-membered chelate rings with the Zn atoms. The

Table 2. Selected bond distances (Å) and angles (°) for the complexes

		1	
Zn1–O1	2.005(7)	Zn1–O2	2.017(6)
Zn1–N1	2.066(9)	Zn1–N2	2.027(9)
Zn1–I1	2.5854(15)	Zn2–O2	2.073(7)
Zn2–O1	2.086(5)	Zn2–O3	2.099(9)
O1–Zn1–O2	79.0(2)	O1–Zn1–N2	133.9(3)
O2–Zn1–N2	88.4(3)	O1–Zn1–N1	87.7(3)
O2–Zn1–N1	148.2(4)	N2–Zn1–N1	80.2(4)
O1–Zn1–I1	115.9(2)	O2–Zn1–I1	104.6(2)
N2–Zn1–I1	110.2(3)	N1–Zn1–I1	107.2(3)
O2–Zn2–O2A	88.0(4)	O2–Zn2–O1	75.9(2)
O2–Zn2–O1A	98.2(2)	O1–Zn2–O1A	172.0(4)
O2–Zn2–O3	172.5(3)	O2–Zn2–O3A	91.6(4)
O1–Zn2–O3	96.7(3)	O1–Zn2–O3A	89.0(3)
O3–Zn2–O3A	89.9(7)		
		2	
Zn1–O1	2.021(5)	Zn1–O2	2.018(5)
Zn1–N1	2.080(8)	Zn1–N2	2.035(9)
Zn1–N5	2.000(10)	Zn2–O1	2.092(5)
Zn2–O2	2.140(5)	Zn2–O4	2.080(5)
Zn2–O5	2.057(6)	Zn2–O6	2.133(6)
Zn2–O3	2.156(5)	Zn3–O3	2.010(5)
Zn3–O4	2.028(5)	Zn3–N6	1.992(9)
Zn3–N3	2.054(7)	Zn3–N4	2.043(7)
N5–Zn1–O2	110.9(3)	N5–Zn1–O1	105.7(3)
O2–Zn1–O1	81.7(2)	N5–Zn1–N2	102.9(4)
O2–Zn1–N2	89.9(3)	O1–Zn1–N2	151.3(3)
N5–Zn1–N1	113.8(3)	O2–Zn1–N1	135.3(3)
O1–Zn1–N1	87.6(3)	N2–Zn1–N1	79.2(3)
O5–Zn2–O4	96.6(2)	O5–Zn2–O1	171.9(2)
O4–Zn2–O1	90.0(2)	O5–Zn2–O6	86.0(2)
O4–Zn2–O6	167.2(2)	O1–Zn2–O6	88.6(2)
O5–Zn2–O2	97.1(2)	O4–Zn2–O2	99.1(2)
O1–Zn2–O2	77.2(2)	O6–Zn2–O2	93.0(2)
O5–Zn2–O3	87.3(2)	O4–Zn2–O3	75.6(2)
O1–Zn2–O3	98.9(2)	O6–Zn2–O3	92.0(2)
O2–Zn2–O3	173.6(2)	N6–Zn3–O3	110.0(3)
N6–Zn3–O4	104.8(3)	O3–Zn3–O4	80.1(2)
N6–Zn3–N4	113.0(3)	O3–Zn3–N4	137.1(3)
O4–Zn3–N4	89.0(2)	N6–Zn3–N3	106.4(3)
O3–Zn3–N3	88.4(3)	O4–Zn3–N3	148.8(3)
N4–Zn3–N3	79.9(3)		

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

Zn2 atom is six-coordinated in an octahedral coordination. The equatorial plane of the octahedral coordination is defined by three phenolate O (O1, O2, O3) from two Schiff base ligands, and one O (O5) atom of DMF ligand. The axial positions of the octahedral coordination are are occupied by one phenolate O (O4) of a Schiff base ligand, and one O (O6) atom of the methanol ligand. There forms a four-membered chelate ring Zn1-O1-Zn2-O2 with the phenolate bridging groups. The dihedral angle between the two benzene rings C1-C6 and C15-C20 is 11.5(4)°, and that between the benzene rings C21-C26 and C35-C40 is 7.5(4)°. The bond distances subtended at the Zn atoms are comparable to those observed in similar zinc(II) complexes with Schiff bases.[18] There present weak Br···S (3.649(3) Å) interactions in the crystal structure (Figure 4).

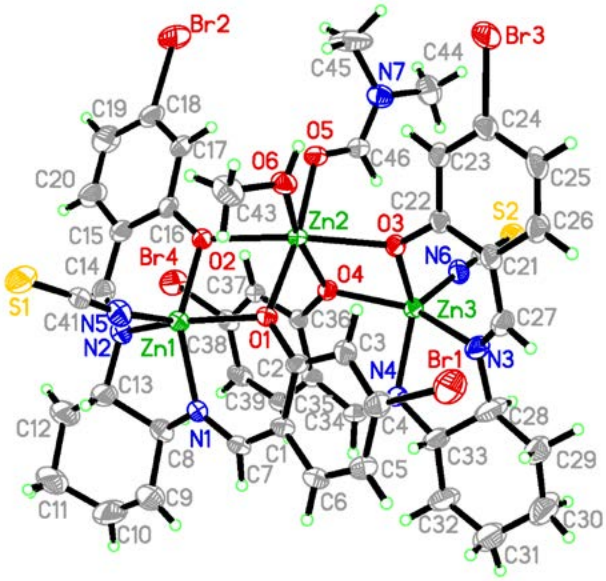


Figure 3. Molecular structure of complex **2**.

3. 5. Thermal Analysis

Thermal analysis of complex **1** is shown in Figure 5, which revealed that the complex is stable up to 286 °C. The complex decomposed from 286 to 618 °C, and conversed into ZnO. The agreement between calculated (83.07%) and experimental (83.55%) mass losses is within experimental errors.

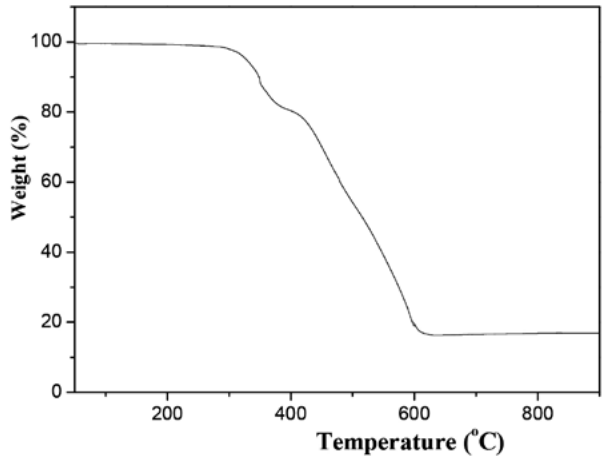


Figure 5. Thermal analysis of complex **1**.

3. 6. Antimicrobial Activity

The results of the antimicrobial activity are summarized in Table 4. A comparative study of minimum inhibitory concentration (MIC) values of the Schiff base and the zinc complexes indicated that the complexes have more effective activity against *Staphylococcus aureus*, *Escherichia coli* and *Candida albicans* than the free Schiff base H_2L. Generally, this is caused by the greater lipophilic nature of the complex than the ligand. Such increased activity of

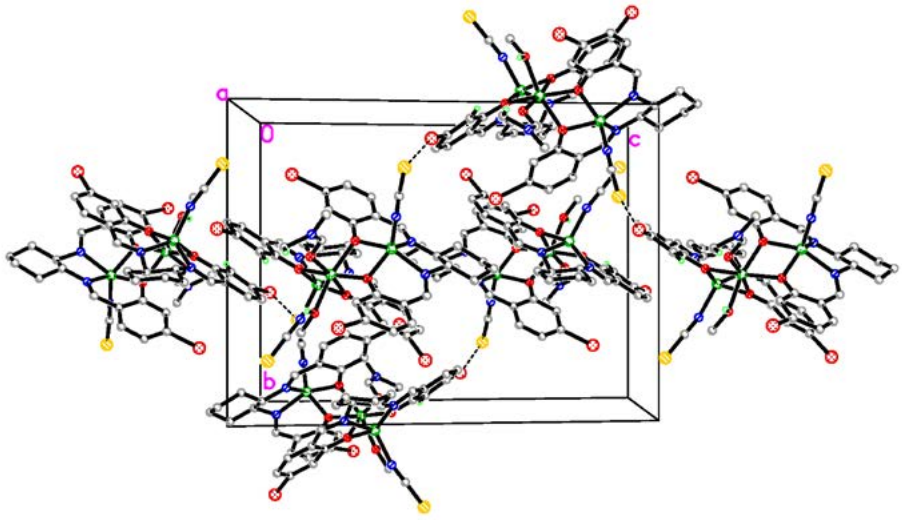


Figure 4. Molecular packing structure of complex **2**, viewed along the *a* axis. The weak Br···S interactions are shown as dashed lines.

the metal chelates can be explained on the basis of chelating theory.[21] On chelating, the polarity of the metal atoms will be reduced to a greater extent due to the overlap of the ligand orbital and partial sharing of positive charge of the metal atoms with donor atoms. Further, it increases the delocalization of π-electrons over the whole chelate ring and enhances the lipophilicity of the complex. This increased lipophilicity enhances the penetration of the complexes into lipid membrane and blocks the metal binding sites on enzymes of micro-organisms.

The two zinc complexes have strong activities against *Staphylococcus aureus*, medium activity against *Escherichia coli*, and weak activity against *Candida albicans*. For *Staphylococcus aureus* and *Escherichia coli*, the activities of both complexes are less than the control drug Tetracycline. While for *Candida albicans*, the complexes have stronger activities than Tetracycline. Complex **1** has the most activity against *Staphylococcus aureus* with MIC value of 1.0 $\mu g \cdot mL^{-1}$. Both complexes have higher activities against *Staphylococcus aureus* and lower activities against *Escherichia coli* and *Candida albicans* than the zinc(II) and manganese(II) complexes with the ligand *N*'-(1-(pyridin-2-yl)ethylidene)isonicotinohydrazide.[22] Further work needs to be carried out to investigate the structure-activity relationship.

Table 4. MIC values ($\mu g \cdot mL^{-1}$) for the antimicrobial activities of the tested compounds

Compound	*Staphylococcus aureus*	*Escherichia coli*	*Candida albicans*
H_2L	64	128	> 1024
1	1.0	8.0	64
2	2.0	8.0	64
Tetracycline	0.25	2.0	> 1024

4. Conclusion

In summary, two new trinuclear zinc(II) complexes with the tetradentate Schiff base ligand *N*,*N*'-bis(5-bromosalicylidene)-1,2-cyclohexanediamine have been prepared and characterized. The structures of both complexes are confirmed by single crystal X-ray crystallographic determination. The Zn atoms in the complexes are in square pyramidal and octahedral coordination. The complexes have effective activities on the bacteria *Staphylococcus aureus* and *Escherichia coli*. Moreover, both complexes have stronger activities against *Candida albicans* than Tetracycline. The iodide coordinated complex **1** has the most activity against *Staphylococcus aureus* with MIC value of 1.0 $\mu g \cdot mL^{-1}$.

Acknowledgments

This research was supported by the National Sciences Foundation of China (nos. 20676057 and 20877036) and Top-class foundation of Pingdingshan University (no. 2008010).

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Povzetek

Sintetizirali smo dva nova trijedrna kompleksa cinka(II), $[Zn_3I_2L_2(H_2O)_2]$ (**1**) in $[Zn_3(CH_3OH)(DMF)L_2(NCS)_2]$ (**2**), pri čemer je L dianionska oblika *N,N'*-bis(5-bromosaliciliden)-1,2-cikloheksandiamina (H_2L). Spojini smo karakterizirali z elementno analizo, IR in UV spektroskopijo, strukturi obeh spojin pa smo določili z monokristalno rentgensko difrakcijo. Obe spojini sta trijedrni in solvatirani, z vodnimi ligandi v primeru spojine **1** in metanolom v primeru spojine **2**. Zunanja atoma Zn sta v kvadratno piramidalni koordinaciji, notranji pa v oktaedrični koordinaciji. Ocenili smo protimikrobno učinkovitost obeh kompleksov na bakterije *Staphylococcus aureus*, *Escherichia coli* in *Candida albicans*, pri čemer smo dobili zanimive rezultate.