Scientific paper

Syntheses, Crystal Structures and Antimicrobial Activity of Copper(II) Complexes with the Ligand *N,N'*-Bis(4-bromosalicylidene)propane-1,2-diamine

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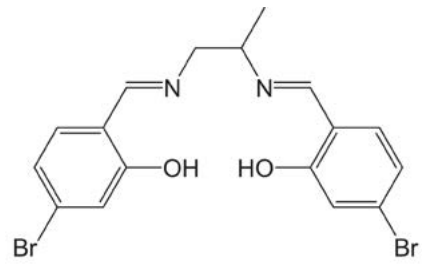
Abstract

Two new copper(II) complexes, [CuL] (**1**) and $[Cu_4Cl_2L_2(N_3)_2]\cdot CH_3OH$ (**2**), derived from the bis-Schiff base *N,N'*-bis(4-bromosalicylidene)propane-1,2-diamine (H_2L) have been prepared and characterized by spectroscopy methods, as well as single crystal X-ray determination. The Cu atom in the mononuclear complex **1** is in square planar coordination. The outer and inner Cu atoms in the phenolate and azide co-bridged tetranuclear complex **2** are in square planar and square pyramidal coordination, respectively. The antibacterial activities of the Schiff base and the two copper complexes have been assayed on the bacteria *Staphylococcus aureus* and *Escherichia coli*, and the yeast *Candida parapsilosis*.

Keywords: Schiff base; Copper complex; Crystal structure; Antibacterial

1. Introduction

Schiff bases due to their easy preparation and good metal-binding ability have been widely used as preferred ligands in the construction of complexes with various metal salts.[1] The compounds with N, O, S donor atoms have structure similarities with some natural biological enzymes. Schiff bases and their metal complexes have a broad range of applications in pharmaceutical and biological fields.[2] In the last few years, Schiff bases have been widely known for their remarkable biological activities, such as antifungal, antibacterial, anti-proliferative, antimalarial, antiviral, antipyretic, and anti-inflammatory activities.[3] Copper complexes with Schiff bases have been extensive studied and are considered as excellent alternatives for classic organic antibacterial agents.[4] Despite the presence of considerable research on the antibacterial activities of Schiff base complexes, it is still necessary to search for new samples to find more effective agents as well as to better understand the biological mechanisms of this type of compounds. With an interest in the chemistry of biologically active compounds, this study aimed to synthesize Schiff base and its copper(II) complexes. The newly synthesized complexes, [CuL] (**1**) and $[Cu_4Cl_2L_2(N_3)_2]\cdot CH_3OH$ (**2**), where L is the deprotonated form of *N,N'*-bis(4-bromosalicylidene)propane-1,2-diamine (H_2L; Scheme 1), are presented and examined for their antimicrobial activities.



Scheme 1. The Schiff base H_2L

2. Experimental

2. 1. Materials and Methods

4-Bromosalicylaldehyde and 1,2-diaminopropane were purchased from TCI Chemical Reagent Co. Ltd. $CuCl_2\cdot 2H_2O$ and NaN_3 were purchased from Aladdin Chemical Reagent Co. Ltd. The methanol was purchased from Kemiou Chemical Reagent Co. Ltd. IR spectra were recorded on a Jasco FT/IR-4000 spectrometer as KBr pellets in the 4000–400 cm^{-1} region. Elemental analyses were performed on a Perkin-Elmer 240C elemental analyzer. UV-Vis spectra were recorded on a Lambda 900 spectrometer. Single crystal X-ray diffraction was carried out on a Bruker SMART 1000 CCD diffractometer.

Caution! The azide complexes are potentially explosive. Although no problem was encountered in the present study, only small amounts of the materials should be prepared and they must be handled with care.

2. 2. Syntheis of H_2L

4-Bromosalicylaldehyde (0.84 g, 4.0 mmol) was dissolved by methanol (20 mL). Then, it was added dropwise to the methanol solution (30 mL) of 1,2-diaminopropane (0.15 g, 2.0 mmol). The reaction mixture was stirred and heated to reflux for 30 min. The solvent was removed by evaporation under reduced pressure. The yellow solid was recrystallized from ethanol to give yellowish crystalline product. Yield: 0.77 g (87%). IR data (KBr, cm^{-1}): 3434, 1623, 1489, 1374, 1243, 1200, 1133, 1040, 985, 905, 853, 798, 587. UV-Vis λ_{max}/nm (1.2×10^{-5} mol L^{-1}, MeOH; ε, L mol^{-1} cm^{-1}): 225 (25,600), 262 (19,750), 315 (7,150), 400 (2,900). Anal. Calcd. (%) for $C_{17}H_{16}Br_2N_2O_2$: C, 46.39; H, 3.66; N, 6.36. Found (%): C, 46.25; H, 3.73; N, 6.45.

2. 3. Synthesis of [CuL] (1)

H_2L (44 mg, 0.10 mmol) and $CuCl_2{\cdot}2H_2O$ (34 mg, 0.20 mmol) were mixed in methanol (30 mL). The mixture was stirred at room temperature for 30 min to give a brown solution. Single crystals of the complex, suitable for X-ray diffraction, were obtained after 5 days. Yield: 27 mg (54%). IR data (cm^{-1}): 1610, 1512, 1407, 1285, 1190, 1132, 1053, 972, 913, 855, 772, 523, 471. UV-Vis (1.3×10^{-5} mol L^{-1}, MeOH; ε, L mol^{-1} cm^{-1}): 232 (21,780), 250 (20,910), 275 (14,810), 357 (6,100). Anal. Calcd. (%) for $C_{17}H_{14}Br_2CuN_2O_2$: C, 40.70; H, 2.81; N, 5.58. Found (%): C, 40.55; H, 2.87; N, 5.66.

2. 4. Synthesis of $[Cu_4Cl_2L_2(N_3)_2]{\cdot}CH_3OH$ (2)

H_2L (44 mg, 0.10 mmol), $CuCl_2{\cdot}2H_2O$ (34 mg, 0.20 mmol) and NaN_3 (13 mg, 0.20 mmol) were mixed in methanol (30 mL). The mixture was stirred at room temperature for 30 min to give a deep brown solution. Single crystals of the complex, suitable for X-ray diffraction, were obtained after 5 days. Yield: 32 mg (48%). IR data (cm^{-1}): 3421, 2072, 1635, 1585, 1534, 1517, 1470, 1415, 1383, 1282, 1265, 1203, 1133, 1067, 1009, 913, 838, 791, 620, 590, 532, 458. UV-Vis (1.3×10^{-5} mol L^{-1}, MeOH; ε, L mol^{-1} cm^{-1}): 230 (20,540), 250 (18,500), 276 (12,610), 355 (5,050). Anal. Calcd. (%) for $C_{35}H_{33}Br_4Cl_2Cu_4N_{10}O_5$: C, 31.88; H, 2.52; N, 10.62. Found (%): C, 31.72; H, 2.43; N, 10.75.

2. 5. X-ray Crystallography

Single crystal X-ray data for the complexes were collected on a Bruker SMART 1000 CCD diffractometer using the SMART/SAINT software.[5] Intensity data were collected using graphite-monochromatized MoK_α radia-

Table 1. Crystallographic data and refinement parameters for the complexes

	1	**2**
Chemical Formula	$C_{17}H_{14}Br_2CuN_2O_2$	$C_{35}H_{33}Br_4Cl_2Cu_4N_{10}O_5$
Fw	501.66	1318.41
T (K)	298(2)	298(2)
Crystal system	Monoclinic	Orthorhombic
Space group	$P2_1/c$	$Pbca$
a (Å)	11.6858(18)	12.3951(11)
b (Å)	11.4195(17)	16.5472(13)
c (Å)	13.0760(18)	22.3770(15)
α (°)	90	90
β (°)	100.562(2)	90
γ (°)	90	90
V (Å^3)	1715.4(4)	4589.6(6)
Z	4	4
m (Mo Kα) (cm^{-1})	5.944	5.478
D_c (g cm^{-3})	1.942	1.908
Reflections collected	8892	23307
Unique reflections	3181	4256
Observed reflections [I ≥ 2σ(I)]	1964	2466
Parameters	218	283
Restraints	0	0
Goodness of fit on F^2	1.047	1.090
R_{int}	0.0458	0.0782
R_1, wR_2 [$I \geq 2\sigma(I)$]	0.0519, 0.1221	0.0698, 0.1757
R_1, wR_2 (all data)	0.0953, 0.1396	0.1270, 0.2058

tion (0.71073 Å) at 298(2) K. The structures were solved by direct methods using SHELX.[6] Multi-scan absorption corrections were applied with SADABS.[7] All non-hydrogen atoms were refined with anisotropic displacement coefficients. The hydrogen atoms bonded to carbon were included in geometric positions and given thermal parameters equivalent to 1.2 and 1.5 times those of the atom to which they were attached. The anisotropic displacement parameters for atoms C8, C9 and C10 in complex **2** are a little larger than usual, which is caused by the slight disorder of the group. Crystallographic data and refinement parameters are given in Table 1, and important interatomic distances and angles are given in Table 2.

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

	1		
C7–N1	1.289(10)	C11–N2	1.279(10)
C8–N1	1.461(10)	C9–N2	1.477(10)
Cu1–O1	1.894(4)	Cu1–O2	1.887(5)
Cu1–N1	1.924(6)	Cu1–N2	1.933(6)
O2–Cu1–O1	88.82(18)	O2–Cu1–N1	174.4(2)
O1–Cu1–N1	93.4(2)	O2–Cu1–N2	93.8(2)
O1–Cu1–N2	176.0(2)	N1–Cu1–N2	84.2(3)
	2		
C7–N1	1.221(14)	C11–N2	1.287(15)
C8–N1	1.531(15)	C9–N2	1.487(16)
Cu1–O1	1.946(6)	Cu1–O2	1.893(6)
Cu1–N1	1.935(9)	Cu1–N2	1.911(10)
Cu2–O1	1.982(5)	Cu2–O2	2.375(6)
Cu2–N3	1.962(7)	Cu2–N3A	2.013(8)
Cu2–Cl1	2.240(3)		
O2–Cu1–N2	95.3(4)	O2–Cu1–N1	175.6(4)
N2–Cu1–N1	85.4(4)	O2–Cu1–O1	85.9(2)
N2–Cu1–O1	172.7(4)	N1–Cu1–O1	92.8(3)
N3–Cu2–O1	165.9(3)	N3–Cu2–N3A	76.5(3)
O1–Cu2–N3A	91.0(3)	N3–Cu2–Cl1	98.1(3)
O1–Cu2–Cl1	95.9(2)	N3A–Cu2–Cl1	155.8(2)
N3–Cu2–O2	104.7(3)	O1–Cu2–O2	73.2(2)
N3A–Cu2–O2	109.7(3)	Cl1–Cu2–O2	94.55(19)

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

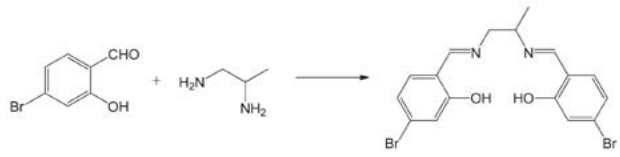
2. 6. Biological Assay

The antibacterial property of the compounds was evaluated by a macro-dilution method using *Staphylococcus aureus*, *Escherichia coli*, and the yeast *Candida parapsilosis*. The cultures of bacteria and yeasts were incubated under vigorous shaking. The compounds were dissolved in small amount of DMSO. Concentration of the tested compounds ranging from 0.010 to 2.5 mmol L^{-1} for the bacteria and yeasts was used in all experiments. The antibacterial activity was characterized by IC_{50} and MIC values. MIC experiments on subculture dishes were used to assess the minimal microbicidal concentration (MMC). Subcultures were prepared separately in Petri dishes containing competent agar medium and incubated at 30 °C for 48 h. The MMC value was taken as the lowest concentration, which showed no visible growth of microbial colonies in the subculture dishes.

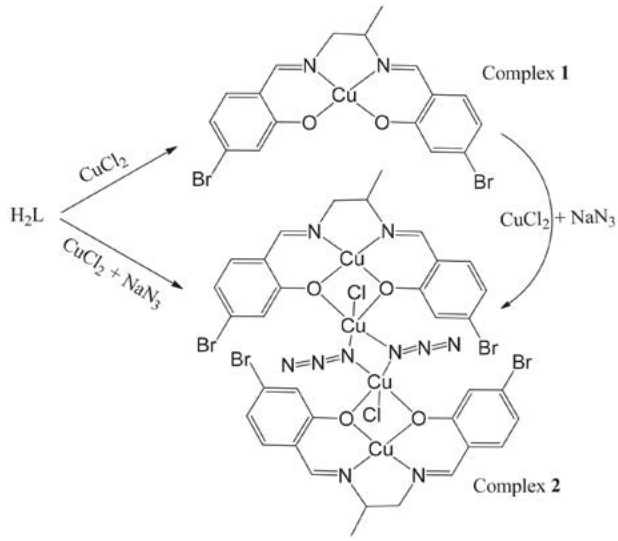
3. Results and Discussion

3. 1. Chemistry

The Schiff base *N,N'*-bis(4-bromosalicylidene)propane-1,2-diamine was prepared by the reaction of 2:1 molar ratio of 4-bromosalicylaldehyde and 1,2-diaminopropane in methanol (Scheme 2). Complex **1** was facile synthesized by the reaction of the Schiff base H_2L with copper chloride in methanol (Scheme 3). Complex **2** was obtained by the reaction of the Schiff base H_2L with copper chloride and sodium azide in methanol (Scheme 3). Single crystals of the complexes were formed by slow evaporation of the solvent at room temperature.



Scheme 2. The synthetic procedure of the Schiff base H_2L



Scheme 3. The synthetic procedure of the complexes

3. 2. Crystal Structure Description of Complex 1

The molecular structure of complex **1** is shown in Fig. 1. The complex is a mononuclear copper(II) species. The Cu atom is coordinated by two imine nitrogen and two phenolate oxygen of the Schiff base ligand, forming

a square planar geometry. The *trans* and *cis* bond angles are 174.4(2)–176.0(2)° and 84.2(3)–93.8(2)°, respectively. Thus, the square planar coordination is slightly distorted, which is mainly caused by the five-membered chelate ring Cu1–N1–C8–C9–N2. The Cu–O and Cu–N bonds are 1.887(5)–1.894(4) Å and 1.924(6)–1.933(6) Å, respectively, which are comparable to those observed in Schiff base copper(II) complexes.[8] The two benzene rings form a dihedral angle of 5.2(5)°.

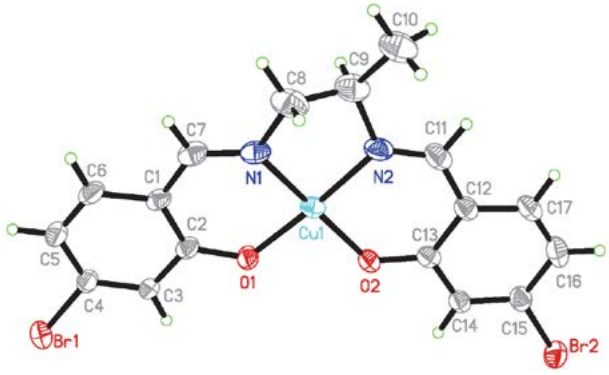


Fig. 1. A perspective view of the molecular structure of complex **1** with the atom labeling scheme. Thermal ellipsoids are drawn at the 30% probability level.

3. 3. Crystal Structure Description of Complex 2

The molecular structure of complex **2** is shown in Fig. 2. The complex is a phenolate and end-on azide co-bridged tetranuclear copper(II) species, with the two [Cu_2ClL] units connected by two bridging azide ligands. Molecule of the complex possesses a crystallographic inversion center symmetry. The phenolate bridged Cu···Cu distance is 3.148(2) Å, and the azide bridged Cu···Cu distance is 3.122(2) Å. The outer Cu atom (Cu1) is coordinated by two imine nitrogen and two phenolate oxygen of the Schiff base ligand, forming a square planar geometry. The *trans* and *cis* bond angles are 172.7(4)–175.6(4)° and 85.4(4)–95.3(4)°, respectively. Thus, the square planar coordination is slightly distorted, which is mainly caused by the strain created by the five-membered chelate ring Cu1–N1–C8–C9–N2. The Cu–O and Cu–N bonds are 1.893(6)–1.946(6) Å and 1.911(10)–1.935(9) Å, respectively, which are comparable to those observed in complex **1** and other Schiff base copper(II) complexes.[9] The inner Cu atom (Cu2) is in a distorted square pyramidal coordination geometry, with the phenolate oxygen (O1), two azide nitrogen (N3 and N3A, symmetry code for A: $-x$, 1 $-y$, $-z$), and one Cl ligand, define the basal plane, and with the other phenolate oxygen (O2) occupies the apical position. The Cu2 atom deviates from the best coordination plane defined by the four basal donor atoms by 0.242(3) Å in direction of the apical donor atom. The *cis* and *trans* bond angles in the basal plane are in the ranges of 76.5(3)–98.1(3)° and 155.8(2)–165.9(3)°, and those among the apical and basal donor atoms are in the range of 73.2(2)–109.7(3)°. Thus, the coordination is distorted from the ideal geometry of a square pyramid. The distortion is mainly caused by the strain created by the four-membered chelate rings Cu1–O1–O2–Cu2 and Cu2–N3–Cu2A–N3A. The bond lengths related to Cu2 atom are 1.982(5)–2.375(6) Å for Cu–O, 1.962(7)–2.013(8) Å for Cu–N and 2.240(3) Å for Cu–Cl, which are comparable to those observed in Schiff base copper(II) complexes with bridging azide ligands.[10] The two benzene rings of the Schiff base ligand form a dihedral angle of 10.4(5)°. The dihedral angle between the two planes formed by the four-membered chelate rings is 69.7(3)°. In the crystal structure, the molecules are linked through intermolecular hydrogen bonds of O–H···Br and intramolecular hydrogen bonds of O–H···Cl, to form a three-dimensional network (Fig. 3).

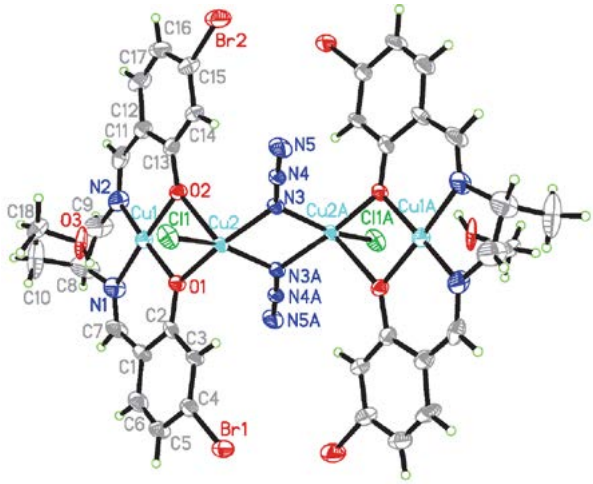


Fig. 2. A perspective view of the molecular structure of complex **2** with the atom labeling scheme. Thermal ellipsoids are drawn at the 30% probability level. Atoms labelled with the suffix A or unlabelled are at the symmetry position $-x$, 1 $-y$, $-z$.

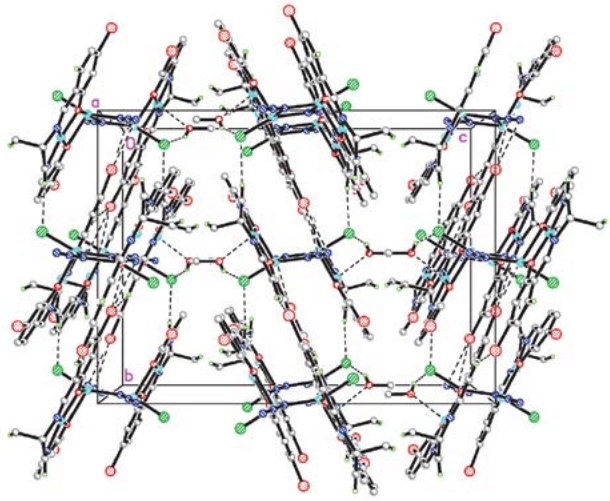


Fig. 3. Molecular packing diagram of complex **2**, viewed along the *a* axis. Hydrogen bonds are shown as dashed lines.

Table 3. Geometrical parameters for hydrogen bonds for complex **2**

D–H···A	Distance, Å		Angle, °	
	D–H	H···A	D···A	D–H···A
O3–H3···Cl1	0.82	2.72	3.208(4)	120(4)
C6–H6···Br1^{i}	0.93	2.87	3.707(4)	150(5)
C10–H10C···N2	0.96	2.61	3.001(4)	105(4)

Symmetry transformation used to generate the symmetry related atoms: ^{i} 1/2 + *x*, 3/2 – *y*, –*z*.

3. 4. IR and UV-Vis Spectra

The weak and broad absorptions centered at 3434 and 3421 cm^{-1} in the spectra of H_2L and complex **2** substantiate the presence of O–H groups. The strong absorption band at 1623 cm^{-1} for H_2L is assigned to the azomethine groups, ν(C=N),[10] which is shifted to lower wave number 1610 cm^{-1} for complex **1** and higher wave number 1635 cm^{-1} for complex **2**. This is in accordance with the variation of the bond lengths of C=N in the compounds. The intense band at 2072 cm^{-1} for complex **2** can be assigned to the stretching vibration of azide ligands.[11] The weak bands in the low wave numbers 450–540 cm^{-1} are due to the vibration of the Cu–O and Cu–N bonds.[12]

In the electronic spectra of H_2L and the complexes, the intense bands observed at about 230–280 nm for the compounds are assigned to intra-ligand π–π^* transitions. The copper complexes displayed bands centered at about 275 nm, which can be assigned to the n–π^* transition.[13] The charge transfer LMCT bands are located at about 355 nm.[14]

3. 5. Antibacterial Activity

The antimicrobial results are summarized in Table 4. The free Schiff base H_2L showed medium activity against *E. coli*, while no activity on *S. aureus* and *C. parapsilosis*. Obviously, the two copper complexes have higher activities than H_2L. The mononuclear copper complex (**1**) showed strong activity against *S. aureus* and *E. coli*, and weak activity against *C. parapsilosis*. The tetranuclear copper complex (**2**) showed strong activity against *S. aureus*, medium activity against *E. coli* and weak activity against *C. parapsilosis*. Interestingly, complex **2** has the most activity against *S. aureus*, with IC_{50} and MIC values of 0.17 and 0.13 mmol L^{-1}, which deserves further study. As a comparison, the copper complexes have similar antibacterial activities against *S. aureus* and *E. coli* to the Schiff base manganese(III) complex[15] and the Schiff base copper(II) complexes.[16]

Table 4. Antibacterial activity of H_2L and the copper complexes

Compound	*S. aureus*		*E. coli*		*C. parapsilosis*	
	IC_{50}*	MIC*	IC_{50}	MIC	IC_{50}	MIC
H_2L	>2.50	>2.50	1.45	2.50	>2.50	>2.50
1	0.38	0.23	0.75	0.42	2.03	1.70
2	0.17	0.13	1.16	0.95	2.27	1.89

* mmol L^{-1}

4. Conclusion

In summary, *N,N'*-bis(4-bromosalicylidene)propane-1,2-diamine was prepared. With the bis-Schiff base, a mononuclear copper(II) complex and a phenolate and azide co-bridged tetranuclear copper(II) complex were obtained. The Schiff base and the two complexes were characterized by physico-chemical methods. Structures of the complexes were confirmed by single crystal X-ray determination. The Schiff base ligand coordinates to the metal atoms through the phenolate oxygen and imine nitrogen. The complexes have effective antibacterial activities on the bacteria *Staphylococcus aureus* and *Escherichia coli*, and the yeast *Candida parapsilosis*.

Supplementary Material

CCDC 2244579 (**1**) and 2244580 (**2**) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge at http://www.ccdc.cam.ac.uk/const/retrieving.html or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44(0)1223-336033 or email: deposit@ccdc.cam.ac.uk.

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Povzetek

Sintetizirali smo dva nova kompleksa bakra(II), [CuL] (**1**) in $[Cu_4Cl_2L_2(N_3)_2]\cdot CH_3OH$ (**2**), z ligandom *N,N'*-bis(4-bromosaliciliden)propan-1,2-diamin (H_2L). Spojini smo karakterizirali s spektroskopskimi metodami in monokristalno rentgensko difrakcijo. Bakrov atom v enojedrnem kompleksu **1** je kvadratno planarno koordiniran. Zunanji in notranji bakrovi atomi v štirijedrnem kompleksu **2**, ki jih povezujejo mostovni fenolatni in azidni ligandi, so kvadratno planarno in kvadratno piramidalno koordinirani. Protibakterijsko učinkovitost liganda in obeh kompleksov smo preizkusili na bakterijah *Staphylococcus aureus* in *Escherichia coli* ter na glivi *Candida parapsilosis*.