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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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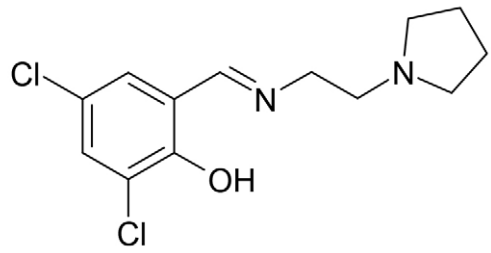
Abstract

Three new copper(II) complexes, [CuClL] (**1**), $[CuBrL]_n$ (**2**) and $[CuL(NCS)]_n$ (**3**), derived from the Schiff base 2,4-dichloro-6-((2-pyrrolidin-1-ylethylimino)methyl)phenol (HL) have been prepared and characterized by spectroscopic methods, as well as single crystal X-ray determination. The Cu atom in complex **1** is in square planar coordination, and those in complexes **2** and **3** are in square pyramidal coordination. The Schiff base ligand coordinates to the Cu atoms through phenolate oxygen, imino nitrogen and pyrrolidine nitrogen. The antibacterial activities of the Schiff base and the three 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 activity

1. Introduction

Schiff base compounds have been widely used as preferred ligands in the construction of metal complexes with versatile structures due to their easy preparation and good metal-binding ability.[1] The compounds with N, O and S 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] They have shown remarkable antifungal, antibacterial, anti-proliferative, antimalarial, antiviral, antipyretic, and anti-inflammatory activities.[3] Copper complexes with Schiff base ligands have been extensively 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 new Schiff base copper(II) complexes. The newly synthesized complexes, [CuClL] (**1**), $[CuBrL]_n$ (**2**) and $[CuL(NCS)]_n$ (**3**), where L is the deprotonated form of 2,4-dichloro-6-((2-pyrrolidin-1-ylethylimino)methyl)phenol (HL; Scheme 1), are presented and examined for their antimicrobial activities.



Scheme 1. The Schiff base HL

2. Experimental

2. 1. Materials and Methods

3,5-Dichlorosalicylaldehyde and *N*-(2-aminoethyl) pyrrolidine were purchased from TCI Chemical Reagent Co. Ltd. $CuCl_2{\cdot}2H_2O$, $CuBr_2$, $Cu(CH_3COO)_2$ and NH_4NCS were purchased from Aladdin Chemical Reagent Co. Ltd. The solvent methanol was purchased from Kemiou Chemical Reagent Co. Ltd. The Schiff base HL was prepared by

reaction of 1:1 molar ratio of 3,5-dichlorosalicylaldehyde with *N*-(2-aminoethyl)pyrrolidine in methanol, which was used to prepare the copper complexes without isolation. 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.

2. 2. Synthesis of [CuClL] (1)

HL (29 mg, 0.10 mmol) and $CuCl_2{\cdot}2H_2O$ (17 mg, 0.10 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 a week. Yield: 27 mg (71%). IR data (cm^{-1}): 1638, 1550, 1462, 1396, 1312, 1263, 1187, 1122, 1054, 1038, 985, 963, 870, 823, 751, 580, 530, 513, 455. UV-Vis (MeOH; ε, L mol^{-1} cm^{-1}): 220 (20,330), 240 (18,720), 270 (13,730), 305 (9,890), 380 (3,210). Anal. Calcd. (%) for $C_{13}H_{15}Cl_3CuN_2O$: C, 40.54; H, 3.93; N, 7.27. Found (%): C, 40.38; H, 4.01; N, 7.36. Λ_M (1×10^{-3} mol L^{-1} in methanol): 31 Ω^{-1} cm^2 mol^{-1}.

2. 3. Synthesis of $[CuBrL]_n$ (2)

HL (29 mg, 0.10 mmol) and $CuBr_2$ (23 mg, 0.10 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 a week. Yield: 33 mg (77%). IR data (cm^{-1}): 1638, 1550, 1461, 1420, 1360, 1313, 1305, 1260, 1187, 1130, 1072, 1045, 980, 867, 831, 750, 715, 580, 535, 517, 496, 445. UV-Vis (MeOH; ε, L mol^{-1} cm^{-1}): 220 (21,150), 240 (17,035), 272 (14,020), 312 (10,720), 385 (4,055). Λ_M (1×10^{-3} mol L^{-1} in methanol): 28 Ω^{-1} cm^2 mol^{-1}.

2. 4. Synthesis of $[CuL(NCS)]_n$ (3)

HL (29 mg, 0.10 mmol), $Cu(CH_3COO)_2{\cdot}H_2O$ (20 mg, 0.10 mmol) and ammonium thiocyanate (7.6 mg, 0.10 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 a week. Yield: 26 mg (63%). IR data (cm^{-1}): 2120, 1639, 1589, 1510, 1453, 1416, 1321, 1208, 1162, 1079, 1023, 966, 887, 860, 756, 686, 569, 511, 475. UV-Vis (MeOH; ε, L mol^{-1} cm^{-1}): 236 (19,370), 270 (8,935), 297 (5,053), 380 (4,650). Λ_M (1×10^{-3} mol L^{-1} in in methanol): 36 Ω^{-1} cm^2 mol^{-1}.

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 radiation

Table 1. Crystallographic data and refinement parameters for the complexes

	1	**2**	**3**
Chemical Formula	$C_{13}H_{15}Cl_3CuN_2O$	$C_{13}H_{15}BrCl_2CuN_2O$	$C_{14}H_{15}Cl_2CuN_3OS$
Fw	385.16	429.62	407.79
T (K)	298(2)	298(2)	298(2)
Crystal system	Orthorhombic	Orthorhombic	Orthorhombic
Space group	$Pca2_1$	$Pca2_1$	$Pbca$
a (Å)	8.614(2)	8.8300(13)	11.7060(15)
b (Å)	11.068(2)	11.1081(17)	12.3149(15)
c (Å)	15.461(3)	15.322(2)	24.367(2)
α (°)	90	90	90
β (°)	90	90	90
γ (°)	90	90	90
V (Å^3)	1474.0(5)	1502.9(4)	3512.8(7)
Z	4	4	8
μ (Mo Kα) (cm^{-1})	2.020	4.463	1.669
D_c (g cm^{-3})	1.736	1.899	1.542
Reflections collected	16379	19017	40889
Unique reflections	3489	3760	4333
Observed reflections [$I \geq 2\geq(I)$]	3228	3491	2634
Parameters	181	182	199
Restraints	1	1	0
R_{int}	0.0378	0.0273	0.0732
R_1, wR_2 [$I \geq 2\sigma(I)$]	0.0259, 0.0636	0.0206, 0.0534	0.0387, 0.0912
R_1, wR_2 (all data)	0.0300, 0.0659	0.0241, 0.0565	0.0839, 0.1092

(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. Complex **2** has been refined as a twin model. 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			
Cu1–Cl1	2.250(1)	Cu1–O1	1.943(3)
Cu1–N1	1.958(3)	Cu1–N2	2.078(3)
O1–Cu1–Cl1	92.62(7)	O1–Cu1–N1	91.41(11)
O1–Cu1–N2	174.47(9)	N1–Cu1–Cl1	162.19(9)
N1–Cu1–N2	84.29(11)	N2–Cu1–Cl1	92.62(7)
2			
Cu1–Br1	2.3915(6)	Cu1–O1	1.934(2)
Cu1–N1	1.955(3)	Cu1–N2	2.085(3)
Cu1–Br1A	3.051(3)		
O1–Cu1–N1	91.17(11)	O1–Cu1–N2	175.10(11)
N1–Cu1–N2	84.28(11)	O1–Cu1–Br1	92.19(7)
N1–Cu1–Br1	162.73(9)	N2–Cu1–Br1	92.69(8)
N1–Cu1–Br1A	88.85(10)	N2–Cu1–Br1A	85.65(10)
O1–Cu1–Br1A	92.50(10)	Br1–Cu1–Br1A	107.80(10)
Symmetry code for A: ½ + *x*, 1 – *y*, *z*.			
3			
Cu1–O1	1.9188(19)	Cu1–N1	1.937(2)
Cu1–N2	2.063(2)	Cu1–N3	1.952(3)
Cu1–S1A	2.927(3)		
O1–Cu1–N1	92.84(9)	O1–Cu1–N3	89.21(10)
N1–Cu1–N3	165.57(11)	O1–Cu1–N2	177.08(9)
N1–Cu1–N2	84.27(9)	N3–Cu1–N2	93.69(10)
N1–Cu1–S1A	93.41(10)	N2–Cu1–S1A	94.33(10)
N3–Cu1–S1A	100.99(10)	O1–Cu1–S1A	85.43(10)

Symmetry code for A: ½ + *x*, *y*, ½ – *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.

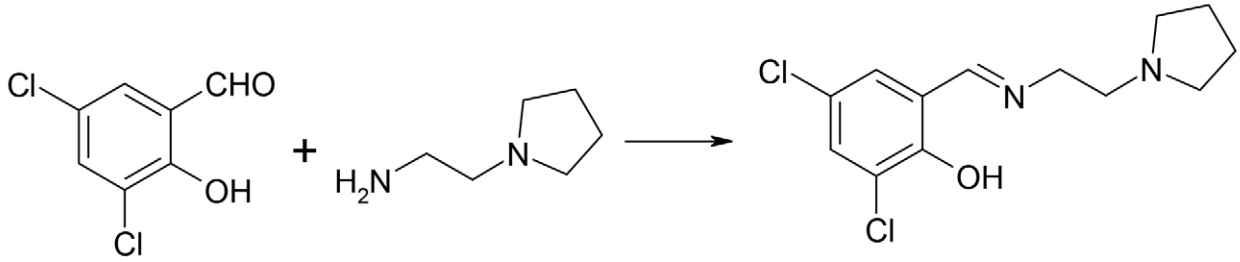
3. Results and Discussion

3. 1. Chemistry

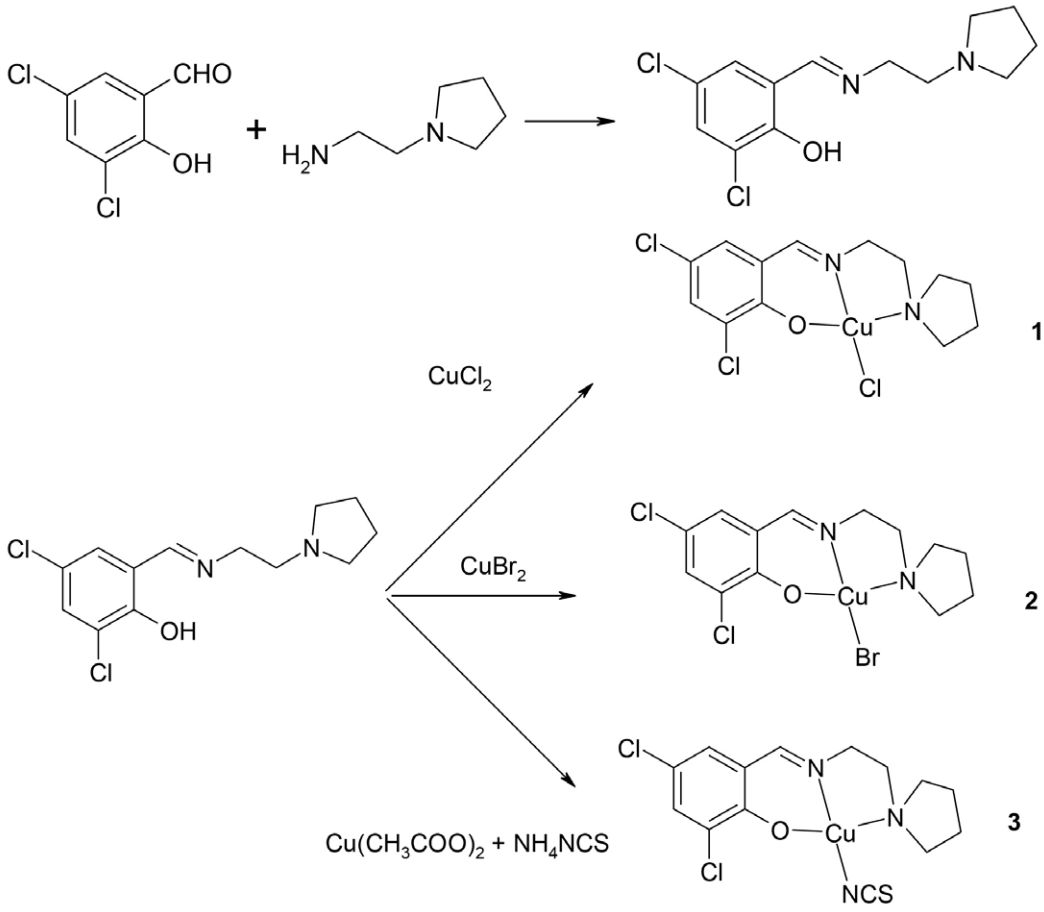
The Schiff base HL was prepared by reaction of 1:1 molar ratio of 3,5-dichlorosalicylaldehyde and *N*-(2-aminoethyl)pyrrolidine in methanol (Scheme 2). The three copper complexes were facile synthesized by reaction of equimolar quantities of the Schiff base HL with copper chloride, copper bromide and copper acetate, respectively in the presence of ammonium thiocyanate in methanol (Scheme 3). Complexes **1** and **2** have no thiocyanate ligands, while complex **3** has one. In fact, complexes **1** and **2** can also be obtained by reaction of HL with copper chloride and copper bromide, respectively. Single crystals of the complexes were formed by slow evaporation of their methanol solution at room temperature.

3.2. Crystal Structure Description of the Complex 1

Molecular structure of complex **1** is shown in Fig. 1. The complex is a mononuclear copper(II) species. The Cu atom in the complex is coordinated by one phenolate oxygen, one imino nitrogen and one pyrrolidine nitrogen of the Schiff base ligand, and one Cl ligand, forming a square planar geometry. The *trans* and *cis* bond angles are 162.19(9)–174.47(9)° and 84.29(11)–92.62(7)°, 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 in



Scheme 2. The synthetic procedure of the Schiff base HL



Scheme 3. The synthetic procedure of the complexes

the complex are comparable to those observed in Schiff base copper(II) complexes.[8] In the crystal structure, the molecules are linked through intermolecular hydrogen bonds of C–H···Cl (Table 3), to form two-dimensional sheets parallel to the *ab* plane (Fig. 2).

3. 3. Crystal Structure Description of the Complex 2

Molecular structure of complex **2** is shown in Fig. 3. The complex is a bromide bridged polymeric copper(II)

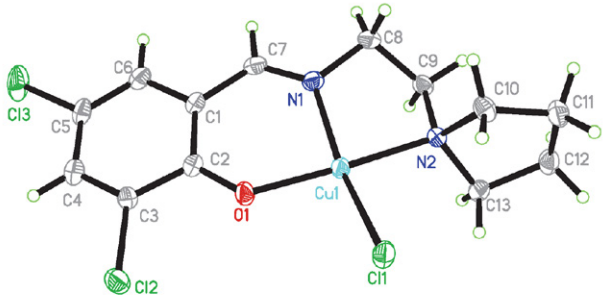


Fig. 1. A perspective view of the molecular structure of complex **1** with the atom labeling scheme. Thermal ellipsoids are drawn at the 30% probability level.

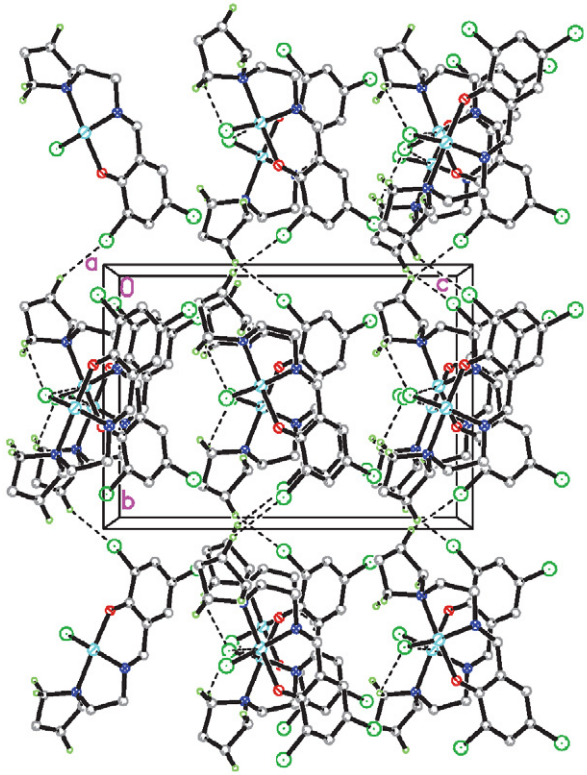


Fig. 2. Molecular packing diagram of complex **1**, viewed along the *a* axis. Hydrogen bonds are shown as dashed lines.

species. The Cu atom in the complex is in a square pyramidal coordination, with the basal plane defined by one phenolate oxygen, one imino nitrogen and one pyrrolidine nitrogen of the Schiff base ligand, and one Br ligand, and with the apical position occupied by one symmetry related Br ligand at the symmetry position ½ + *x*, 1 – *y*, *z*. The *trans* and *cis* bond angles in the basal plane are 162.73(9)–175.10(11)° and 84.28(11)–92.69(8)°, respectively. The bond angles among the apical and basal donor atoms are 107.80(10)°. Thus, the square pyramidal 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 in the complex are comparable to those observed in Schiff base copper(II) complexes.[9] In the crystal structure, the molecules are liked by Br ligand and intermolecular hydrogen bonds of C-H···Br (Table 3), to form one-dimensional chains running along the *a* axis (Fig. 4).

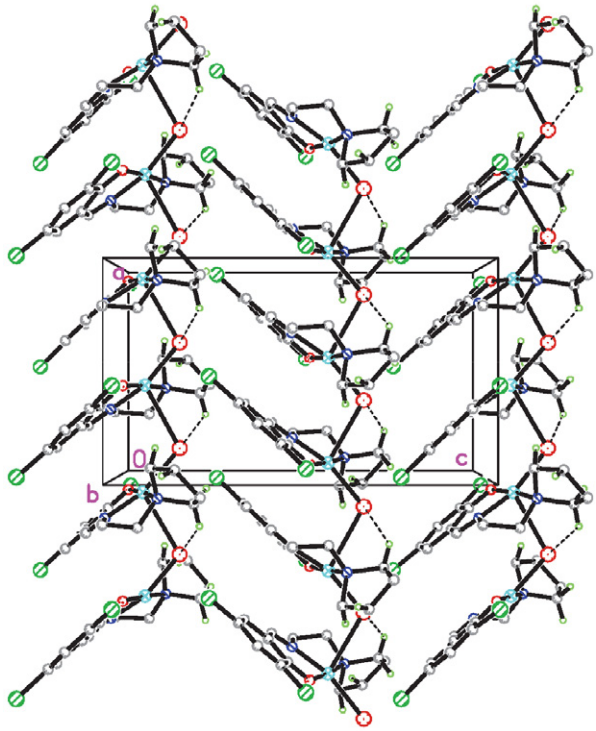


Fig. 4. Molecular packing diagram of complex **2**, viewed along the *b* axis. Hydrogen bonds are shown as dashed lines.

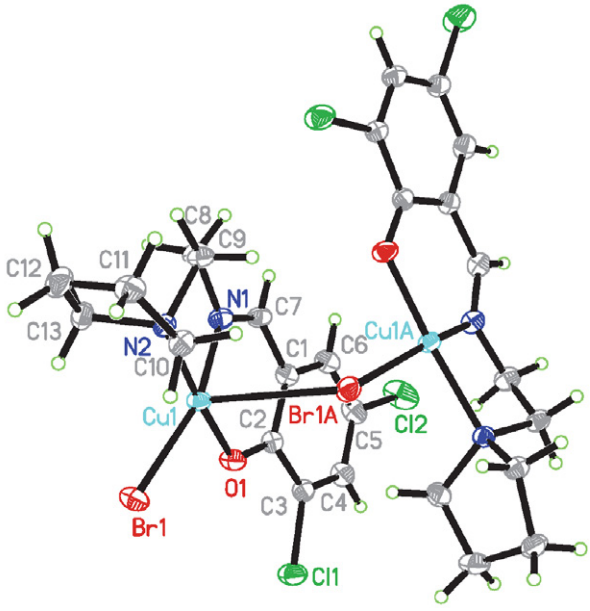


Fig. 3. A perspective view of the polymeric structure of complex **2** with the atom labeling scheme. Thermal ellipsoids are drawn at the 30% probability level. Repeated molecule is related to the symmetry operation ½ + *x*, 1 – *y*, *z*.

3. 4. Crystal Structure Description of the Complex 3

Molecular structure of complex **3** is shown in Fig. 5. The complex is a thiocyanate bridged polymeric copper(II) species. The Cu atom in the complex is in a square pyramidal coordination, with the basal plane defined by one phenolate oxygen, one imino nitrogen and one pyrrolidine nitrogen of the Schiff base ligand, and one N atom of a thiocyanate ligand, and with the apical position occupied by one S atom of a symmetry related thiocyanate ligand. The *trans* and *cis* bond angles in the basal plane are 165.57(11)–177.08(9)° and 84.27(9)–93.69(10)°, respectively. The bond angles among the apical and basal donor atoms are 85.43(10)–100.99(10)°. Thus, the square pyramidal 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 in the complex are

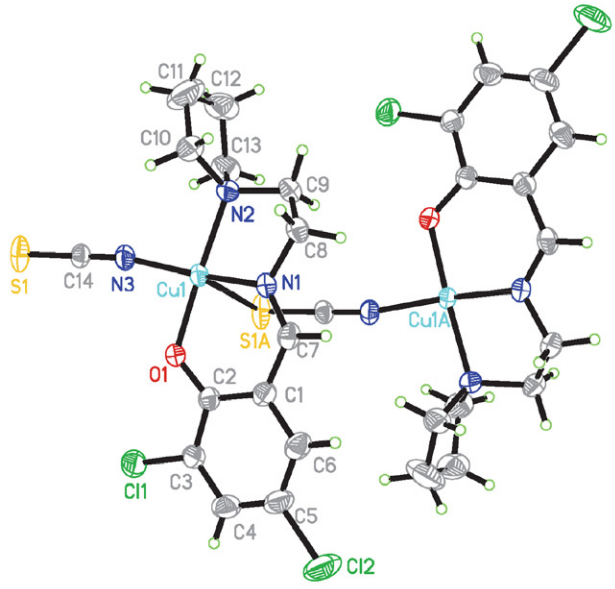


Fig. 5. A perspective view of the polymeric structure of complex **3** with the atom labeling scheme. Thermal ellipsoids are drawn at the 30% probability level. Repeated molecule is related to the symmetry operation ½ + *x*, *y*, ½ – *z*.

comparable to those observed in Schiff base copper(II) complexes.[10] In the crystal structure, the molecules are linked by thiocyanate ligand, to form one-dimensional chains running along the *a* axis. The chains are further linked through intermolecular hydrogen bonds of C-H···S (Table 3) at the *b* axis, to form two-dimensional sheets parallel to the *ab* plane (Fig. 6).

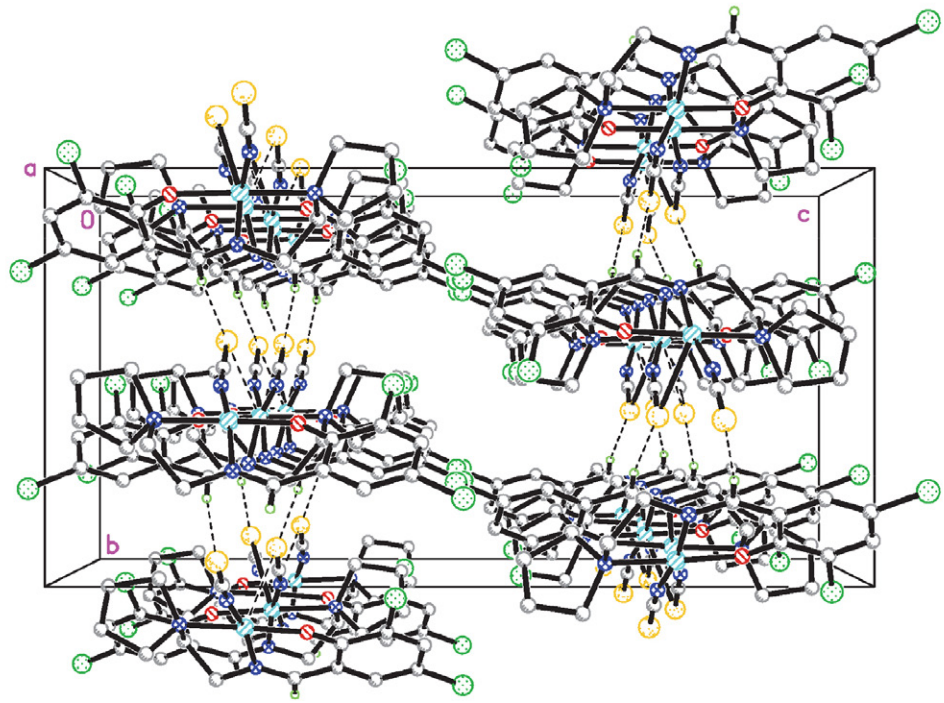


Fig. 6. Molecular packing diagram of complex **3**, viewed along the *a* axis. Hydrogen bonds are shown as dashed lines.

Table 3. Geometrical parameters for hydrogen bonds for the complexes

D–H···A	Distance, Å D–H	H···A	D···A	Angle D–H···A, °
1				
C11–H11B···Cl2^{i}	0.97	2.80(3)	3.715(4)	157(5)
C13–H13A···Cl1^{ii}	0.97	2.79(3)	3.420(4)	123(5)
2				
C10–H10A···Br1^{iii}	0.97	2.88(2)	3.500(4)	122(4)
C12–H12B···Cl1^{iv}	0.97	2.80(2)	3.709(4)	156(4)
3				
C7–H7···S1^{iii}	0.93	2.86(3)	3.785(5)	171(5)

Symmetry transformation used to generate the symmetry related atoms: ^{i} –½ + *x*, 1 – *y*, *z*; ^{ii} *x*, 1 + *y*, *z*; ^{iii} –½ + *x*, 1 – *y*, *z*; ^{iv} *x*, –1 + *y*, *z*; ^{v} ½ – *x*, ½ + *y*, *z*.

3. 5. IR and UV-Vis Spectra

The strong absorption bands at 1638–1639 cm^{-1} for the complexes are assigned to the azomethine groups, *ν*(C=N).[11] The intense band at 2120 cm^{-1} for complex **3** can be assigned to the stretching vibration of thiocyanate ligand.[12] The weak bands in the low wave numbers 440–550 cm^{-1} are due to the vibration of the Cu–O and Cu–N bonds.[13]

In the UV-Vis spectra of the complexes, the intense bands observed at 220–280 nm are assigned to intra-ligand π–π^* transitions. The complexes displayed bands centered at 290–315 nm, which can be assigned to the n–π^* transition.[14] The charge transfer LMCT bands are located at 380–385 nm.[15]

3. 6. Antibacterial Activity

The antimicrobial results are summarized in Table 4. The free Schiff base HL showed medium activity against *E. coli*, while no activity on *S. aureus* and *C. parapsilosis*. Interestingly, the three copper complexes have higher activities than HL. This is caused by the greater lipophilic nature of the complexes than the free ligands. The increased biological activity of the complexes can be explained with the chelating theory.[16] The chloride coordinated mononuclear copper complex **1** and bromide bridged polymeric complex **2** have similar activities, which showed strong activity against *S. aureus* and *E. coli*, and medium activity against *C. parapsilosis*. The thiocyanate bridged polymeric copper complex **3** showed strong activity against *S. aureus* and *E. coli*, while weak activity against *C. parapsilosis*. In general,

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	
HL	>2.50	>2.50	1.57	1.23	>2.50	>2.50	
1	0.41	0.25	0.26	0.13	1.81	1.53	
2	0.45	0.27	0.28	0.16	1.92	1.60	
3	0.72	0.49	0.55	0.31	2.20	1.87	
Cu-1	0.38	0.23	0.75	0.42	2.03	1.70	[ref. 17]
Cu-2	0.17	0.13	1.16	0.95	2.27	1.89	[ref. 17]
Cu-3	0.27	0.16	0.56	0.32	2.17	1.25	[ref. 18]
Zn-1	1.15	0.62	1.83	1.25	3.39	2.50	[ref. 18]

* mmol L^{-1}

complex **3** has a litter weaker activity against the bacteria and yeast than the other two. Notably, complex **1** has the most activity against *E. coli*, with IC_{50} and MIC values of 0.26 and 0.13 mmol L^{-1}, which deserves further study. The present three complexes have similar antibacterial activities against *S. aureus* and *C. parapsilosis*, while stronger activities against *E. coli* as compared to the copper(II) complexes (Cu-1, Cu-2) with the Schiff base ligand *N,N'*-bis(4-bromosalicylidene)propane-1,2-diamine.[17] The present three complexes have similar antibacterial activities against the bacteria strains with the copper(II) complex (Cu-3) with the Schiff base *N,N'*-bis(4-bromosalicylidene)propane-1,3-diamine, while have stronger activities against the bacteria strains with the zinc(II) complex (Zn-1) with the same ligand.[18]

4. Conclusion

In summary, three new copper(II) complexes derived from 2,4-dichloro-6-((2-pyrrolidin-1-ylethylimino) methyl)phenol with different co-ligands were prepared. The 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, imino nitrogen and pyrrolidine nitrogen. The complexes have effective antibacterial activities on the bacteria *Staphylococcus aureus* and *Escherichia coli*, and the yeast *Candida parapsilosis*.

Supplementary Material

CCDC 2333475 (**1**), 2333476 (**2**) and 2333477 (**3**) 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 tri nove bakrove(II) komplekse [CuClL] (**1**), $[CuBrL]_n$ (**2**) in $[CuL(NCS)]_n$ (**3**) s Schiffovo bazo 2,4-dikloro-6-((2-pirolidin-1-iletilimino)metil)fenol (HL) in jih okarakterizirali s spektroskopskimi metodami ter z rentgensko monokristalno analizo. Atom Cu v kompleksu **1** je v kvadratno planarni koordinaciji, v kompleksih **2** in **3** pa v kvadratno piramidalni koordinaciji. Ligand Schiffove baze je na Cu centralne atome koordiniran preko fenolatnega kisikovega atoma, imino dušikovega atoma in pirolidinskega dušikovega atoma. Antibakterijske aktivnosti Schiffove baze in treh bakrovih kompleksov smo določili na bakterijah *Staphylococcus aureus* in *Escherichia coli* ter kvasovkah *Candida parapsilosis*.