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Scientific paper

# Synthesis, Crystal Structures and Catalytic Oxidation Property of Two Copper(II) Complexes Derived from 5-Bromo-2-((2-piperazin-1-ylethylimino)methyl)phenol

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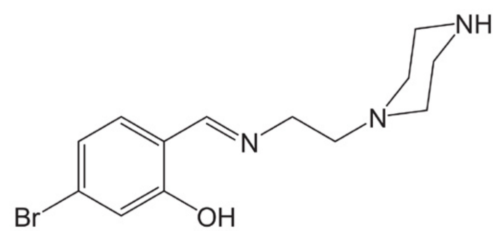
## Abstract

Two new copper(II) complexes, $[Cu(HL)(ONO_2)]NO_3$ (**1**) and $[CuCl(HL)]Cl \cdot H_2O$ (**2**), where HL is the zwitterionic form of the Schiff base 5-bromo-2-((2-piperazin-1-ylethylimino)methyl)phenol (HL), have been prepared. The Schiff base and two complexes were characterized by IR and UV-Vis spectroscopy. In addition, the free Schiff base was characterized by $^1$H NMR spectroscopy. Structures of the complexes were further confirmed by single crystal X-ray diffraction. The Cu atoms in the complexes are in square planar coordination. Crystal structures of both copper complexes are stabilized by hydrogen bonds. The complexes have high catalytic activity for the epoxidation of styrene, with conversions and selectivity over 90%.

**Keywords:** Copper complexes, Schiff base, crystal structure, catalytic property

## 1. Introduction

Schiff bases are a kind of compounds containing –CH=N– functional group, which have received much attention in coordination chemistry. The compounds can easily coordinate to many metal ions, to form mononuclear or polynuclear complexes with interesting structures.[1] Schiff base complexes have wide applications in the fields of biological and medicinal chemistry, catalysis, magnetic materials, etc.[2] A number of Schiff base complexes with different metal ions have fascinating catalytic epoxidation applications in the fields of organic synthesis, medicinal chemistry, and also in environmental protection[3] To date, a large number of Schiff base copper complexes have been reported with catalytic properties.[4] However, the catalytic epoxidation of styrene with Schiff base copper complexes is rare.[5] In pursuit of new Schiff base copper complexes with catalytic epoxidation properties, we report herein two new copper(II) complexes, $[Cu(HL)(ONO_2)]NO_3$ (**1**) and $[CuCl(HL)]Cl \cdot H_2O$ (**2**), where HL is the zwitterionic form of the Schiff base 5-bromo-2-((2-piperazin-1-ylethylimino)methyl)phenol (HL, Scheme 1).




**Scheme 1.** The Schiff base HL.

## 2. Experimental

### 2. 1. Materials and Physical Measurements

4-Bromosalicylaldehyde, *N*-(2-aminoethyl)piperazine, copper nitrate and copper chloride were purchased from Aldrich. The remaining reagents (AR grade) were purchased from Xiya Chemical Co. Ltd. Infrared spectra were recorded as KBr discs with a FTS-40 BioRad FT-IR spectrophotometer. Electronic spectra were recorded on a Lambda 35 spectrometer. Microanalyses for C, H and N of HL and the complexes were carried out on a Carlo-Erba 1106 elemental analyzer. Solution electrical conductivity was measured at 298(2) K using a DDS-11 conductivity meter. GC analyses were performed on a Shimadzu GC-2010 gas chromatograph.

## 2. 2. X-ray Crystallography

Crystallographic data of the two copper complexes were collected on a Bruker SMART CCD area diffractometer with graphite monochromated Mo K$\alpha$ radiation ($\lambda$ = 0.71073 Å) at 298(2) K. Absorption corrections were applied by using the multi-scan program.[6] Structures of both complexes were solved by direct methods and successive Fourier difference syntheses, and anisotropic thermal parameters for all nonhydrogen atoms were refined by full-matrix least-squares procedure against $F^2$.[7] All non-hydrogen atoms were refined anisotropically. The amino H atoms (H3A and H3B) of both complexes were located from difference Fourier maps and refined isotropically, with N–H and H···H distances restrained to 0.90(1) and 1.37(2) Å, respectively. The remaining hydrogen atoms were located at calculated positions, and refined isotropically with $U_{iso}$(H) values constrained to 1.2$U_{iso}$(C). Crystallographic data and experimental details for the structural analysis are summarized in Table 1.

## 2. 3. Synthesis of [Cu(HL)($ONO_2$)]$NO_3$ (1)

4-Bromosalicylaldehyde (1.0 mmol, 0.20 g) and *N*-(2-aminoethyl)piperazine (1.0 mmol, 0.13 g) were mixed in 50 mL methanol. The mixture was stirred for 30 min at room temperature to give a clear yellow solution. Then, copper nitrate trihydrate (1.0 mmol, 0.24 g) was added to the mixture and stirred for 10 min to give a brown solution. The solution was evaporated to remove three quarters of the solvent under reduced pressure, yielding deep brown solid. Yield: 0.37 g (74%). Well-shaped single crystals suitable for X-ray diffraction were obtained by re-crystallization of the solid from methanol. Analysis calculated for $C_{13}H_{18}BrCuN_5O_7$: C, 31.24; H, 3.63; N, 14.01%; found: C, 31.11; H, 3.72; N, 13.87%. IR data (KBr, cm$^{-1}$): 1637 (s, $\nu_{C=N}$), 1422, 1303, 1068 (m, $\nu_{ONO2}$), 1385 (s, $\nu_{NO3}$). UV-Vis data ($\lambda_{max}$, nm): 222, 249, 278, 305, 370.

## 2. 4. Synthesis of [CuCl(HL)]Cl·$H_2O$ (2)

4-Bromosalicylaldehyde (1.0 mmol, 0.20 g) and *N*-(2-aminoethyl)piperazine (1.0 mmol, 0.13 g) were mixed in 50 mL methanol. The mixture was stirred for 30 min at room temperature to give a clear yellow solution. Then, copper chloride dihydrate (1.0 mmol, 0.17 g) was added to the mixture and stirred for 10 min to give a brown solution. The solution was evaporated to remove three quarters of the solvent under reduced pressure, yielding deep brown solid. Yield: 0.31 g (67%). Well-shaped single crystals suitable for X-ray diffraction were obtained by re-crystallization of the solid from methanol. Analysis cal-

**Table 1.** Crystallographic data for the single crystals of both complexes

| | 1 | 2 |
|---|---|---|
| Empirical formula | $C_{13}H_{18}BrCuN_5O_7$ | $C_{13}H_{20}BrCl_2CuN_3O_2$ |
| Formula weight | 499.77 | 464.57 |
| Crystal system | Monoclinic | Orthorhombic |
| Space group | $P2_1/c$ | *Pbca* |
| $a$ (Å) | 21.8899(16) | 12.8838(6) |
| $b$ (Å) | 6.6682(8) | 13.3881(6) |
| $c$ (Å) | 12.3922(12) | 19.9142(10) |
| $\alpha$ (°) | 90 | 90 |
| $\beta$ (°) | 90.1890(10) | 90 |
| $\gamma$ (°) | 90 | 90 |
| $V$ (Å$^3$) | 1808.8(3) | 3435.0(3) |
| Z | 4 | 8 |
| $F$(000) | 1004 | 1864 |
| $\mu$, mm$^{-1}$ | 3.464 | 3.918 |
| $R_{int}$ | 0.0454 | 0.0306 |
| Collected data | 40256 | 35102 |
| Unique data | 3354 | 3193 |
| Observed data [$I > 2\sigma(I)$] | 2891 | 2718 |
| Restraints | 3 | 3 |
| Parameters | 250 | 208 |
| Goodness-of-fit on $F^2$ | 1.036 | 1.039 |
| $R_1$, $wR_2$ indices [$I > 2\sigma(I)$] | 0.0272, 0.0637 | 0.0233, 0.0549 |
| $R_1$, $wR_2$ indices (all data) | 0.0348, 0.0673 | 0.0321, 0.0591 |

culated for $C_{13}H_{20}BrCl_2CuN_3O_2$: C, 33.60; H, 4.34; N, 9.04%; found: C, 33.73; H, 4.38; N, 8.96%. IR data (KBr, $cm^{-1}$): 3478 (m, $\nu_{OH}$), 1635 (s, $\nu_{C=N}$). UV-Vis data ($\lambda_{max}$, nm): 225, 275, 365.

## 2. 5. Styrene Epoxidation

The epoxidation reaction was carried out at room temperature in acetonitrile under $N_2$ atmosphere with constant stirring. The composition of the reaction mixture was 2.00 mmol of styrene, 2.00 mmol of chlorobenzene (internal standard), 0.10 mmol of the complexes (catalyst) and 2.00 mmol iodosylbenzene or sodium hypochlorite (oxidant) in 5.00 mL freshly distilled acetonitrile. When the oxidant was sodium hypochlorite, the solution was buffered to pH 11.2 with $NaH_2PO_4$ and NaOH. The composition of reaction medium was determined by GC with styrene and styrene epoxide quantified by the internal standard method (chlorobenzene). All other products detected by GC were mentioned as others. The reaction time for maximum epoxide yield was determined by withdrawing periodically 0.1 mL aliquots from the reaction mixture. This time was used to monitor the efficiency of the catalyst on performing at least two independent experiments. Blank experiments with each oxidant and using the same experimental conditions except catalyst were also performed.

# 3. Results and Discussion

## 3. 1. Chemistry

Complexes **1** and **2** were facile synthesized from the Schiff base HL with copper nitrate and copper chloride, respectively in methanol (Scheme 2). Complex **1** is a nitrate coordinated copper compound, while complex **2** is a chloride coordinated copper compound. The molar conductivities are 137 $\Omega^{-1}$ $cm^2$ $mol^{-1}$ (**1**) and 152 $\Omega^{-1}$ $cm^2$ $mol^{-1}$ (**2**), which are consistent with the values expected for 1:1 electrolyte.[8] Powder XRD confirms the purity of both complexes (Figs. 1 and 2).

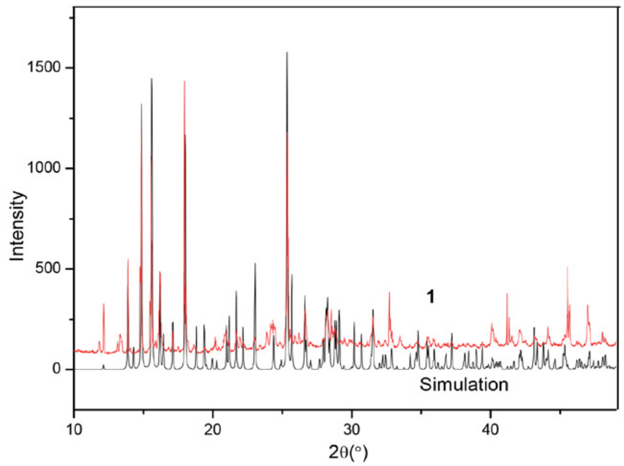



**Fig. 1.** Powder XRD of complex **1**.

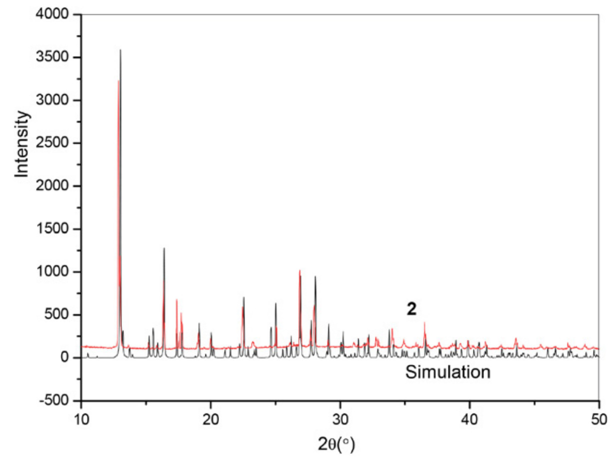
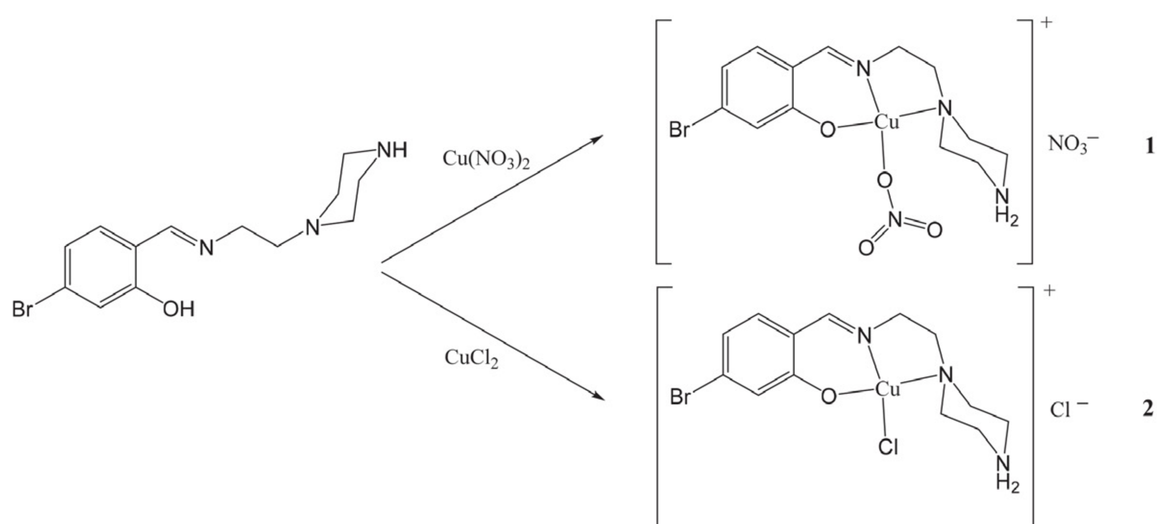



**Fig. 2.** Powder XRD of complex **2**.




**Scheme 2.** Synthetic procedure of both copper complexes.

## 3. 2. Crystal Structure Description of [Cu(HL)($ONO_2$)]$NO_3$ (1)

The coordinate bond lengths and bond angles are given in Table 2. Molecular structure of the compound is shown in Fig. 3. The compound contains a mononuclear [Cu(HL)($ONO_2$)]$^+$ cation and a nitrate anion. The Schiff base is in a zwitterionic form, with the phenol hydrogen atom transferred to NH group. The Cu atom is coordinated by phenolate oxygen (O1), imino nitrogen (N1) and piperazine nitrogen (N2) of the Schiff base ligand, and one oxygen (O2) of a nitrate ligand, to form square planar geometry. The distortion of the coordination can be observed from the coordinate bond angles. The *cis* and *trans* angles are 85.33(8)–95.31(8)° and 166.97(8)–174.96(8)°. The deviation from the ideal square planar geometry is mainly origin from the strain created by the five-membered chelate ring Cu1–N1–C8–C9–N2. The bond lengths of Cu1–O1 (1.8899(17) Å), Cu1–N1 (1.911(2) Å) and Cu1–N2 (2.082(2) Å) are comparable to other Schiff base copper(II) complexes.[9] The piperazine ring is in a chair conformation.

Two adjacent [Cu(HL)($ONO_2$)]$^+$ cations are linked by two nitrate anions through intermolecular hydrogen bonds of types N–H···O (Table 3), to form a dimer. The dimers are further linked through intermolecular hydrogen bonds of types N–H···O to form one dimensional chains running along *b* axis (Fig. 4).

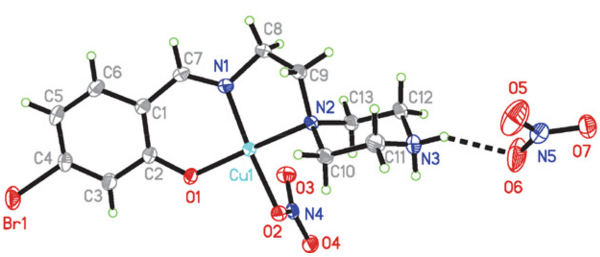



**Fig. 3.** ORTEP diagram of complex **1** with 30% thermal ellipsoid.

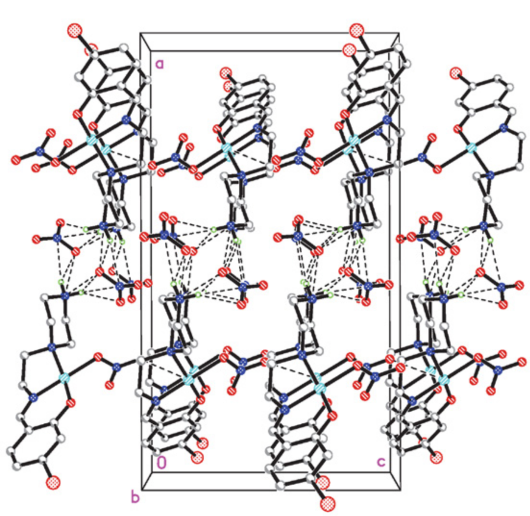



**Fig. 4.** Molecular packing structure of complex **1** linked by hydrogen bonds (dashed lines).

## 3. 3. Crystal Structure Description of [CuCl(HL)]Cl·$H_2O$ (2)

The coordinate bond lengths and bond angles are given in Table 2. Molecular structure of the compound is shown in Fig. 5. The compound contains a mononuclear [CuCl(HL)]$^+$ cation, a chloride anion, and a water molecule of crystallization. The Schiff base is in a zwitterionic form, with the phenol hydrogen atom transferred to NH group. The Cu atom is coordinated by phenolate oxygen (O1), imino nitrogen (N1) and piperazine nitrogen (N2) of the Schiff base ligand, and one Cl ligand, to form square planar geometry. The distortion of the coordination can be observed from the coordinate bond angles. The *cis* and *trans* angles are 84.35(8)–93.32(5)° and 163.37(6)–175.42(7)°. The deviation from the ideal square planar geometry is mainly origin from the strain created by the five-membered chelate ring Cu1–N1–C8–C9–N2. The bond lengths of Cu1–O1 (1.9257(16) Å), Cu1–N1 (1.9545(19) Å) and Cu1–N2 (2.0954(18) Å) are longer than those observed in complex **1**, and comparable to other Schiff base copper(II) complexes.[10] The piperazine ring is in a chair conformation.

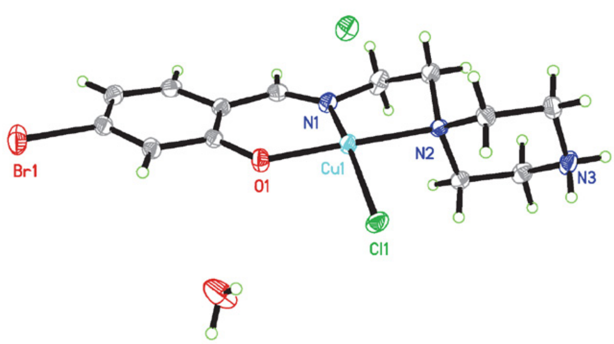



**Fig. 5.** ORTEP diagram of complex **2** with 30% thermal ellipsoid.

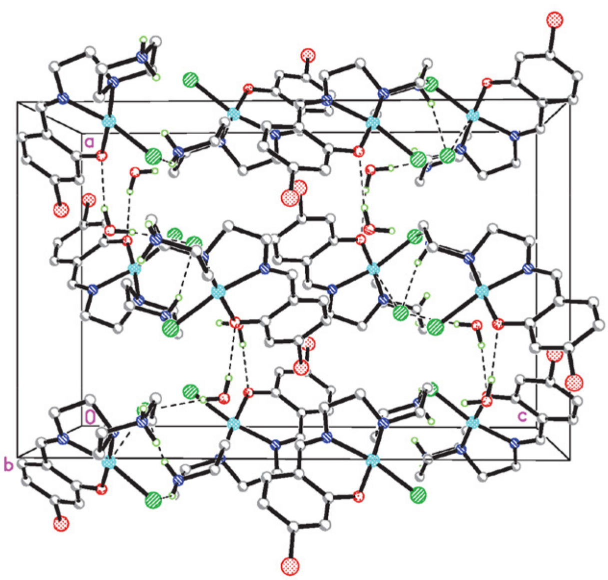



**Fig. 6.** Molecular packing structure of complex **2** linked by hydrogen bonds (dashed lines).

The $[CuCl(HL)]^+$ cations, chloride anions and water molecules are linked through intermolecular hydrogen bonds of types N–H···Cl (Table 3), to form one dimensional chains running along *a* axis (Fig. 6).

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

| | | | |
|---|---|---|---|
| **1** | | | |
| Cu1-O1 | 1.8899(17) | Cu1-N1 | 1.911(2) |
| Cu1-O2 | 2.0158(17) | Cu1-N2 | 2.082(2) |
| O1-Cu1-N1 | 95.31(8) | O1-Cu1-O2 | 88.60(7) |
| N1-Cu1-O2 | 174.96(8) | O1-Cu1-N2 | 166.97(8) |
| N1-Cu1-N2 | 85.33(8) | O2-Cu1-N2 | 91.58(7) |
| **2** | | | |
| Cu1-O1 | 1.9257(16) | Cu1-N1 | 1.9545(19) |
| Cu1-N2 | 2.0954(18) | Cu1-Cl1 | 2.2701(6) |
| O1-Cu1-N1 | 92.03(7) | O1-Cu1-N2 | 175.42(7) |
| N1-Cu1-N2 | 84.35(8) | O1-Cu1-Cl1 | 89.35(5) |
| N1-Cu1-Cl1 | 163.37(6) | N2-Cu1-Cl1 | 93.32(5) |

**Table 3.** Hydrogen bond distances (Å) and bond angles (°) for the complexes

| ***D*–H···*A*** | ***d*(*D*–H)** | ***d*(H···*A*)** | ***d*(*D*···*A*)** | **Angle (*D*–H···*A*)** |
|---|---|---|---|---|
| **1** | | | | |
| N3–H3A···O6 | 0.90(1) | 1.98(2) | 2.764(3) | 145(3) |
| N3–H3B···O6$^{\#1}$ | 0.90(1) | 2.15(2) | 2.918(4) | 142(3) |
| N3–H3B···O7$^{\#1}$ | 0.90(1) | 2.13(2) | 2.971(3) | 155(3) |
| **2** | | | | |
| N3–H3B···Cl2$^{\#2}$ | 0.90(1) | 2.27(2) | 3.135(2) | 159(3) |
| N3–H3A···Cl1$^{\#2}$ | 0.90(1) | 2.38(2) | 3.151(2) | 143(3) |

Symmetry codes: #1: 1 – *x*, – *y*, 1 – *z*; #2: 1 – *x*, ½ + *y*, ½ – *z*.

## 3. 4. IR and UV-vis Spectra of the Complexes

The medium absorption at 3478 cm$^{-1}$ of complex **2** is assigned to the stretching vibration of O-H bonds of water molecules. The strong bands at 1637 cm$^{-1}$ for **1** and 1635 cm$^{-1}$ for **2** are attributed to the vibration of C=N groups.[11] Complex **1** shows IR bands at 1422 ($\nu_5$), 1303 ($\nu_1$) and 1068 cm$^{-1}$ ($\nu_2$) due to NO stretches. The value of $\Delta(\nu_5–\nu_1)$, 119 cm$^{-1}$, suggests monodentate coordination. For monodentate coordination of $NO_3$, the separation of first NO stretching vibrations is low (100–130 cm$^{-1}$), whereas these bands show larger separation (180–225 cm$^{-1}$) when $NO_3$ is bidentate.[12] IR spectrum of complex **1** also shows a band at 1385 cm$^{-1}$ due to ionic nitrate.[13]

In the UV-Vis spectra of both complexes, the bands at 360–370 nm are attributed to the azomethine chromophore π-π* transitions. The bands at higher energy (220–280 nm) are associated with the benzene π-π* transitions.[14]

## 3. 5. Catalytic Properties of the Complexes

The conversion of styrene, selectivity for styrene oxide, yield of styrene oxide and reaction time to obtain maximum yield using both complexes as oxidants are listed in Table 4. The results reveal that both copper complexes as catalysts convert styrene most efficiently in the presence of PhIO and NaOCl as oxidants. Nevertheless, the catalysts are selective towards the formation of styrene epoxides despite the formation of by-products benzaldehyde, phenylacetaldehyde, styrene epoxides derivative, alcohols *etc.* The results also show that both complexes have excellent efficiency for styrene epoxide yields. When the reactions were performed with the oxidants PhIO and NaOCl, complexes **1** and **2** have styrene conversions of 93% and 91%, and 91% and 90%, respectively. Thus, PhIO acts as a better oxidant than NaOCl with respect to both styrene conversion and styrene epoxide selectivity. The epoxide yields for complexes **1** and **2** using both PhIO and NaOCl as oxidants are 90% and 87%, and 85% and 81%, respectively.

**Table 4.** Catalytic epoxidation results of both complexes*

| | 1 | | 2 | |
|---|---|---|---|---|
| Oxidant | PhIO | NaOCl | PhIO | NaOCl |
| Conversion (%) | 93 | 91 | 91 | 90 |
| Epoxide yield (%) | 90 | 87 | 85 | 81 |
| Selectivity (%) | 95 | 93 | 93 | 90 |

* The time is 2 h for PhIO, and 3 h for NaOCl.

# 4. Conclusion

Two new mononuclear copper(II) complexes derived from the Schiff base ligand 5-bromo-2-((2-piperazin-1-ylethylimino)methyl)phenol were obtained. Single crystal X-ray analysis indicates that the Cu atoms in both complexes are in square planar coordination. The anions of metal salts like nitrate and chloride can coordinate to the Cu ions. In addition, they also play as counter anions in the complexes. Both complexes have efficient catalytic property for the epoxidation of styrene, with conversions and selectivity over 90%.

### Supplementary Material

CCDC 2420082 for **1** and 2420083 for **2** contain the supplementary crystallographic data for this paper. These data can be obtained free of charge *via* http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk.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## Povzetek

Pripravili smo dva nova bakrova(II) kompleksa, $[Cu(HL)(ONO_2)]NO_3$ (**1**) in $[CuCl(HL)]Cl \cdot H_2O$ (**2**), kjer je HL zwitterionska oblika Schiffove baze 5-bromo-2-((2-piperazin-1-yletilimino)metil)fenol (HL). Schiffovo bazo in dva kompleksa smo okarakterizirali z IR in UV-Vis spektroskopijo. Poleg tega smo prosto Schiffovo bazo okarakterizirali z $^1H$ NMR spektroskopijo. Strukture kompleksov smo določili z monokristalno rentgensko difrakcijo. Cu atomi v kompleksih so v kvadratno planarni koordinaciji. Kristalni strukturi obeh bakrovih kompleksov sta stabilizirani z vodikovimi vezmi. Kompleksa imata visoke katalitične aktivnosti pri epoksidaciji stirena, s pretvorbami in selektivnostjo nad 90 %.