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# Synthesis, crystal structure, and electrochemical properties of Ni(II) and Cu(II) complexes with two unsymmetrical $N_2O_2$ Schiff base ligands

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Abstract Nickel(II) and copper(II) complexes of two unsymmetrical tetradentate Schiff base ligands [Ni(Me-salabza)] (1), [Cu(Me-salabza)] (2) and [Ni(salabza)] (3), {H<sub>2</sub>salabza = N,N'-bis[(salicylidene)-2-aminobenzylamine] and H<sub>2</sub>Me-salabza = N,N'-bis[(methylsalicylidene)-2-aminobenzylamine]}, have been synthesized and characterized by elemental analysis and spectroscopic methods. The crystal structures of **2** and **3** complexes have been determined by single crystal X-ray diffraction. Both copper(II) and nickel(II) ions adopt a distorted square planar geometry in [Cu(Me-salabza)] and [Ni(salabza)] complexes. The cyclic voltammetric studies of these complexes in dichloromethane indicate the electronic effects of the methyl groups on redox potential.

Keywords Unsymmetrical  $N_2O_2$  Schiff base  $\cdot$  Ni(II) and Cu(II) complexes  $\cdot$  Crystal structure  $\cdot$  Cyclic voltammetry

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#### Introduction

Schiff base complexes have remained an important area of research due to their simple synthesis, versatility, and diverse range of applications such as antimicrobial [1-3], anticancer [4], diuretic [5], antitumor, and biological activities [6–10].

Many symmetrical tetradentate Schiff bases of 1,2-diamines with o-hydroxyaldehydes/ketones have been prepared and studied intensively. However, much less attention has been focused on unsymmetrical tetradentate, but in recent years, there is a growing interest in the coordination chemistry of transition metal complexes of unsymmetrical Schiff base ligands, due to the fact that the ligands around central metal ions in natural systems are unsymmetrical [11–13]. The unsymmetrical Schiff bases are obtained by the mono-condensation of diamines with aldehydes/ketones, or by stepwise condensation of the appropriate diamine with two different carbonyl compounds [11, 13–16]. Corresponding complexes may serve as biological models [11, 17-20] or as catalysts in some chemical processes [21-23] and promising materials for optoelectronic applications [24-27] and the design of biosensors [28].

Recently, the ease of preparation of metal salen-based modified electrodes by oxidative electro-polymerisation of metal complexes in poor coordinating solvents has prompted their use in heterogeneous electrocatalysis [29]. The role played by structural electronic effects to control the redox chemistry of these metal complexes may prove to be critical in the design of new catalysts [30]. The electronic properties and steric demands of these Schiff base ligands can be tuned selectively via appropriate tailoring and modification of the ligand structure.



Scheme 1 The chemical formula of the Schiff base ligands

In continuation of our work on the synthesis of the unsymmetrical Schiff base metal complexes [31, 32], we report the synthesis, spectral and electrochemical properties of Cu(II) and Ni(II) complexes of the unsymmetrical tetradentate Schiff base ligands  $H_2$ Me-salabza and  $H_2$ salabza (Scheme 1). The electronic effects of methyl groups on the redox potentials and the X-ray crystal structures of [Cu(Me-salabza)] complex (2) and [Ni(salabza)] (3) are also reported and discussed.

## **Experimental setup**

## Materials and general methods

All solvents and chemicals were of commercial reagent grade and used as received from Aldrich and Merck. Infrared spectra from KBr pellets were collected on a FT-IR JASCO 680 plus spectrometer in the range  $4.000-400 \text{ cm}^{-1}$ . UV-Vis absorption spectra were recorded on a JASCO V-570 spectrophotometer. <sup>1</sup>H-NMR spectra were measured with a BRUKER AVANCEDEX (400 MHz) spectrometer. Proton chemical shifts are reported in ppm relative to Me<sub>4</sub>Si as internal standard. Elemental analyses were performed using a Perkin-Elmer 2400 II CHNS-O elemental analyzer. Electrochemical properties of these complexes were studied by cyclic voltammetry. Cyclic voltammograms were recorded using a SAMA 500 Research Analyzer using a three-electrode system, a glassy carbon working electrode (Metrohm 6.1204.110 with 2.0  $\pm$  0.1 mm diameter), a platinum disk auxiliary electrode and Ag wire as reference electrode. CV measurements were performed in dichloromethane with tetrabutylammonium hexafluorophosphate (TBAH) as supporting electrolyte. The solutions were deoxygenated by purging with Ar for 5 min. All electrochemical potentials were calibrated versus internal  $Fc^{+/0}$  (E<sup>0</sup> = 0.46 V versus SCE) couple under the same conditions [33].

# Synthesis

# Synthesis of H<sub>2</sub>Me-salabza ligand

The new ligand H<sub>2</sub>Me-salabza was synthesized by adding a solution of 2-hydroxy acetophenone (0.272 g, 2 mmol) in ethanol (5 ml) to a stirring solution of 2-aminobenzylamine (0.122 g, 1 mmol) in ethanol (5 ml). The mixture was stirred for 1 h at room temperature to give a yellow precipitate. The product was filtered off and washed with cold ethanol. Yield 86 %. Anal. Calc. for C<sub>23</sub>H<sub>22</sub>N<sub>2</sub>O<sub>2</sub>: C, 77.07; H, 6.19; N, 7.82; found: C, 76.84; H; 6.06, N; 7.9 %. FT-IR (KBr, cm<sup>-1</sup>)  $\nu_{max}$ : O–H (3830–3109), C = N (1607, 1571). UV–Vis:  $\lambda_{max}$  (nm) ( $\varepsilon$ , L mol<sup>-1</sup> cm<sup>-1</sup>) (CH<sub>2</sub>Cl<sub>2</sub>): 229 (101890), 257 (7150), 324 (330). <sup>1</sup>H-NMR (CDCl<sub>3</sub>, 400 MHz): 2.28 (s, 3H, H<sub>c</sub>), 2.32 (s, 3H, H<sub>d</sub>), 4.71 (s, 2H, H<sub>c</sub>), 7.57–6.65 (m, 12H, *H*<sub>aromatic</sub>), 14.50 (s, 1H, *H*<sub>enolic</sub>), 16.14 (s, 1H, *H*<sub>enolic</sub>).

# Synthesis of H<sub>2</sub>salabza ligand

The H<sub>2</sub>salabza ligand was synthesized by a procedure reported in the literature [34]. The crystalline product obtained was characterized by elemental analysis, <sup>1</sup>H-NMR, and spectroscopic methods, and the results are in good agreement with the literature values.

# Synthesis of [Ni(Me-salabza)] complex (1)

To a solution of Ni(CH<sub>3</sub>COO)<sub>2</sub>·4H<sub>2</sub>O (0.0248 g, 0.1 mmol) in ethanol (10 mL), a solution of H<sub>2</sub>Me-salabza (0.0353 g, 0.1 mmol) in dichloromethane (15 ml) was added, and the mixture was stirred for 4 h at room temperature and filtered. Brown crystals were obtained by slow evaporation of solvents. Yield 78 %. Anal. Calc. for C<sub>23</sub>H<sub>20</sub>N<sub>2</sub>NiO<sub>2</sub>: C, 66.55; H, 4.86; N, 6.75; Found: C, 66.19; H, 4.74; N, 6.69 %. FT-IR (KBr, cm<sup>-1</sup>)  $\nu_{max}$ : 1597, 1568 (C = N). UV–Vis:  $\lambda_{max}$  (nm) ( $\varepsilon$ , L mol<sup>-1</sup> cm<sup>-1</sup>) (CH<sub>2</sub>Cl<sub>2</sub>): 237 (70050), 260 (72890), 350 (123230), 438 (8520), 600 (265).

# Synthesis of [Cu(Me-salabza)] complex (2)

This complex was prepared by the same procedure used for 1 except that  $Cu(CH_3COO)_2$ ·  $H_2O$  (0.0199 g, 0.1 mmol) was used as the metal salt. Brown crystals suitable for X-ray diffraction were obtained by slow evaporation of solvents. Yield 80 %. Anal. Calc. for  $C_{23}H_{20}N_2CuO_2$ : C, 65.78; H, 4.80; N, 6.67; found: C, 65.88; H, 4.65; N,

6.67 %. FT-IR (KBr, cm<sup>-1</sup>)  $v_{max}$ : 1598, 1567 (C = N). UV–Vis:  $\lambda_{max}$  (nm) ( $\epsilon$ , L mol<sup>-1</sup> cm<sup>-1</sup>) (CH<sub>2</sub>Cl<sub>2</sub>): 229 (61010), 262 (29400), 344 (9430), 572 (290).

# Synthesis of [Ni(salabza)] complex (3)

The complex **3** was prepared by a procedure similar to that of complex **1** except that H<sub>2</sub>salabza (0.033 g, 0.1 mmol) was used instead of H<sub>2</sub>Me-salabza ligand. Dark green crystals suitable for X-ray diffraction were obtained by slow evaporation of solvents. Yield 86 %. Anal. Calc. for NiC<sub>21</sub>H<sub>16</sub>N<sub>2</sub>O: C, 65.16; H, 4.17; N, 7.24. found. C, 65.31; H, 4.32; N, 6.92 %. FT-IR (KBr, cm<sup>-1</sup>),  $v_{max}$ : 1609, 1579 (C = N). UV–Vis:  $\lambda_{max}$  (nm) ( $\varepsilon$ , L mol<sup>-1</sup> cm<sup>-1</sup>) (CH<sub>2</sub>Cl<sub>2</sub>): 268 (52910), 310 (16360), 342 (119120), 444 (75030), 614 (160).

## Crystal structure determination and refinement

### X-ray crystallography for 2 and 3

Suitable single crystals of **2** and **3** were obtained by slow evaporation of a methanol–dichloromethane (1:1 v/v) solution at room temperature.

X-ray data of **2** were collected at T = 294 K on a STOE IPDS II diffractometer with graphite monochromatic Mo K $\alpha$  ( $\lambda = 0.71073$  Å) radiation. Cell refinement and data reduction were performed with the help of the X-Area (1.36) program [35], and a numerical absorption correction was performed with the program XRED32 (1.31) [36].

X-ray data of **3** were collected at T = 100 K, on a Bruker APEX-II CCD diffractometer with graphite monochromatic Mo K $\alpha$  ( $\lambda = 0.71073$  Å) radiation. Cell refinement and data reduction were performed with the program SAINT [37]. Correction for absorption was carried out with the multi-scan method and program SADABS [37]. The structures of **2** and **3** were solved with direct methods using the program SHELXS97 [38] and structure refinement on  $F^2$  was carried out with the program SHELXL-97. The crystallographic and refinement data of these complexes are summarized in Table 1.

#### **Results and discussion**

#### Synthesis and characterization

The tetradentate Schiff base ligands,  $H_2$ salabza or  $H_2$ Mesalabza, have been derived from condensation of 2-aminobenzylamine with aldehyde/ketone. All the complexes (Scheme 2) have been prepared by the direct reaction of the corresponding ligand,  $H_2$ salabza or  $H_2$ Me-salabza with metal salt at room temperature. Crystals of these

Table 1 Crystal data and structure refinement for (2) and (3)

Compound	(2)	(3)	
Empirical formula	C23H20CuN2O2	C <sub>21</sub> H <sub>16</sub> NiN <sub>2</sub> O <sub>2</sub>	
Formula weight	419.95 387.07		
Temperature (K)	294(2) 100(2)		
Crystal system	Monoclinic	Monoclinic	
Space group	P21/n	P21/c	
a (Å)	15.526(3)	11.4528(13)	
<i>b</i> (Å)	8.0264(16)	8.6351(10)	
<i>c</i> (Å)	16.517(3) 16.8094(19)		
α (°)	90.00	90.00	
β (°)	115.12(3)	99.6860(10)	
γ (°)	90.00	90.00	
$V(\text{\AA}^3)$	1863.5(6)	1638.69	
Ζ	4	4	
$D_{\text{calc}} (\text{Mg/m}^3)$	1.497	1.569	
$\mu \text{ (mm}^{-1})$	1.19	1.202	
Crystal size (mm)	$0.52\times0.14\times0.10$	$0.45 \times 0.35 \times 0.20$	
F (000)	868	800	
$\theta$ range (°)	2.7–27.8	2.66-30.00	
Index ranges	$-21 \le h \le 21,$ $-9 \le k \le 10,$ $-22 \le l \le 22$	$-14 \le h \le 16,$ $-12 \le k \le 12,$ $-23 \le l \le 23$	
Absorption correction	Integration	Multi-scan	
Maximum and minimum transmission	0.6786 and 0.9083	0.78 and 0.65	
Reflections collected	13168/4936	25804/4759	
R <sub>int</sub>	0.0606	0.0211	
Data/restraints/ parameters	4936/0/254	4759/0/235	
Goodness of fit on $F^2$	0.981	1.035	
Final <i>R</i> indices $[I > 2\sigma(I)]$	$R_1 = 0.055,$ $wR_2 = 0.152$	$R_1 = 0.0222,$ $wR_2 = 0.0598$	
Largest difference peak and hole $(e.Å^{-3})$	0.369 and -0.611	0.445 and -0.458	

complexes suitable for X-ray crystallography were obtained in good yield (about 80 %).

Description of structures

#### Crystal structure of [Cu(Me-salabza)] (2)

The crystal structure of [Cu(Me-salabza)] complex is shown in Fig. 1. The crystallographic and refinement data for complex **2** are summarized in Table 1, and selected bond distances and angles are listed in Table 2.



Scheme 2 The chemical formula of 1, 2, and 3 complexes



Fig. 1 The molecular structure of  $\left[\text{Cu}(\text{Me-salabza})\right]$  with its atom labeling scheme

Table 2 Selected bond lengths (Å) and angles (°) for (2) and (3)

Complex	(2)		(3)
Bond lengths			
Cu1–O1	1.908(3)	Ni1-O1	1.8435(8)
Cu1–O2	1.857(3)	Ni1-O2	1.8563(8)
Cu1–N1	1.975(3)	Ni1-N1	1.8800(9)
Cu1–N2	1.976(3)	Ni1-N2	1.8919(9)
Bond angles			
O2-Cu1-O1	87.85(13)	O1-Ni1-O2	82.53(3)
O2-Cu1-N1	168.51(13)	O1-Ni1-N1	94.39(4)
O1–Cu1–N1	87.46(13)	O2-Ni1-N1	164.35(4)
O2-Cu1-N2	92.89(12)	O1-Ni1-N2	164.36(4)
O1-Cu1-N2	164.76(15)	O2-Ni1-N2	93.16(4)
N1-Cu1-N2	94.39(12)	N1-Ni1-N2	93.63(4)



Fig. 2 The molecular structure of [Ni(salabza)] with its atom labeling scheme

Complex **2** crystallizes in monoclinic space group P 21/n in this complex, the Schiff base ligand acts as tetradentate chelating ligand coordinated to Cu(II) center. The central Cu(II) ion has the distorted square planar geometry. However, the whole molecule is not planar since the two halves are folded with an angle of 18.08°. The average Cu–O bond length of 1.882 Å and Cu–N distance of 1.975 Å agree well with the corresponding distances in related copper(II) complexes [13].

## Crystal structure of [Ni(salabza)] (3)

The crystal structure of [Ni(salabza)] complex is shown in Fig. 2. The crystallographic and refinement data for complex **3** are summarized in Table 1, and selected bond distances and angles are listed in Table 2.

Complex **3** crystallizes in monoclinic space group P21/c in this complex, the Schiff base ligand acts as tetradentate chelating ligand coordinated to Ni(II) center. The angle O–Ni–N = 164.35(4)° indicates that the coordination geometry of the nickel atom is distorted square planar. The two halves of this complex are folded with respect to each other with an angle of 21.18°. The average Ni–O and Ni–N bond lengths are 1.845(2) and 1.885(9) Å, in agreement with the corresponding distances reported for the related Ni(II) complexes [39].

#### Spectral characterization

The prepared metal complexes are all air-stable solids and have good elemental analyses. The FT-IR data of the ligands and their complexes are listed in "Experimental setup". The FT-IR spectra of the free ligands exhibit the characteristic bands of two different imine (C=N) groups in the unsymmetrical Schiff base, which appear at 1,607 and  $1,571 \text{ cm}^{-1}$  for H<sub>2</sub>Me-salabza and at 1,633 and 1,614 cm<sup>-1</sup> for H<sub>2</sub>salabza. Similar features are observed in the IR spectra of the **1–3** complexes, with the C=N stretching vibrations appearing in the 1,567–1,598 cm<sup>-1</sup> region. The v(C=N) bands are shifted to lower wave numbers with respect to the free ligand, indicating a decrease in the C=N bonds order due to the coordination of the imine nitrogen to the metal and back bonding from the metal to the  $\pi^*$  orbital of the azomethine groups.

The UV-visible spectra of the ligands and their complexes were recorded in dichloromethane solution in the 200–800 nm region. The UV-Vis data of compounds are presented in "Experimental setup". The absorption spectra of H<sub>2</sub>Me-salabza and H<sub>2</sub>salabza ligands consist of an intense band centered at 324 nm, attributing to  $n \rightarrow \pi^*$  transitions. Further, two intense bands at 257 and 229 nm for H<sub>2</sub>Mesalabza and at 261 and 231 nm for H<sub>2</sub>salabza were related to  $\pi \rightarrow \pi^*$  transitions of aromatic rings and the azomethine groups. These transitions are also found in the spectra of the complexes, but they shifted toward lower frequencies, confirming the coordination of the ligand to the metal ion.

The UV-visible spectra of the Ni(II) complexes show two absorption bands corresponding to  ${}^{1}A_{1g} \rightarrow {}^{1}B_{1g}$  and  ${}^{1}A_{1g} \rightarrow {}^{1}A_{2g}$  transitions and appear at 600 and 438 nm for [Ni(Me-salabza)], and 614 and 448 nm for [Ni(salabza)]. The blue shift in the position of the ligand field transitions in [Ni(Me-salabza)] relative to the [Ni(salabza)] is due to the higher ligand field strength of Me-salabza as compared to that of salabza, originating from the inductive electrondonating properties of methyl substituents. The copper(II) complex shows two bands at 572 and 344 nm, assignable to  ${}^{2}B_{1g} \rightarrow {}^{2}A_{1g}$  and  ${}^{2}B_{1g} \rightarrow {}^{2}B_{2g}$  transitions [40]. These data are indicative of an approximate square planar geometry of the ligand around the Ni(II) and Cu(II) ions.

The <sup>1</sup>H NMR spectral measurements were performed in CDCl<sub>3</sub> solution for H<sub>2</sub>Me-salabza and H<sub>2</sub>salabza ligands and the corresponding data are given in "Experimental setup". The chemical formula of ligands is presented in Scheme 1. The main features of the <sup>1</sup>H NMR spectra of the unsymmetrical Schiff base ligands are the signals due to two different CH<sub>imine</sub> groups in H<sub>2</sub>salabza appearing at 8.47 and 8.58 ppm and the signals due to two different CH<sub>3</sub> substituents in H<sub>2</sub>Me-salabza appearing at 2.28 and 2.32 ppm. The enolic signals appear at 14.50 and 16.14 ppm for H<sub>2</sub>Me-salabza and at 13.13 and 13.35 ppm for H<sub>2</sub>salabza. The aromatic protons of these compounds appear in the appropriate region (5.8–7.8 ppm).

### Electrochemical studies

The cyclic voltammograms of the [Ni(Me-salabza)] and [Ni(salabza)] complexes in CH<sub>2</sub>Cl<sub>2</sub> solution are shown in

Figs. 3, 4 respectively. The irreversible oxidation process observed at 1.03 and 1.285 V is attributed to the oxidation of the Ni<sup>II</sup>/Ni<sup>III</sup> center in the [Ni(Me-salabza)] and [Ni(salabza)] complexes, respectively. The oxidation potential in [Ni(Me-salabza)] is less positive than that of the [Ni(salabza)].

The cyclic voltammogram of the [Cu(Me-salabza)] complex in  $CH_2Cl_2$  solution is shown in Fig. 5. The quasireversible reduction process observed at -1.20 V is attributed to the reduction of the Cu<sup>II</sup>/Cu<sup>I</sup> center. The reduction potential for Cu<sup>II</sup>/Cu<sup>I</sup> in [Cu(salabza)] is -1.014 V [33], which is less negative than that of the [Cu(Me-salabza)].

The variation in the redox potentials of Me-salabza complexes relative to those of salabza analogs correlates



Fig. 3 Cyclic voltammogram of the [Ni(Me-salabza)] in CH<sub>2</sub>Cl<sub>2</sub> at 298 K, c  $\approx 4 \times 10^{-3}$ , scan rate = 100 mV s<sup>-1</sup>



Fig. 4 Cyclic voltammogram of the [Ni(salabza)] in CH\_2Cl\_2 at 298 K, c  $\approx\!\!4\times10^{-3},$  scan rate = 100 mV s^{-1}



Fig. 5 Cyclic voltammogram of the [Cu(Me-salabza)] in CH<sub>2</sub>Cl<sub>2</sub> at 298 K, c  $\approx 4 \times 10^{-3}$ , scan rate = 100 mV s<sup>-1</sup>

well with the shifts observed in the energies of the ligand field transitions in these complexes. The inductive effect of the methyl groups apparently renders the Me-salabza a stronger electron donor which in turn leads to a blue shift in the position of the LF bands and ease of oxidation or harder reduction of the metal centers.

## Conclusions

In this work, we have reported the synthesis of new Ni(II) and Cu(II) complexes with two unsymmetric Schiff base ligands,  $H_2$ Me-salabza and  $H_2$ salabza. The crystal structures of [Cu(Me-salabza)] and [Ni(salabza)] complexes have been determined by single crystal X-ray diffraction. Both copper(II) and nickel(II) ions adopt a distorted square planar geometry in these complexes. The inductive effect of the methyl substituents in making Me-salabza a stronger ligand relative to salabza is documented, and on this basis, the correlation between the variation in the redox potentials and the shift in the position of the ligand field bands is rationalized.

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