

## Short Note

# *N,N'*-4,5-Dimethoxy-1,2-phenylenebis(salicylideneiminato)nickel(II)

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**Abstract:** The title compound, which is potentially interesting as a building block for electrochemically active metallopolymer, was synthesized and characterized by single-crystal X-ray diffraction, IR and NMR spectroscopies.

**Keywords:** crystal structure; 4,5-dimethoxy-1,2-phenylenediamine; N<sub>2</sub>O<sub>2</sub> type Schiff bases; nickel(II) complexes



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## 1. Introduction

Nickel complexes with tetradentate N<sub>2</sub>O<sub>2</sub> Schiff bases are used as monomers to prepare polymeric functional materials for advanced electrochemical energy storage devices, electrocatalytic systems, sensors, and electrochromic devices [1–6]. The properties of these materials depend on the presence of electron-donating or electron-withdrawing substituents in the Schiff base [7–10] and on the structure of the metal complex [7,8,11]. The 1,2-phenylenediimine bridging unit of the Salphen-type Schiff base complexes is a prospective structural site for modulating properties that are insufficiently explored in the literature.

Introducing vicinal hydroxyl groups in *para* positions to imine units of 1,2-phenylenediimine provide additional metal binding sites in Salphen-type complexes [12]. Crown-etherification of these sites further tunes the coordination ability of complexes [13,14]. Etherification of the hydroxyls has been reported with the intent of solubility enhancement for supramolecular [15] and polymeric [16] systems and for control of the supramolecular assembly of tris(salphen)-type trinickel(II) metallocryptands encapsulated a guanidinium ion [17] or alkali metal cations [18]. Oxidative chemistry of protected (unpolymerizable) nickel complex with a doubly methoxylated bridging unit of the Salphen complex has also been reported [19].

The structure of the simplest dimethoxylated Salphen nickel complex has not been reported so far. Thus, we perform a synthesis and investigations of structural and spectral properties of the novel complex *N,N'*-(4,5-dimethoxy-1,2-phenylene)bis(salicylideneaiminato)-nickel(II) ([Ni(Salphen(CH<sub>3</sub>O)<sub>2</sub>)] bearing two strongly donating methoxy substituents in the bridging phenylenediimine unit as a first step in exploring the influence of such monomer structural modification on metallopolymer electrochemistry.

## 2. Results and Discussion

### 2.1. X-ray Structural Analysis

The crystal structures of the [Ni(Salphen(CH<sub>3</sub>O)<sub>2</sub>)] was determined by the X-ray structural analysis. Samples of the complex suitable for studying were isolated by crystallization from a saturated acetonitrile solution (Avantor Performance Materials).

According to the X-ray diffraction data, the compound under study crystallizes in two forms (**a** and **b**) that differ in the way of the complex molecule solvation by acetonitrile (Figure 1) (Table 1).

**Table 1.** Crystallographic data, experimental parameters, and refinement of the [NiSalphen(CH<sub>3</sub>O)<sub>2</sub>] $\cdot$ CH<sub>3</sub>CN complex structure.

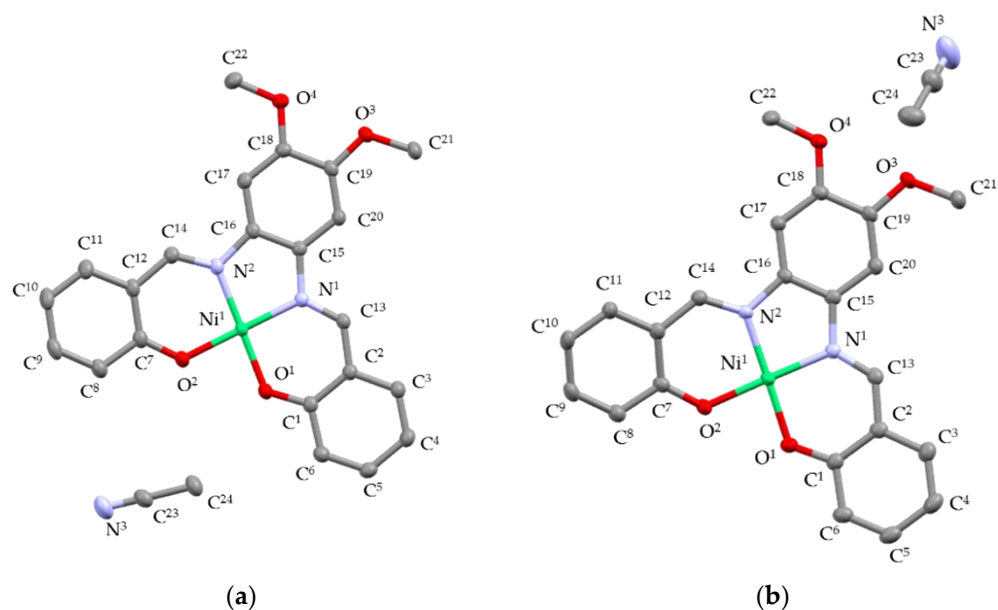
Parameter	Value	
Structure	<b>a</b>	<b>b</b>
Empirical formula	C <sub>24</sub> H <sub>21</sub> NiN <sub>3</sub> O <sub>4</sub>	C <sub>24</sub> H <sub>21</sub> NiN <sub>3</sub> O <sub>4</sub>
M	474.15	474.15
Temperature/K	100(2)	100(2)
Crystal system	monoclinic	trigonal
Space group	<i>P</i> 2 <sub>1</sub> / <i>n</i>	<i>R</i> 3 <i>c</i>
<i>a</i> /Å	7.95760(10)	38.7229(7)
<i>b</i> /Å	12.98650(10)	38.7229(7)
<i>c</i> /Å	20.1006(2)	7.2027(2)
$\alpha$ /°	90	90
$\beta$ /°	99.9830(10)	90
$\gamma$ /°	90	120
Volume/Å <sup>3</sup>	2045.77(4)	9353.2(4)
<i>Z</i>	4	18
$\rho_{calc}$ /g/cm <sup>3</sup>	1.539	1.515
$\mu$ /mm <sup>−1</sup>	1.694	1.668
<i>F</i> (000)	984.0	4428.0
Crystal size/mm	0.1 × 0.08 × 0.05	0.16 × 0.04 × 0.02
Radiation/ $\lambda$ /Å	CuK $\alpha$ 1.54184	CuK $\alpha$ (1.54184)
2 $\Theta$ range for data collection/°	8.142–138.212	4.564 to 139.994
Index ranges	−9 ≤ <i>h</i> ≤ 9, −10 ≤ <i>k</i> ≤ 15, −24 ≤ <i>l</i> ≤ 24	−46 ≤ <i>h</i> ≤ 32, −47 ≤ <i>k</i> ≤ 41, −8 ≤ <i>l</i> ≤ 8
Reflections collected	11517	18976
Independent reflections	3812 [ <i>R</i> <sub>int</sub> = 0.0261, <i>R</i> <sub>sigma</sub> = 0.0245]	3872 [ <i>R</i> <sub>int</sub> = 0.0453, <i>R</i> <sub>sigma</sub> = 0.0347]
GOOF by <i>F</i> <sup>2</sup>	1.074	1.039
<i>R</i> factors [ <i>I</i> > 2 $\sigma$ ( <i>I</i> )]	<i>R</i> <sub>1</sub> = 0.0278, <i>wR</i> <sub>2</sub> = 0.0721	<i>R</i> <sub>1</sub> = 0.0310, <i>wR</i> <sub>2</sub> = 0.0773
<i>R</i> factors [all reflections]	<i>R</i> <sub>1</sub> = 0.0304, <i>wR</i> <sub>2</sub> = 0.0737	<i>R</i> <sub>1</sub> = 0.0325, <i>wR</i> <sub>2</sub> = 0.0783
Largest diff. peak/hole, e Å <sup>−3</sup>	0.23/−0.29	0.29/−0.28

Figures 1 and 2 represent the molecular structures of the complex structures **a** and **b**. The Ni atom in the complex is bound to two phenolate oxygen atoms and two nitrogen atoms located at the vertices of a distorted square. The distortion degree can be characterized with the values of the torsion angles Ni<sup>1</sup>O<sup>1</sup>C<sup>1</sup>C<sup>2</sup> [3.1(2)°] (structure **a**), [12.5(5)°] (**b**) and Ni<sup>1</sup>N<sup>2</sup>C<sup>14</sup>C<sup>12</sup> [0.6(2)°] (**a**), [5.5(5)°] (**b**). The values of the torsion angles show that the distortion of structure (**b**) (Figure 2b) is higher than structure (**a**) (Figure 2a).

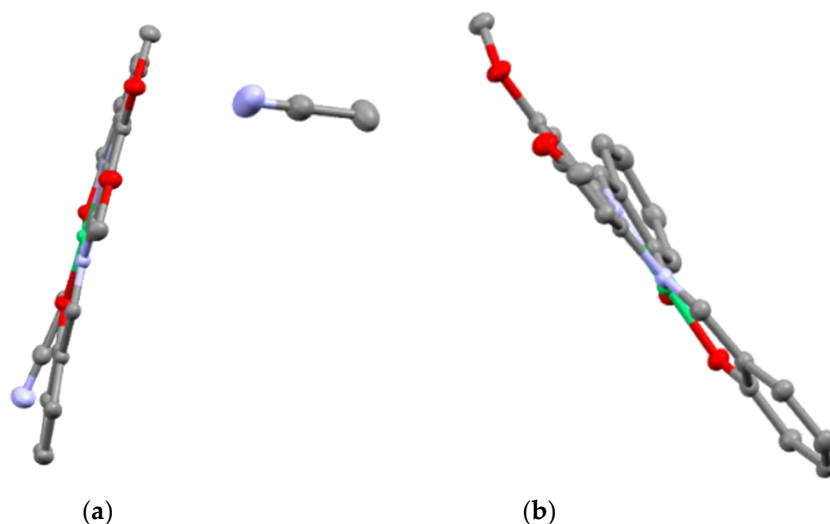
At the same time, the bond angles characterizing nickel bonds with donor atoms are approximately the same for both structures: O<sup>1</sup>Ni<sup>1</sup>N<sup>2</sup> [179.47(5)°] (**a**) and [179.17(12)°] (**b**), N<sup>1</sup>Ni<sup>1</sup>O<sup>2</sup> [178.57(5)°] (**a**) and [179.03(11)°] (**b**).

The different nature of the solvation of structures (**a**) and (**b**), as well as a higher degree of distortion of the structure (**b**), lead to the following differences in crystal lattices: monoclinic (**a**) and trigonal (**b**), and a much larger unit cell volume of (**b**) (see Table 1).

In general, the [Ni(Salphen(CH<sub>3</sub>O)<sub>2</sub>)] complex, as well as most nickel complexes of the salen- and salphen types, has a distorted square-planar geometry, which suggests the possibility of obtaining on its basis functional polymeric materials for electrochemical devices.



**Figure 1.** Molecular view of  $[\text{Ni}(\text{Salphen}(\text{CH}_3\text{O})_2)] \cdot \text{CH}_3\text{CN}$  in representation of atoms with thermal ellipsoids ( $p = 50\%$ ), structures (a,b).



**Figure 2.** Comparison of the distortion of structures (a,b) with thermal ellipsoids at a 50% probability.

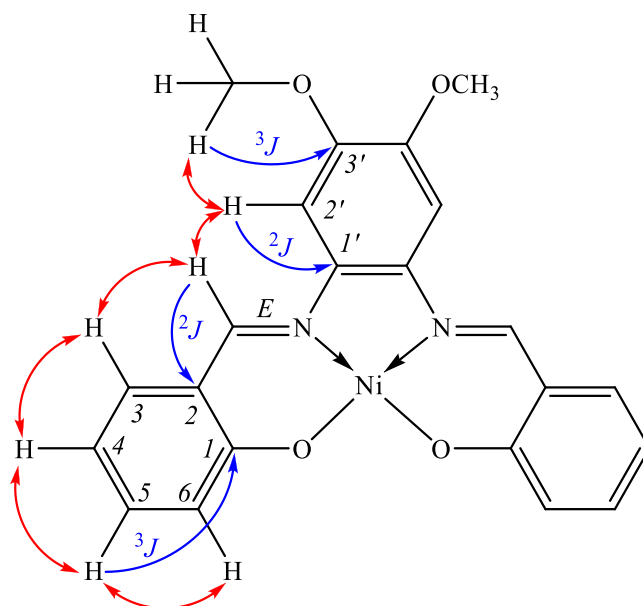
## 2.2. Infrared Spectroscopic and Nuclear Magnetic Resonance Studies

IR spectra (Figure S1) were registered on Shimadzu IRPrestige-21 spectrometers with samples in KBr pellets. The main experimental IR bands and their assignment are shown in Table 2.  $^1\text{H}$ ,  $^{13}\text{C}\{-^1\text{H}\}$  NMR spectra (Figures S2 and S3),  $^1\text{H}\text{--}^{13}\text{C}$  HMQC (Figure S4),  $^1\text{H}\text{--}^{13}\text{C}$  HMBC (Figure S5),  $^1\text{H}\text{--}^1\text{H}$  dqf-COSY (Figure S6) as well as  $^1\text{H}\text{--}^1\text{H}$  NOESY (mixing time from 0.5 to 2 s) (Figure S7) experiments were acquired on a Jeol ECX400A spectrometer (400 MHz for  $^1\text{H}$  nuclei and 100 MHz for  $^{13}\text{C}$  nuclei) in  $\text{DMSO-}d_6$ . The residual signals of the solvent ( $\text{DMSO-}d_6$ : 2.50 ppm for  $^1\text{H}$  nuclei and 39.6 ppm for  $^{13}\text{C}$  nuclei) were used as internal standard.

$^1\text{H}$  NMR: 3.83 (3H,  $\text{OCH}_3$ ), 6.61 (1H, ddd,  $\text{H}^4$ ,  $^3J$  7.9, 6.8,  $^4J$  0.9 Hz), 6.82 (1H, br.d,  $\text{H}^6$ ,  $^3J$  8.5 Hz), 7.23 (1H, ddd,  $\text{H}^5$ ,  $^3J$  8.5, 6.8,  $^4J$  1.7 Hz), 7.52 (1H, dd,  $\text{H}^3$ ,  $^3J$  7.9,  $^4J$  1.7 Hz), 7.59 (1H, s,  $\text{H}^{2'}$ ), 8.64 (1H, s,  $\text{CH}=\text{N}$ ) (Atom labeling shown on Figure 3).

**Table 2.** IR band assignment according to [20].

IR band, $\text{cm}^{-1}$	Assignment
<i>ligand core molecule</i>	
1585–1608	C = N str in azomethine and C–C str in phenolic ring
1518	C–C str in phenolic ring
1450–1465	C–O, C=N and C–C str in phenolic and six-membered chelate ring
1367	C = N in six-membered chelate ring and C–C str in phenolic ring
1333	C–O and C–C str in phenolic ring
1244	C–C str in phenolic and six-membered chelate ring
1109–1189	C–N str in five-membered chelate ring
729–753	ring vib and C–H out-of-plane def in phenolic ring
<i>metal chelate</i>	
594	asym O–Ni–O str
459	asym N–Ni–N str
<i>substituents in phenolate moieties</i>	
2831	sym and asym C–H str in $-\text{O}-\text{CH}_3$
1090–1100	sym C–O–C str in $\text{phenyl}-\text{O}-\text{CH}_3$
<i>diimine backbone</i>	
3042–3012	sym and asym C–H str in <i>o</i> -phenylene
1146	C–H in-plane def in <i>o</i> -phenylene
753	ring vib and C–H out-of-plane def in <i>o</i> -phenylene

**Figure 3.** Key correlations in  $^1\text{H}$ - $^{13}\text{C}$  HMBC (blue arrows) and  $^1\text{H}$ - $^1\text{H}$  NOESY (double-edged red arrows) spectra.

$^{13}\text{C}\{^1\text{H}\}$  NMR: 56.73 ( $\text{OCH}_3$ ), 99.05 ( $\text{C}^{2'}$ ), 115.58 ( $\text{C}^4$ ), 120.57 ( $\text{C}^6$ ), 120.91 ( $\text{C}^2$ ), 134.29 ( $\text{C}^3$ ), 135.10 ( $\text{C}^5$ ), 136.12 ( $\text{C}^{1'}$ ), 149.75 ( $\text{C}^{3'}$ ), 155.20 ( $\text{C}=\text{N}$ ), 164.95 ( $\text{C}^1$ ).

Assignment of signals of protons and carbon atoms in  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra was carried out using homo- ( $^1\text{H}$ - $^1\text{H}$  COSY,  $^1\text{H}$ - $^1\text{H}$  NOESY) and heteronuclear ( $^1\text{H}$ - $^{13}\text{C}$  HMQC,  $^1\text{H}$ - $^{13}\text{C}$  HMBC) experiments. Notably, the presence of cross peaks in the  $^1\text{H}$ - $^1\text{H}$  COSY spectrum due to long-range spin–spin interactions through 5 bonds ( $\text{CH}_3/\text{H}^{2'}$ ,  $\text{H}^{2'}/\text{CH}=\text{N}$ ,  $\text{CH}=\text{N}/\text{H}^6$ ), as well as typical for *ortho*-substituted benzene ring *J*-coupling constant through 4 bonds ( $\text{H}^3/\text{H}^5$ ,  $\text{H}^4/\text{H}^6$ ). The key cross peaks used for interpretation in the  $^1\text{H}$ - $^{13}\text{C}$  HMBC spectrum are 3.83 ( $\text{OCH}_3$ )/149.75 ( $\text{C}^{3'}$ ); 7.59 ( $\text{H}^{2'}$ )/136.12 ( $\text{C}^{1'}$ ); 8.64 ( $\text{CH}=\text{N}$ )/120.91 ( $\text{C}^2$ ); 7.52 ( $\text{H}^3$ )/155.20 ( $\text{C}=\text{N}$ ); 7.23 ( $\text{H}^5$ )/164.95 ( $\text{C}^1$ ) (Figure 3).

Analysis of the  $^1\text{H}$ - $^1\text{H}$  NOESY spectrum (mixing time variation) indicates the planar structure of the complex. Thus, the cross-peaks  $\text{H}^{2'}/\text{CH}=\text{N}$ ,  $\text{CH}=\text{N}/\text{H}^3$ , due to the

nuclear Overhauser effect, indicate the spatial proximity of the azomethine proton simultaneously with two protons of different aromatic rings, which is possible with the coplanar organization of the (*E*)-azomethine block.

### 3. Materials and Methods

All chemicals used in the synthesis were of “reagent-grade” purity and were purchased from local suppliers.

4,5-Dimethoxy-1,2-phenylenediamine was obtained in two steps, as reported in [21]. 1,2-Dimethoxybenzene was converted into 4,5-dimethoxy-1,2-dinitrobenzene (86% yield) by the reaction with concentrated nitric acid (65%) followed by the reduction with hydrazine monohydrate and Pd/C catalyst yielding 4,5-dimethoxy-1,2-phenylenediamine (55% yield).

The ligand was prepared by the standard method of refluxing an ethanolic solution containing salicylaldehyde (Aldrich) and a 4,5-dimethoxy-1,2-phenylenediamine in stoichiometric amounts (95% yield). Nickel(II) complex [Ni(Salphen(CH<sub>3</sub>O)<sub>2</sub>)] was prepared by refluxing ethanolic solutions of nickel(II) acetate (Aldrich) with the Schiff base ligand, as described in [22]. Obtained complexes were recrystallized from acetonitrile and dried at 60 °C for several hours (70% yield).

X-ray diffraction analysis was performed at 100 K on a XtaLAB Synergy-S diffractometer (Rigaku, Japan) equipped with a HyPix-6000HE CCD detector (Rigaku, Tokyo, Japan), CuK $\alpha$  radiation ( $\lambda$  1.54184 Å). The structure was solved using the ShelXT-2013 software package [23] and refined using the ShelXL-2013 package [24] included in the OLEX2 interface [25]. The crystallographic parameters have been deposited in the Cambridge X-Ray Database (CCDC 2189692-2189693).

**Supplementary Materials:** Figure S1: IR spectrum of *N,N'*-4,5-dimethoxy-1,2-phenylenebis(salicylideneiminato)nickel(II) in KBr; Figure S2: <sup>1</sup>H NMR spectrum of *N,N'*-4,5-dimethoxy-1,2-phenylenebis(salicylideneiminato)nickel(II) in DMSO-*d*<sub>6</sub>; Figure S3: <sup>1</sup>H{<sup>13</sup>C} NMR spectrum of *N,N'*-4,5-dimethoxy-1,2-phenylenebis(salicylideneiminato)nickel(II) in DMSO-*d*<sub>6</sub>; Figure S4: <sup>1</sup>H-<sup>13</sup>C HMQC spectrum of *N,N'*-4,5-dimethoxy-1,2-phenylenebis(salicylideneiminato)nickel(II) in DMSO-*d*<sub>6</sub>; Figure S5: <sup>1</sup>H-<sup>13</sup>C HMBC spectrum of *N,N'*-4,5-dimethoxy-1,2-phenylenebis(salicylideneiminato)nickel(II) in DMSO-*d*<sub>6</sub>; Figure S6: <sup>1</sup>H-<sup>1</sup>H dqf-COSY spectrum of *N,N'*-4,5-dimethoxy-1,2-phenylenebis(salicylideneiminato)nickel(II) in DMSO-*d*<sub>6</sub>; Figure S7: <sup>1</sup>H-<sup>1</sup>H NOESY spectrum of *N,N'*-4,5-dimethoxy-1,2-phenylenebis(salicylideneiminato)nickel(II) in DMSO-*d*<sub>6</sub>. CCDC 2189692-2189693 contains 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 CCDC, 12 Union Road, Cambridge CB2 1EZ, UK; Fax: +44 1223 336033; E-mail: [deposit@ccdc.cam.ac.uk](mailto:deposit@ccdc.cam.ac.uk)).

**Author Contributions:** Conceptualization, M.K. A.T. and E.S.; synthesis, N.V. and E.S.; methodology, M.K.; investigation, R.B. and D.S.; writing—original draft preparation, E.S., A.T. and R.B.; writing—review and editing, M.K. All authors have read and agreed to the published version of the manuscript.

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**Conflicts of Interest:** The authors declare no conflict of interest.

**Sample Availability:** Samples of the compounds are available from the authors upon request.

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