



Short Note

N,N'-1,2-Phenylene-bis(3-tert-butylsalicylideneiminato)nickel (II)

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Abstract: A nickel complex with a Schiff base Ni(3-tert-butyl-salophen) was synthesized and structurally characterized by single-crystal X-ray diffraction, ¹H, ¹³C{¹H}, ¹H-¹³C HMQC, ¹H-¹³C HMBC, ¹H-¹H dqc-COSY and ¹H-¹H NOESY NMR spectroscopy, and IR and UV-vis spectroscopy.

Keywords: N₂O₂ type Schiff bases; crystal structure; nickel(II) complexes; salophen; X-ray diffraction

1. Introduction

Schiff bases are compounds containing an imine or azomethine functional group formed by the reaction of a primary amine with an aldehyde or ketone [1–3]. They form stable complexes with various metal ions. They are commonly used as ligands in coordination chemistry due to their ability to form stable complexes with metal ions. Schiff base complexes have a wide range of applications, including catalysis [4–8], energy storage devices [9], sensors [10], and medicinal chemistry [11–14]. The catalytic activity strongly depends on the structure and properties of the complexes, particularly the presence of substituents in the ligand part of the molecule and the nature of the metal ion. One example of a Schiff base ligand is salophen, a tetradeятate ligand containing a central aromatic ring substituted with two phenolic groups via the azomethine linkers. Salophen ligands have been extensively studied due to their unique properties, including high selectivity and catalytic activity. Recently, there has been increasing interest in the synthesis and characterization of sterically hindered Schiff base complexes [15–18]. The introduction of bulky tert-butyl groups into the ortho- and para-positions of the ligand rings provides efficient enantioselective catalysis, affects the coordination geometry of the metal center, and can change the properties of the formed complex [15]. Crystal structure determination is an important tool for the characterization of sterically hindered Schiff base complexes, because it provides valuable information on the impact of steric hindrance on the structure and properties of metal complexes. The structures of tert-butyl-substituted salophen complexes reported previously include relatively simple ortho- and para-di-tert-butyl- or ortho-tert-butyl- and other functional groups para-substituted complexes [19–24], di-tert-butyl-substituted crown ethers bearing complexes [25,26], as well as more sophisticated supramolecular structures [27–32]. The structures of complexes containing tert-butyl-substituents in the ortho- and para-positions of the phenyl rings of the ligand and various substituents the bridge, such as HOOC-, CH₃-, CH₃O-, N(CH₃)₂-, have also been described [21,24,30,31]. Introduction of N(CH₃)₂ substituent in the para-position of the ligand moiety leads to monoclinic packing [23], while the complex with CH₃-groups in phenyl bridge leads to triclinic packing [21]. Meanwhile, the simplest ortho-tert-butyl-salophen Ni complex has not been structurally characterized yet. In this article, we will focus on the synthesis and NMR



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(nuclear magnetic resonance), FTIR (Fourier-transform infrared spectroscopy), and UV-VIS (ultraviolet and visible) characterization of a new Ni(salophen) complex that contains a sterically hindered Schiff base ligand. This study will contribute to our understanding of the properties of sterically hindered Schiff base complexes and may have important implications for the design of new catalysts and materials.

2. Results

2.1. X-ray Structural Analysis

The crystal structure of the Ni(3-tert-butyl-salophen) was determined by X-ray diffraction. Brick red needle-like crystals were grown from a saturated acetonitrile solution by slow solvent evaporation.

The title compound crystallizes in the orthorhombic space group *Pbca*. Figure 1 presents the molecular structures of Ni(3-tert-butyl-salophen). Nickel with two oxygen and two nitrogen atoms form a square-planar coordination skeleton. The distortion of the square-planar geometry for the Ni(3-t-butyl-salophen) is low and can be characterized by torsion angles Ni¹-O¹-C²⁴-C¹⁹ (1.5(2) $^{\circ}$) and Ni¹-N²-C⁷-C⁸ (10.9(2) $^{\circ}$) and by valence angles O¹-Ni¹-N² (176.05(6) $^{\circ}$) and N¹-Ni¹-O² (175.57(6) $^{\circ}$).

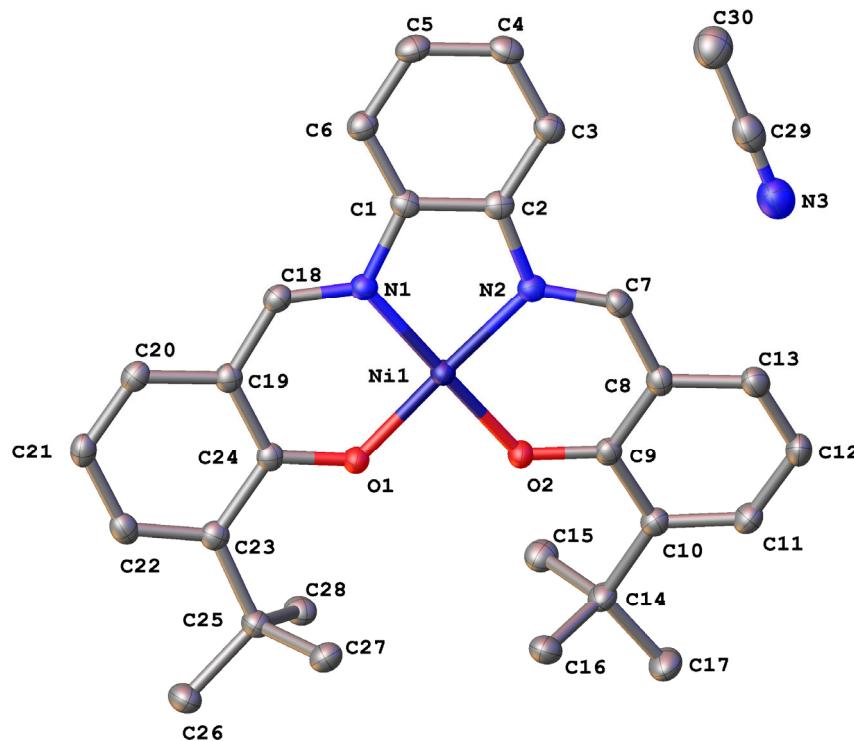


Figure 1. Molecular view of the Ni(3-tert-butyl-salophen) \times CH₃CN in representation of atoms with thermal ellipsoids ($p = 50\%$).

The packing of Ni(3-tert-butyl-salophen) is quite compact (Figure 2) with the closest Ni-Ni distance of 4.706 Å. As anticipated, the bulky complex is packed into a crystal without any stacking motif. The steric bulkiness induced by the tert-butyl groups in the phenol rings prevents close molecular contacts in the crystal.

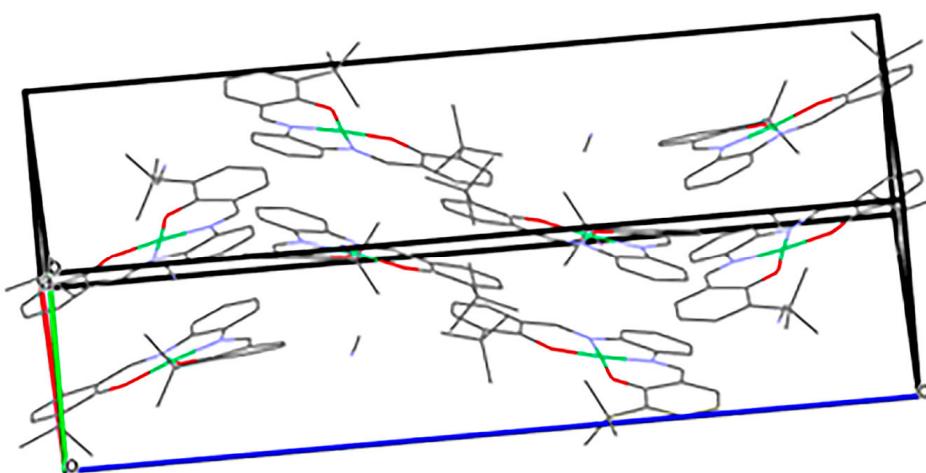


Figure 2. Packing of the Ni(3-tert-butyl-Salophen) \times CH₃CN in crystals.

Crystallographic data, experimental parameters, and refinement of the complex structure are shown in Table 1.

Table 1. Crystallographic data, experimental parameters, and refinement of the Ni(3-tert-butyl-Salophen) \times CH₃CN complex structure.

Parameter	Value
Empirical formula	C ₃₀ H ₃₃ N ₃ NiO ₂
Molecular weight	526.30
Temperature/K	100(2)
Crystal system	orthorhombic
Space group	Pbca
<i>a</i> /Å	24.9825(6)
<i>b</i> /Å	6.9558(2)
<i>c</i> /Å	29.1851(7)
Volume/Å ³	5071.6(2)
<i>Z</i>	8
ρ_{calc} /g/cm ³	1.379
μ/mm^{-1}	1.357
<i>F</i> (000)	2224.0
Crystal size/mm	0.16 \times 0.02 \times 0.02
Radiation/λ/Å	CuKα (1.54184)
2Θ range for data collection/°	6.056–139.978
Index ranges	$-30 \leq h \leq 29, -8 \leq k \leq 4,$ $-35 \leq l \leq 3$
Reflections collected	18,181
Independent reflections	4709 [$R_{\text{int}} = 0.0344$, $R_{\text{sigma}} = 0.0323$]
GOOF on F^2	1.057
<i>R</i> factors [$I \geq 2\sigma(I)$]	$R_1 = 0.0334, wR_2 = 0.0897$
<i>R</i> factors [all reflections]	$R_1 = 0.04124, wR_2 = 0.0944$
Largest diff. peak/hole, e Å ⁻³	0.31/−0.28

2.2. Infrared Spectroscopic and Nuclear Magnetic Resonance Studies

IR spectra (Figure S1) were registered on a Shimadzu IR Prestige-21 spectrometer with samples in KBr pellets. The main experimental IR bands and their assignment are given in Table S1. ¹H, ¹³C NMR spectra (Figures S2 and S3), ¹H–¹³C HMQC (Figure S4), ¹H–¹³C HMBC (Figure S5), ¹H–¹H dqc-COSY (Figure S6), as well as NOESY (mixing time from 0.5 to 2 s) (Figure S7) experiments were performed on a Jeol ECX400A spectrometer (400 MHz for ¹H nuclei and 100 MHz for ¹³C nuclei) in DMSO-*d*6. Residual solvent

signals ($\text{DMSO}-d_6$: 2.50 ppm for ^1H nuclei and 39.6 ppm for ^{13}C nuclei) were used as an internal standard.

^1H NMR: 1.36 (9H, s, 3CH_3), 6.58 (1H, t, H^4 , 3J 7.6 Hz), 7.23 (1H, dd, H^5 , 3J 7.2, 4J 1.5 Hz), 7.28 (1H, dd, $\text{H}^{3'}$, 3J 6.2, 4J 3.2 Hz), 7.47 (1H, dd, H^3 , 3J 7.9, 4J 1.5 Hz), 8.12 (1H, dd, $\text{H}^{2'}$, 3J 6.2, 4J 3.2 Hz), 8.78 (1H, s, $\text{CH}=\text{N}$) (Atom labeling is shown in Figure 3).

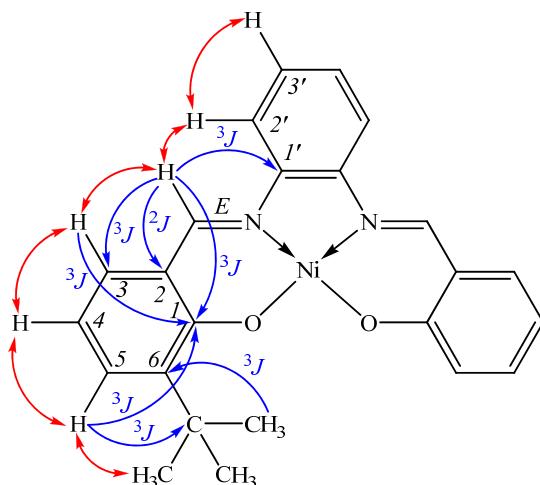


Figure 3. Key correlations in ^1H - ^{13}C HMBC (blue arrows) and ^1H - ^1H NOESY (red double-edged arrows) spectra.

$^{13}\text{C}\{^1\text{H}\}$ NMR: 30.06 [$\text{C}(\text{CH}_3)_3$], 35.77 [$\underline{\text{C}}(\text{CH}_3)_3$], 115.40 (C^4), 116.51 ($\text{C}^{2'}$), 121.12 (C^2), 127.80 ($\text{C}^{3'}$), 131.88 (C^5), 133.44 (C^3), 140.31 (C^6), 142.93 (C^1), 157.32 ($\text{C}=\text{N}$), 165.11 (C^1).

The assignment of signals of protons and carbon atoms in ^1H and ^{13}C NMR spectra was carried out using homonuclear (^1H - ^1H COSY, ^1H - ^1H NOESY) and heteronuclear (^1H - ^{13}C HMQC, ^1H - ^{13}C HMBC) experiments. Noteworthy is the presence of cross peaks in the ^1H - ^1H COSY spectrum due to long-range spin-spin interactions through five bonds (CH_3/H^5 , $\text{CH}=\text{N}/\text{H}^{2'}$, $\text{CH}=\text{N}/\text{H}^3$), as well as the characteristic of ortho-substituted benzene ring J -coupling constant (4J 1.5 Hz) through four bonds (H^3/H^5). The key cross peaks used for interpretation in the ^1H - ^{13}C HMBC spectra were 1.36 (CH_3)/140.31 (C^6); 8.78 ($\text{CH}=\text{N}$)/142.93 (C^1)/121.12 (C^2)/133.44 (C^3)/165.11 (C^1); 7.47 (H^3)/157.32 ($\text{C}=\text{N}$); 7.23 (H^5), 7.47 (H^3)/165.11 (C^1); 7.23 (H^5)/35.77 [$\underline{\text{C}}(\text{CH}_3)_3$] (Figure 3).

Analysis of the ^1H - ^1H NOESY spectrum (mixing time variation) indicates the planar structure of the complex. Thus, the cross peaks $\text{CH}=\text{N}/\text{H}^{2'}$ (a very important cross peak for identifying the $\text{H}^{2'}$ proton) and $\text{CH}=\text{N}/\text{H}^3$, 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.

2.3. UV-Vis Spectroscopic Studies

The spectrum of the Ni(3-tert-butyl-Salophen) complex (Figure S8) contains three main bands: 263, 378, and 475 nm (Table S2). The absorption band of the complex at 263 nm is due to the $\pi \rightarrow \pi^*$ transition of benzene. Another band at 378 nm is attributed to the $n \rightarrow \pi^*$ transition of the non-bonding electron located on the azomethine nitrogen atom of the ligand. The bands 475 nm and the weakly resolved band at 563 nm correspond to π -d and d-d transitions in the nickel center [33,34].

3. Materials and Methods

All reagents were of commercial grade and were used without purification.

The complex Ni(3-tert-butyl-salophen) was prepared according to previously published procedures [35]. The ligand N,N' -1,2-phenylene-bis(3-tert-butylsalicylideneamine) was obtained by condensation of o-phenylenediamine with 3-tert-butylbenzaldehyde [18].

The nickel (II) complex Ni(3-tert-butyl-Salophen) was prepared by refluxing ethanolic solutions of Ni(II) acetate (Aldrich) with the ligand. The obtained complex was recrystallized from acetonitrile and dried at 60 °C for 12 h (70% yield).

X-ray diffraction analysis was performed at 100 K on a Rigaku XtaLAB Synergy-S diffractometer equipped with a HyPix-6000HE CCD detector with CuK α radiation (λ 1.54184 Å). The structure was solved using the ShelXT software package [36] and refined using the ShelXL package [37] included in the OLEX2 interface [38]. The crystallographic parameters were deposited in the Cambridge X-ray Database (CCDC 2236037).

4. Conclusions

N,N'-1,2-phenylene-bis(3-tert-butylsalicylideneiminato)nickel (II) was synthesized and characterized by NMR and by IR and UV-vis spectroscopy.

Supplementary Materials: The following supporting information can be downloaded online. Figure S1: IR spectrum of *N,N'*-1,2-phenylene-bis(3-tert-butyl-salicylideneiminato)nickel (II) in KBr; Table S1: The main experimental IR bands of the metal complexes Ni(3-tert-butyl-Salophen) and their assignment; Figure S2: 1H NMR spectrum of *N,N'*-1,2-phenylene-bis(3-tert-butyl-salicylideneiminato)nickel (II) in DMSO-d6; Figure S3: 1H{13C} NMR spectrum of *N,N'*-1,2-phenylene-bis(3-tert-butyl-salicylideneiminato)nickel (II) in DMSO-d6; Figure S4: 1H-13C HMQC spectrum of *N,N'*-1,2-phenylene-bis(3-tert-butyl-salicylideneiminato)nickel (II) in DMSO-d6; Figure S5: 1H-13C HMBC spectrum of *N,N'*-1,2-phenylene-bis(3-tert-butyl-salicylideneiminato)nickel (II) in DMSO-d6; Figure S6: 1H-1H dqf-COSY spectrum of *N,N'*-1,2-phenylene-bis(3-tert-butyl-salicylideneiminato)nickel (II) in DMSO-d6; Figure S7: 1H-1H NOESY spectrum of *N,N'*-1,2-phenylene-bis(3-tert-butyl-salicylideneiminato)nickel (II) in DMSO-d6. Figure S8: UV-vis spectrum of *N,N'*-1,2-phenylene-bis(3-tert-butyl-salicylideneiminato)nickel (II) in acetonitrile; Table S2: The main experimental UV-vis spectra maximums of the metal complexes [Ni(3-tert-butyl-Salophen)] and their assignment. CCDC 2236037 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via <http://www.ccdc.cam.ac.uk/structures>, accessed on 19 June 2023 (or from the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK; Fax: +44 1223 336033; E-mail: deposit@ccdc.cam.ac.uk).

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

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