# The synthesis, crystal structure and spectroscopic properties of a dinuclear $\mu$–pyrazolato–*N*,*N'*–bridged dinickel(II) complex of 1,3–bis(saliscylideneamino)propan–2–ol

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# The synthesis, crystal structure and spectroscopic properties of a dinuclear $\mu$–pyrazolato–*N,N'*–bridged dinickel(II) complex of 1,3–bis(saliscylideneamino)propan–2–ol

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The title compound with formally pentadentate ligand L = 1,3-bis(saliscylidene amino)propan-2-ol), [$Ni_2$(L)(3,5 prz)], prz = pyrazolate was synthesized and identified using elemental analysis, IR spectroscopy and it is crystal structure determined by X-ray diffraction technique. The title compound crystallizes in monoclinic space group $P2_1/c$ with cell parameters a = 29.873(4), b = 11.131(2), c = 13.166(3) Å, β = 107.770(10)°, V = 4169.0(13) Å$^3$, Z = 4, $D_{cal}$ = 1.618 Mg/m$^3$. The nickel ions are bridged by the alkoxo group of the ligand and the N atoms of $\mu$-pyrazolate ($\mu$-prz) group. Each nickel(II) ion is coordinated by two N atoms two O atoms, forming a square with *trans*-$N_2O_2$ geometry. The Ni···Ni distance and Ni–O–Ni angle are 3.371(1) Å and 126.4(1)°, respectively.

## 1 Introduction

Binucleating Schiff base ligands are highly inclined to give homo and hetero polynuclear complexes with many transition metal ions and their complexes have been interest for many years [1-6]. This is partly because of the interest in relating structures to magnetic exchange effects in homo-and hetero-binuclear metal complexes of those ligands [7,8] and partly because of the use of such complexes to mimic aspects of two-metal biosites in various proteins and enzymes [9,10]. These complexes play an important role in the developing of coordination chemistry related to catalysis and enzymatic reactions, magnetism and molecular architectures [11-14]. In addition, these Schiff base complexes provide structural models for dinuclear sites in several proteins involved in oxygen storage of hemerytherin and oxygen activation of methanemonooxygenase [15-18]. Especially, dinuclear nickel(II) complexes play an important role in the structure and catalytic function of the active site of the nickel-containing enzyme urease [19, 20]. The biophysical and biochemical properties of nickel-containing urease from microbial organisms and plants have been extensively reviewed and dinuclear nickel(II) complexes have been reported so far as models for the nickel site of urease [21-31].

Although a large number of unsymmetric doubly-bridged dinuclear copper(II) complexes have been reported [32-38], relatively few structures of unsymmetric doubly-bridged nickel(II) complexes have been reported [39-41]. In the course of our studies on transition metal Schiff base complexes [32, 42-46] and in connection with these investigations, we have therefore synthesized and characterized a dinuclear nickel(II) complex bridged by an alkoxide and $\mu$-pyrazolate ($\mu$-prz) with a pentadentate Schiff-base ligand.

## 2 Experimantal

## Synthesis

All starting materials were of reagent grade and were used without further purification and purchased from the Aldrich Company. The Schiff base ligand was prepared by reaction of 1,3-diaminopropan-2-ol with salicylaldehyde (1:2 mol ratio) in methanol. The yellow Schiff bases precipitated from solution on cooling. The dinuclear Ni(II)complex was obtained when a sample of the ligand (1 mmol) in methanol (50 ml) was added drop wise to a stirred mixture containing 3,5-dimethylpyrazole (1 mmol) and nickel(II) perchlorate hexahydrate (2 mmol) in methanol (25 ml). Triethylamine (3 mmol) was added to the solution. The mixture was stirred and thin green crystals suitable for X-ray structure determination collected and washed with methanol. The chemical diagram for the complex is given in Fig. 1. *Caution!* Perchlorate salts of metal complexes with organic ligands are potentially explosive. Even small amounts of material should be handled with caution.

## Elemental Analysis

[$C_{22}H_{22}N_4Ni_2O_3$] (507.86), M.P. > 310 $^{o}$C (decomposition), yield %45 (0.182 g)

Calculated %; C: 52.03, H: 4.36 , N: 11.03, Ni: 23.12

Found %; C:51.98, H: 4.42 , N:11.15, Ni: 23.20

## IR data

IR spectra were measured with a Perkin-Elmer Bx FT-IR instrument with the samples as KBr pellets in the 4000-400 cm$^{-1}$ range. Electronic spectra in acetone solutions in the 900 – 200 nm range were recorded on Perkin-Elmer Lambda 2.

## Data Collection and Reduction

A crystal of dimension 0.07x0.15x0.45 mm$^3$ was mounted on an Enraf-Nonius CAD-4 diffractometer [47] equipped with a graphite monochromatized MoK$_\alpha$ radiation ($\lambda$=0.71073 Å). Precise unit cell dimensions were determined by least-squares refinement on the setting angles of 25 reflections (2.41° ≤ $\theta$ ≤ 30.20°) carefully centered on the diffractometer. The standard reflections ($\bar{1}$ 1 2, $\bar{2}$ 2 2, 8 2 7) were measured every 7200 s and the orientation of the crystal was checked after every 600 reflections. A total of 5750 reflections were recorded, with Miller indices $h_{min}$ = - 41, $h_{max}$ = 39, $k_{min}$ = 0, $k_{max}$= 15, $l_{min}$ = 0, $l_{max}$ = 18. Data reduction and corrections for absorption and decomposition were achieved using the Nonius Diffractometer Control Software [47].

### Structure Solution and Refinement

The structure was solved by direct methods using the program SHELXS-97 [48] in the WinGX package [49] and refined by full matrix least square method using SHELXL-97 [48]. The positions of the H atoms bonded to C atoms were calculated (C-H distance 0.96 Å), and refined using a riding model, and H atom displacement parameters were restricted to be 1.2U$_{eq}$ of the parent atom. The crystal data, intensity data collection details and refinement parameters are summarized in Table 1. The final positional parameters are presented in Table 2. Fig. 1 shows the chemical diagram and a perspective ORTEP drawing of the molecule is shown in Fig. 2 [50]. Selected bond lengths and angles are summarized in Table 3 and Table 4. Crystallographic data (excluding structure factors) for the structure reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC–232690 [51].

## 3 **Results and Discussion**

## X-ray crystal structure

The title complex consists of dinuclear molecules in which two nickel atoms are linked by the alkoxide and $\mu$-pyrazolate nitrogen atoms. Each nickel(II) ion is coordinated by two nitrogen and two oxygen atoms forming a coordination plane. The average distances Ni–N and Ni–O are 1.971(1) and 1.874(1) Å, respectively. These distances are comparable of with the bond lengths reported in other nickel(II) complexes [52-56]. The Ni1–O2–Ni2 bridging angle is 126.4(1)° which are in the range of similar binuclear nickel(II) complexes [57, 58], but it is larger than the angle of the hydroxo bridge [25]. The distance between the two nickel(II) centres is 3.347(1) Å.

In the solid state, planar nickel(II) Schiff base complex have been found to appear in a "stepped" centrosymmetric conformation [59, 60-62]. Due to the non-planarity of the chelate rings in solution, two chiral "umbrella" conformations are also possible. In the title compound, the atoms with the greatest deviation from the coordination planes Ni1, N1, O1, N4, O2 and Ni2, N2, O3, N3, O2 are O2 atom at 0.116(1) Å and O2 atom at 0.127(1) Å, respectively. The maximum deviation from the plane defined by atoms O1, C1-C7, N1, C8 and C9 is 0.095(2) Å for the C9 atom, and for C9, C10, N2, C11-C17 and O3 it is –0.169(2) Å for the C9 atom. The deviation of C9 atom from the plane defined by atoms Ni1, O2, Ni2 and C9 is -0.013(1) Å. The maximum deviation from the same plane is 0.034(1) Å for the O2 atom. The dihedral angles between Ni1, N1, O1 and Ni2, N2, O3 planes with respect to the NiONi plane 6.9(1)° and 9.7(1)°. Another important feature is the geometry of the bridging O atom, O2, the bond angles around O2 of 117.1(1), 126.4(1) and 116.3(1)° indicate the pyramidal stereochemistry at this atom. The remaining five-membered rings are not planar, as seen from the torsion angles N1–C8–C9–O2 of 10.3(2) so, the whole molecule is not planar since the two halves are folded with respect to one another. The least-squares planes through each half of the molecule are inclined at an angle of 10.9(1)°, forming a shallow umbrella form as reported in other planar monomer nickel(II) structures [44, 63].

The packing diagram for the complex is shown in Fig. 3 as a along the c axis projection. The molecules parallel to b are stacked with each Ni atom almost directly above the other. The closest intermolecular Ni···Ni distance is 3.865(1) Å and there is an infinite Ni···Ni···Ni chain along the b axis.

Although, some octahedral coordination of nickel(II) complexes exhibit an intramolecular weak antiferromagnetic interaction or ferromagnetic interaction [64], the investigated compound is diamagnetic which is consistent with the planar geometry around the Ni(II) ions.

## Spectroscopic properties

The IR spectra of the free Schiff base ligand show a broad band at 3250-3420 $cm^{-1}$, which is likely to be a superposition of bands from alcohol-OH and phenol-OH. The $\gamma$(OH) band is absent in the IR spectra of the complex. This indicates that the alcholic and phenolic protons are lost upon complexation. The $\gamma$(C=N) band (ca. 1634 $cm^{-1}$) of the free ligand is shifted to a little lower frequency (ca. 1625 $cm^{-1}$) upon complexation, suggesting that the imino nitrogen is coordinated to the nickel ion [65]. The X-ray structure also confirms this coordination.

Electronic spectra of the complex show a strong band 330 nm is assigned to the intraligand charge transfer transitions ($\pi \rightarrow \pi^*$), moderately intense peak at 420 nm is due to ligand to metal charge transfer transitions and a weak band due to *d-d* transitions at around

550-650 nm, which is characteristic of the diamagnetic square planar Ni(II) complexes [66].

## Acknowledgements

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**Table 1.** Crystallographic data for the investigated complex

| | |
|---|---|
| CCDC Number | CCDC-232690 |
| Empirical formula | $C_{22}H_{22}N_4Ni_2O_3$ |
| Formula weight (g.mol$^{-1}$) | 507.86 |
| Wavelength (Å) | 0.71073 |
| Crystal system | Monoclinic |
| Space group | $P2_1/c$ |
| Unit cell dimension | |
| a = 29.873(4) Å | $\alpha = 90^o$ |
| b = 11.131(2) Å | $\beta = 107.77(1)^o$ |
| c = 13.166(3) Å | $\gamma = 90^o$ |
| Volume (Å$^3$) | 4169 (1) |
| Z | 4 |
| Calculated density (Mg.m$^{-3}$) | 1.618 |
| Absorption coefficient (mm$^{-1}$) | 1.839 |
| F(000) | 2096 |
| Index ranges | $-41 \leq h \leq 39$, $0 \leq k \leq 15$, $0 \leq l \leq 18$ |
| Reflections collected | 5793 |
| Independent reflections | 5746 [$R$(int) = 0.013] |
| Data / restraints / parameters | 5793/ 0 /280 |
| Goodness-of-fit on $F^2$ | 1.028 |
| Final $R$ indices [$I$>2$\sigma$(I)] | $R$ = 0.0261, $wR$ = 0.0698 |
| Largest diff. peak and hole | 0.318 and -0.363 e. Å$^{-3}$ |

Table 2: Atomic coordinates and equivalent isotropic displacement parameters (Å$^2$) involving non-hydrogen atoms

| Atom | x | y | z | $^*$U(eq) |
|---|---|---|---|---|
| C1 | 0.35428(5) | 0.10791(14) | 0.61624(13) | 0.0346(3) |
| C2 | 0.39950(6) | 0.11781(14) | 0.68164(14) | 0.0389(3) |
| C3 | 0.43437(7) | 0.09926(17) | 0.63520(15) | 0.0499(5) |
| C4 | 0.42469(7) | 0.06416(17) | 0.52731(14) | 0.0492(5) |
| C5 | 0.37903(6) | 0.05291(18) | 0.46210(15) | 0.0473(4) |
| C6 | 0.34228(5) | 0.07118(14) | 0.50908(13) | 0.0369(3) |
| C7 | 0.29678(6) | 0.05533(19) | 0.43747(18) | 0.0541(5) |
| C8 | 0.21426(6) | 0.04069(19) | 0.38605(15) | 0.0459(4) |
| C9 | 0.17684(7) | 0.04828(19) | 0.43118(14) | 0.0481(4) |
| C10 | 0.12824(6) | 0.04351(18) | 0.37776(13) | 0.0441(4) |
| C11 | 0.05736(5) | 0.08382(16) | 0.41283(13) | 0.0378(3) |
| C12 | 0.02812(5) | 0.13464(15) | 0.46964(13) | 0.0372(3) |
| C13 | -0.02033(7) | 0.14402(17) | 0.41949(17) | 0.0507(5) |
| C14 | -0.05113(8) | 0.19604(18) | 0.46298(19) | 0.0568(5) |
| C15 | -0.03436(6) | 0.23109(16) | 0.57123(15) | 0.0444(4) |
| C16 | 0.01232(5) | 0.21952(15) | 0.62578(13) | 0.0366(3) |
| C17 | 0.04223(5) | 0.16333(13) | 0.57462(13) | 0.0320(3) |
| C18 | 0.18448(6) | 0.26973(17) | 0.79509(14) | 0.0431(4) |
| C19 | 0.22973(7) | 0.29882(19) | 0.85562(14) | 0.0487(4) |
| C20 | 0.26126(6) | 0.24991(17) | 0.80979(14) | 0.0428(4) |
| C21 | 0.14004(8) | 0.3002(2) | 0.81482(18) | 0.0574(5) |
| C22 | 0.31291(7) | 0.25645(19) | 0.85083(17) | 0.0546(5) |
| N1 | 0.25909(5) | 0.06409(13) | 0.46268(11) | 0.0374(3) |
| N2 | 0.10107(5) | 0.08446(13) | 0.44510(10) | 0.0361(3) |
| N3 | 0.18811(5) | 0.20608(11) | 0.71010(10) | 0.0321(2) |
| N4 | 0.23608(5) | 0.19456(11) | 0.72053(9) | 0.0300(2) |
| O1 | 0.32015(4) | 0.12320(10) | 0.66204(10) | 0.0384(2) |
| O2 | 0.18957(4) | 0.09548(9) | 0.53277(8) | 0.0298(2) |
| O3 | 0.08860(4) | 0.15716(10) | 0.63311(9) | 0.0383(2) |
| Ni1 | 0.253822(6) | 0.120521(17) | 0.600073(13) | 0.02861(6) |
| Ni2 | 0.140186(7) | 0.140417(17) | 0.585013(14) | 0.03106(6) |

$^*$U(eq) = $(1/3)\,\Sigma_i\,\Sigma_j\,U_{ij}a_i^*a_j^*a_i\,a_j$

Table 3: Selected bond lengths (Å)

| | |
|---|---|
| Ni1···Ni2 | 3.347(1) |
| N1–Ni1 | 1.966(2) |
| N2–Ni2 | 1.958(1) |
| N3–N4 | 1.403(2) |
| N3–Ni2 | 1.964(1) |
| N4–Ni1 | 1.997(1) |
| O1–Ni1 | 1.899(1) |
| O2–Ni1 | 1.873(1) |
| O2–Ni2 | 1.876(1) |
| O3–Ni2 | 1.846(1) |

Table 4: Selected bond angles (°)

| | |
|---|---|
| Ni1–O2–Ni2 | 126.4(1) |
| C17–O3–Ni2 | 128.4(1) |
| O2–Ni1–O1 | 171.8(1) |
| O2–Ni1–N1 | 82.8(1) |
| O1–Ni–1N1 | 92.0(1) |
| O2–Ni1–N4 | 87.4(1) |
| O1–Ni1–N4 | 98.5(1) |
| N1–Ni1–N4 | 167.9(1) |
| O3–Ni2–O2 | 170.1(1) |
| O3–Ni2–N2 | 91.9(1) |
| O2–Ni2–N2 | 83.1(1) |
| O3–Ni2–N3 | 98.4(1) |
| O2–Ni2–N3 | 87.3(1) |
| N2–Ni2–N3 | 168.7(1) |

## Figure Captions

Fig. 1: Chemical diagram of the investigated compound.

Fig. 2: The molecular structure of the compound. Displacement ellipsoids are plotted at the 50 % probability level.

Fig. 3: Packing of the molecules viewed down the c axis.

Fig. 1

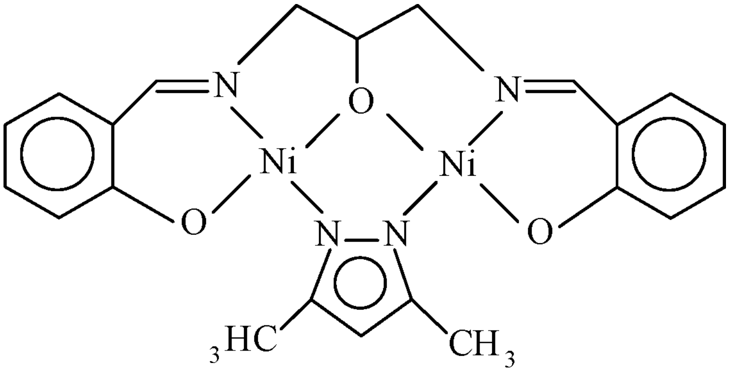
N
O
N
Ni
Ni
O
N
N
O
$_3$HC
CH$_3$

Fig. 2

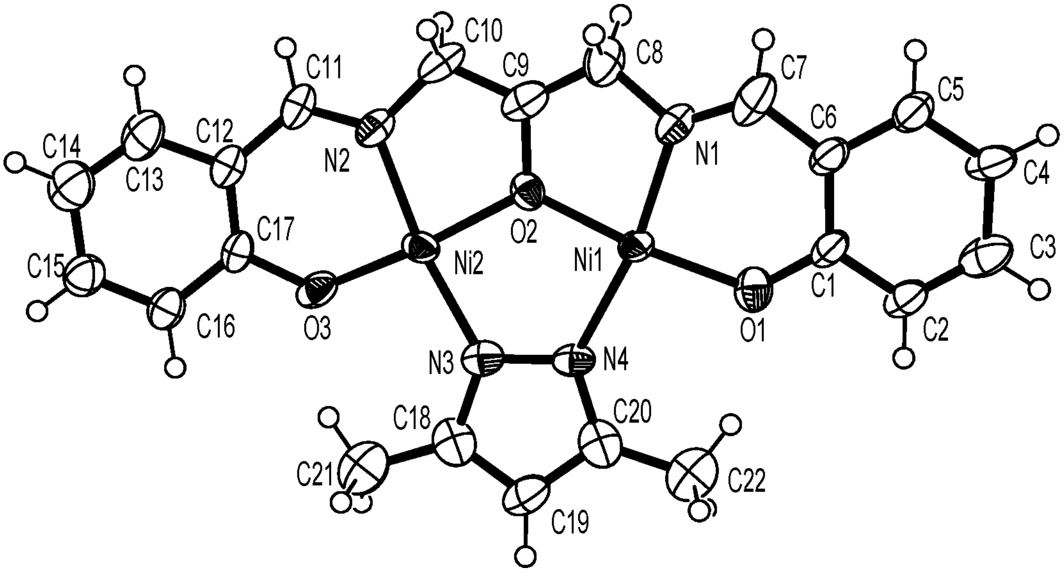
C10
C9
C8
C11
C7
C5
C6
C12
C14
N2
N1
C4
C13
C17
O2
C3
C15
Ni2
Ni1
C1
C16
O3
O1
C2
N3
N4
C18
C20
C21
C22
C19

Fig 3.