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SYNTHESIS AND CHARACTERIZATION OF DIAQUA(3-*p*-TOLYLIMINO-2-BUTANONE OXIMATO)(3-*p*-TOLYLIMINO-2-BUTANONE OXIME)NICKEL(II) PERCHLORATE

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SYNTHESIS AND CHARACTERIZATION OF DIAQUA(3-*p*-TOLYLIMINO-2-BUTANONE OXIMATO)(3-*p*-TOLYLIMINO-2-BUTANONE OXIME)NICKEL(II) PERCHLORATE

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[Ni(L⁻¹)(HL)(H₂O)₂]·ClO₄ with a Schiff base ligand L (HL = 3-*p*-tolylimino-2-butanone oxime) was prepared and structurally characterized by IR, cyclic voltammetry and X-ray diffraction methods. The nickel atom has distorted octahedral coordination consisting of four nitrogen atoms and two oxygen atoms. The equatorial plane is formed by two oxime nitrogen atoms and two imine nitrogen atoms of two Schiff base ligand (L⁻¹ and HL) with Ni—N bond distances between 2.01(1) and 2.11(1) Å. Water oxygen atoms occupy axial positions with Ni—O bond distances of 2.06(1) and 2.15(1) Å. The oxime groups in the Schiff base ligands are coordinated to Ni atom through their nitrogen atoms. One asymmetric intramolecular hydrogen bridge between the two oxime groups is found in the title complex.

Keywords: Nickel complex; Imine–oxime complex; Crystal structure; Spectroscopy

INTRODUCTION

Synthesis of imine–oxime complexes is one of the challenging areas in the field of coordination chemistry since researchers confirmed the validity of cobaloximes as vitamin B₁₂ model compounds [1]. The oxime group can function as a bridging ligand between two metal ions through nitrogen and deprotonated oxygen in monoanionic form, offering a binuclear

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or polynuclear complex [2]. The oxime group can also function as a monodentate ligand through nitrogen in neutral form or in monoanionic form, or through deprotonated oxygen in monoanionic form, offering a mononuclear complex. In most mononuclear complexes, two imine–oxime ligands have the same state, either in the same monoanionic form or neutral form [3–11]. Two asymmetric intramolecular hydrogen bridges exist between the two oxime groups when the two oxime groups are in dioxime ligands and in the same anionic form [3, 5].

Only a few complexes with imine–oxime ligands in which the two imine–oxime ligands are in two different oxidation states have been studied [7, 12, 13]. There is only one asymmetric intramolecular hydrogen bridge between the two oxime groups in one complex molecule. One imine–oxime ligand is present as such while the second imine–oxime ligand is present as the conjugate base; the single hydrogen atom is then shared in the asymmetric (O—H...O) bridge between the two oxime groups. The formation of intramolecular hydrogen bonds in these complexes determines the *cis*-arrangement of the two imine–oxime ligands. Herein we report a nickel complex $[\text{Ni}(\text{L}^{-1})(\text{HL})(\text{H}_2\text{O})_2] \cdot \text{ClO}_4$ (HL = 3-*p*-tolylimino-2-butanone oxime), in which two imine-oxime ligands are in an anionic form and a neutral form, respectively.

EXPERIMENTAL

Preparation

One mmol of $\text{Ni}(\text{ClO}_4)_2 \cdot 6\text{H}_2\text{O}$ (0.256 g) and 1 mmol of butane-2,3-dione oxime (0.101 g) were dissolved in 25 cm³ of methanol to yield a light green solution. An excess of *p*-toluidine was added, causing a colour change to red. The resulting solution was heated and stirred for 15–20 mins, filtered and allowed to crystallize. Red-brown rectangular crystals were obtained in 6–8 days. Yield: 64%. *Anal.* Calc. for $\text{C}_{22}\text{H}_{31}\text{ClN}_4\text{NiO}_8$ (%): C, 46.14; H, 5.42; N, 9.79; O, 22.37. Found: C, 46.42; H, 5.38; N, 9.58; O, 22.48.

Measurements

IR spectra were measured on a Perkin–Elmer 200 Fourier transform IR spectrometer in KBr pellets. Cyclic voltammetry was performed on a BAS 100 A electrochemical instrument. A three electrode system was employed

with a platinum disc working electrode, an Ag/AgCl reference electrode and a platinum wire auxiliary electrode, $(\text{Et}_4\text{N})\text{ClO}_4$ (0.1 M in methanol) being the supporting electrolyte. ESR spectra were measured on a Bruker-ER-420 ESR spectrometer (X-band).

X-ray Structure Determination

X-ray diffraction data for a crystal of size $0.48 \times 0.20 \times 0.13$ mm were recorded on an Enraf-Nonius CAD4 diffractometer with graphite-monochromated MoK_α radiation ($\lambda = 0.71069 \text{ \AA}$), the scan mode being $\omega - 2\theta$. Cell dimensions were refined using 25 reflections in the range $20^\circ < 2\theta < 30^\circ$. Diffraction data were corrected for Lorentz and polarization effects. Crystallographic data are given in Table I.

The structure was solved by direct methods and refined by a full-matrix least-squares procedures. Most non-hydrogen atoms were refined with anisotropic thermal parameters, except for the four oxygen atoms in the ClO_4 group (isotropic parameters). Partial occupancy of the four oxygen atoms was proposed on the basis of peak heights in the difference Fourier map and thermal parameters. These occupancy factors were also refined in the earlier refinement and then fixed at 1/2 (see Tab. II). All hydrogen

TABLE I Crystallographic data for the complex

Formula	$\text{C}_{22} \text{H}_{31} \text{Cl} \text{N}_4 \text{Ni} \text{O}_8$
Formula weight	573.67
Crystal system	monoclinic
Space group	Cc
$a(\text{\AA})$	11.594(2)
$b(\text{\AA})$	32.781(7)
$c(\text{\AA})$	7.820(2)
$\beta(^\circ)$	117.63(3)
$V(\text{\AA}^3)$	2633.3(9)
Z	4
$D_{\text{calc}} (\text{Mg m}^{-3})$	1.447
$\mu (\text{mm}^{-1})$	0.890
$F(000)$	1200
$2\theta_{\text{max}} (^\circ)$	51.94
Index range h	$-9 \rightarrow 14$
k	$0 \rightarrow 40$
l	$-9 \rightarrow 9$
No. of independent reflections	2728 [$R_{\text{int}} = 0.042$]
No. of observed reflections	2177
No. of variables	321
R	0.0521
wR	0.1321
GOF	0.974

TABLE II Atomic coordinates and U_{eq} ($\text{\AA}^2 \times 10^3$) values for the non-hydrogen atoms

Atom	x/a	y/b	z/c	$U(eq)$
Ni(1)	0.3621 (3)	0.5735 (1)	0.1761 (4)	31 (1)
N(1)	0.247 (1)	0.5299 (4)	0.210 (2)	36 (3)
N(2)	0.230 (1)	0.6094 (3)	0.225 (2)	37 (3)
N(3)	0.476 (1)	0.5315 (3)	0.142 (2)	33 (3)
N(4)	0.490 (1)	0.6087 (4)	0.123 (2)	34 (3)
O(1)	0.262 (1)	0.4904 (3)	0.184 (1)	35 (2)
O(2)	0.470 (1)	0.4898 (3)	0.172 (2)	48 (3)
O(3)	0.495 (1)	0.5755 (3)	0.480 (1)	41 (3)
O(4)	0.2392 (9)	0.5760 (3)	-0.117 (2)	40 (3)
C(1)	0.040 (1)	0.5177 (5)	0.216 (2)	51 (4)
C(2)	0.146 (1)	0.5429 (5)	0.224 (2)	46 (4)
C(3)	0.150 (2)	0.5871 (5)	0.254 (2)	43 (4)
C(4)	0.052 (1)	0.6057 (4)	0.310 (3)	58 (5)
C(5)	0.239 (1)	0.6515 (5)	0.252 (2)	46 (4)
C(6)	0.146 (2)	0.6762 (5)	0.108 (3)	73 (6)
C(7)	0.148 (2)	0.7174 (5)	0.148 (3)	90 (7)
C(8)	0.252 (3)	0.7348 (7)	0.313 (4)	98 (8)
C(9)	0.339 (2)	0.7086 (5)	0.456 (3)	73 (5)
C(10)	0.331 (2)	0.6692 (6)	0.406 (3)	70 (6)
C(11)	0.253 (3)	0.7812 (6)	0.348 (4)	120 (10)
C(12)	0.685 (2)	0.5156 (5)	0.145 (3)	60 (5)
C(13)	0.575 (1)	0.5431 (4)	0.126 (2)	35 (3)
C(14)	0.576 (1)	0.5894 (5)	0.101 (2)	38 (3)
C(15)	0.666 (2)	0.6060 (5)	0.031 (3)	80 (6)
C(16)	0.489 (2)	0.6531 (3)	0.099 (2)	42 (4)
C(17)	0.385 (2)	0.6699 (5)	-0.066 (3)	59 (5)
C(18)	0.385 (2)	0.7139 (5)	-0.064 (3)	69 (5)
C(19)	0.483 (2)	0.7366 (4)	0.050 (4)	65 (6)
C(20)	0.571 (2)	0.7199 (6)	0.204 (3)	94 (8)
C(21)	0.583 (2)	0.6778 (6)	0.231 (3)	65 (5)
C(22)	0.482 (2)	0.7829 (5)	0.030 (3)	122 (10)
Cl(1)	0.3631 (6)	-0.0955 (1)	0.1752 (9)	50 (1)
O(5)	0.266 (2)	-0.0745 (5)	0.035 (3)	66 (4)*
O(6)	0.402 (2)	-0.0640 (6)	0.342 (3)	73 (5)*
O(7)	0.251 (2)	-0.1150 (5)	0.139 (2)	71 (4)*
O(8)	0.474 (2)	-0.0985 (6)	0.158 (3)	82 (5)*
O(9)	0.353 (2)	-0.0726 (7)	0.011 (3)	95 (8)*
O(10)	0.483 (2)	-0.0878 (8)	0.328 (4)	102 (8)*
O(11)	0.405 (2)	-0.1242 (8)	0.065 (4)	116 (7)*
O(12)	0.331 (3)	-0.1361 (8)	0.228 (4)	114 (8)*

* Refined isotropically with occupancy factor 1/2.

$U(eq)$ is defined as one third of the trace of the orthogonalized U_{ij} tensor.

atoms were located by difference Fourier maps or geometrically and added to the structure factor calculations, but their positions were not refined. Full-matrix least-squares refinement resulted in an R factor of 0.052 for the present structure and of 0.053 for the other enantiomorph. The absolute structure was determined and is in accordance with the selected setting [Flack χ parameter = -0.0093(542)] [14]. Positional parameters and equivalent isotropic thermal parameters are listed in Table II; selected bond distances and angles are shown in Table III.

TABLE III Selected bond lengths (Å) and angles (°) for the complex

Ni(1)—N(1)	2.05 (1)
Ni(1)—N(2)	2.11 (1)
Ni(1)—N(3)	2.01 (1)
Ni(1)—N(4)	2.07 (1)
Ni(1)—O(3)	2.15 (1)
Ni(1)—O(4)	2.06 (1)
N(1)—C(2)	1.30 (2)
N(1)—O(1)	1.33 (2)
N(2)—C(3)	1.28 (2)
N(2)—C(5)	1.39 (2)
N(3)—C(13)	1.26 (2)
N(3)—O(2)	1.39 (1)
N(4)—C(14)	1.26 (2)
N(4)—C(16)	1.47 (2)
C(1)—C(2)	1.46 (2)
C(2)—C(3)	1.47 (2)
C(3)—C(4)	1.52 (2)
C(8)—C(11)	1.54 (3)
C(12)—C(13)	1.51 (2)
C(13)—C(14)	1.53 (2)
C(14)—C(15)	1.49 (2)
C(19)—C(22)	1.53 (2)
O(1)···H(02)	1.39
O(2)—H(02)	1.07
O(1)···O(2)	2.46 (3)
O(4)···O(1a)	2.76 (1)
O(3)···O(2b)	2.70 (1)
N(1)—Ni(1)—N(2)	78.2 (5)
N(1)—Ni(1)—N(3)	92.7 (2)
N(1)—Ni(1)—N(4)	169.4 (5)
N(1)—Ni(1)—O(3)	93.1 (4)
N(1)—Ni(1)—O(4)	90.8 (4)
N(2)—Ni(1)—N(3)	170.7 (4)
N(2)—Ni(1)—N(4)	112.1 (2)
N(2)—Ni(1)—O(3)	89.5 (5)
N(2)—Ni(1)—O(4)	89.6 (5)
N(3)—Ni(1)—N(4)	77.1 (5)
N(3)—Ni(1)—O(3)	89.3 (5)
N(3)—Ni(1)—O(4)	92.3 (4)
N(4)—Ni(1)—O(3)	89.6 (4)
N(4)—Ni(1)—O(4)	86.8 (4)
O(3)—Ni(1)—O(4)	175.7 (2)
C(2)—N(1)—O(1)	121 (1)
O(1)—N(1)—Ni(1)	121.3 (9)
C(3)—N(2)—C(5)	123 (1)
C(13)—N(3)—O(2)	115 (1)
C(14)—N(4)—C(16)	116 (1)
N(1)—C(2)—C(1)	126 (2)
N(2)—C(3)—C(2)	120 (2)
C(2)—C(3)—C(4)	118 (1)
C(10)—C(5)—N(2)	123 (2)
C(9)—C(8)—C(11)	121 (2)
N(3)—C(13)—C(12)	125 (1)
N(4)—C(14)—C(15)	127 (1)
C(21)—C(16)—N(4)	122 (2)
C(18)—C(19)—C(22)	122 (2)
O(2)—H(02)···O(1)	177
O(4)—H(04B)···O(1a)	174
O(3)—H(03B)···O(2b)	165

Symmetry codes; a: $x, 1-y, -1/2+z$; b: $x, 1-y, 1/2+z$.

RESULTS AND DISCUSSION

IR Spectra

A strong band at 1554 cm^{-1} and a very strong band at 1097 cm^{-1} are assigned to $\nu(\text{C}=\text{N})$ and $\nu(\text{N}-\text{O})$ in the oxime group. This shows that the oxime group is coordinated to nickel atom through its nitrogen atom forming a five-membered chelate ring [13,15]. The band at 1508 cm^{-1} is attributed to $\nu_{\text{C}=\text{N}}$ of the imine group and $\nu_{\text{C}=\text{C}}$ of the aromatic ring. The medium strong band at 1369 cm^{-1} is assigned to stretching vibration of the C—N bond in the aromatic amine. The $\nu(\text{Ni}-\text{N})$ band in the title complex appears at 623 cm^{-1} [16]. A weak shoulder near 1740 cm^{-1} is observable and is characteristic of $\delta(\text{O}-\text{H}\cdots\text{O})$ for intramolecular hydrogen bonding [13]. The title complex shows an absorption band at 1101 cm^{-1} (antisymmetric stretch) of the perchlorate anion [17].

ESR Spectra

The X-band ESR spectrum of the title complex was recorded at room temperature. The g value of 2.2039 obtained from the spectrum is in agreement with distorted octahedral geometry for Ni^{2+} [18].

Cyclic Voltammetry

There is *quasi*-reversible redox behaviour corresponding to the $\text{Ni}^{\text{II}}/\text{Ni}^{\text{III}}$ couple [19] in methanol solution for the title complex, with $E_{pa}=833$, $E_{pc}=683$, $E_{1/2}=758$ and $\Delta E_p=150\text{ mV}$. An irreversible redox potential is found at $E_{pa}=183$, $E_{pc}=383$, $E_{1/2}=100$ and $\Delta E_p=566\text{ mV}$. This may be caused by a $\text{Ni}^{\text{II}}/\text{Ni}^{\text{I}}$ couple.

Description of the Structure

The molecular structure of the title complex, $[\text{Ni}(\text{C}_{11}\text{H}_{13}\text{N}_2\text{O})(\text{C}_{11}\text{H}_{14}\text{N}_2\text{O})(\text{H}_2\text{O})_2]\text{ClO}_4$, is illustrated in Figure 1. The nickel atom has distorted octahedral geometry with the equatorial plane defined by the two oxime nitrogen atoms (N(1) and N(3)) and two imine nitrogen atoms (N(2) and N(4)) from the two Schiff base ligands (L^{-1} and HL). The Ni atom lies $0.009(9)\text{ \AA}$ out of the equatorial plane. The two axial positions are occupied by two oxygen atoms (O(3) and O(4)) from the two coordinated water molecules. Ni—O (water) bonds are $2.15(1)$ and $2.06(1)\text{ \AA}$, within the

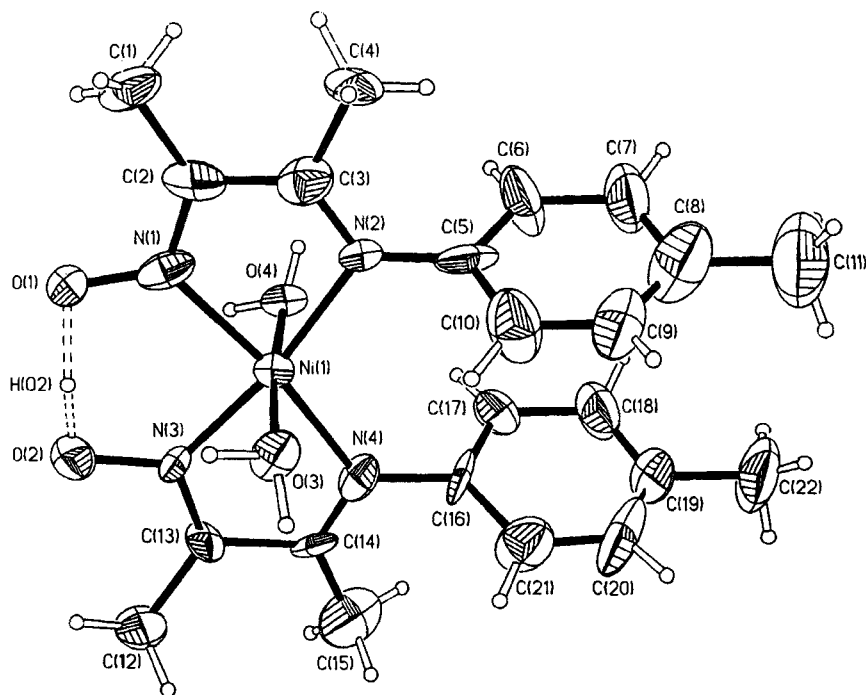


FIGURE 1 Molecular structure of the title complex showing atom numbering and 50% probability displacement ellipsoids.

normal range of 2.00–2.20 Å observed in known structures [20]. The chelate angles $N(1)-Ni(1)-N(2)$ and $N(3)-Ni(1)-N(4)$ are 78.2(5) and 77.1(5)°, respectively.

The two five-membered chelate rings $N(1), C(2), C(3), N(2), Ni(1)$ and $N(3), C(13), C(14), N(4), Ni(1)$ are planar with mean deviations of 0.05 Å and 0.04 Å, respectively. The dihedral angle between the two chelate rings is 6.1(5)°. However, the dihedral angles between the chelate rings and the neighbouring benzene ring are 72.6(4) and 68.1(4)°, respectively.

Most of the structural parameters in 3-*p*-tolylimino-2-butanone oxime ligands of the title complex are similar to those in free 3-*p*-tolylimino-2-butanone oxime [21]. For example, the free ligand and the ligands of the title complex are *syn*-methyl, *syn*-methyl isomers related to the presence of the $N(1)=C(2)$ and $N(2)=C(3)$ bonds [21], the torsion angles for $N(1)-C(2)-C(3)-N(2)$ and $N(3)-C(13)-C(14)-N(4)$ (13(2) and 8(2)°) show delocalization of electrons through the heterodiene groups $N=C-C=N$. However, there are some differences between the

3-*p*-tolylimino-2-butanoneoxime ligands (L^{-1} and HL) in the title complex. The bond lengths N(1)—O(1) and N(2)—C(5) (1.33 and 1.39 Å) in ligand L^{-1} are shorter than the corresponding bond lengths (1.39 and 1.47 Å) in ligand HL. According to the Bernstein equation [6, 22], the calculated bond orders for bonds N(1)—O(1) and N(3)—O(2) in the title complex and bond N—O in free 3-*p*-tolylimino-2-butanoneoxime [22] are 1.47, 1.20 and 1.20, respectively, indicating that partial double-bond contribution in the N(1)—O(1) bond is larger than that in the N(3)—O(2) bond. These facts confirm that the title complex is of the $M(L^{-1})(HL)$ type, not of the ML_2 type.

The O(1) \cdots O(2) distance between the two oxime groups of the two Schiff base ligands is 2.46(3) Å. The O(2)—H(02) distance is 1.07 Å, while the O(1) \cdots H(02) distance is 1.39 Å. There are intermolecular hydrogen bonds between the OH groups of coordinated water molecules and the oxygen atoms of the oxime groups in the Schiff base ligands, as shown in Table III. Therefore, the molecules in the title complex crystal are held together by intramolecular hydrogen bonds, forming a chain along the *c* axis.

Supplementary Material

Full lists of bond distances and angles, anisotropic thermal parameters, torsion angles and structure factors are available from the authors upon request.

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