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NOTE

Synthesis and Characterization of Single Crystal of *bis*-(L-Histidinato)nickel(II) Monohydrate

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The complex *bis*-(L-histidinato)nickel(II) monohydrate has been synthesized and structurally characterized by single-crystal X-ray diffraction. This complex was solved in the monoclinic system, space group C2, with the following unit-cell parameters: a = 29.313(6) Å, b = 8.227 (2)Å, c = 6.264(1) Å, α = 90º, β = 90.189(5)º, γ = 90º, Z = 4, V= 1510.5(5)ºA^3. The final R value was R1 = 0.0301 for 7558 measured reflections.

Key Words: Synthesis, Characterization, Single crystal, *Bis*-(L-histidinato)nickel(II) monohydrate.

Amino acids are low molecular weight ligands in biological systems. Among the amino acids, L-histidine is one of the strongest metal coordinating ligands and plays an important role in the binding of metal ions by proteins, therefore, histidine is an essential amino acid, has a positively charged imidazole functional group[1]. The coordination between Ni(II) ions and peptides containing one or two histidine residues in different positions has been extensively reported[2]. The imidazol makes it a common participant in enzyme catalyzed reactions[3]. Unprotonated imidazole is nucleophilic and can serve as a general base. The imidazole nitrogen of L-histidine residues often provides the primary means by which the metal ions are bound to proteins.

The complex (Fig. 1) was prepared by reaction of nitrilotriacetic acid, $NiCl_2$ and histidine with a molar ratio of 1: 1: 1 in water, respectively. The mixture was stirred for *ca.* 1 h at room temperature. This solution yielded purple crystals of the complex after 2 months.

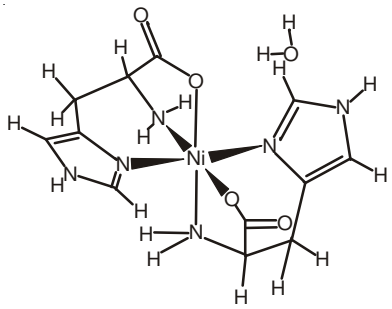

Fig. 1. Structure of *bis*-(L-histidinato)nickel(II) monohydrate

Data were collected on a Bruker APEX II CCD. The structures were solved using direct methods (SHELXS-97) and refined against F^2 using the SHELXL-97 software[4]. All hydrogen atoms were treated in riding model with the equivalent thermal parameters of the atoms to which corresponding H atoms are bonded. The crystal and experimental data are given in Table-1.

TABLE-1
CRYSTAL AND EXPERIMENTAL DATA

Empirical formula	$C_{12}H_{18}N_6NiO_5$
Formula weight	385.05
Temperature	120(2) K
Crystal system	Monoclinic
Wavelength	0.71073 Å
Space group	C2
Unit cell dimensions	
	a = 29.313(6) Å; α = 90º
	b = 8.227(2) Å; β = 90.189(5)º
	c = 6.264(1) Å; γ = 90°
V	1510.5(5) °A^3
Z	4
D_x	1.693 mg/m^3
Absorption coefficient	1.324 mm^{-1}
$2\theta_{max}$	56.0°
R1 indices [6168 refs I > 2σ (I)]	R1 = 0.0301, wR2 = 0.0636
Reflections collected	7558
Diffractometer	Bruker APEX II CCD
Program system	SHELXL-97
Structure determination	SHELXS-97
Refinement	Full matrix least

The atomic coordinates and equivalent isotropic displacement parameters for the non-hydrogen atoms are listed in Table-2.

TABLE-2
ATOMIC COORDINATES ($\times 10^4$) AND EQUIVALENT ISOTROPIC DISPLACEMENT PARAMETERS ($Å^2 \times 10^3$) FOR THE COMPLEX

Atom	X	Y	Z	U(eq)
Ni(1)	8678910	2821(1)	2303(1)	12(1)
O(1)	9072(1)	-417(2)	6814(3)	20(1)
O(3)	8732(1)	1710(2)	5306(3)	15(1)
O(3)	8206(1)	7428(2)	2195(3)	17(1)
O(4)	8627(1)	5303(2)	3168(3)	16(1)
N(1)	8754(1)	411(2)	1407(3)	14(1)
N(2)	10067(1)	4114(3)	2526(3)	18(1)
N(3)	9381(1)	3048(3)	2328(3)	15(1)
N(4)	8615(1)	3937(2)	-656(3)	15(1)
N(5)	7304(1)	2760(3)	4240(3)	16(1)
N(6)	7968(1)	2811(3)	2597(3)	13(1)
C(1)	9846(1)	375(3)	5235(4)	14(1)
C(2)	9067(1)	-305(3)	3004(4)	15(1)
C(3)	9562(1)	54(3)	2393(4)	18(1)
C(4)	9697(1)	1806(3)	2430(4)	16(1)
C(5)	10120(1)	2484(3)	2548(4)	18(1)
C(6)	9617(1)	4426(3)	2415(4)	18(1)
C(7)	8386(1)	6081(3)	1839(4)	13(1)
C(8)	8299(1)	5299(3)	-340(4)	14(1)
C(9)	7803(1)	4709(3)	-455(4)	17(1)
C(10)	7665(1)	3770(3)	1494(4)	14(1)
C(11)	7253(1)	3747(3)	2502(4)	16(1)
C(12)	7736(1)	2234(3)	4241(4)	16(1)
O(1S)	9197(1)	6278(2)	6669(3)	20(1)

$Ueq = 8/3\ \pi^2(U11(aa^*)^2 + U22(bb^*)^2 + U33(cc^*)^2 + 2U12(aa^*bb^*)\cos\gamma + 2U13(aa^*cc^*)\cos\beta + 2U23[(bb^*cc^*)\cos\alpha]$

Ni is hexacoordinated in an approximately octahedral geometry, chelated by four N atoms and two O atoms from two L-histidine ligands [Ni(1)-N(1)= 2.073(2) Å, Ni(1)-N(3)= 2.066(2) Å, Ni(1)-N(4)= 2.076(2) Å, Ni(1)-N(6)= 2.091(2) Å, Ni(1)-O(2)= 2.097(2) Å, Ni(1)-O(4)= 2.117(2) Å]. Hydrogen bonds between the water molecules, the O atoms of the carboxylate groups and the imidazole nitrogen of L-histidine link the molecules into a three-dimensional network (Table-3). Fig. 2 shows the ORTEP structure of the title complex.

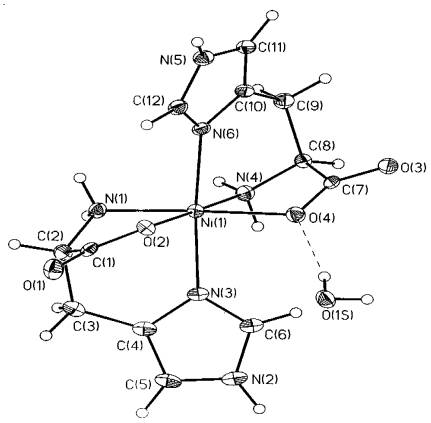

Fig. 2. ORTEP structure of the title complex with partial atom labeling, the ellipsoids enclose 50 % of the electronic density

TABLE-3
SELECTED BOND LENGTHS [Å] AND ANGLES [°] FOR THE COMPLEX

Ni(1)-N(3)	2.067(2)	C(70)-C(8)	1.530(3)
Ni(1)-N(1)	2.073(2)	C(8)-C(9)	1.534(3)
Ni(1)-N(4)	2.076(2)	C(8)-H(8A)	1.000
Ni(1)-N(6)	2.091(2)	C(9)-C(10)	1.501(3)
Ni(1)-O(2)	2.097(2)	C(9)-H(9A)	0.9900
Ni(1)-O(4)	2.1174(2)	C(9)-H(9B)	0.9900
O(2)-C(1)	1.266(3)	C(11)-H(11A)	0.9500
O(3)-C(7)	1.248(3)	C(12)-H(12A)	0.9500
O(4)-C(7)	1.264(3)	O(1S)-H(1S)	0.9000
N(1)-C(2)	1.477(3)	O(1S)-H(2S)	0.8500
N(1)-H(1A)	0.9000		
N(1)-H(1B)	0.9000	N(3)-Ni(1)-N(1)	88.88(8)
N(2)-C(6)	1.347(3)	N(3)-Ni(1)-N(4)	93.04(8)
N(2)-C(5)	1.350(4)	N(1)-Ni(1)-N(4)	100.96(8)
N(2)-H(2)	0.9001	N(3)-Ni(1)-N(6)	172.64(9)

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