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NOTE

Hydrothermal Synthesis and Crystal Structure of a Ni(II) Complex with Diacetyl Dihydrazone Ligand

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A novel nickel(II) complex $NiL_3 \cdot (ClO_4)_2$ (L = diacetyl dihydrazone) has been synthesized by hydrothermal method and characterized by IR spectra and single-crystal X-ray method. The crystal is trigonal, space P-3c1 with unit cell parameters: a = 9.5922(5) Å, b = 9.5922(5) Å, c = 15.1871(14) Å, α = 90º, β = 90º, γ = 120º, V = 1210.16(14) Å^3, Z = 2, M_r = 600.09, D_c = 1.647 Mg/cm^3, μ = 1.087 mm^{-1}, $F_{(000)}$ =624, T = 293(2) K, R = 0.0474, wR = 0.1276 for 8580 reflections with I > 2σ(I).

Key Words: Nickel(II) complex, Diacetyl dihydrazone, Crystal structure.

In recent years, the transition metal coordination chemistry of hydrazone Schiff base ligands has got enormous significance due to its diversity of molecular structures[1-4] and good biological activity of insecticidal, sterilization and weeding[4-7] and excellent anticancer, catalytic, spectral and magnetic properties[1-3,6].

Here we report the hydrothermal synthesis and crystal structure of a novel mononuclear nickel(II) complex $NiL_3 \cdot (ClO_4)_2$ (L=diacetyl dihydrazone).

$Ni(ClO_4)_2 \cdot 6H_2O$ was prepared in our laboratory. Diacetyl dihydrazone was prepared by similar procedure in the literature[8]. All reagents were of AR grade and used without further purification. IR spectra were recorded on a Nexus-870 spectrophotometer. The crystal structure was determined by Siemens SMART CCD area-detector diffractometer.

Synthesis: A mixture of 7 mL acetonitrile solution of diacetyl dihydrazone (3 mmol) and 14 mL H_2O solution of $NiL_3 \cdot (ClO_4)_2$ (1 mmol), was carried out in a autoclave and heated to 80 ºC for 48 h. After cooling, the well-shaped scarlet four edges column single crystals were obtained. Yield 37 %. IR spectrum (KBr, ν_{max}, cm^{-1}): 3420, 3320 (N-H); 1610 (C=N); 1090, 623 ($C1O_4^-$).

Crystal structure determination: A scarlet colour single crystal 0.45 mm × 0.35 mm × 0.30 mm was selected for crystallographic data collection at 293(2) K and structure determinated with graphite monochromatic MoK$_\alpha$ radiation (λ = 0.71073Å). A total of 8580 reflections were collected in the range of 2.45 ≤ θ ≤ 27.48º, of which 935 reflections were unique with R_{int} = 0.0193 and R = 0.0474 and wR = 0.1276, where w = 1/[$s^2(F_0^2)$ + (0.0643P)^2 + 1.0654], P = (F_0^2 + 2F_0^2)/3. The maximum and minimum peaks on the final difference Fourier map are corresponding to 0.324 and -0.496e/Å^3, respectively. The CCDC numbers was 646143.

The atomic coordinates and thermal parameters are listed in Table-1 and the selected bond lengths and bond angles in Table-2, respectively. Fig. 1 shows diagram of the molecular structure of the complex $NiL_3 \cdot (ClO_4)_2$. Fig. 2 shows a perspective view of the crystal packing in the unit cell.

TABLE-1
NON-HYDROGEN ATOMIC COORDINATES (× 10^4)
AND THERMAL PARAMETERS (× 10^{3}Å^2)

Atom	X	Y	Z	U (eq)
Ni	0	0	2500	32(1)
N(1)	2094(3)	800(2)	1770(1)	38(1)
N(2)	2324(3)	1507(3)	960(2)	54(1)
C(1)	3122(3)	431(3)	2072(1)	35(1)
C(2)	4643(3)	807(4)	1617(2)	53(1)

The molecular structure of $NiL_3 \cdot (ClO_4)_2$ is shown in Fig. 1. It is easy to see that the center nickel(II) ion is coordinated with six N atoms of the three diacetyl dihydrazone and shows a distorted octahedral geometry. As shown in the molecular packing diagram (Fig. 2), there are positive negative charge interactions between $[NiL_3]^{2+}$ cation and and ClO_4^-.

TABLE-2
SELECTED BOND LENGTHS (Å) AND BOND ANGLES (°)

Bond	Length	Angle	(°)	Angle	(°)
Ni-N(1)#1	2.076(2)	C(1)#3-C(1)-C(2)	120.57(15)	N(1)#1- Ni-N(1)	96.11(11)
Ni-N(1)#2	2.076(2)	N(1)#1- Ni-N(1)#2	166.87(11)	N(1)#2- Ni-N(1)	94.16(7)
N(1)-C(1)	1.287(3)	N(1)#2- Ni-N(1)#3	96.11(11)	N(1)#4- Ni-N(1)	166.87(11)
N(1)-N(2)	1.369(3)	N(1)-C(1)-C(2)	124.2(2)	C(1)#3-C(1)-C(2)	120.57(15)
CL-O(1)	1.399(4)	O(2)-CL-O(1)	108.05(16)	O(1)-CL-O(2)#7	109.04(15)

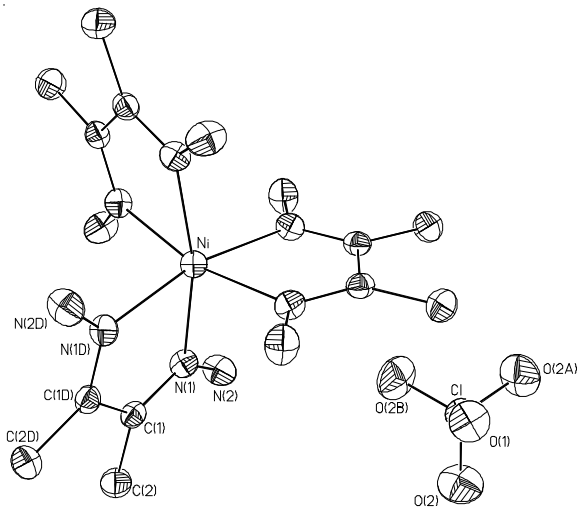

Fig. 1. Molecular structure of the complex $NiL_3 \cdot (ClO_4)_2$

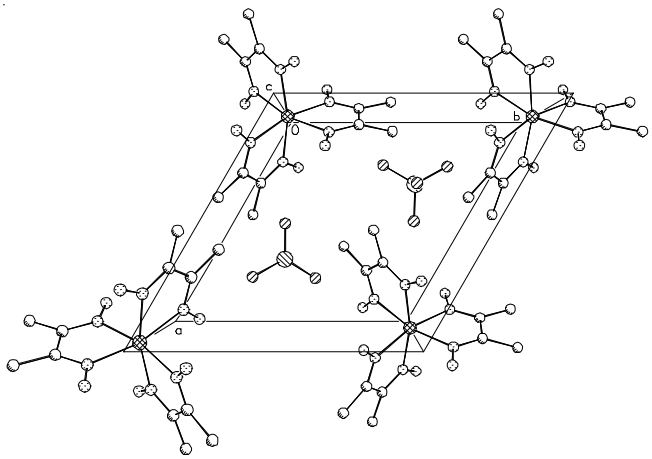

Fig. 2. Molecular packing arrangement in the unit cell

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