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NOTE

Synthesis and Crystal Structure of Nickel(II) Complex: $[Ni(phen)_2(N_3)_2]$

WEN-JIE BI* and HUA-ZE DONG*

School of Chemistry and Chemical Engineering, Hefei Normal University, Hefei, P.R. China

*Corresponding authors: E-mail: 501178740@qq.com; dapdong@163.com

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A novel nickel(II) complex $[Ni(phen)_2(N_3)_2]$ has been synthesized, with the 1,10-phenanthroline (phen) and the nitrine (N_3^-) as ligands and characterized by IR spectra and single-crystal X-ray diffraction measurements. The crystal is triclinic, space group P-1 with cell dimensions of a = 8.2079(7) Å, b = 11.0042(10) Å, c = 12.4317(11) Å, α = 82.259(2)°, β = 82.240(2)°, γ = 72.5150(10)°, V = 1055.88(16) Å^3, Z = 2, Mr = 503.18, Dc = 1.583 mg/cm^3, μ = 0.957 mm^{-1}, F(000) = 516, T = 298(2) K, R = 0.0372, wR = 0.0948 for 5278 reflections with I > 2(I). In the molecular structure unit, nickel(II) cation is coordinated by six donor atoms and has a slightly distorted octahedron configuration.

Keywords: Nickel(II) complex, Phenanthroline, Azium, Crystal structure.

1,10-Phenanthroline is a kind of good metal chelating agent and provide two donor atoms. Their complexes have stable chemical property and excellent performance[1]. Nickel is one of necessary trace elements in organisms, nickel(II) complexes have broad application prospects in molecular magnets[2,3], superoxide dismutase[4,5], biological activity[6,7] and catalytic aspects[8,9]. Herein, the synthesis and crystal structure of a mononuclear complex $[Ni(phen)_2(N_3)_2]$ are reported.

All reagents were of AR grade and used without further purification. IR spectra were recorded on a Nicolet 870 spectrophotometer. The X-ray structure was determined by Bruker Smart-1000 CCD area-detector diffractometer.

Synthesis: 20 mL ethanol solution of phen (10 mmol) was respectively added to 20 mL H_2O solution of $NiCl_2$ (5 mmol) and NaN_3 (30 mmol) under stirring for 4 h. After being filtered, the solution was stand at room temperature for 1 week, crystals were obtained. The product was green cubic-shaped crystals. Yield 46 %. IR spectrum (KBr, cm^{-1}) : 3430, 2070, 1630, 1500, 1429, 852, 719, 636.

Structure determination: A single crystal (0.44 mm × 0.42 mm × 0.40 mm) was selected for crystallographic data collection at 298(2) K and structure determinated with graphite monochromatic MoK_α radiation (λ = 0.71073 Å). A total of 5278 reflections were collected in the range of 2.61° ≤ θ ≤ 25.02°, of which 3655 reflections were unique with R_{int} = 0.0230. Lp effects and empirical absorption were applied in data corrections. The structure was solved by direct methods and expanded using fourier techniques and SHELXS-97 program system was used in the solution and refinement of the structure. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were added according to theoretical model.

$$R_1 = \Sigma(||F_0|-|F_c||)/\Sigma|F_0| = 0.0372 \quad (1)$$

and

$$wR_2 = \{\Sigma[w(F_0^2-F_C^2)^2]/\Sigma w(F_0^2)^2\}^{1/2} = 0.0948 \quad (2)$$

where $w = 1/[s^2(F_0^2) + (0.0588\ P)^2 + 0.3204\ P]$, $P = (F_0^2 + 2F_0^2)/3$. The maximum and minimum peaks on the final difference fourier map are corresponding to 0.7008 and 0.6782e/Å^3 (CCDC No. 937456), respectively.

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 $[Ni(phen)_2(N_3)_2]$. Fig. 2 shows the packing diagram of the complex in the unit cell. As shown in the Fig. 1, the center nickel(II) cation is six-coordinated with four nitrogen atoms of the two 1,10-phenanthroline and two nitrogen atoms of the two N_3-anions. Ni(II) is in a slightly distorted octahedral geometry environment.

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

Bond	Length	Angle	(°)	Angle	(°)
NI(1)-N(8)	2.073(2)	N(4)-Ni(1)-N(2)	92.21(8)	N(8)-Ni(1)-N(4)	94.36(9)
Ni(1)-N(5)	2.095(2)	N(4)-Ni(1)-N(3)	79.03(8)	N(1)-Ni(1)-N(3)	93.04(8)
Ni(1)-N(3)	2.117(2)	N(5)-Ni(1)-N(3)	88.89(9)	C(1)-N(1)-Ni(1)	128.29(18)
N(2)-C(10)	1.322(3)	N(7)-N(6)-N(5)	177.3(3)	N(6)-N(5)-Ni(1)	126.04(19)
N(4)-C(22)	1.328(3)	C(13)-N(3)-Ni(1)	129.24(19)	N(2)-C(6)-C(5)	117.1(2)
N(6)-N(7)	1.166(3)	C(1)-N(1)-Ni(1)	128.29(18)	N(3)-C(13)-C(14)	122.9(3)

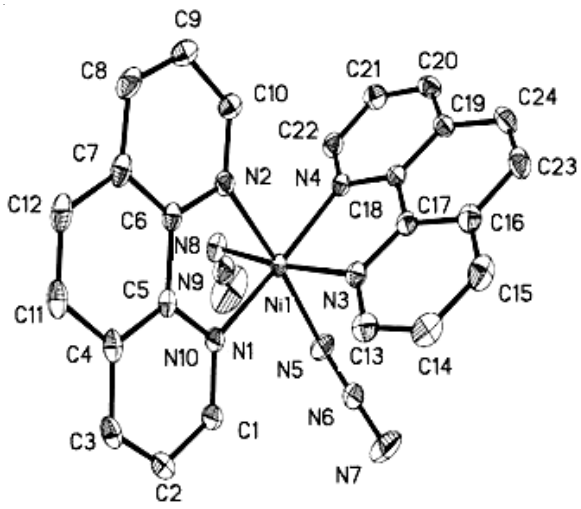

Fig. 1. Molecular structure of the complex $[Ni(phen)_2(N_3)_2]$

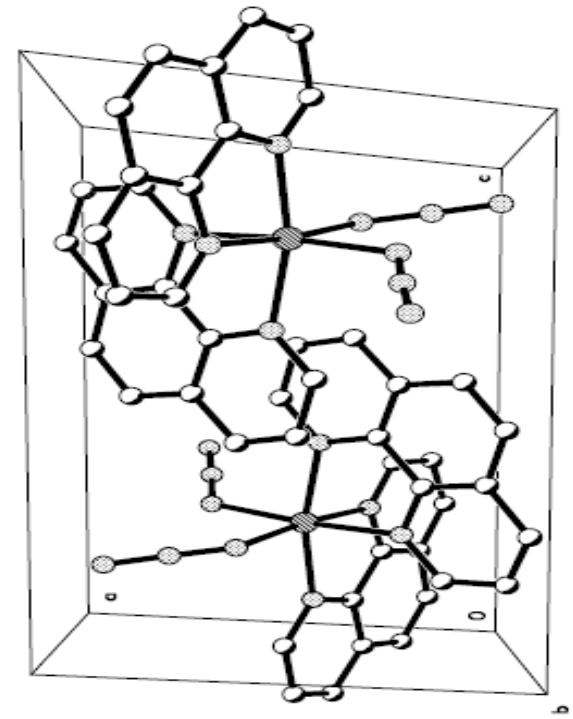

Fig. 2. Molecular packing arrangement in the unit cell

TABLE-1
NON-HYDROGEN ATOMIC COORDINATES ($\times 10^4$)
AND THERMAL PARAMETERS ($\times 10^3 Å^2$)

Atom	X	Y	Z	U(eq)
NI(1)	5172(1)	7118(1)	7652(1)	27(1)
N(2)	7337(3)	5481(2)	7685(2)	30(1)
N(3)	6727(3)	8376(2)	7339(2)	29(1)
N(4)	5378(3)	7446(2)	9234(2)	30(1)
N(5)	3008(3)	8702(2)	7470(2)	41(1)
N(6)	2966(3)	9661(2)	6912(2)	36(1)
N(7)	2863(4)	10634(3)	6382(3)	67(1)
N(8)	3653(3)	5901(2)	8147(2)	42(1)

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