Asian Journal of Chemistry; Vol. 27, No. 7 (2015), 2739-2740

ASIAN JOURNAL OF CHEMISTRY

http://dx.doi.org/10.14233/ajchem.2015.17928

NOTE

Hydrothermal Synthesis and Crystal Structure of Ni(II) Complex with Azamacrocycle

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Received: 1 May 2014; *Accepted*: 22 July 2014; *Published online*: 30 March 2015; AJC-17119

A new azamacrocyclic Ni(II) complex with the m.f. $C_{24}H_{44}N_8O_2Ni_2$ was synthesized by hydrothermal reaction and characterized by IR spectra and single-crystal X-ray diffraction. The crystal is in a Triclinic system, space group P-1 with unit cell parameters: a = 9.4238(3)Å, b = 9.44760(10) Å, c = 9.5006(3)Å, α = 77.706(2)º, β = 65.597(2)º, γ = 71.132(2)º, V = 725.81(3)Å^3, Z = 1, Mr = 594.09, Dc = 1.359 Mg/cm^3, μ = 1.332 mm^{-1}, F(000) = 316, T = 298(2) K, R = 0.0621, wR = 0.1534 for 3825 reflections with I > 2σ(I). The crystal structure analysis shows that the nickel(II) ion was coordinated by four nitrogen atoms of the azamacrocycle.

Keywords: Ni(II) complex, Azamacrocycle, Hydrothermal synthesis, Structure.

Recently, the coordination constructions toward metal ions of macrocyclic ligands have attracted much interest[1-3]. In our laboratory, a series of macrocyclic metal complexes were synthesized[4-9]. In this paper, a tetraazamacrocyclic nickel complex [NiL]·Ni(CN)$_4$ (L = 5,7,2,14-tetraethyl-7,14-dimethyl-1,4,8,11-tetraazamacrocyclic-4,11-diene) is reported.

All reagents were of AR grade and used without further purification. L·(ClO$_4$)$_2$ was prepared in our laboratory. IR spectrum was recorded on a Nexus-870 spectrophotometer. The crystal structure was determined by Siemens SMART CCD area-detector diffractometer.

Synthesis: An acetonitrile solution (10 mL) of L·(ClO$_4$)$_2$ (10 mmol) and aqueous solution (15 mL) of a mixed Ni(ClO$_4$)$_2$ (5 mmol) and K$_2$[Ni(CN)$_4$] (5 mmol) were carried out in a autoclave and heated to 80 ºC for 72 h. After cooling, the cubic dark green crystals were resulted. Yield 41 %. IR spectrum (KBr, ν_{max}, cm^{-1}): 3400, 2980, 1650, 1370, 1070, 980.

Crystal structure determination: A single crystal (1.10 mm × 0.36 mm × 0.08 mm) was selected for crystallographic data collection at 298(2) K and structure determinated with graphite monochromatic MoK$_\alpha$ radiation (λ = 0.71073Å). A total of 3825 reflections were collected in the range of 2.29º ≤ θ ≤ 25º, of which 11405 reflections were unique with Rint = 0.0381 and R = 0.0621 and wR = 0.1534, where w = 1/[s^2(F$_0^2$) + (0.0699P)^2 + 1.7665P], P = (F$_0^2$ + 2F$_0^2$)/3. The maximum and minimum peaks on the final difference Fourier map are corresponding to 0.731 and -0.486 e/Å^3 (CCDC No. 607358), respectively.

The atomic coordinates and thermal parameters are listed in Table-1 and the selected bond lengths and bond angles in Table-2. Respecting, Fig. 1 shows the molecular structure of the title compound. Fig. 2 shows the packing diagram of the title compound. From the Fig. 1, it is easy to see that the nickel (II) ion is four-coordinated with four nitrogen atoms of the tetraazamacrocycle.

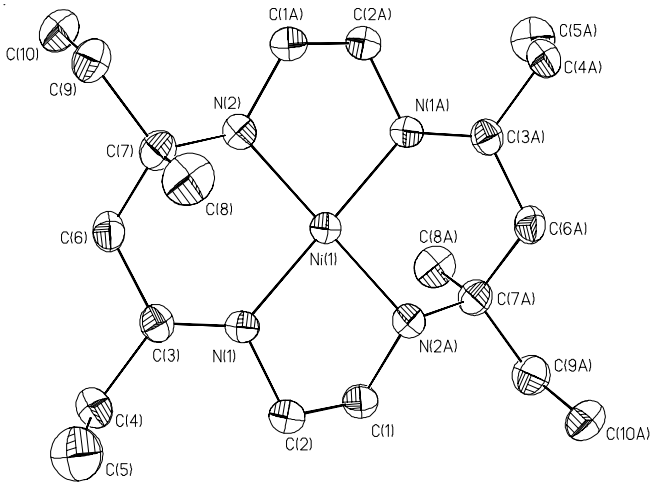

Fig. 1. Molecular structure of [NiL]·Ni(CN)$_4$

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

Atom	X	Y	Z	U(eq)
NI(1)	0	0	5000	42(1)
NI(2)	5000	5000	0	51(1)
N(1)	705(5)	1250(4)	5787(4)	46(1)
N(2)	836(5)	855(4)	2879(4)	50(1)
N(11)	2765(10)	7621(8)	-1115(8)	108(2)
N(12)	3570(8)	6163(7)	3147(6)	88(2)
C(11)	3598(8)	6609(7)	-671(7)	72(2)
O	-2177(5)	3188(7)	3695(6)	96(2)

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

Bond	Length	Angle	(°)	Angle	(°)
NI(1)-N(1)	1.920(4)	N(1)#1-NI(1)-N(1)	180.0(2)	N(1)#1-NI(1)-N(2)#1	93.73(16)
NI(1)-N(2)	1.934(4)	N(1)-NI(1)-N(2)	93.73(16)	C(12)#2-NI(2)-C(12)	180.00(1)
NI(2)-C(11)	1.857(7)	N(1)-NI(1)-N(2)#1	86.27(16)	C(12)#2-NI(2)-C(11)	89.4(3)
NI(2)-C(12)	1.856(6)	N(2)-NI(1)-N(2)#1	180.0	C(3)-N(1)-NI(1)	129.1(3)
N(1)-C(3)	1.280(6)	C(12)-NI(2)-C(11)	90.6(3)	C(1)#1-N(2)-NI(1)	107.9(3)
N(11)-C(11)	1.151(8)	N(11)-C(11)-NI(2)	178.1(8)	N(2)#1-C(1)-C(2)	108.1(4)

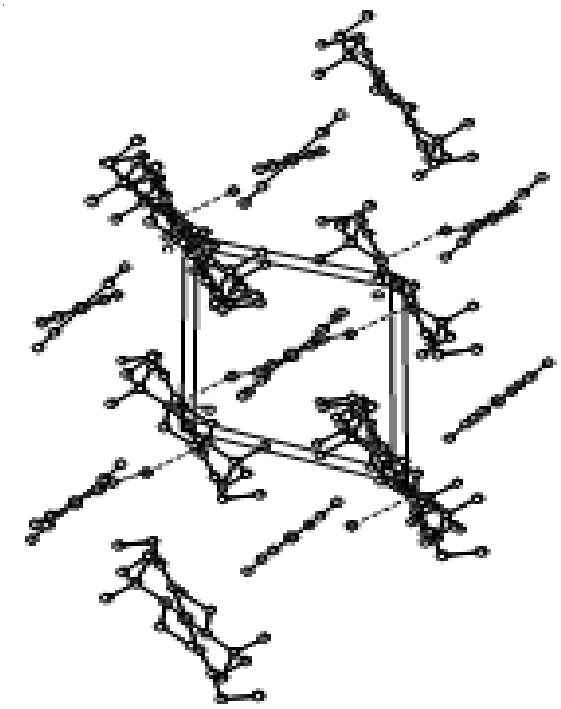

Fig. 2. Molecular packing arrangement in unit cell

ACKNOWLEDGEMENTS

This work is financially supported by the Natural Science Foundation of Anhui Province (No.1308085MB23), the National Natural Science Foundation of China (No. 20871039 and 21101053) and Key Discipline Foundation of Hefei Normal University.

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