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Research Article

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Study on a structure of Tris-(4,4'-dimethyl-2,2'-dipyridyl)-Ni iodine hydrate, $Ni(C_{12}H_{12}N_2)_3 \bullet 2I \bullet H_2O$

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ABSTRACT

A novel Ni metal complex $Ni(C_{12}H_{12}N_2)_3 \bullet 2I \bullet H_2O$ has been synthesized from a solution reaction and the crystal structure has been determined by means of single-crystal X-ray diffraction. Monoclinic, C2/c. a=12.5920(14) Å, b = 25.350(3) Å, c =12.9771(13) Å, α= 90°, β=100.912(3)°, γ=90°. V=4067.5(8). Z=4. R_1 =0.1152, wR_2 = 0.3725, T=293(2) K. The Ni atom is six-coordinated by six N atoms. The crystal packing is stabilized by O-H...I hydrogen bonding interactions.

Key words: Ni complex, structure analysis

INTRODUCTION

Inorganic–organic materials with 1-D, 2-D, and 3-D frameworks have attracted a lot of attention because of structural flexibility and applications in molecular absorption, catalysis, photochemistry, and electromagnetism [1-7]. The use of transition metal complexes of chelating 2,2-bipyridine to generate multidimensional infinite arrays or networks by way of hydrogen bonding is an area of ordinary interest in metal-coordination chemistry [8,9]. These hydrogen-bonding interactions have long-time been considered to be of importance in biological systems and in crystal engineering [10,11]. There are some reports that Ni complexes can accelerate the cleavage of double-stranded DNA, while other Ni complexes have excellent catalytic activity [12-14]. The presence of pyridine groups in the complexes provides the possibility to obtain highly stable metal complexes [15]. In this paper, the new Ni complex is reported.

EXPERIMENTAL SECTION

All commercially obtained reagent-grade chemicals were used without further purification. A mixture of $NiCl_2$ (0.1 mmol, 0.014g), 4,4'-dimethyl-2,2'-dipyridyl (0.01 mmol, 0.002g), KI (0.1 mmol, 0.02g) and LiOH (0.2 mmol, 0.005g) were added into 10 mL water and heated for 6h at 343K. The solution was obtained by filtration after cooling the reaction to room temperature. Red block single crystals suitable for X-ray measurements were obtained after a few weeks.

The crystal data was collected on a Bruker smart CCD Area Detector.

A red block single crystal with dimensions of 0.43 x 0.40 x 0.38 mm was selected for measurement. Diffraction data of the single crystal were collected by ψ-ω scan mode using a graphite-monochromatic Mo *K*α rediation (λ=0.71073 Å) at 293(2) K on a Bruker Smart Apex CCD diffractometer.

RESULTS AND DISCUSSION

The title crystal structure (Fig.1) is built up of one Ni cation, 4,4'-dimethyl-2,2'-dipyridyl, iodine and water molecular. The crystal data and structure refinement is shown in Table 1. The Ni atom is six-coordinated by six N atom from three 4,4'-dimethyl-2,2'-dipyridyl moleculars. The Ni-N bond lengths are in the range of 2.079-2.083 Å. The bond angles of N1-Ni-N1#, N1-Ni-N3, N2-Ni-N2# are 96.1, 92.9, 175.2°, respectively. The torsion angles of N1-Ni-N2-C6, N2-Ni-N1-C1, N3-Ni-N1-C5 are 15.2, 174.2, 76.2, respectively. Selected bond lengths and bond angles are shown in Table 2.

The crystal packing is stabilized by O-H...I bonding interactions.

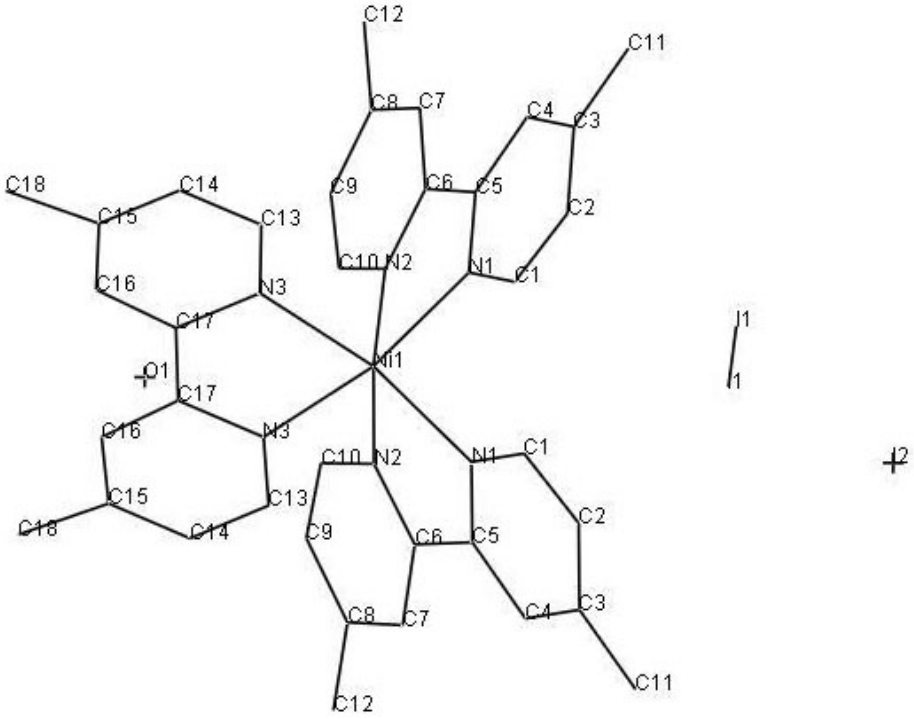

Fig.1 The molecular structure of C36H38I2N6NiO

Table 1. Crystal data and structure refinement for the title complex

Empirical formula	$C_{36}H_{38}I_2N_6NiO$
Formula weight	883.23
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system, space group	Monoclinic, C2/c
Unit cell dimensions	a = 12.5920(14) Å alpha = 90°.
	b = 25.350(3) Å beta = 100.912(3)°.
	c = 12.9771(13) Å gamma = 90°.
Volume	4067.5(8) $Å^3$
Z, Calculated density	4, 1.442 Mg/m^3
Absorption coefficient	2.028 mm^{-1}
F(000)	1752
Crystal size	0.43 x 0.40 x 0.38 mm
Theta range for data collection	3.18 to 25.02°.
Limiting indices	-14<=h<=14, -29<=k<=30, -15<=l<=15
Reflections collected / unique	15975 / 3584 [R(int) = 0.1251]
Completeness to theta = 25.02	99.8 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.5129 and 0.4760
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3584 / 0 / 222
Goodness-of-fit on F^2	1.249
Final R indices [I>2sigma(I)]	R1 = 0.1152, wR2 = 0.3322
R indices (all data)	R1 = 0.1844, wR2 = 0.3725
Largest diff. peak and hole	1.935 and -1.066 e.$Å^{-3}$

Table 2. Select bond lengths [Å] and angles [°] for the title complex

Ni(1)-N(1)	2.079(8)
Ni(1)-N(1)#1	2.079(8)
Ni(1)-N(3)#1	2.081(8)
Ni(1)-N(3)	2.081(8)
Ni(1)-N(2)	2.083(8)
Ni(1)-N(2)#1	2.083(8)
N(1)-C(1)	1.325(13)
N(1)-C(5)	1.340(12)
N(2)-C(6)	1.337(13)
N(2)-C(10)	1.359(14)
N(3)-C(13)	1.310(14)
N(3)-C(17)	1.365(13)
I(1)-I(1)#2	2.591(5)
C(1)-C(2)	1.360(15)
C(1)-H(1)	0.9300
C(2)-C(3)	1.399(15)
C(3)-C(4)	1.382(15)
C(3)-C(11)	1.476(15)
C(4)-C(5)	1.412(14)
C(5)-C(6)	1.470(14)
C(6)-C(7)	1.425(14)
C(7)-C(8)	1.359(16)
C(8)-C(9)	1.384(16)
C(8)-C(12)	1.504(15)
C(9)-C(10)	1.357(15)
C(13)-C(14)	1.379(16)
C(14)-C(15)	1.281(17)
C(15)-C(16)	1.336(18)
C(15)-C(18)	1.526(16)
C(16)-C(17)	1.411(15)
N(1)-Ni(1)-N(1)#1	96.1(4)
N(1)-Ni(1)-N(3)#1	168.9(3)
N(1)#1-Ni(1)-N(3)#1	92.9(3)
N(1)-Ni(1)-N(3)	92.9(3)
N(1)#1-Ni(1)-N(3)	168.9(3)
N(3)#1-Ni(1)-N(3)	79.0(5)
N(1)-Ni(1)-N(2)	78.1(3)
N(1)#1-Ni(1)-N(2)	98.7(3)
N(3)#1-Ni(1)-N(2)	94.2(3)
N(3)-Ni(1)-N(2)	89.6(3)
N(1)-Ni(1)-N(2)#1	98.7(3)
N(1)#1-Ni(1)-N(2)#1	78.1(3)
N(3)#1-Ni(1)-N(2)#1	89.6(3)
N(3)-Ni(1)-N(2)#1	94.2(3)
N(2)-Ni(1)-N(2)#1	175.2(4)
C(1)-N(1)-C(5)	119.1(9)
C(1)-N(1)-Ni(1)	126.3(7)
C(5)-N(1)-Ni(1)	114.3(7)
C(6)-N(2)-C(10)	116.8(9)
C(6)-N(2)-Ni(1)	113.9(7)
C(10)-N(2)-Ni(1)	127.3(7)
C(13)-N(3)-C(17)	118.6(9)
C(13)-N(3)-Ni(1)	127.4(7)
C(17)-N(3)-Ni(1)	113.7(7)
N(1)-C(1)-C(2)	123.5(10)
N(1)-C(1)-H(1)	118.3
C(1)-C(2)-C(3)	119.8(11)
C(4)-C(3)-C(2)	116.9(10)
C(4)-C(3)-C(11)	120.0(11)
C(2)-C(3)-C(11)	123.1(11)
C(3)-C(4)-C(5)	120.3(10)
N(1)-C(5)-C(4)	120.4(10)
N(1)-C(5)-C(6)	115.5(9)
C(4)-C(5)-C(6)	124.0(9)
N(2)-C(6)-C(7)	121.0(10)
N(2)-C(6)-C(5)	115.3(9)
C(7)-C(6)-C(5)	123.7(10)
C(8)-C(7)-C(6)	120.5(11)
C(7)-C(8)-C(9)	118.0(10)

C(7)-C(8)-C(12)	118.6(11)
C(9)-C(8)-C(12)	123.4(11)
C(10)-C(9)-C(8)	119.2(11)
C(9)-C(10)-N(2)	124.5(11)
N(2)-C(10)-H(10)	117.7
N(3)-C(13)-C(14)	121.0(12)
N(3)-C(13)-H(13)	119.5
C(15)-C(14)-C(13)	124.1(14)
C(14)-C(15)-C(16)	115.4(12)
C(14)-C(15)-C(18)	127.6(14)
C(16)-C(15)-C(18)	116.8(13)
C(15)-C(16)-C(17)	123.8(12)
N(3)-C(17)-C(16)	116.8(11)
N(3)-C(17)-C(17)#1	116.5(6)

CONCLUSION

A novel Ni metal complex $Ni(C_{12}H_{12}N_2)_3 \bullet 2I \bullet H_2O$ has been synthesized from a solution reaction and the crystal structure has been determined by means of single-crystal X-ray diffraction.

Acknowledgments
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REFERENCES

[1] O.M. Yaghi, M. O'Keeffe, N.W. Ockwig, H.K. Chae, M. Eddaoudi, *J. Kim. Nature*, 423, 705, **2003**.
[2] L. Han, M.C. Hong, R.H. Wang, J.H. Luo, Z.Z. Lin, D.Q. Yuan. *Chem. Commun.*, 2580, **2003**.
[3] N. Calin, S.C. Sevov. *Inorg, Chem.*, 42, 7304, **2003**.
[4] D. Drewes, E.M. Limanski, B. Krebs. Eur. *J. Inorg. Chem.*, 4849, **2004**.
[5] M.X. Yang, S. Lin, X.H. Chen, M.H. Luo, J.H. Liu. *J. Coord. Chem.*, 63, 406, **2010**.
[6] M.X. Yang, L.J. Chen, S. Lin, X.H. Chen, H. Huang. *Dalton Trans.*, 40, 1866, **2011**.
[7] XIAO-HUA CHEN, SHEN LIN, JIAO-HUA LIU, HUA HUANG, MING-XING YANG and LI-JUAN CHEN, *Journal of Coordination Chemistry*, 64, 3482, **2011**.
[8] A.J. Blake, N.R. Champness, P. Hubberstey, W.S. Li, M.A. Withersby and M. Schro¨ der, *Coord. Chem. Revs*, 183, 117, **1999**.
[9] M. Munakata, L.P. Wu, M. Yamamoto, T.K. Sowa and M. Maekawa, *J. Am. Chem. Soc*., 118, 3117, **1996**.
[10] X.F. Chen, P. Cheng, X. Liu, B. Zhao, D.Z. Liao, S.P. Yan and Z.H. Jiang, *Inorg. Chem.*, 40, 2652, **2001**.
[11] YOLANDA RODRIGUEZ-MARTIN, PABLO A. LORENZO-LUIS, PEDRO GILI and CATALINA RUIZ-PE REZ, *J. Coord. Chem.*, 56, 181, **2003**.
[12] G.W. Yang, X.P. Xia. *Chin. J. Appl. Chem.*, 12, 13, **1995**.
[13] G.W. Yang, D.F. Wang. *Chem. Res. Chin. Univ.*, 16, 55, **1995**.
[14] J.C. Xian, R.T. Li, G.W. Cao, H.K. Lin, S.R. Zhu. *Chin. J. Inorg. Chem.*, 16, 833, **2000**.
[15] BEI LIANG, XIANGRONG LIU, JIANJUN CHEN and KANSHE LI, *Journal of Coordination Chemistry*, 63, 1951, **2010**.