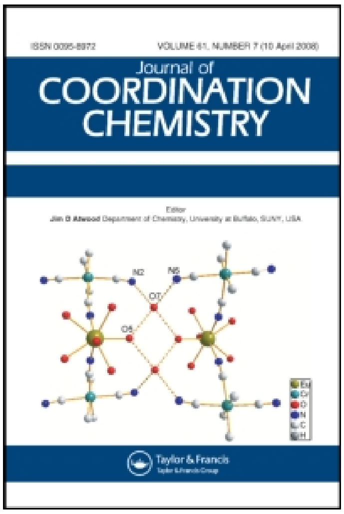


Journal of Coordination Chemistry
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713455674

Synthesis and characterization of picolinate-containing nickel(II) complexes with tridentate and tripodal tetradentate ligands

Feng-Mei Nie[a]; Jing Chen[a]; Zhen Li[a]; Fei Lu[a]
[a] Department of Chemistry, Capital Normal University, Beijing 100048, China

To cite this Article Nie, Feng-Mei , Chen, Jing , Li, Zhen and Lu, Fei(2010) 'Synthesis and characterization of picolinate-containing nickel(II) complexes with tridentate and tripodal tetradentate ligands', Journal of Coordination Chemistry, 63: 10, 1711 — 1719, First published on: 11 June 2010 (iFirst)

To link to this Article: DOI: 10.1080/00958972.2010.488690

URL: http://dx.doi.org/10.1080/00958972.2010.488690

Journal of Coordination Chemistry
Vol. 63, No. 10, 20 May 2010, 1711–1719

Taylor & Francis
Taylor & Francis Group

Synthesis and characterization of picolinate-containing nickel(II) complexes with tridentate and tripodal tetradentate ligands

FENG-MEI NIE*, JING CHEN, ZHEN LI and FEI LU

Department of Chemistry, Capital Normal University, Beijing 100048, China

(*Received 21 June 2009; in final form 19 February 2010*)

Two picolinate-containing nickel(II) complexes $[Ni(bbma)(pic)(H_2O)]ClO_4 \cdot CH_3OH$ (**1**) and $[Ni(ntb)(pic)]Cl \cdot CH_3OH \cdot 3H_2O$ (**2**) were synthesized and characterized by infrared, elemental analysis, UV-Vis, and X-ray diffraction analyses, where bbma is bis(benzimidazol-2-yl-methyl)amine, ntb is tris(2-benzimidazolylmethyl)amine, pic is the anion of picolinic acid. X-ray analysis shows that both complexes are mononuclear with picolinate coordinated to Ni(II) in a μ_2-N,O chelating mode. Both complexes adopt distorted octahedral geometry. Intermolecular N–H $\cdots$ O and O–H $\cdots$ O hydrogen bonds and π–π interactions in **1** and **2** are important in stabilization of the crystal structures.

Keywords: Nickel(II) complex; Picolinic acid; Benzimidazole; Crystal structure

1. Introduction

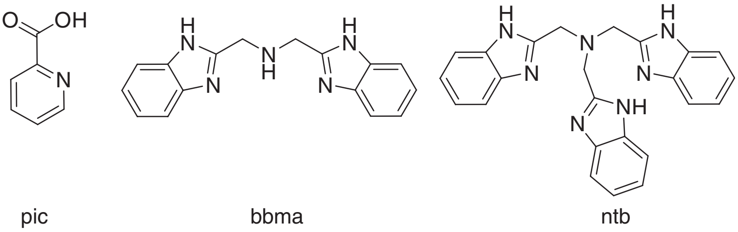
Histidine imidazole is present at the active site of numerous enzymes and plays a key role in their catalytic functions [1–3]. Therefore, complexes with imidazole-containing ligands are models for the active sites of the relevant metalloenzymes. Synthetically accessible poly-benzimidazole ligands bbma and ntb are often used to construct models of such enzymes [4, 5]; some nickel(II) complexes of bbma and ntb have been reported. For bbma, only the structures of type $[Ni(bbma)_2]X_2$ ($X = Cl^-$, ClO_4^-, and SO_4^{2-}) have been established by X-ray analysis [6–8]. For ntb, several nickel(II) complexes containing azide, water, and isonicotinate as coligands have been characterized *via* X-ray diffraction method [9–11]. To our knowledge, nickel(II) complexes of bbma and ntb with picolinate as coligand have not been reported.

Picolinic acid is a biologically important molecule and is involved in biochemical and physical applications (scheme 1). The anion of picolinic acid contains two carboxylate oxygens and one pyridyl nitrogen as potential donors. A number of picolinate transition

---

*Corresponding author. Email: niefm@mail.cnu.edu.cn

Journal of Coordination Chemistry
ISSN 0095-8972 print/ISSN 1029-0389 online © 2010 Taylor & Francis
DOI: 10.1080/00958972.2010.488690



Scheme 1. Chemical diagram of picolinic acid, bbma and ntb.

metal complexes have been reported [12–16]. Compared with other picolinate transition metal complexes, only a few nickel(II) complexes containing picolinates have been reported [17–20].

We are interested in preparation of nickel(II) picolinate complexes containing bbma and ntb for two reasons. First, we would like to see how the structural change of the main ligand from linear tridentate bbma to tetradentate tripodal ntb affects the coordination modes of picolinate as coligand. Second, each bbma and ntb contains two or three benzimidazole groups. When complexes of bbma and ntb were formed, the uncoordinated NH on the benzimidazole rings may act as hydrogen donors, the uncoordinated oxygen of picolinate and the counter anions as hydrogen acceptors, versatile intermolecular interactions, such as hydrogen bonds and strong π–π interactions may afford interesting supramolecular coordination assembly. Here we report two new complexes $[Ni(bbma)(pic)(H_2O)]ClO_4 \cdot CH_3OH$ (**1**) and $[Ni(ntb)(pic)]Cl \cdot CH_3OH \cdot 3H_2O$ (**2**), which were characterized by infrared (IR), elemental analysis (EA), UV-Vis, and X-ray diffraction analyses.

2. Experimental

2.1. *Reagents and measurements*

All chemicals used in this study were purchased from commercial sources. Bbma and ntb were synthesized according to the literature procedures [21, 22]. IR spectra were recorded on a Bruker TENSOR 27 FT-IR spectrophotometer with KBr pellets from 4000 to 400 cm^{-1}. UV-Vis spectra were measured on a TU-1800PC Ultraviolet-Visible spectrophotometer. EA (C, H, N) was performed using a Perkin-Elmer 240 elemental analyzer.

2.2. *Synthesis of the complexes*

2.2.1. Synthesis of $[Ni(bbma)(pic)(H_2O)]ClO_4 \cdot CH_3OH$ (1). Bbma (0.055 g, 0.2 mmol) in 10 mL of methanol was dropped carefully into a stirred methanol solution (20 mL) containing $Ni(ClO_4)_2 \cdot 6H_2O$ (0.073 g, 0.2 mmol), and a methanol solution (10 mL) of picolinic acid (0.025 g, 0.2 mmol) and piperidine (0.017 g, 0.2 mmol) was added. After stirring for 1 h, the filtrate was allowed to stand for several weeks to give blue crystals of **1**. Yield: 0.062 g (51%). FT-IR (KBr pellet, cm^{-1}): 3374 s, 3296 s, 2919 m, 2780 m, 1628 s, 1567 w, 1470 s, 1455 s, 1121 s, 1109 s, 750 m. Anal. Calcd for $C_{23}H_{25}NiN_6O_8Cl$ (%): C, 45.46; H, 4.15; N, 13.83. Found (%): C, 45.45; H, 4.18; N, 13.84.

2.2.2. Synthesis of [Ni(ntb)(pic)]Cl • CH_3OH • $3H_2O$ (2). To a stirred methanol solution (20 mL) containing $NiCl_2 \cdot 6H_2O$ (0.058 g, 0.2 mmol) and ntb (0.080 g, 0.2 mmol), a methanol solution (10 mL) of picolinic acid (0.025 g, 0.2 mmol) and piperidine (0.017 g, 0.2 mmol) was added dropwise. After stirring for half an hour, the filtrate of the resulting solution was allowed to stand for several weeks to give blue crystals of **2**. Yield: 0.075 g (53%). FT-IR (KBr pellet, cm^{-1}): 3362 s, 3187 s, 2924 m, 2740 m, 1626 s, 1595 s, 1452 s, 1390 s, 740 s. EA suggests that one lattice MeOH and H_2O are lost upon storage in air. Anal. Calcd for $C_{30}H_{29}NiN_8O_4Cl$ (%): C, 54.69; H, 4.44; N, 17.02. Found (%): C, 54.45; H, 4.44; N, 17.02.

2.3. *X-ray crystallography*

Crystals of **1** and **2** were mounted on Bruker P4 and Saturn724+ diffractometers, respectively. Determination of unit cell parameters and data collection were performed with Mo-Kα radiation ($\lambda = 0.71073$ Å). The structures were solved by direct methods and Fourier difference techniques and refined by full matrix least-squares using SHELXL-97 [23]. All non-hydrogen atoms were refined anisotropically. The hydrogens were located on calculated positions and refined isotropically. The crystal parameters and the details of data collection and refinement for the two complexes are listed in table 1, and selected bond lengths and angles are listed in tables 2, 3, and 4. Because the crystal size of **1** is small and the crystal is also not of good quality which might lead to decay during data collection, the *R*(int) value of **1** is high.

3. Results and discussion

3.1. *Description of the structures*

3.1.1. [Ni(bbma)(pic)(H_2O)](ClO_4) • CH_3OH (1). Reaction of $Ni(ClO_4)_2 \cdot 6H_2O$, bbma, and picolinic acid (depronated with piperidine) in the ratio of 1 : 1 : 1 in methanol led to the formation of **1**. The cation structure of **1** is shown in figure 1.

The structure of **1** consists of one $[Ni(bbma)(pic)(H_2O)]^+$, one perchlorate, and one methanol. The Ni(II) is coordinated with three nitrogens of bbma, one oxygen of water molecule, one pyridine nitrogen, and one oxygen of bidentate picolinate. The Ni(II) is in distorted octahedral coordination geometry. The equatorial plane is formed by O(1) from carboxylate of picolinate, O(3) of water, N(1) and N(4) from bbma. The bond lengths are 2.075(4) Å for Ni(1)–O(1), 2.080(4) Å for Ni(1)–O(3), 2.076(4) Å for Ni(1)–N(1), and 2.107(4) Å for Ni(1)–N(4). The equatorial bond angles are from 88.43(17)° to 93.34(16)°. The Ni(II) is displaced out of the mean equatorial plane by 0.136 Å toward N(6). The axial sites are occupied by amine N(3) from bbma and the pyridine N(6) of picolinate with N(3)–Ni(1)–N(6) angle of 170.38(19)°. The bond distance of Ni(1)–N(3) is 2.131(5) Å, longer than Ni(1)–N(6) (2.071(4) Å).

Table 1. Crystal data and refinement details for **1** and **2**.

Compound	**1**	**2**
Empirical formula	$C_{23}H_{25}ClN_6O_8Ni$	$C_{31}H_{35}ClN_8O_6Ni$
Formula weight	607.65	709.83
Temperature (K)	113(2)	93(2)
System, space group	Monoclinic, $P\ 2_1/n$	Monoclinic, $P\ 2_1/c$
Unit cell dimensions (Å, °)		
a	9.4841(19)	9.2509(16)
b	10.326(2)	18.791(3)
c	26.742(5)	18.973(3)
α	90	90
β	90.45(3)	102.390(3)
γ	90	90
Volume (Å^3), Z	2618.8(9), 4	3221.3(9), 4
Crystal size (mm^3)	0.16 × 0.14 × 0.12	0.47 × 0.37 × 0.23
Density (g m^{-3})	1.541	1.464
μ (mm^{-1})	0.902	0.742
$F(000)$	1256	1480
θ range (°)	1.52–25.01	3.09–27.49
Limiting indices	$-11 \le h \le 11$	$-8 \le h \le 12$
	$-12 \le k \le 12$	$-24 \le k \le 19$
	$-31 \le l \le 31$	$-19 \le l \le 24$
Reflections collected/unique	20,947/4528 [R(int) = 0.1663]	18,476/7198 [R(int) = 0.0248]
Goodness-of-fit on F^2	1.039	1.004
Data/restraints/parameters	4528/100/406	7198/7/450
R_1 and wR_2 [$I > 2\sigma(I)$]	$R_1 = 0.0842$ $wR_2 = 0.1972$	$R_1 = 0.0359$ $wR_2 = 0.0808$
R indices (all data)	$R_1 = 0.1047$ $wR_2 = 0.2123$	$R_1 = 0.0411$ $wR_2 = 0.0843$
Largest difference peak and hole (e Å^{-3})	1.509/−0.930	0.371/−0.291

Table 2. Selected bond lengths (Å) and angles (°) for **1**.

Ni(1)–N(6)	2.071(4)	Ni(1)–O(3)	2.080(4)
Ni(1)–O(1)	2.075(4)	Ni(1)–N(4)	2.107(4)
Ni(1)–N(1)	2.076(4)	Ni(1)–N(3)	2.131(5)
N(6)–Ni(1)–O(1)	80.55(16)	N(1)–Ni(1)–N(4)	158.88(18)
N(6)–Ni(1)–N(1)	99.31(16)	O(3)–Ni(1)–N(4)	88.58(17)
O(1)–Ni(1)–N(1)	91.69(16)	N(6)–Ni(1)–N(3)	170.38(19)
N(6)–Ni(1)–O(3)	93.74(18)	O(1)–Ni(1)–N(3)	89.88(17)
O(1)–Ni(1)–O(3)	174.23(16)	N(1)–Ni(1)–N(3)	79.78(16)
N(1)–Ni(1)–O(3)	88.43(17)	O(3)–Ni(1)–N(3)	95.81(18)
N(6)–Ni(1)–N(4)	101.74(17)	N(4)–Ni(1)–N(3)	79.74(17)
O(1)–Ni(1)–N(4)	93.34(16)		

Adjacent molecules of **1** are interlinked by intermolecular H-bonds through methanol forming a 1-D chain. Two H-bonds are formed for each methanol, one between the uncoordinated carboxylate O(8) of picolinate and OH of methanol [O(8) ⋯ O(2) 2.717(6) Å, O(8)–H(8) ⋯ O(2) 157.00°] and the other between methanol O(8) and coordinated water [O(3) ⋯ O(8) 2.736(6) Å, O(3)–H(3C) ⋯ O(8) 162(6)°]. Neighboring antiparallel 1-D chains are interlinked by intermolecular H-bond interactions involving NH of bbma and uncoordinated carboxylate oxygen of

Table 3. Selected bond lengths (Å) and angles (°) for **2**.

Ni(1)–N(2)	2.0375(15)	Ni(1)–N(4)	2.0745(15)
Ni(1)–N(6)	2.0401(15)	Ni(1)–N(8)	2.1147(15)
Ni(1)–O(1)	2.0452(12)	Ni(1)–N(1)	2.2412(14)
N(2)–Ni(1)–N(6)	88.71(6)	O(1)–Ni(1)–N(8)	79.53(5)
N(2)–Ni(1)–O(1)	94.71(5)	N(4)–Ni(1)–N(8)	91.93(6)
N(6)–Ni(1)–O(1)	104.41(5)	N(2)–Ni(1)–N(1)	80.88(5)
N(2)–Ni(1)–N(4)	92.05(6)	N(6)–Ni(1)–N(1)	78.38(5)
N(6)–Ni(1)–N(4)	157.03(6)	O(1)–Ni(1)–N(1)	174.79(5)
O(1)–Ni(1)–N(4)	98.40(5)	N(4)–Ni(1)–N(1)	79.08(5)
N(2)–Ni(1)–N(8)	173.43(6)	N(8)–Ni(1)–N(1)	105.02(5)
N(6)–Ni(1)–N(8)	89.66(6)		

Table 4. Selected hydrogen bond parameters in **1** and **2**.

Compound	D–H···A	D···A (Å)	DHA (°)	Symmetry code of A
1	O3–H3B···O8	2.736(6)	162(6)	$-x+3/2,\ y+1/2,\ -z+3/2$
	N5–H5A···O2	2.746(6)	165(6)	$-x+3/2,\ y+1/2,\ -z+3/2$
	O8–H8···O2	2.717(6)	157.00	$-x+3/2,\ y+1/2,\ -z+3/2$
2	O3–H3A···O2	2.6685(19)	164.00	
	N3–H3N···O5	2.797(2)	158.00	$1-x,\ 1-y,\ 1-z$
	O4–H4A···O2	2.832(2)	163(3)	
	O4–H4B···Cl1	3.2247(17)	158(2)	$-x,\ -1/2+y,\ 3/2-z$
	O5–H5A···O6	2.778(2)	168(2)	$1-x,\ 1-y,\ 1-z$
	O5–H5B···Cl1	3.2671(17)	169(2)	
	N5–H5N···O3	2.774(2)	170.00	$x,\ 1/2-y,\ -1/2+z$
	O6–H6A···O3	2.829(2)	165(3)	$1-x,\ 1/2+y,\ 3/2-z$
	O6–H6B···Cl1	3.1294(18)	175(3)	
	N7–H7N···O4	2.809(2)	167.00	$-x,\ 1/2+y,\ 3/2-z$

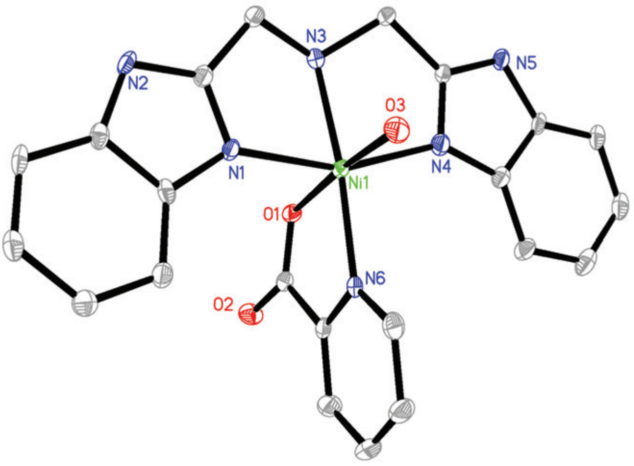


Figure 1. Perspective view of the cation structure of **1** with 30% probability thermal ellipsoids.

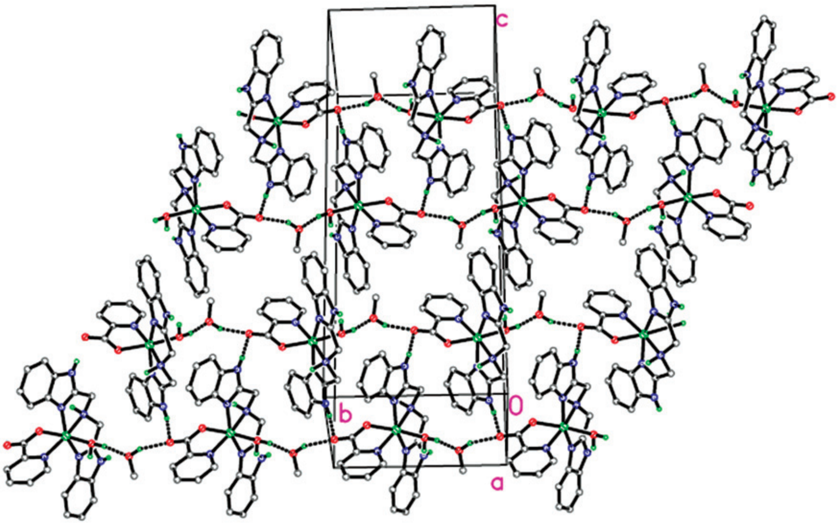


Figure 2. 1-D chain structure of **1** with hydrogen bonds. The broken lines indicate intermolecular hydrogen bonds.

picolinate [N(5)$\cdots$O(2) 2.746(6) Å, N(5)–H(5A)$\cdots$O(2) 165(6)°] to form a new 1-D chain structure (figure 2).

This new 1-D chain is further expanded to a 2-D network through offset π–π stacking interactions of adjacent aromatic benzimidazole rings of bbma ($\pi\cdots\pi = 3.614$ Å) and hydrogen bonding interactions involving NH of bbma and water. These hydrogen bonds and π–π stacking interactions enhance the stability of **1**.

3.1.2. [Ni(ntb)(pic)]Cl•CH_3OH•$3H_2O$ (2).

Complex **2** was prepared by $NiCl_2 \cdot 6H_2O$, ntb and deprotonated picolinic acid in 1 : 1 : 1 ratio in methanol. The perspective drawing for **2** with atom labeling scheme is illustrated in figure 3. Complex **2** contains discrete monomeric $[Ni(ntb)(pic)]^+$, one methanol, three water molecules, and a chloride (figure 3). The Ni(II) is octahedrally coordinated by four nitrogens of ntb, one carboxylate oxygen, and pyridine nitrogen of bidentate picolinate. The equatorial plane is defined by three benzimidazoles N(2), N(4), N(6) of ntb and the pyridine nitrogen N(8) of picolinate with bond angles in the range 88.71(6)°–92.05(6)°. The bond lengths of the three Ni–N (benzimidazole) bonds are 2.0375(15) Å for Ni(1)–N(2), 2.0745(15) Å for Ni(1)–N(4), and 2.0401(15) Å for Ni(1)–N(6). The Ni(1)–N(8) (pyridine) distance is 2.1147(15) Å, slightly longer than those observed for benzimidazole nitrogens. The axial sites are occupied by the tertiary amine nitrogen N(1) of ntb and one carboxylate O(1) of picolinate with O(1)–Ni(1)–N(1) angle of 174.79(5)°. The bond length of Ni(1)–O(1) is 2.0452(12) Å and the bond distance of Ni(1)–N(1) is 2.2412(14) Å, 0.1905(15) Å longer than those for benzimidazole. Other bond lengths and angles associated with ntb and pic are as expected [9–11, 24]. As shown in figure 4, extensive hydrogen bonds exist in **2**. Hydrogen bonds involve lattice methanol, water, and NH group of ntb as well as lattice chloride, generating a 3-D architecture.

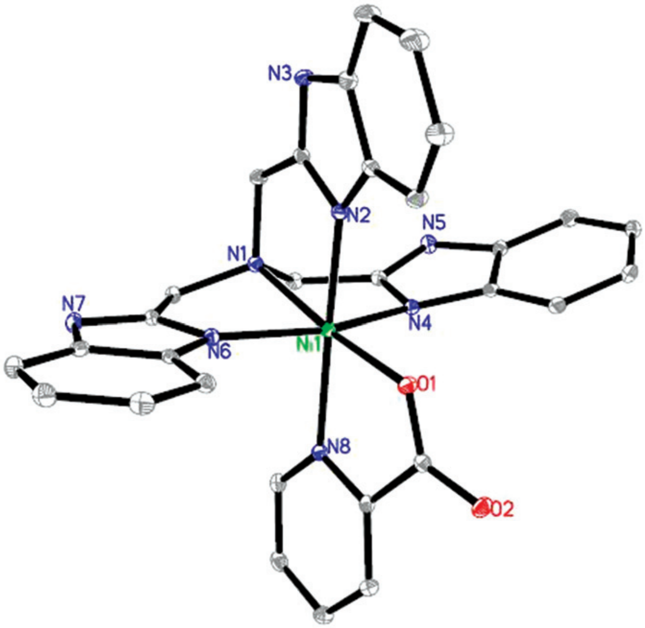


Figure 3. Perspective view of the cation structure of **2** with 30% probability thermal ellipsoids.

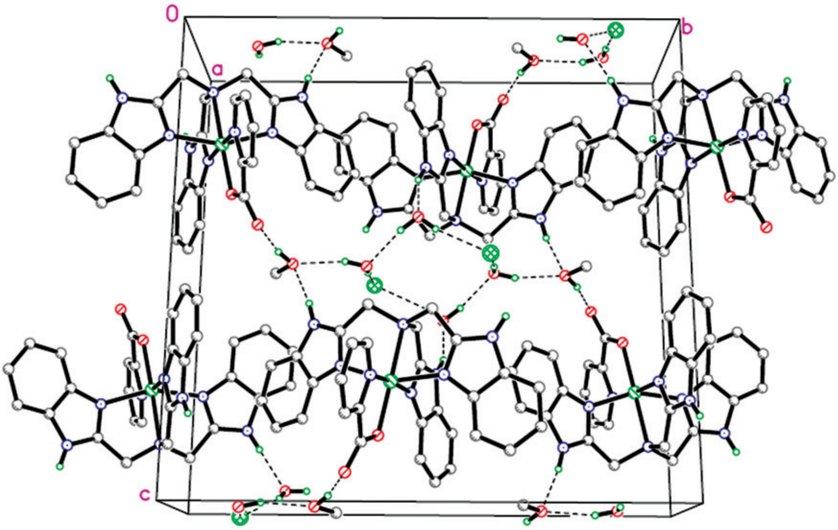


Figure 4. 3-D network structure of **2** with hydrogen bonds. The broken lines indicate intermolecular hydrogen bonds.

3.2. *Characterizations of the complexes*

3.2.1. IR spectra. Rather broad absorptions from 3100 to 3500 cm^{-1} of **1** and **2** are associated with the N–H stretching vibration of the benzimidazole ring and hydrogen-bonded water. The strong absorption at 1109 cm^{-1} indicates that perchlorate is present in **1** [25]. Strong bands at 1626, 1452 cm^{-1} and 1628, 1455 cm^{-1} could be attributed to anti-symmetric and symmetric vibrations of carboxylate.

3.2.2. UV-Vis spectra. The electronic spectra of **1** and **2** in DMF display three bands in the region of 300–1100 nm at *ca* 356, 593, and 1066 nm for **1** and at 337, 570, and 1044 nm for **2**, consistent with octahedral nickel(II) complexes [26, 27]. The high energy band can be assigned as $^3A_{2g} \rightarrow {}^3T_{1g}(P)$ and the other two as $^3A_{2g} \rightarrow {}^3T_{2g}(F)$, $^3A_{2g} \rightarrow {}^3T_{1g}(F)$ [9]. These findings were consistent with the X-ray results.

4. Conclusion

Two new picolinate-containing nickel(II) complexes $[Ni(bbma)(pic)(H_2O)](ClO_4) \cdot CH_3OH$ (**1**) and $[Ni(ntb)(pic)]Cl \cdot CH_3OH \cdot 3H_2O$ (**2**) with poly-benzimidazole tridentate and tripodal tetradentate ligand systems were synthesized and structurally characterized. Both are mononuclear with picolinate coordinated to nickel(II) in N,O-bidentate chelating mode and adopt distorted octahedral geometry. As bbma and ntb contain two or three NH groups from their benzimidazole side arm and the picolinate anion contains carboxylate oxygen, extensive hydrogen bonds are formed in the above two complexes.

Supplementary material

CCDC 716918 and 716919 contain the supplementary crystallographic data for **1** and **2** in CIF format. These data can be obtained free of charge *via* http://www.ccdc.cam.ac.uk or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB21EZ, UK; Fax: (+44) 1223-336-033; or E-mail: deposit@ccdc.cam.ac.uk.

Acknowledgments

This study was supported by Beijing Natural Science Foundation (Grant no. 2092010), Beijing Municipal Education Commission research program (KM200810028009) and the Scientific Research Foundation for Returned Overseas Chinese Scholars, Beijing Municipal Government.

References

[1] S.E. Castillo-Blum, N. Barba-Behrens. *Coord. Chem. Rev.*, **196**, 3 (2000).
[2] L.-H. Yin, P.-Y. Bu, P. Cheng, J. Li, S.-P. Yan, Z.-H. Jiang, D.-Z. Liao. *J. Coord. Chem.*, **55**, 537 (2002).

[3] L.J. Garcia, C.J. Server, E. Coret, J.V. Folgado, E. Escriva, R. Ballesteros. *Inorg. Chim. Acta*, **245**, 75 (1996).
[4] F.-M. Nie, F. Lu, J. Chen, Y.-L. Yang. *Inorg. Chim. Acta*, **362**, 4198 (2009).
[5] A.W. Addison, H.M.J. Hendriks, J. Reedijk, L.K. Thompson. *Inorg. Chem.*, **20**, 103 (1981).
[6] J.-Y. Xu, W. Gu, L. Li, S.-P. Yan, P. Cheng, D.-Z. Liao, Z.-H. Jiang. *J. Mol. Struct.*, **644**, 23 (2003).
[7] (a) T. Pandiyan, S. Bernes, C. Duran de Bazua. *Polyhedron*, **16**, 2819 (1997); (b) M. Velusamy, M. Palaniandavar, K.R.J. Thomas. *Polyhedron*, **17**, 2179 (1998).
[8] Y.-G. Chen, Z.-K. He, L. Zhang, Q. Chen, J.-L. Lu, Z.-L. Deng, L. Chen, W.-D. Wang. *Huaxue Xuebao*, **66**, 1651 (2008).
[9] J.-L. Tian, M.-J. Xie, Z.-Q. Liu, S.-P. Yan, D.-Z. Liao, Z.-H. Jiang. *J. Coord. Chem.*, **58**, 833 (2005).
[10] F.-M. Miao, W.-H. Zhou, J.-G. Han, Z. Fan, J.-L. Wang, H.-G. Wang, X.-K. Yao, J.-Z. Huo, Y.-H. Qiao. *Wuji Huaxue Xuebao*, **12**, 234 (1996).
[11] F.-M. Nie, Z. Li, Z.-Y. Dong, Y.-L. Yang. *Z. Kristallogr. NCS*, **224**, 437 (2009).
[12] D.D.D. Hepburn, J.P. Vicente. *J. Inorg. Biochem.*, **94**, 86 (2003).
[13] M.S. Kanth, A. Mishra. *Orient. J. Chem.*, **21**, 537 (2005).
[14] S.L. Jain, P. Bhattacharyya. *J. Chem. Soc., Dalton Trans.*, **16**, 2691 (2005).
[15] K.S. Yamaguchi, L. Spencer, D.T. Sawyer. *FEBS*, **197**, 249 (1986).
[16] Z. Heren, H. Pasaoglu, G. Kastas, C. Keser, O.Z. Yesilel. *Z. Anorg. Allg. Chem.*, **632**, 1578 (2006).
[17] H. Loiseleur. *Acta Crystallogr.*, **28**, 816 (1972).
[18] A.-Q. Wu, G.-H. Guo, F.-K. Zheng, G.-C. Guo, J.-S. Huang. *Acta Crystallogr.*, **E60**, m571 (2004).
[19] A.M. Barrios, S.J. Lippard. *J. Am. Chem. Soc.*, **121**, 11751 (1999).
[20] K.-F. Ma, Q. Shi, M.-L. Hu, X.-Q. Cai, S.-M. Huang. *Inorg. Chim. Acta*, **362**, 4926 (2009).
[21] U. Sivagnanam, T. Pandiyan, M. Palaniandavar. *Indian J. Chem.*, **32B**, 572 (1993).
[22] C. Nazikkol, R. Wegner, J. Bremer, B. Krebs. *Z. Anorg. Allg. Chem.*, **662**, 329 (1996).
[23] G.M. Sheldrick. *SHELX-97*, *Program for Crystal Structure Refinement*, University of Göttingen, Germany (1997).
[24] C. Boskovic, A. Sieber, G. Chaboussant, H.U. Gdel, J. Ensling, W. Wernsdorfer, A. Neels, G. Labat, H. Stoeckli-Evans, S. Janssen. *Inorg. Chem.*, **43**, 5053 (2004).
[25] H.-L. Wu, W.-K. Dong, Y. Chen. *J. Coord. Chem.*, **60**, 1269 (2007).
[26] L.K. Thompson, B.S. Ramaswamy, R.D. Dawe. *Can. J. Chem.*, **56**, 1311 (1978).
[27] A.B.P. Lever. *Coord. Chem. Rev.*, **3**, 119 (1968).