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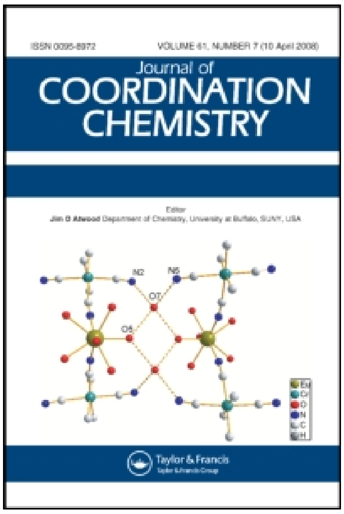

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Synthesis and characterization of picolinato and nicotinato cobalt(II) complexes containing tris(2-benzimidazolylmethyl)amine

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Synthesis and characterization of picolinato and nicotinato cobalt(II) complexes containing tris(2-benzimidazolylmethyl)amine

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Two new mononuclear cobalt(II) complexes $[Co(ntb)(pic)](ClO_4) \cdot (CH_3OH)_{2.35}$ (**1**) and $[Co(ntb)(nic)](ClO_4) \cdot CH_3OH$ (**2**) were synthesized and structurally characterized, where ntb is tris(2-benzimidazolylmethyl)amine, pic is the anion of picolinic acid, and nic is the anion of nicotinic acid. The X-ray analysis indicates that the Co(II) center is six-coordinate in distorted octahedral and five-coordinate in distorted trigonal bipyramidal geometry for **1** and **2**, respectively. In **1**, the picolinate anion coordinates to Co(II) in a bidentate μ_2-N,O chelating mode. In **2**, the nicotinate anion coordinates with Co(II) through a monodentate carboxylate oxygen. 1-D chain structures were formed by intermolecular hydrogen bonds in the two complexes and $\pi - \pi$ interactions are important for the stabilization of the structures.

Keywords: Cobalt(II) complex; Picolinic acid; Nicotinic acid; Crystal structure; Tris(2-benzimidazolylmethyl)amine

1. Introduction

The imidazole group of histidine plays a key role in the active sites of many metalloproteins [1, 2]. Ligands containing imidazole and benzimidazole are of great interest due to their similarity to histidine imidazole, one of them is the benzimidazole-derived ligand tris(2-benzimidazolylmethyl)amine (ntb) (scheme 1). Various metal complexes containing ntb have been studied [3–22] since the first report of the synthesis of ntb in 1977 [23]; their synthesis and structures have also been reviewed [24, 25]. To our knowledge, only a few structurally characterized cobalt(II) complexes with ntb were reported in which H_2O [26], NCS^- [27], and Cl^- [28–31] were used as coligands, but that with picolinic acid and nicotinic acid as coligands have not been reported. In this study, two new mononuclear cobalt(II) complexes with ntb $[Co(ntb)(pic)](ClO_4) \cdot (CH_3OH)_{2.35}$ (**1**) and $[Co(ntb)(nic)](ClO_4) \cdot CH_3OH$ (**2**) were synthesized and characterized for the first time.

Picolinic acid and nicotinic acid were chosen as coligands in our study mainly due to the following reasons: (1) Picolinic acid and nicotinic acid are isomers of pyridine

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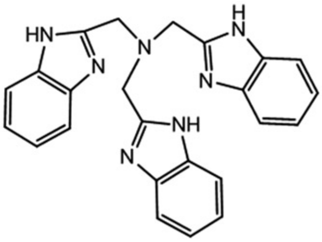
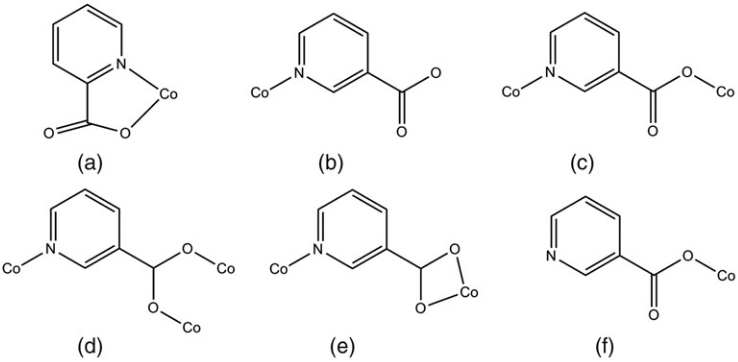


Scheme 1. Structure of ntb ligand.



Scheme 2. Bonding modes of picolinic acid (a) and nicotinic acid (b–f) in cobalt(II) complexes.

carboxylic acid with both N and O donors and exhibit different coordination modes in previously published cobalt(II) complexes as shown in scheme 2(a–e). Picolinic acid usually coordinates to Co(II) in a chelating μ_2-N,O mode (scheme 2a) [32–35]; nicotinic acid coordinates to Co(II) in four bonding modes, monodentate with pyridine nitrogen (scheme 2b) [36–38], bidentate with both N and O (scheme 2c) [39] and tridentate μ_3-N,O,O (scheme 2d, e) [40–42]. The bonding of picolinic acid and nicotinic acid may be affected by CoN_4 coordination environment formed by Co(II) and ntb. (2) Picolinic acid and nicotinic acid are able to link metals to form various 0-D, 1-D, 2-D, or 3-D structures through hydrogen bonds, $\pi-\pi$ stacking, or other weak interactions [43–45].

In the two cobalt(II) complexes of our study, picolinate coordinates to Co(II) in the bidentate μ_2-N,O chelating mode and the nicotinate in the monodentate mode with carboxylate oxygen (scheme 2f). Co(II) ion in **1** is six-coordinate in distorted octahedral coordination geometry; this is a new example comparing with the previously reported five-coordinate cobalt(II) complexes of ntb [26–31].

2. Experimental

2.1. *Reagents and measurements*

All chemicals and solvents were purchased from commercial sources and used without purification. Sodium nicotinate was readily prepared by reacting nicotinic acid with a stoichiometric amount of NaOH in water. Ligand ntb was synthesized according to the published procedures [46]. Elemental C, H, and N analyses were performed using an Elemental Vario MICRO CUBE (Germany) elemental analyzer. IR spectra were taken as KBr pellets and recorded on a Bruker TENSOR 27 FT-IR spectrophotometer from 4000 to 400 cm^{-1}. The UV-Vis spectrum was measured on a TU-1800PC UV-Vis spectrophotometer from 200 to 900 nm.

2.2. *Synthesis of the complexes*

2.2.1. Synthesis of $[Co(ntb)(pic)](ClO_4)\cdot(CH_3OH)_{2.35}$ (1). Solid ntb (0.081 g, 0.2 mmol) was added to a stirred methanol (15 mL) solution of $Co(ClO_4)_2\cdot 6H_2O$ (0.073 g, 0.2 mmol) to give a clear pink solution; to this solution, a methanol (15 mL) solution of picolinic acid (0.025 g, 0.2 mmol) and piperidine (0.017 g, 0.2 mmol, to deprotonate picolinic acid) was added dropwise. The resulting solution was stirred for 4 h and then filtered. Dark red crystals of the quality of X-ray structure analysis were formed by slow evaporation of the filtrate at room temperature for 1 week. Yield: 0.089 g (59%). FT-IR (KBr pellet, cm^{-1}): 3420 s/br, 3206 s, 2924 m, 1644 s, 1621 s, 1595 s, 1474 s, 1454 s, 1371 s, 1121 s, 746 s. The elemental analysis shows that 1.35 lattice methanols were replaced by one water during storage in air. Anal. Calcd for $C_{31}H_{31}ClCoN_8O_8$: C, 50.45; H, 4.23; N, 15.18. Found: C, 50.67; H, 3.89; N, 15.57.

2.2.2. Synthesis of $[Co(ntb)(nic)](ClO_4)\cdot CH_3OH$ (2). To a stirred methanol (20 mL) solution of $Co(ClO_4)_2\cdot 6H_2O$ (0.073 g, 0.2 mmol), solid ntb (0.081 g, 0.2 mmol) was added to form a clear pink solution. To this solution, a methanol (25 mL) solution of sodium nicotinate (0.029 g, 0.2 mmol) was added dropwise; the purple solution was stirred for 4 h and then filtered. After 2 weeks, purple crystals were obtained by slow evaporation of the filtrate at room temperature. Yield: 0.092 g (63%). FT-IR (KBr pellet, cm^{-1}): 3420 s/br, 3231 s/br, 2915 m, 2767 m, 1617 s, 1598 s, 1472 s, 1455 s, 1375 s, 1120 s, 745 s. Anal. Calcd for $C_{31}H_{29}CoN_8O_7Cl$: C, 51.70; H, 4.07; N, 15.57. Found: C, 51.36; H, 3.81; N, 15.81.

2.3. *X-ray crystallography*

The crystals of **1** and **2** were mounted on the Rigaku Saturn724+ diffractometer and Bruker Smart 1000 area detector, respectively, both equipped with graphite-monochromated Mo-Kα radiation ($\lambda = 0.71073$ Å). The determination of unit cell parameters and data collection were performed at 93(2) K and 293(2) K for **1** and **2**, respectively. Empirical absorption correction was applied to the collected reflections [47]. First, the structures were solved by direct methods, and second by Fourier difference techniques and refined using full-matrix least-squares methods *via* SHELXL-97 [48]. The non-hydrogen atoms were subjected to anisotropic refinement and the hydrogens were located geometrically. One methanol in **1** was disordered and modeled to locate at three positions with occupancy of 0.608 for C(33), O(9), 0.395 for C(34), O(10), 0.347 for C(32), O(8) atoms; the total occupancy of this disordered methanol molecule was 1.35. The crystal parameters and details of the data collection and refinement for the two complexes are listed in table 1.

Table 1. Crystallographic data and structures refinement for **1** and **2**.

Compound	**1**	**2**
Empirical formula	$C_{32.35}H_{34.40}ClCoN_8O_{8.35}$	$C_{31}H_{29}ClCoN_8O_7$
Formula weight	763.26	720.00
Temperature (K)	93(2)	293(2)
Crystal system	Triclinic	Triclinic
Space group	$P\bar{1}$	$P\bar{1}$
Unit cell dimensions (Å, °)		
a	9.815(2)	9.886(4)
b	13.324(3)	12.550(5)
c	14.642(3)	13.581(6)
α	84.740(10)	82.289(7)
β	74.780(9)	80.810(7)
γ	69.511(7)	76.316(7)
Volume (Å^3), Z	1730.8(7), 2	1608.0(11), 2
Calculated density (g m^{-3})	1.465	1.487
$F(000)$	791	742
Crystal size (mm^3)	0.43 × 0.37 × 0.23	0.30 × 0.25 × 0.20
Absorption coefficient (mm^{-1})	0.637	0.677
θ range (°)	3.01–27.00	1.68–25.03
Limiting indices	$-12 \le h \le 12$; $-16 \le k \le 17$; $-14 \le l \le 18$	$-11 \le h \le 9$; $-12 \le k \le 14$; $-16 \le l \le 16$
Reflections collected	13,581	6679
Independent reflection	7275 [R(int) = 0.0309]	5648 [R(int) = 0.0277]
Data/restraints/parameters	7275/0/499	5648/0/435
Goodness-of-fit on F^2	1.001	1.007
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0490$, $wR_2 = 0.1205$	$R_1 = 0.0523$, $wR_2 = 0.1225$
R indices (all data)	$R_1 = 0.0570$, $wR_2 = 0.1272$	$R_1 = 0.0947$, $wR_2 = 0.1391$
Largest difference peak and hole (e Å^{-3})	0.437 and −0.596	0.625 and −0.395

3. Results and discussion

3.1. *Description of the structures*

3.1.1. [Co(ntb)(pic)](ClO_4)·(CH_3OH)$_{2.35}$ (1). Complex **1** was obtained by the reaction of $Co(ClO_4)_2 \cdot 6H_2O$, ntb, and picolinate in the ratio of 1 : 1 : 1 in methanol. The cation structure of **1** is shown in figure 1 and the main bond distances and angles are listed in table 2. The structure of **1** is composed of $[Co(ntb)(pic)]^+$, one perchlorate anion, and 2.35 methanols in which one is disordered with the total occupancy of 1.35. The central Co(II) is six-coordinate in a distorted octahedral N_5O ligand set formed by four nitrogens of ntb and one pyridine N(8) and one carboxylate O(1) of picolinate in bidentate μ_2-N,O chelating mode. Two benzimidazolyl nitrogens N(4) and N(6), tertiary amine N(1), and O(1) define the equatorial plane and Co(1) lies 0.114 Å above the plane toward N(2). Equatorial bond angles are in the range 73.88(7)–114.90(8)°, deviating from 90° remarkably. The axial positions are occupied by N(2) and N(8) with bond lengths of 2.091(2) Å for Co(1)–N(2) and 2.213(2) Å for Co(1)–N(8). The bond distance between Co(1) and tertiary amine N(1) (0.367 Å) is significantly longer than Co(1)–N(benzimidazolyl) (average 2.082(2) Å); this elongation could also be observed in other Co(II) complexes containing ntb [27, 29–31]. The *trans* angles of N(2)–Co(1)–N(8), 169.10(8)°, N(1)–Co(1)–O(1), 166.48(7)°, and N(6)–Co(1)–N(4), 136.30(8)° deviated from 180° for the ideal octahedron.

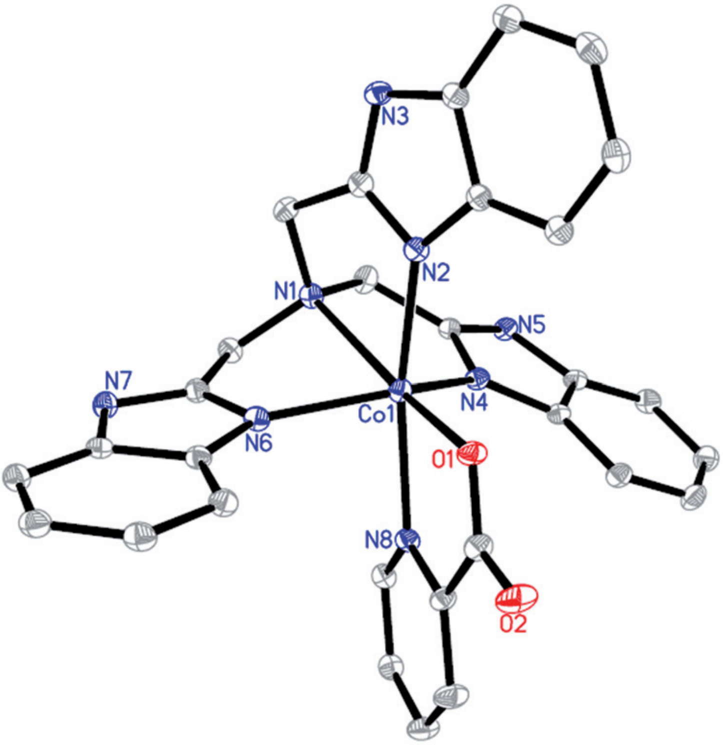


Figure 1. The cation structure of **1** (30% probability thermal ellipsoids).

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

Co(1)–O(1)	2.0441(18)	Co(1)–N(4)	2.064(2)
Co(1)–N(2)	2.091(2)	Co(1)–N(6)	2.092(2)
Co(1)–N(8)	2.213(2)	Co(1)–N(1)	2.449(2)
O(1)–Co(1)–N(4)	114.90(8)	O(1)–Co(1)–N(2)	93.82(8)
O(1)–Co(1)–N(6)	102.07(8)	N(4)–Co(1)–N(2)	98.48(8)
N(4)–Co(1)–N(6)	136.30(8)	O(1)–Co(1)–N(8)	76.24(7)
N(2)–Co(1)–N(6)	101.58(8)	N(4)–Co(1)–N(8)	82.06(8)
N(2)–Co(1)–N(8)	169.10(8)	N(6)–Co(1)–N(8)	85.09(8)
O(1)–Co(1)–N(1)	166.48(7)	N(4)–Co(1)–N(1)	74.63(8)
N(8)–Co(1)–N(1)	115.68(7)	N(2)–Co(1)–N(1)	74.74(7)
N(6)–Co(1)–N(1)	73.88(7)		

As shown in figure 2, two antiparallel 1-D chains were formed by intermolecular hydrogen bonds N(5)–H(5N)$\cdots$O(2) [N(5)$\cdots$O(2) 2.694(3) Å, N(5)–H(5N)$\cdots$O(2) 142.00°] between NH of ntb and uncoordinated oxygen of picolinate. In addition, $\pi-\pi$ stacking interactions ($\pi\cdots\pi = 3.664$ Å) of imidazolyl groups and benzene rings of ntb exist between the two adjacent antiparallel 1-D chains, contributing to the stabilization of the crystal.

3.1.2. [Co(ntb)(nic)](ClO_4)·CH_3OH (2). Complex **2** was synthesized by the reaction of $Co(ClO_4)_2\cdot 6H_2O$, ntb, and sodium nicotinate in ratio of 1 : 1 : 1 in methanol; crystals

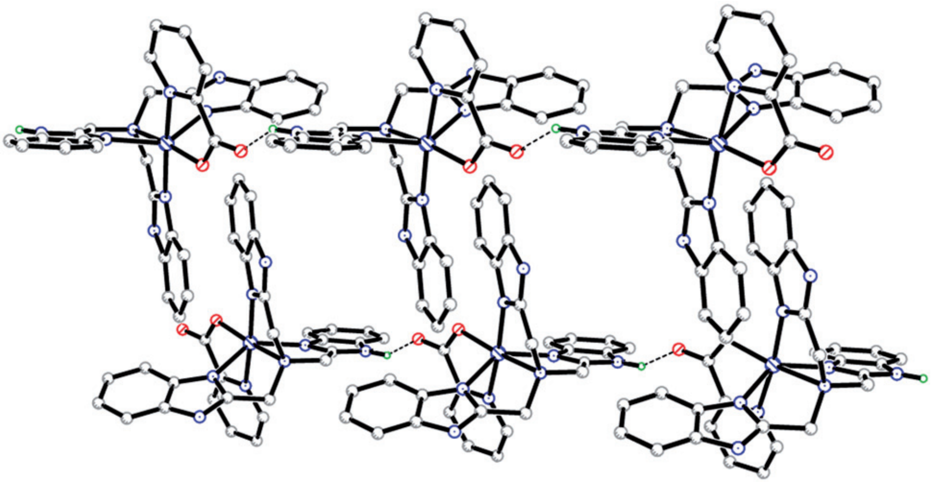


Figure 2. 1-D chain structure formed by intermolecular hydrogen bonds in **1** along the *b*-axis. N(5)···(2) 2.694(3) Å, N(5)–H(5N)···O(2) 142.00°, symmetry codes: $-1+x$, y, z.

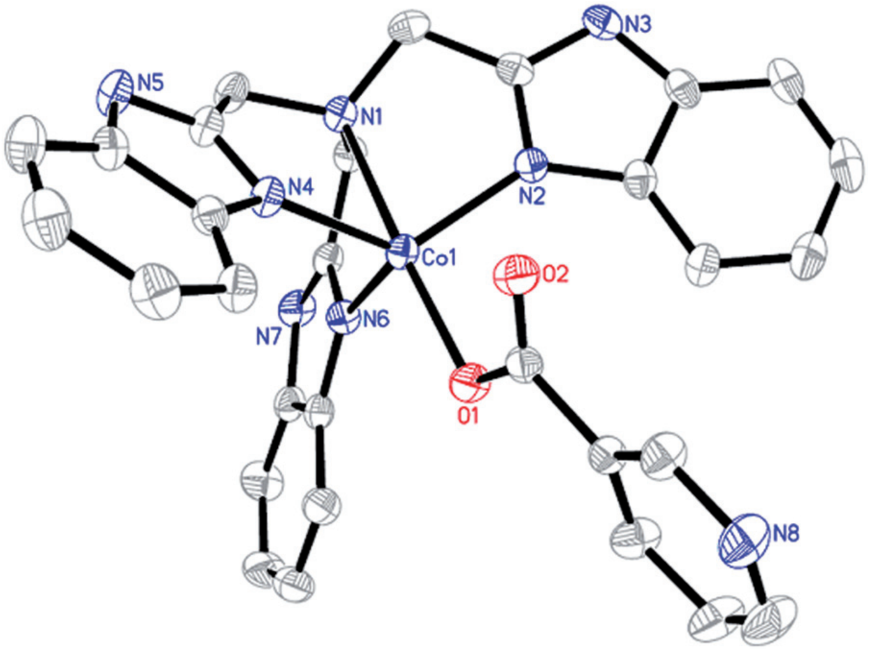


Figure 3. The cation structure of **2** (30% probability thermal ellipsoids).

of the product are relatively stable. The cation of **2** is illustrated in figure 3 and the selected bond lengths and angles are given in table 3. Complex **2** contains discrete monomeric $[Co(ntb)(nic)]^{+}$, one perchlorate, and one lattice methanol. The central Co(II) is five-coordinate in a distorted trigonal bipyramidal with the value of Addison τ parameter being 0.75 [$\tau = 0$ (square pyramid) and 1 (trigonal bipyramid)] [49]. The three

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

Co(1)–O(1)	2.003(3)	Co(1)–N(4)	2.059(3)
Co(1)–N(2)	2.053(3)	Co(1)–N(6)	2.051(3)
Co(1)–N(1)	2.389(3)		
O(1)–Co(1)–N(4)	112.56(12)	O(1)–Co(1)–N(2)	101.77(12)
O(1)–Co(1)–N(6)	98.92(12)	N(4)–Co(1)–N(2)	125.49(13)
N(4)–Co(1)–N(6)	104.55(12)	N(2)–Co(1)–N(6)	110.56(12)
O(1)–Co(1)–N(1)	170.72(11)	N(4)–Co(1)–N(1)	76.11(11)
N(6)–Co(1)–N(1)	75.02(12)	N(2)–Co(1)–N(1)	74.42(11)

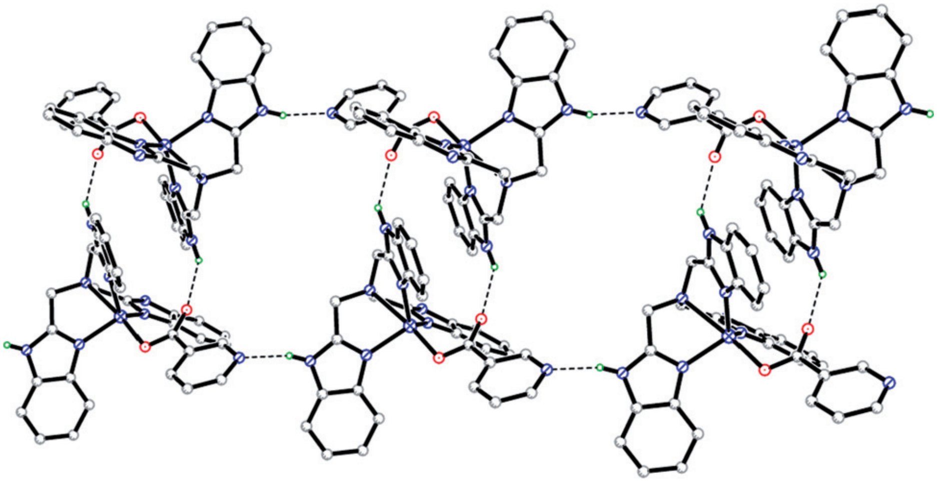


Figure 4. 1-D chain structure formed by intermolecular hydrogen bonds in **2** along the *a*-axis. N(3) ··· O(2) 2.802(4), N(3)–H(3) ··· O(2) 141.86°; N(7) ··· N(8) 2.799(5), N(7)–H(7) ··· N(8) 162.21°, symmetry codes: $1-x$, $-y$, $1-z$; x, $y+1$, z.

benzimidazolyl nitrogens N(2), N(4), and N(6) of ntb make up the trigonal plane with an average Co(1)–N(benzimidazolyl) bond length of 2.054(3) Å, shorter than that between Co(1) and amine N(1) (2.389(3) Å); similar bond length of $Co–N_{amine}$ is found in other reported Co(II) complexes with ntb [27, 29–31]. Co(1) is displaced with a bond distance of 0.525 Å out of the mean plane of N(2), N(4), and N(6) and toward O(1) with the Co(1)–O(1) bond distance of 2.003(3) Å. Bond angles of the trigonal plane are in the range 104.55(12)–125.49(13)°, deviating from 120° of ideal trigonal bipyramid. The axial sites are taken up by N(1) and carboxylate O(1) of nicotinate; the bond angle of O(1)–Co(1)–N(1) is 170.72(11)°.

Figure 4 depicts the 1-D chain structure formed by intermolecular hydrogen bonds in **2** along the *a*-axis. The N–H ··· O [N(3) ··· O(2) = 2.802(4) Å and N(3)–H(3) ··· O(2) = 141.86°] intermolecular hydrogen bonds between NH of ntb and uncoordinated carboxylate oxygen of nicotinate bridge the two adjacent molecules to form a double molecular unit. The double molecular units are further connected through hydrogen bonds N–H ··· N [N(7) ··· N(8) = 2.799(5) Å and N(7)–H(7) ··· N(8) = 162.21°] between the NH of ntb and nitrogen of nicotinate to give a 1-D chain structure. Strong $\pi-\pi$ interactions between imidazolyl groups ($\pi \cdots \pi = 3.551$ Å) of ntb exist in the double

Table 4. Hydrogen bond geometry for **1** and **2**.

D–H···A	d(H···A) (Å)	∠(DHA) (°)	d(D···A) (Å)	Symmetry codes
Complex **1**				
N(5)–H(5N)···O(2)	1.9500	142.00	2.694(3)	$-1+x, y, z$
Complex **2**				
N(3)–H(3)···O(2)	2.0800	141.86	2.802(4)	$1-x, -y, 1-z$
N(7)–H(7)···N(8)	1.9700	162.21	2.799(5)	$x, y+1, z$

molecular units and stabilize the chain structure. Bond parameters of hydrogen bonds of the two complexes are summarized in table 4.

3.2. *Characterizations of the complexes*

3.2.1. IR spectra. Complexes **1** and **2** show broad absorptions in the range 3100–3500 cm^{-1} which could be assigned to ν(O–H) for methanol and ν(N–H) for the benzimidazole ring of ntb. The strong absorption band at 1121 cm^{-1} indicates that ionic perchlorates (Td) are present in the two complexes [12]. High intensity bands at 1644, 1595, and 1371 cm^{-1} for **1** are associated with $\nu_{as}(COO^-)$ and $\nu_s(COO^-)$ of picolinate [32]; strong bands at 1617, 1595, and 1375 cm^{-1} for **2** are attributed to $\nu_{as}(COO^-)$ and $\nu_s(COO^-)$ of nicotinate, suggesting that the carboxylate of picolinate and nicotinate are coordinated to Co(II).

3.2.2. *UV-Vis spectra*

Electronic spectra are measured in DMF in the 200–900 nm range. The UV band of ntb (281 nm) in **1** and **2** is blue-shifted to 296 and 287 nm, showing C=N coordination to Co(II) [4, 10, 12, 50]. Complex **1** exhibits a broad d–d transition around 531 nm, similar to the reported six-coordinate Co(II) complex of ntb [30]. Bands at 516 and 589 nm in **2** are assigned to d–d transitions of Co(II). The difference between the visible spectra of the two complexes may be attributed to the different coordination number and coordination geometry of Co(II) ion in **1** and **2**.

4. Conclusion

Two new mononuclear cobalt(II) complexes $[Co(ntb)(pic)](ClO_4)\cdot(CH_3OH)_{2.35}$ (**1**) and $[Co(ntb)(nic)](ClO_4)\cdot CH_3OH$ (**2**) containing tripodal tetradentate ntb were synthesized. Picolinate coordinates to Co(II) in a bidentate μ_2-N,O chelating mode in **1** and nicotinate coordinates with Co(II) through monodentate carboxylate oxygen in **2**. Intermolecular hydrogen bonds form remarkably different 1-D chain structures in the two complexes and π–π interactions also exist in both complexes.

Supplementary data

CCDC 749298 and 749297 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 336033; or Email: deposit@ccdc.cam.ac.uk.

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