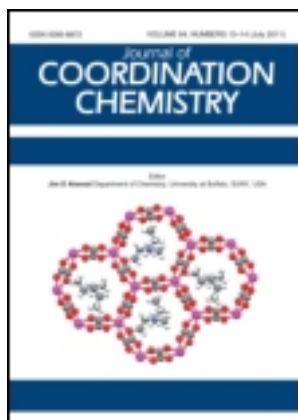


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Supramolecular architecture built of Co(II) and a tripodal ligand containing 1-D water tapes with (H₂O)₁₆ cluster units

Zhenpeng Li^a, Liang Huang^a, Pinxian Xi^a, Hongyan Liu^a, Yanjun Shi^a, Guoqiang Xie^a, Min Xu^a, Fengjuan Chen^a & Zhengzhi Zeng^a

^a Key Laboratory of Nonferrous Metals Chemistry and Resources Utilization of Gansu Province and State Key Laboratory of Applied Organic Chemistry, College of Chemistry and Chemical Engineering, Lanzhou University, Lanzhou 730 000, P.R. China
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Supramolecular architecture built of Co(II) and a tripodal ligand containing 1-D water tapes with (H₂O)₁₆ cluster units

ZHENPENG LI, LIANG HUANG, PINXIAN XI, HONGYAN LIU,
YANJUN SHI, GUOQIANG XIE, MIN XU, FENGJUAN CHEN and
ZHENGZHI ZENG*

Key Laboratory of Nonferrous Metals Chemistry and Resources Utilization of Gansu Province and State Key Laboratory of Applied Organic Chemistry, College of Chemistry and Chemical Engineering, Lanzhou University, Lanzhou 730 000, P.R. China

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The reaction of Co(NO₃)₂·6H₂O with a tripodal ligand leads to a new complex {[Co(L)]·2NO₃·8H₂O} (**1**) confirmed by single-crystal X-ray diffraction, infrared spectroscopy, and elemental analysis. The particular interest of **1** is in the formation of a 1-D water tape consisting of (H₂O)₁₆ cluster units, the neighboring water tapes are connected by free nitrate anions *via* hydrogen bonds into a 2-D guest layer. These guest layers are alternately packed face-to-face with the 2-D host layers along the *a*-axis to form a 3-D supramolecular architecture. There exist C–H···N and C–H···O weak hydrogen bonds between the guest layer and host layer. These weak hydrogen bonds and water–nitrate, water–water hydrogen bonds are important for the stability of the overall structure.

Keywords: Cobalt complex; (H₂O)₁₆ cluster; Water tape; Hydrogen bond; Supramolecular architecture

1. Introduction

Water plays an important role in many biological, chemical, and physical processes [1, 2]. Water clusters, groups of water molecules held together by hydrogen bonds, are believed to be a model for connecting isolated molecules to bulk water [1]. The exploration of structural information of water clusters is key to understanding the behavior of water molecules [3]. Considerable attention has been paid to theoretical and experimental studies of water clusters [4–8] to understand the nature of water–water interactions in bulk water or ice; a number of self-assembled water clusters including tetramers [9, 10], pentamers [11, 12], hexamers [13, 14], octamers [15, 16], decamers [17, 18], undecamers [19], dodecamers [20, 21], tetradecamers [22], hexadecamers [23], 1-D chain [24–26], 1-D tape [27–29], 2-D layer [30–34], and 3-D

*Corresponding author. Email: zengzhzh@yahoo.com.cn

networks [35] have been reported. This work presents structural configurations of water and also provides new insights into the role of water in framework structures.

Among water clusters, 1-D water morphologies draw considerable interest because of their important roles in biological processes. Structural studies have shown that 1-D water morphologies exist in gramicidin A membrane channels [36], bacteriorhodopsin [37], and α -amylase [38] for rapid transport of protons and act as “proton wires”. Very recently, significant progress has been made with respect to structural characterization of 1-D aggregates. For example, Lee *et al.* [27] reported an ice-like self-assembled T4(2)6(2) water tape trapped in a proton-transfer salt host. Neogi and Bharadwaj [24] synthesized a porous coordination polymeric structure with infinite interlinked chains of Zn(II) metallocycles with an infinite water chain passing through the metallocycles like a thread. Pang and coworkers [26] characterized a saturated Keggin POM/lanthanide compound, which contains a water chain with trimeric and hexameric water clusters. However, there are few examples [39] of 1-D water tapes containing large water cluster subunits.

To obtain 1-D water morphologies, it is important to construct host frameworks suitable for water molecules extending into chains or tapes. In this article, we introduce a tripodal ligand (L) and expected to construct a new compound containing 1-D water morphologies. Fortunately, we obtained such a new compound $\{[\text{Co}(\text{L})] \cdot 2\text{NO}_3 \cdot 8\text{H}_2\text{O}\}$ (**1**) [$\text{L} = \text{N}(\text{CH}_2\text{CH}_2\text{OC}_9\text{H}_6\text{N})_3$] (figure 1), in which a 1-D water tape consisting of $(\text{H}_2\text{O})_{16}$ cluster units is observed. The structural elucidation of this novel water morphology provides new insight into water with implications in biological environments.

2. Experimental

2.1. Methods and materials

Carbon, hydrogen, and nitrogen were analyzed on an Elemental Vario EL analyzer. Infrared (IR) spectra ($4000\text{--}400\text{cm}^{-1}$) were determined with KBr disks on a Thermo Mattson FTIR spectrometer. ^1H NMR spectra were measured on a Varian

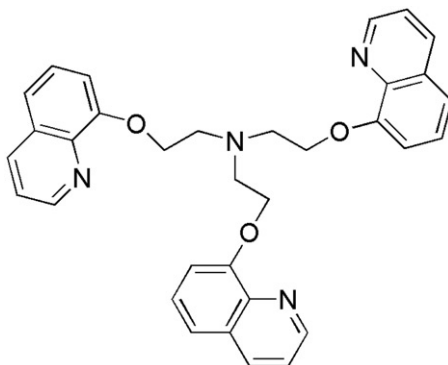


Figure 1. Structure of the tripodal ligand.

VR300-MHz spectrometer using TMS as reference. Mass spectra were performed on a VG ZAB-HS fast-atom bombardment (FAB) instrument.

All materials and solvents employed in this study were analytical grade reagents. Tris(2-chloroethyl) amine hydrochloride was prepared according to the literature [40]. Other reagents and solvents were purchased from commercial suppliers and used without purification.

2.2. Preparation of the tripodal ligand (L)

The tripodal ligand (figure 1) was prepared according to the method previously reported [41]. Yellowish, viscous oil; yield 53%; ^1H NMR (CDCl_3) δ 3.45 (t, 6, $J=7$ Hz, CH_2N), 4.44 (t, 6, $J=7$ Hz, CH_2O). FAB-MS: $m/z = 531.3$ $[\text{M} + \text{H}]^+$. Anal. Calcd for $\text{C}_{33}\text{H}_{30}\text{N}_4\text{O}_3$ (%): C, 74.70; H, 5.70; N, 10.56. Found (%): C, 75.00; H, 6.01; N, 10.63.

2.3. Preparation of 1

The tripodal ligand L (0.53 g, 1.0 mmol) was dissolved in 10 mL ethanol, and then 10 mL aqueous solution of $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (0.291 g, 1.0 mmol) was added to the ethanol solution. The reaction mixture was stirred at room temperature for 8 h and then filtered. The filtrate was allowed to evaporate at RT for 2 weeks, when red crystals were obtained in ~60% yield. Anal. Calcd for $\text{C}_{33}\text{H}_{46}\text{CoN}_6\text{O}_{17}$ (%): C, 46.17; H, 5.41; N, 9.80. Found (%): C, 46.43; H, 5.28; N, 9.67. IR (KBr, cm^{-1}): $\nu(\text{O-H})$ 3048; $\nu(\text{C=C}) + \nu(\text{C=N})$ 1624, 1590, 1509, 1472; $\nu(\text{C-N})$ 1318; $\nu(\text{NO}_3^-)$ 1384.

2.4. X-ray crystallographic study

Crystallographic data and refinement parameters for **1** are presented in table 1, obtained at 296 K on a Rigaku RAXIS-RAPID diffractometer using a $\text{Mo-K}\alpha$ radiation ($\lambda = 0.71073 \text{ \AA}$). The structures were solved with direct methods and refined with full-matrix least-squares based on F^2 using the SHELXTL-97 program package [42, 43]. Anisotropic thermal parameters were applied to all non-hydrogen atoms. The organic hydrogens were generated geometrically. Hydrogen bonding was inferred by considering $\text{O}\cdots\text{O}$ close contacts of water molecules involved ($\text{O}\cdots\text{O}$ distance less than the sum of van der Waals radii 3.04 \AA) [12].

3. Results and discussion

3.1. X-ray structural characterization

Single-crystal X-ray structural analysis reveals that **1** consists of one $[\text{Co}(\text{L})]^{2+}$, two uncoordinated nitrates, and eight lattice waters. The key bond lengths and angles are summarized in table 2. The structure of $[\text{Co}(\text{L})]^{2+}$ is shown in figure 2a; the Co is coordinated with seven donors (three oxygens and four nitrogens), all of which belong to one L including one bridgehead nitrogen ($\text{Co}(1)-\text{N}(2)\cdots 2.183(4) \text{ \AA}$) and three ethereal oxygens ($\text{Co}(1)-\text{O}(1)\cdots 2.376(3) \text{ \AA}$, $\text{Co}(1)-\text{O}(2)\cdots 2.373(3) \text{ \AA}$,

Table 1. Crystal data and structure refinement for **1**.

Empirical formula	C ₃₃ H ₄₆ CoN ₆ O ₁₇
Formula weight	857.69
Temperature (K)	296(2)
Wavelength (Å)	0.71073
Crystal system	Triclinic
Space group	<i>P</i> $\bar{1}$
Unit cell dimensions	
<i>a</i>	11.247(3)
<i>b</i>	13.254(3)
<i>c</i>	14.172(4)
α	66.979(3)°
β	89.204(3)°
γ	87.905(3)°
Volume (Å ³), <i>Z</i>	1943.0(8), 2
Calculated density (g cm ⁻³)	1.438
Absorption coefficient (mm ⁻¹)	0.524
<i>F</i> (000)	898
Crystal size (mm ³)	0.22 × 0.20 × 0.18
Crystal color	Red
θ range for data collection	2.39–25.20°
Max. and min. transmission	0.9116/0.8935
Refinement method	Full-matrix least-squares on <i>F</i> ²
Reflection collected/unique	13904/6910 [<i>R</i> (int) = 0.0352]
Data/restraints/parameters	6910/27/562
Goodness-of-fit on <i>F</i> ²	1.039
Final <i>R</i> indices [<i>I</i> > 2(<i>I</i>)]	<i>R</i> 1 = 0.0600, <i>wR</i> 2 = 0.1539
<i>R</i> indices (all data)	<i>R</i> 1 = 0.0862, <i>wR</i> 2 = 0.1701
Largest difference peak and hole (e Å ⁻³)	0.712 and –0.706
CCDC number	794393

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

Bond lengths	
Co(1)–N(2) ... 2.183(4)	Co(1)–N(6) ... 2.123(4)
Co(1)–N(5) ... 2.141(3)	Co(1)–N(7) ... 2.135(3)
Co(1)–O(2) ... 2.373(3)	Co(1)–O(9) ... 2.360(3)
Co(1)–O(1) ... 2.376(3)	
Bond angles	
N(6)–Co(1)–N(7) ... 96.08(13)	N(6)–Co(1)–N(2) ... 119.82(14)
N(6)–Co(1)–N(5) ... 96.80(13)	N(7)–Co(1)–N(5) ... 94.85(13)
N(7)–Co(1)–N(2) ... 121.57(14)	N(5)–Co(1)–N(2) ... 121.50(13)
N(6)–Co(1)–O(9) ... 81.97(12)	N(7)–Co(1)–O(9) ... 165.73(12)
N(5)–Co(1)–O(9) ... 71.46(12)	N(2)–Co(1)–O(9) ... 70.77(13)
N(6)–Co(1)–O(2) ... 167.06(11)	N(7)–Co(1)–O(2) ... 71.37(12)
N(5)–Co(1)–O(2) ... 81.56(12)	N(2)–Co(1)–O(2) ... 71.08(12)
O(9)–Co(1)–O(2) ... 109.39(11)	N(6)–Co(1)–O(1) ... 71.59(12)
N(7)–Co(1)–O(1) ... 80.16(12)	N(5)–Co(1)–O(1) ... 166.61(11)
N(2)–Co(1)–O(1) ... 71.22(12)	O(9)–Co(1)–O(1) ... 112.27(11)
O(2)–Co(1)–O(1) ... 108.20(11)	

Co(1)–O(9) ... 2.360(3) Å) from sidegroups. The other three nitrogens (Co(1)–N(5) ... 2.141(3) Å, Co(1)–N(6) ... 2.123(4) Å, Co(1)–N(7) ... 2.135(3) Å) are from three quinoline rings. In this structure, the average Co–O distance (2.369 Å) is longer than the average Co–N distance (2.145 Å), indicating stronger Co–N bonds.

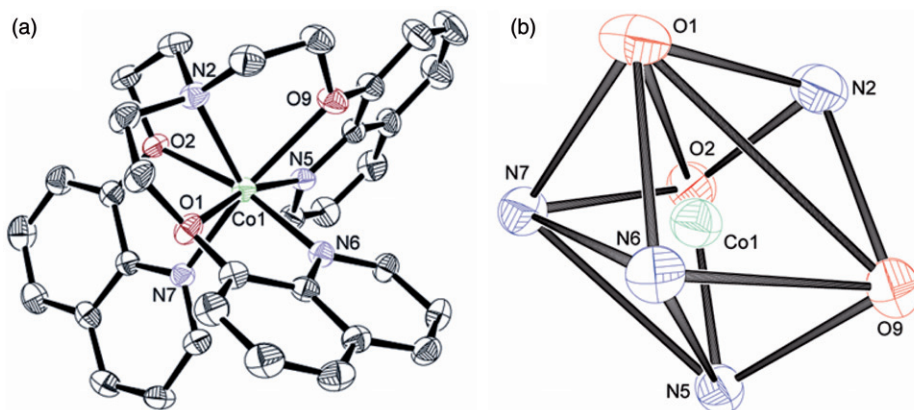


Figure 2. (a) ORTEP diagram showing the structure of the $[\text{Co}(\text{L})]^{2+}$ motif in **1** (at 30% probability level). All hydrogens are omitted for clarity. (b) The coordination polyhedron of Co (50% probability ellipsoids).

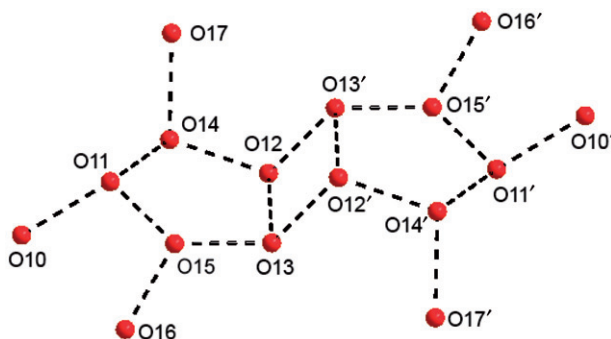


Figure 3. Ball and stick representation of the $(\text{H}_2\text{O})_{16}$ cluster. Dotted black lines show hydrogen bonding between water molecules.

The coordination polyhedron around Co is distorted pentagonal bipyramidal (figure 2b). O1 and N5 are at apical positions and the other five donors (O2, O9, N2, N6, N7) comprise the distorted pentagon. Moreover, C–H $\cdots\pi$ interactions are found between the quinoline rings of adjacent $[\text{Co}(\text{L})]^{2+}$ cations. The $[\text{Co}(\text{L})]^{2+}$ cations self-assemble into a 2-D host layer *via* C–H $\cdots\pi$ interactions in the *bc* crystallographic plane (figure S1).

The hydrogen-bonding association of lattice waters in **1** lead to the formation of a 16-membered water cluster (figure 3). Geometrical parameters of the water clusters are summarized in table 3. The $(\text{H}_2\text{O})_{16}$ cluster consists of one $(\text{H}_2\text{O})_4$ subunit and two $(\text{H}_2\text{O})_8$ subunits. Each $(\text{H}_2\text{O})_8$ cluster is formed by eight types of water molecules (O10–O17). O12, O13, O12', O13' atoms of the two adjacent $(\text{H}_2\text{O})_8$ clusters are hydrogen bonded to form a planar cyclic water tetramer. In the $(\text{H}_2\text{O})_8$ cluster, five waters (O11–O15) form a cyclic water pentamer. The five oxygens within the pentamer are non coplanar. Adjacent pentamers and tetramers connect by sharing an edge and form a staircase structure (figure 3).

Table 3. Geometrical parameters of hydrogen bonds (\AA , $^\circ$) for $(\text{H}_2\text{O})_{16}$ clusters.

O10...O11	2.840	O10...O11...O14	119.46
O11...O14	2.769	O10...O11...O15	109.90
O11...O15	2.699	O14...O11...O15	103.18
O12...O13	2.845	O14...O12...O13	107.91
O12...O13'	2.756	O13...O12...O13'	93.01
O12...O14	2.787	O14...O12...O13'	116.23
O13...O15	2.713	O12...O13...O15	102.39
O14...O17	2.829	O15...O13...O12'	118.47
O15...O16	2.688	O12...O13...O12'	86.99
O12...O14...O17	107.23	O11...O15...O16	108.00
O11...O14...O12	109.83	O11...O15...O13	116.58
O11...O14...O17	111.00	O16...O15...O13	122.47

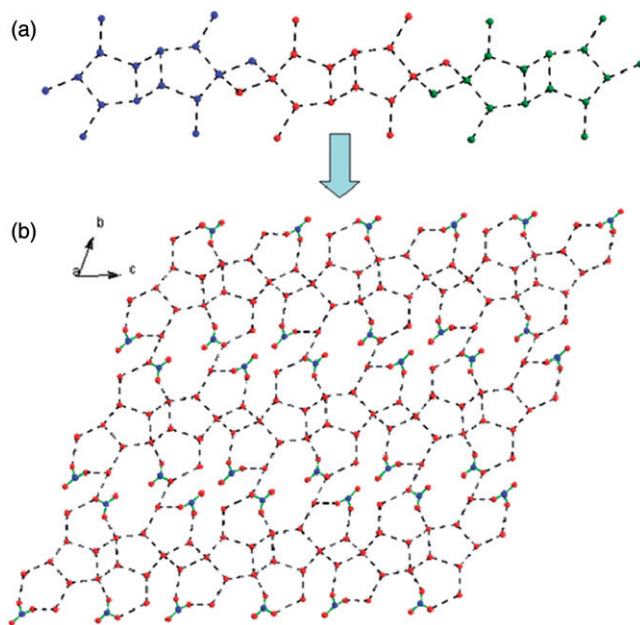


Figure 4. (a) The 1-D water tape containing $(\text{H}_2\text{O})_{16}$ units. Three units are labeled in blue, red, and green. (b) Packing diagram of the 2-D layer of the water tapes with nitrates as connectors.

As shown in figure 4a, adjacent $(\text{H}_2\text{O})_{16}$ clusters self-assemble by O10...O11', O10'...O11 hydrogen bonds into an infinite water tape along the c -axis; four-membered rings occur between two adjacent $(\text{H}_2\text{O})_{16}$ units of the tape. According to the classifying law [12] of Infantes and Motherwell, this tape can be classified as T4(1)5(2)T4(2)5(1). Within the water tape, the O...O distances are 2.688–2.845 \AA with an average distance of 2.773 \AA , shorter than those observed in liquid water (2.854 \AA) [44, 45] and comparable to the value of 2.759 \AA in hexagonal ice [46]. Moreover, the O...O...O angles (from 86.99 to 122.47 $^\circ$) with an average of 106.63 $^\circ$ are slightly smaller than the corresponding value of 109.3 $^\circ$ in hexagonal ice.

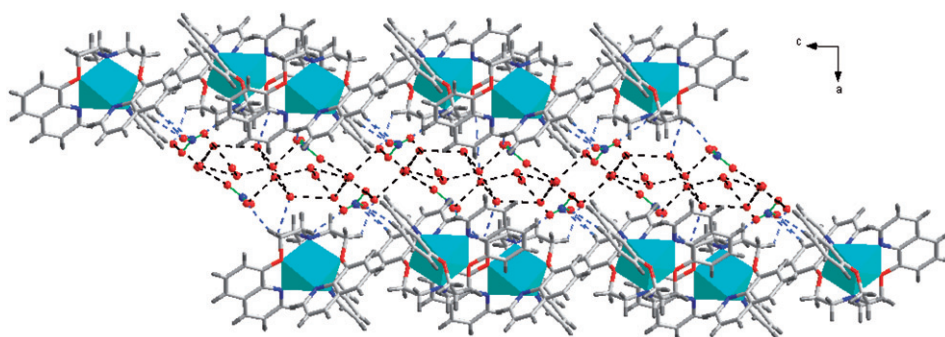


Figure 5. View of the interactions between the host layer and guest layer in **1** along the *b*-axis (gray-25%: carbon; gray-50%: hydrogen; red: oxygen; blue: nitrogen; polyhedron: CoN_4O_3).

The free nitrates are linkers, joining the neighboring water tapes to form a 2-D layered structure through hydrogen bonding of $\text{O10}\cdots\text{O6}$, $\text{O16}\cdots\text{O7}$, $\text{O16}\cdots\text{O4}$, $\text{O13}\cdots\text{O5}$, and $\text{O17}\cdots\text{O4}$ (figure 4b). The distances of $\text{O10}\cdots\text{O6}$, $\text{O16}\cdots\text{O7}$, $\text{O16}\cdots\text{O4}$, $\text{O13}\cdots\text{O5}$, and $\text{O17}\cdots\text{O4}$ are 2.792, 2.901, 2.920, 2.747, and 3.038 Å, respectively. The $\text{O}\cdots\text{O}$ distances are shorter than the sum of van der Waals radii 3.04 Å [12]. The shorter $\text{O}\cdots\text{O}$ distances show stronger interactions between the nitrate anions and water through hydrogen bonds in this compound. These 2-D layered structures as guest layers are alternately packed face-to-face with the 2-D host layers along the *a*-axis (figure S1). They are linked to each other through $\text{C-H}\cdots\text{O}$ and $\text{C-H}\cdots\text{N}$ weak hydrogen bonds between L and free nitrate or lattice waters into a 3-D supramolecular structure (figure 5).

This structure was compared with other structures that contain 2-D guest water layers. Ramanan *et al.* [33] reported a 2-D water layer sandwiched between long fibers of 2,3-phenazinediamine tetrahydrate. In another report from Yang *et al.* [34], the wave-like water layer sandwiched in the space of the wave-like metal crystal building layers is similar to these two structures. In order to provide a stable structure, the guest layer geometry exactly matches the host structure. The packing diagram of the 3-D supramolecular structure viewed along the crystallographic *b*-axis is shown in figure S2. The distance between two adjacent host layers is *ca* 4.601 Å, the guest layer is sandwiched between two adjacent host layers. The distance between two adjacent guest layers is *ca* 11.247 Å and the host layer is sandwiched. There are $\text{C-H}\cdots\text{N}$ and $\text{C-H}\cdots\text{O}$ hydrogen bonds between the adjacent host layer and guest layer. Obviously, the guest layer plays an important role in stabilizing the 3-D supramolecular structure.

3.2. IR spectra

IR spectrum of **1** (figure S3) exhibits strong bands at 1624, 1590, 1509, and 1472 cm^{-1} due to $\text{C}=\text{C}$ and $\text{C}=\text{N}$ stretching vibrations of the quinoline rings of the tripodal ligand. The band at 1318 cm^{-1} is assigned to $\text{C}-\text{N}$ stretching vibration of the tripodal ligand. The band at 579 cm^{-1} for **1** is assigned to $\text{Co}-\text{O}$ stretch [47]. These data strongly indicate that the oxygen has formed a coordinate bond with cobalt. Below 500 cm^{-1} are some weak bands which are assigned to $\text{Co}-\text{N}$ stretches [48], further confirming that

nitrogen bonds to cobalt. The free nitrate of the compound appear at 1384 cm^{-1} in the spectra. The IR spectrum of the complex exhibits a broad band centered at 3408 cm^{-1} attributable to O–H stretching frequency for the water cluster, similar to that of liquid water (3490 cm^{-1}) [49].

4. Conclusion

We have synthesized and characterized a 3-D supramolecular architecture of Co(II) with a tripodal ligand, in which an infinite 1-D water tape is observed. The water tape is a T4(1)5(2)T4(2)5(1) type aggregate that consists of large $(\text{H}_2\text{O})_{16}$ cluster units. Adjacent water tapes are connected by free nitrates *via* hydrogen bonds into a 2-D guest layer, which is sandwiched between the host layers and plays an important role in stabilizing the structure. Such an arrangement of water tapes has not been described so far and provides novel structural aspects of water. It is very important for providing insight into hydrogen-bonding motifs and understanding water clusters of different sizes and shapes in diverse environments.

Supplementary material

CCDC 794393 contains the supplementary crystallographic data for this article. These data can be obtained free of charge *via* <http://www.ccdc.cam.ac.uk/conts/retrieving.html>, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB 21EZ, UK; Fax: (+44) 1223-336-033; or E-mail: deposit@ccdc.cam.ac.uk. Structural figures and IR spectra were available from the author.

Acknowledgments

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