

# catena-(Bis( $\mu_2$ -1,3-bis(4-pyridyl)propane-*N,N'*)-diaqua-cobalt(II)) dinitrate 1,3-bis(4-pyridyl)propane monohydrate

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## Key indicators

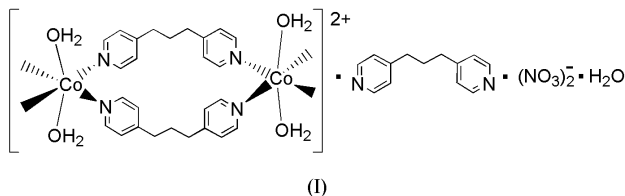
Single-crystal X-ray study  
 T = 294 K  
 Mean  $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$   
 H-atom completeness 96%  
 Disorder in solvent or counterion  
 R factor = 0.047  
 wR factor = 0.123  
 Data-to-parameter ratio = 15.4

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The hydrothermally prepared title compound,  $[\text{Co}(\text{C}_{26}\text{H}_{28}\text{N}_4)(\text{H}_2\text{O})_2]2\text{NO}_3 \cdot \text{C}_{13}\text{H}_{14}\text{N}_2 \cdot \text{H}_2\text{O}$ , (I), consists of infinite one-dimensional chains containing two independent  $\text{Co}^{2+}$  centers, each octahedrally coordinated by two water molecules and four bridging 1,3-bis(4-pyridyl)propane (bpp) ligands. The chains are linked into infinite two-dimensional layers by hydrogen bonding, *via* an uncoordinated bpp molecule, and further into a three-dimensional framework *via* interstitial nitrate anions and water molecules. (I) is isostructural to the Ni analog previously reported [Krishnamohan Sharma *et al.*, (2000). *Cryst. Eng.* **3**, 201–208].

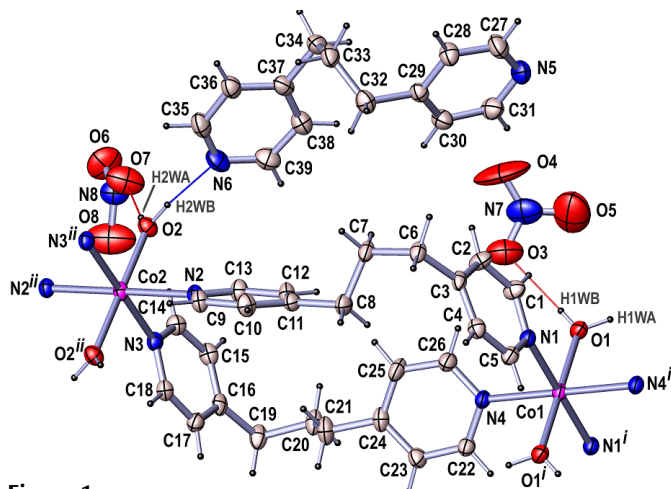
## Comment

The asymmetric unit of (I) consists of two independent  $\text{Co}^{2+}$  centers bridged by two independent 1,3-bis(4-pyridyl)propane ligands (Fig. 1). Both  $\text{Co}^{2+}$  centers lie on crystallographic inversion centers. Two independent, coordinated water molecules, two nitrate anions, a free (uncoordinated) bpp molecule and a water molecule of crystallization complete the asymmetric unit. The interstitial water was modeled as disordered over two nearby positions in the refined proportion O9A/O9B = 0.69 (3)/0.31 (3). Minor positional disorder for both nitrates is reflected in slightly elongated displacement ellipsoids for atoms of these groups.

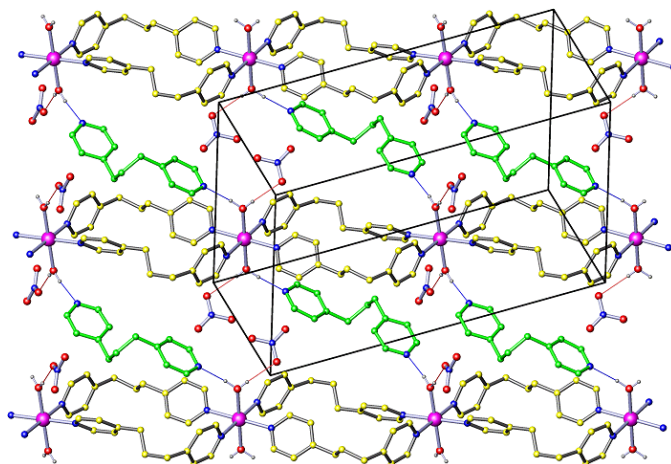


The local coordination of both  $\text{Co}^{2+}$  centers is similar,  $\{\text{CoN}_4\text{O}_2\}$ , with four equatorial pyridyl N atoms from two independent bpp ligands and their inversion-related symmetry equivalents, and a water molecule and its inversion-related equivalent (Table 1). The coordinated bpp ligands bridge the  $\text{Co}^{2+}$  centers into infinite one-dimensional chains along the crystallographic  $[10\bar{1}]$  direction.  $\text{O}-\text{H}\cdots\text{N}$  and  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonding links the one-dimensional chains into two-dimensional layers parallel to the crystallographic (101) plane, and further into a three-dimensional network (see Table 2). The hydrogen-bonding linkage into two-dimensional layers is *via* the uncoordinated bpp molecule (Fig. 2). The linkage into a three-dimensional network is *via* the nitrate anions and, presumably, the disordered  $\text{H}_2\text{O}$  molecules. H atoms could not be located and were not calculated for this disordered species,

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**Figure 1**  
Asymmetric unit of (I), together with additional atoms to complete the metal coordination. Disordered water molecule omitted for clarity. Displacement ellipsoids drawn at the 30% probability level. [Symmetry codes: (i)  $-x, 1-y, 1-z$ ; (ii)  $1-x, 1-y, -z$ .]



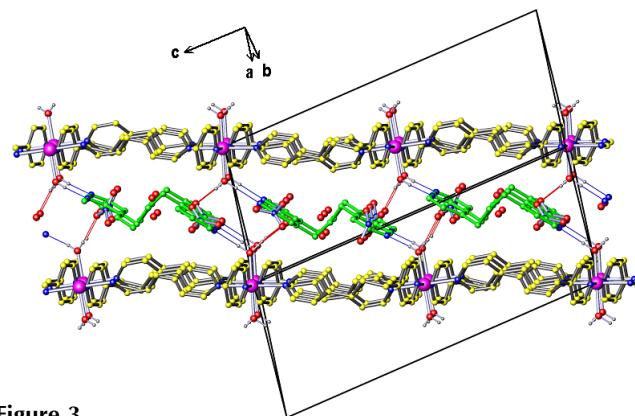
**Figure 2**  
A single layer of (I), showing the one-dimensional chains hydrogen-bonded together *via* the uncoordinated bpp molecules. Hydrogen bonds are shown as thin red or blue lines.

but its position midway between two nitrate anions in two adjacent layers strongly suggests a role in forming a three-dimensional framework (Fig. 3).

Hydrothermally prepared (I) is isostructural to the Ni analog obtained from a room-temperature solution-layering technique (Krishnamohan Sharma *et al.*, 2000), and is very similar to a cobalt perchlorate compound (Plater *et al.*, 2001) also obtained at room temperature. In addition, the infinite one-dimensional chains in (I) are similar to those found in  $M(\text{NO}_3)_2(\text{bpp})_2 \cdot \text{C}_6\text{H}_6$  ( $M = \text{Co}, \text{Cd}$ ), where the nitrate anions are directly bound to the metal chains due to the absence of a coordinating solvent (Bujaci *et al.*, 2002).

## Experimental

The title compound was prepared by hydrothermal reaction of  $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  (0.0350 g, 0.2 mmol) with 1,3-bis(4-pyridyl)propane (0.0402 g, 0.2 mmol) in water (0.80 ml) in an evacuated sealed Pyrex



**Figure 3**  
[110] view of the three-dimensional structure of (I), showing the interstitial nature of the uncoordinated bpp molecules, nitrate ions and disordered  $\text{H}_2\text{O}$  molecules.

tube. The reaction was heated to 403 K at  $10 \text{ K min}^{-1}$  and held at 403 K for 12 h before cooling slowly ( $0.1 \text{ K min}^{-1}$ ) to 298 K. The reaction yielded plentiful orange crystals.

## Crystal data

$[\text{Co}(\text{C}_{26}\text{H}_{28}\text{N}_4)(\text{H}_2\text{O})_2]2\text{NO}_3 \cdot$   
 $\text{C}_{13}\text{H}_{14}\text{N}_2 \cdot \text{H}_2\text{O}$   
 $M_r = 831.78$   
Triclinic,  $P\bar{1}$   
 $a = 10.0177(6) \text{ \AA}$   
 $b = 10.8091(6) \text{ \AA}$   
 $c = 21.8916(12) \text{ \AA}$   
 $\alpha = 76.104(1)^\circ$   
 $\beta = 85.504(1)^\circ$   
 $\gamma = 63.301(1)^\circ$   
 $V = 2054.6(2) \text{ \AA}^3$

$Z = 2$   
 $D_x = 1.345 \text{ Mg m}^{-3}$   
Mo  $K\alpha$  radiation  
Cell parameters from 8261 reflections  
 $\theta = 2.3\text{--}26.4^\circ$   
 $\mu = 0.48 \text{ mm}^{-1}$   
 $T = 294(2) \text{ K}$   
Irregular, orange  
 $0.56 \times 0.38 \times 0.20 \text{ mm}$

## Data collection

Bruker SMART APEX CCD diffractometer  
 $\omega$  and  $\varphi$  scans  
Absorption correction: multi-scan (SADABS, Bruker, 1999)  
 $T_{\min} = 0.629, T_{\max} = 0.909$   
16276 measured reflections

8371 independent reflections  
5361 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.024$   
 $\theta_{\max} = 26.4^\circ$   
 $h = -12 \rightarrow 12$   
 $k = -13 \rightarrow 12$   
 $l = -27 \rightarrow 27$

## Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.047$   
 $wR(F^2) = 0.124$   
 $S = 0.96$   
8371 reflections  
543 parameters

H atoms treated by a mixture of independent and constrained refinement  
 $w = 1/[\sigma^2(F_o^2) + (0.0649P)^2]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 0.50 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -0.34 \text{ e \AA}^{-3}$

**Table 1**

Selected geometric parameters ( $\text{\AA}, ^\circ$ ).

Co1—O1 <sup>i</sup>	2.0871 (19)	Co2—O2 <sup>ii</sup>	2.0897 (19)
Co1—O1	2.0871 (19)	Co2—O2	2.0897 (19)
Co1—N4 <sup>i</sup>	2.1850 (17)	Co2—N2 <sup>ii</sup>	2.1731 (17)
Co1—N4	2.1850 (17)	Co2—N2	2.1731 (17)
Co1—N1 <sup>i</sup>	2.2194 (19)	Co2—N3	2.2202 (19)
Co1—N1	2.2194 (19)	Co2—N3 <sup>ii</sup>	2.2202 (19)
O1—Co1—N4	92.59 (8)	O2—Co2—N2	88.03 (8)
O1—Co1—N1	90.48 (7)	O2—Co2—N3	89.03 (8)
N4—Co1—N1	89.72 (7)	N2—Co2—N3	87.04 (7)

Symmetry codes: (i)  $-x, 1-y, 1-z$ ; (ii)  $1-x, 1-y, -z$ .

**Table 2**  
Hydrogen-bonding geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O1—H1WA $\cdots$ N5 <sup>iii</sup>	0.88 (3)	1.90 (3)	2.767 (3)	170 (3)
O1—H1WB $\cdots$ O3	0.74 (3)	2.15 (3)	2.879 (4)	169 (3)
O2—H2WA $\cdots$ O7	0.71 (3)	2.05 (3)	2.757 (3)	172 (3)
O2—H2WB $\cdots$ N6	0.84 (3)	1.90 (3)	2.736 (3)	173 (3)

Symmetry code: (iii)  $-x, -y, 1 - z$ .

H atoms attached to C were geometrically idealized, with  $C_{\text{arene}}-H = 0.930 \text{ \AA}$ ,  $C_{\text{methylene}}-H = 0.970 \text{ \AA}$  and  $U_{\text{iso}}(H) = 1.2U_{\text{iso}}(C)$ . Coordinated water H atoms were located and freely refined with isotropic displacement parameters. H atoms belonging to the disordered water molecule were not located or calculated.

Data collection: *SMART-NT* (Bruker, 1999); cell refinement: *SAINT-Plus-NT* (Bruker, 1999); data reduction: *SAINT-Plus-NT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997a); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997a);

molecular graphics: *ATOMS* (Dowty, 2001); software used to prepare material for publication: *SHELXTL* (Sheldrick, 1997b).

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