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Triaqua(1,10-phenanthroline-2,9-dicarboxylato)cobalt(II) dihydrate

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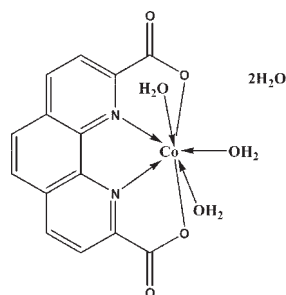
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.035; wR factor = 0.098; data-to-parameter ratio = 14.2.

The title compound, $[\text{Co}(\text{C}_{14}\text{H}_6\text{N}_2\text{O}_4)(\text{H}_2\text{O})_3]\cdot 2\text{H}_2\text{O}$, has two-fold crystallographic symmetry. The Co^{II} atom is in a distorted pentagonal-bipyramidal coordination environment with two N atoms and two O atoms from a tetradentate 1,10-phenanthroline-2,9-dicarboxylate ligand and one O atom from a water molecule forming the pentagonal plane, and two O atoms from two water molecules occupying axial positions. In the crystal, adjacent molecules are linked by $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds, forming a three-dimensional network.

Related literature

For the structures and properties of coordination compounds, see: Zhao *et al.* (2008); Poulsen *et al.* (2005). For the use of multi-carboxylate and heterocyclic carboxylic acids in coordination chemistry, see: Luo *et al.* (2009); Han *et al.* (2009) and for the dicarboxylate ligand H_2PDA (H_2PDA is 1,10-phenanthroline-2,9-dicarboxylic acid), see: Xie *et al.* (2005). For the isotopic structure $[\text{Mg}(\text{PDA})(\text{H}_2\text{O})_3]\cdot 2\text{H}_2\text{O}$, see: Park *et al.* (2001). For the high affinity of the Co^{II} ion to water molecules, see: (Zhang & Chen (2009). For bond distances and angles in other seven-coordinated Co^{II} complexes, see: Newkome *et al.* (1984); Rajput & Biradha (2007). For the synthesis of 1,10-phenanthroline-2,9-dicarboxylic acid, see: De Cian *et al.* (2007).



Experimental

Crystal data

$[\text{Co}(\text{C}_{14}\text{H}_6\text{N}_2\text{O}_4)(\text{H}_2\text{O})_3]\cdot 2\text{H}_2\text{O}$
 $M_r = 415.22$
 Orthorhombic, $Fddd$
 $a = 7.4093$ (5) Å
 $b = 18.9267$ (17) Å
 $c = 46.609$ (4) Å
 $V = 6536.1$ (9) Å³
 $Z = 16$
 Mo $K\alpha$ radiation
 $\mu = 1.11$ mm⁻¹
 $T = 296$ K
 $0.20 \times 0.19 \times 0.17$ mm

Data collection

Bruker SMART CCD diffractometer
 9724 measured reflections
 1877 independent reflections
 1520 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.032$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.035$
 $wR(F^2) = 0.098$
 $S = 1.06$
 1877 reflections
 132 parameters
 2 restraints
 H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.96$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.46$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O3}-\text{H3A}\cdots\text{O1}^{\text{i}}$	0.837 (17)	1.957 (16)	2.778 (2)	167 (2)
$\text{O5}-\text{H5A}\cdots\text{O2}^{\text{ii}}$	0.91	1.95	2.837 (3)	164
$\text{O5}-\text{H5B}\cdots\text{O5}^{\text{iii}}$	0.91	1.93	2.803 (4)	161
$\text{O5}-\text{H5B}'\cdots\text{O5}^{\text{ii}}$	0.90	2.22	3.096 (6)	165
$\text{O4}-\text{H4A}\cdots\text{O2}^{\text{iv}}$	0.74 (4)	2.02 (4)	2.750 (3)	171 (4)
$\text{O4}-\text{H4B}\cdots\text{O5}$	0.79 (4)	2.04 (4)	2.818 (3)	169 (3)

Symmetry codes: (i) $x + \frac{1}{2}, -y + \frac{7}{4}, -z + \frac{5}{4}$; (ii) $-x + \frac{5}{4}, y, -z + \frac{5}{4}$; (iii) $x, -y + \frac{5}{4}, -z + \frac{5}{4}$; (iv) $x + 1, y, z$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINTE* (Bruker, 1997); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *pubCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2639).

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supporting information

Acta Cryst. (2010). E66, m372–m373 [doi:10.1107/S1600536810007567]

Triaqua(1,10-phenanthroline-2,9-dicarboxylato)cobalt(II) dihydrate**Zi-Fa Shi, Zhu-Qing Gao and Jin-Zhong Gu****S1. Comment**

In recent years, the research of coordination compounds has been one of the most attractive fields due to their peculiar structures and properties (Zhao *et al.*, 2008; Poulsen *et al.*, 2005). Many multi-carboxylate or heterocyclic carboxylic acids are used for this purpose (Luo *et al.*, 2009; Han *et al.*, 2009). In the designed synthesis of the coordination compounds, H₂PDA is an excellent dicarboxylate ligand (Xie *et al.*, 2005). In order to extend the investigation, we have prepared the Co^{II} complex of H₂PDA, and report its crystal structure here.

The title compound (Fig. 1) is located on a twofold axis of symmetry which passes through the Co and O3 atoms, which is isomorphous with [Mg(PDA)(H₂O)₃].2H₂O (Park *et al.*, 2001). The seven-coordinated Co atom is in a distorted pentagonal bipyramidal geometry. Two N and two O atoms from PDA and one O atom from a water molecule define the pentagonal plane, and the two axial positions are occupied by O atoms derived from two water molecules.

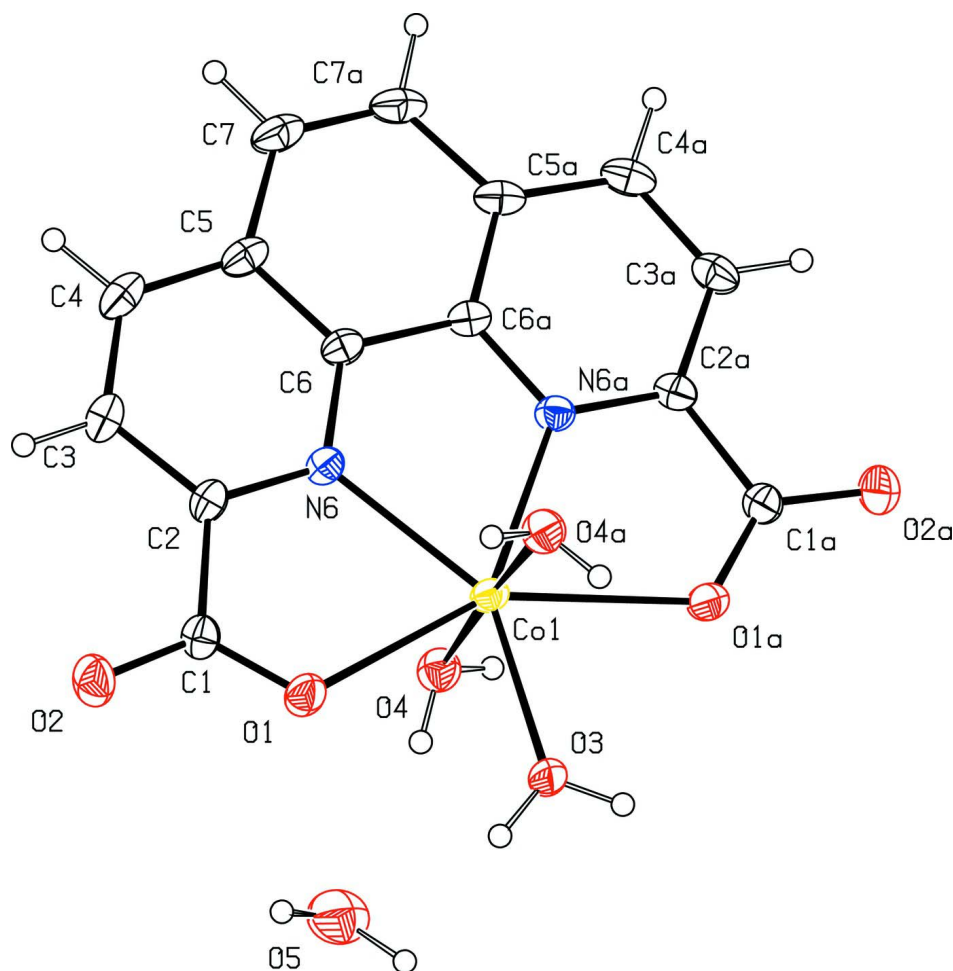
Important bond distances and angles are presented in Table 1. The bond distances between Co and the PDA donor atoms [Co—O1 2.3364 (16) Å and Co—N6 2.1936 (18) Å] are significantly longer than those to the coordinated water molecules [Co—O3 2.072 (2) Å and Co—O4 2.1254 (19) Å]. This is probably due to the high rigidity of PDA as well as the high affinity of the Co^{II} ion to water molecules (Zhang & Chen, 2009). The carboxylate groups of the PDA ligand are almost coplanar with the phenanthroline unit as indicated by the O1—C1—C2—N6 torsion angle of 2.6 (3)°. All bond distances and angles are similar to those observed in other seven-coordinated Co^{II} complexes (Newkome *et al.*, 1984; Rajput & Biradha, 2007). Adjacent molecules are linked by O—H...O hydrogen bonds, forming a three-dimensional network.

S2. Experimental

1,10-Phenanthroline-2,9-dicarboxylic acid was synthesized by using a literature method (De Cian *et al.*, 2007). To a solution of cobalt nitrate hexahydrate (0.145 g, 0.5 mmol) in water (5 ml) was added an aqueous solution (5 ml) of the ligand (0.135 g, 0.5 mmol) and sodium hydroxide (0.04 g, 1.0 mmol). The reactants were sealed in a 25-ml Teflon-lined, stainless-steel Parr bomb. The bomb was heated at 433 K for 3 days. The cool solution yielded single crystals in ca 50% yield. Anal. Calcd for C₁₄H₁₆CoN₂O₉: C, 40.50; H, 3.88; N, 6.75. Found: C, 40.01; H, 4.12; N, 6.45.

S3. Refinement

The coordinated water H atoms were located in a difference Fourier map and refined with distance constraints of O—H = 0.83 (3) Å. The free water H atoms attached to oxygen atoms were placed at calculated positions and refined with the riding model, considering the position of oxygen atoms and the quantity of H atoms. The carbon-bound H atoms were placed in geometrically idealized positions, with C—H = 0.93 Å, and constrained to ride on their respective parent atoms, with $U_{iso}(H) = 1.2 U_{eq}(C)$.

**Figure 1**

The molecular structure of (I) showing 50% probability displacement ellipsoids and the atom-numbering scheme (Symmetry code A: $-x+7/4, -y+7/4, z$).

Triaqua(1,10-phenanthroline-2,9-dicarboxylato)cobalt(II) dihydrate

Crystal data

$[\text{Co}(\text{C}_{14}\text{H}_6\text{N}_2\text{O}_4)(\text{H}_2\text{O})_3] \cdot 2\text{H}_2\text{O}$

$M_r = 415.22$

Orthorhombic, *Fddd*

Hall symbol: $-F\ 2uv\ 2vw$

$a = 7.4093\ (5)\ \text{\AA}$

$b = 18.9267\ (17)\ \text{\AA}$

$c = 46.609\ (4)\ \text{\AA}$

$V = 6536.1\ (9)\ \text{\AA}^3$

$Z = 16$

$F(000) = 3408$

$D_x = 1.688\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 2624 reflections

$\theta = 3.0\text{--}25.2^\circ$

$\mu = 1.11\ \text{mm}^{-1}$

$T = 296\ \text{K}$

Block, yellow

$0.20 \times 0.19 \times 0.17\ \text{mm}$

Data collection

Bruker APEXII CCD

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

9724 measured reflections

1877 independent reflections

1520 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.032$
 $\theta_{\text{max}} = 27.5^\circ$, $\theta_{\text{min}} = 1.8^\circ$
 $h = -9 \rightarrow 9$

$k = -24 \rightarrow 23$
 $l = -45 \rightarrow 60$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.035$
 $wR(F^2) = 0.098$
 $S = 1.06$
 1877 reflections
 132 parameters
 2 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: inferred from
 neighbouring sites
 H atoms treated by a mixture of independent
 and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0494P)^2 + 16.2882P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.96 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.46 \text{ e } \text{\AA}^{-3}$

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) etc. and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Co1	0.8750	0.8750	0.575281 (8)	0.02703 (15)	
C1	0.5006 (3)	0.79758 (12)	0.56914 (5)	0.0303 (5)	
C2	0.5619 (3)	0.81188 (11)	0.53888 (5)	0.0286 (5)	
C3	0.4668 (3)	0.79146 (13)	0.51428 (5)	0.0369 (5)	
H3	0.3573	0.7678	0.5160	0.044*	
C4	0.5356 (4)	0.80639 (13)	0.48763 (5)	0.0403 (6)	
H4	0.4723	0.7936	0.4712	0.048*	
C5	0.7025 (3)	0.84111 (12)	0.48536 (5)	0.0336 (5)	
C6	0.7884 (3)	0.85853 (11)	0.51113 (4)	0.0283 (5)	
C7	0.7938 (4)	0.85890 (13)	0.45911 (5)	0.0426 (6)	
H7	0.7394	0.8479	0.4417	0.051*	
H3A	0.953 (3)	0.8937 (8)	0.6302 (4)	0.063 (10)*	
H5A	0.8692	0.7242	0.6386	0.075*	
H5B	0.8592	0.6518	0.6227	0.075*	0.50
H5B'	0.7138	0.7061	0.6221	0.075*	0.50
H4A	1.088 (5)	0.7728 (17)	0.5770 (7)	0.049 (10)*	
H4B	0.955 (5)	0.7546 (18)	0.5909 (8)	0.060 (11)*	
N6	0.7184 (2)	0.84503 (10)	0.53724 (4)	0.0274 (4)	
O1	0.6029 (2)	0.82142 (9)	0.58824 (4)	0.0380 (4)	
O2	0.3589 (2)	0.76361 (11)	0.57280 (4)	0.0446 (5)	
O3	0.8750	0.8750	0.61974 (5)	0.0459 (7)	

O4	0.9892 (3)	0.77204 (10)	0.57658 (4)	0.0359 (4)
O5	0.8336 (4)	0.69890 (11)	0.62306 (5)	0.0670 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Co1	0.0278 (3)	0.0337 (3)	0.0196 (2)	−0.00067 (18)	0.000	0.000
C1	0.0231 (11)	0.0333 (12)	0.0344 (12)	0.0020 (9)	0.0013 (9)	−0.0046 (9)
C2	0.0256 (11)	0.0298 (11)	0.0305 (11)	0.0043 (9)	−0.0041 (9)	−0.0060 (8)
C3	0.0325 (13)	0.0401 (13)	0.0382 (13)	−0.0006 (10)	−0.0095 (10)	−0.0084 (10)
C4	0.0463 (15)	0.0418 (14)	0.0328 (13)	0.0055 (11)	−0.0153 (11)	−0.0087 (10)
C5	0.0438 (14)	0.0321 (12)	0.0248 (11)	0.0101 (11)	−0.0069 (9)	−0.0048 (9)
C6	0.0313 (12)	0.0296 (11)	0.0239 (10)	0.0057 (9)	−0.0010 (9)	−0.0015 (8)
C7	0.0637 (17)	0.0412 (14)	0.0228 (11)	0.0069 (12)	−0.0065 (11)	−0.0037 (9)
N6	0.0261 (9)	0.0320 (9)	0.0241 (9)	0.0013 (8)	−0.0014 (7)	−0.0023 (7)
O1	0.0327 (9)	0.0544 (10)	0.0268 (8)	−0.0075 (8)	0.0024 (7)	−0.0060 (7)
O2	0.0304 (10)	0.0550 (11)	0.0483 (11)	−0.0086 (8)	0.0052 (8)	−0.0053 (8)
O3	0.0490 (16)	0.0680 (17)	0.0206 (11)	−0.0285 (14)	0.000	0.000
O4	0.0312 (11)	0.0403 (10)	0.0363 (10)	0.0012 (8)	−0.0012 (8)	0.0004 (8)
O5	0.1009 (19)	0.0418 (11)	0.0582 (13)	−0.0027 (12)	0.0008 (13)	−0.0004 (10)

Geometric parameters (Å, °)

Co1—O3	2.072 (2)	C4—C5	1.404 (4)
Co1—O4	2.1254 (19)	C4—H4	0.9300
Co1—O4 ⁱ	2.1254 (19)	C5—C6	1.399 (3)
Co1—N6 ⁱ	2.1936 (18)	C5—C7	1.438 (3)
Co1—N6	2.1936 (18)	C6—N6	1.348 (3)
Co1—O1	2.3364 (16)	C6—C6 ⁱ	1.426 (5)
Co1—O1 ⁱ	2.3364 (16)	C7—C7 ⁱ	1.349 (6)
C1—O2	1.243 (3)	C7—H7	0.9300
C1—O1	1.253 (3)	O3—H3A	0.837 (17)
C1—C2	1.506 (3)	O4—H4A	0.74 (4)
C2—N6	1.320 (3)	O4—H4B	0.79 (4)
C2—C3	1.400 (3)	O5—H5A	0.9066
C3—C4	1.372 (4)	O5—H5B	0.9119
C3—H3	0.9300	O5—H5B'	0.8988
O3—Co1—O4	88.37 (5)	C4—C3—C2	119.8 (2)
O3—Co1—O4 ⁱ	88.37 (5)	C4—C3—H3	120.1
O4—Co1—O4 ⁱ	176.75 (11)	C2—C3—H3	120.1
O3—Co1—N6 ⁱ	143.92 (5)	C3—C4—C5	119.5 (2)
O4—Co1—N6 ⁱ	92.83 (8)	C3—C4—H4	120.3
O4 ⁱ —Co1—N6 ⁱ	89.80 (7)	C5—C4—H4	120.3
O3—Co1—N6	143.92 (5)	C6—C5—C4	116.5 (2)
O4—Co1—N6	89.80 (7)	C6—C5—C7	117.5 (2)
O4 ⁱ —Co1—N6	92.83 (8)	C4—C5—C7	126.0 (2)
N6 ⁱ —Co1—N6	72.16 (10)	N6—C6—C5	123.7 (2)

O3—Co1—O1	75.02 (4)	N6—C6—C6 ⁱ	115.43 (12)
O4—Co1—O1	86.46 (8)	C5—C6—C6 ⁱ	120.83 (14)
O4 ⁱ —Co1—O1	92.69 (7)	C7 ⁱ —C7—C5	121.69 (15)
N6 ⁱ —Co1—O1	141.06 (6)	C7 ⁱ —C7—H7	119.2
N6—Co1—O1	68.91 (6)	C5—C7—H7	119.2
O3—Co1—O1 ⁱ	75.02 (4)	C2—N6—C6	118.72 (18)
O4—Co1—O1 ⁱ	92.69 (7)	C2—N6—Co1	122.73 (14)
O4 ⁱ —Co1—O1 ⁱ	86.46 (8)	C6—N6—Co1	118.50 (15)
N6 ⁱ —Co1—O1 ⁱ	68.91 (6)	C1—O1—Co1	119.62 (15)
N6—Co1—O1 ⁱ	141.06 (6)	Co1—O3—H3A	125.6 (15)
O1—Co1—O1 ⁱ	150.03 (8)	Co1—O4—H4A	112 (3)
O2—C1—O1	126.8 (2)	Co1—O4—H4B	106 (2)
O2—C1—C2	118.4 (2)	H4A—O4—H4B	108 (3)
O1—C1—C2	114.7 (2)	H5A—O5—H5B	118.1
N6—C2—C3	121.7 (2)	H5A—O5—H5B'	104.2
N6—C2—C1	113.83 (18)	H5B—O5—H5B'	110.7
C3—C2—C1	124.5 (2)		
O2—C1—C2—N6	-176.6 (2)	C6 ⁱ —C6—N6—Co1	0.2 (3)
O1—C1—C2—N6	2.6 (3)	O3—Co1—N6—C2	-2.8 (2)
O2—C1—C2—C3	2.2 (3)	O4—Co1—N6—C2	84.14 (17)
O1—C1—C2—C3	-178.6 (2)	O4 ⁱ —Co1—N6—C2	-93.94 (17)
N6—C2—C3—C4	-0.5 (4)	N6 ⁱ —Co1—N6—C2	177.2 (2)
C1—C2—C3—C4	-179.2 (2)	O1—Co1—N6—C2	-2.14 (16)
C2—C3—C4—C5	1.0 (4)	O1 ⁱ —Co1—N6—C2	178.19 (14)
C3—C4—C5—C6	-0.1 (3)	O3—Co1—N6—C6	179.93 (11)
C3—C4—C5—C7	178.5 (2)	O4—Co1—N6—C6	-93.11 (16)
C4—C5—C6—N6	-1.3 (3)	O4 ⁱ —Co1—N6—C6	88.81 (16)
C7—C5—C6—N6	179.9 (2)	N6 ⁱ —Co1—N6—C6	-0.07 (11)
C4—C5—C6—C6 ⁱ	177.6 (2)	O1—Co1—N6—C6	-179.40 (17)
C7—C5—C6—C6 ⁱ	-1.2 (4)	O1 ⁱ —Co1—N6—C6	0.9 (2)
C6—C5—C7—C7 ⁱ	0.1 (4)	O2—C1—O1—Co1	174.59 (19)
C4—C5—C7—C7 ⁱ	-178.6 (3)	C2—C1—O1—Co1	-4.5 (3)
C3—C2—N6—C6	-0.9 (3)	O3—Co1—O1—C1	-176.70 (18)
C1—C2—N6—C6	178.00 (18)	O4—Co1—O1—C1	-87.44 (18)
C3—C2—N6—Co1	-178.11 (16)	O4 ⁱ —Co1—O1—C1	95.71 (18)
C1—C2—N6—Co1	0.7 (3)	N6 ⁱ —Co1—O1—C1	2.7 (2)
C5—C6—N6—C2	1.8 (3)	N6—Co1—O1—C1	3.71 (16)
C6 ⁱ —C6—N6—C2	-177.2 (2)	O1 ⁱ —Co1—O1—C1	-176.70 (18)
C5—C6—N6—Co1	179.17 (16)		

Symmetry code: (i) $-x+7/4, -y+7/4, z$.

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O3—H3A \cdots O1 ⁱⁱ	0.837 (17)	1.957 (16)	2.778 (2)	167 (2)
O5—H5A \cdots O2 ⁱⁱⁱ	0.91	1.95	2.837 (3)	164

O5—H5B···O5 ^{iv}	0.91	1.93	2.803 (4)	161
O5—H5B'···O5 ⁱⁱⁱ	0.90	2.22	3.096 (6)	165
O4—H4A···O2 ^v	0.74 (4)	2.02 (4)	2.750 (3)	171 (4)
O4—H4B···O5	0.79 (4)	2.04 (4)	2.818 (3)	169 (3)

Symmetry codes: (ii) $x+1/2, -y+7/4, -z+5/4$; (iii) $-x+5/4, y, -z+5/4$; (iv) $x, -y+5/4, -z+5/4$; (v) $x+1, y, z$.