

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

Poly[[diaqua[μ -1,4-bis(pyridin-4-yl-methyl)piperazine- κ^2 N:N']{ μ -2,2'-[(1,4-phenylene)bis(oxy)]diacetato- κ^2 O:O'}-cobalt(II)] pentahydrate]

Alexander D. Sample and Robert L. LaDuca*

Lyman Briggs College, Department of Chemistry, Michigan State University, East Lansing, MI 48825, USA

Correspondence e-mail: laduca@msu.edu

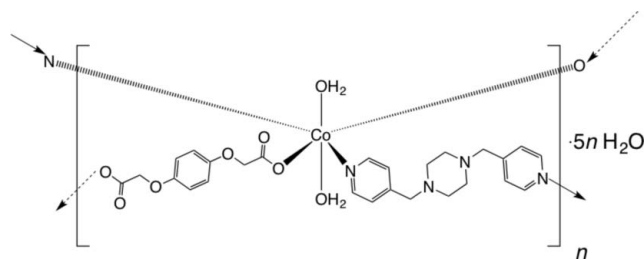
Received 15 April 2014; accepted 17 April 2014

Key indicators: single-crystal X-ray study; $T = 173$ K; mean $\sigma(\text{C}-\text{C}) = 0.012$ Å; some non-H atoms missing; R factor = 0.093; wR factor = 0.228; data-to-parameter ratio = 15.6.

In the title compound, $\{[\text{Co}(\text{C}_{10}\text{H}_8\text{O}_6)(\text{C}_{16}\text{H}_{20}\text{N}_4)(\text{H}_2\text{O})_2] \cdot 5\text{H}_2\text{O}\}_n$, octahedrally coordinated Co^{II} ions on crystallographic inversion centres are bound by *trans* O atoms belonging to two hydroquinone-*O,O'*-diacetate (hqda) anions {systematic name: 2,2'-[(1,4-phenylene)bis(oxy)]diacetate}, two *trans*-pyridine N-donor atoms from two bis(pyridin-4-ylmethyl)piperazine (4-bpmp) ligands, and two *trans* aqua ligands. The exobidentate hqda and 4-bpmp ligands form $[\text{Co}(\text{hqda})(4\text{-bpmp})(\text{H}_2\text{O})_2]_n$ coordination polymer layers parallel to (110) that are anchored into the full crystal structure by $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonding between aqua ligands and ligated hqda O atoms. Disordered water molecules of crystallization occupy incipient channels along [100]. However, these could not be modeled reliably and so they were treated with SQUEEZE in PLATON [Spek (2009)]. *Acta Cryst. D* **65**, 148–155]; the crystal data take the presence of these molecules into account. The crystal under investigation was twinned by non-merohedry, the twin fraction of the components being 53.3% and 46.7%. Only data from the major twin component were used in the refinement.

Related literature

For the preparation of bis(4-pyridylmethyl)piperazine, see: Niu *et al.* (2001). For the preparation of divalent metal terephthalate coordination polymers containing 4-bpmp, see: Farnum *et al.* (2013).



Experimental

Crystal data

$[\text{Co}(\text{C}_{10}\text{H}_8\text{O}_6)(\text{C}_{16}\text{H}_{20}\text{N}_4)(\text{H}_2\text{O})_2] \cdot 5\text{H}_2\text{O}$
 $M_r = 677.57$
 Triclinic, $P\bar{1}$
 $a = 5.7727$ (8) Å
 $b = 10.3421$ (15) Å
 $c = 13.1675$ (19) Å
 $\alpha = 87.175$ (2)°
 $\beta = 78.856$ (2)°
 $\gamma = 81.474$ (2)°
 $V = 762.61$ (19) Å³
 $Z = 1$
 Mo $K\alpha$ radiation
 $\mu = 0.63$ mm⁻¹
 $T = 173$ K
 $0.19 \times 0.17 \times 0.05$ mm

Data collection

Bruker APEXII CCD diffractometer
 Absorption correction: multi-scan (TWINABS; Sheldrick, 2003)
 $T_{\text{min}} = 0.676$, $T_{\text{max}} = 0.745$
 13385 measured reflections
 2786 independent reflections
 2032 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.053$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.093$
 $wR(F^2) = 0.228$
 $S = 1.10$
 2786 reflections
 179 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.71$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.89$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O4}-\text{H4A}\cdots\text{O1}^1$	0.91	2.28	2.945 (7)	130
$\text{O4}-\text{H4B}\cdots\text{O2}$	0.91	1.85	2.636 (7)	143

Symmetry code: (i) $x + 1, y, z$.

Data collection: APEX2 (Bruker, 2012); cell refinement: SAINT (Bruker, 2012); data reduction: SAINT (Bruker, 2012); program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: OLEX2 (Dolomanov *et al.*, 2009); software used to prepare material for publication: OLEX2.

We gratefully acknowledge Lyman Briggs College of Michigan State University for funding this work.

Supporting information for this paper is available from the IUCr electronic archives (Reference: TK5308).

References

- Bruker (2012). APEX2 and SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.
 Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.

Farnum, G. A., Murray, N. H. & LaDuca, R. L. (2013). *Inorg. Chim. Acta*, **406**, 65–72.
Niu, Y., Hou, H., Wei, Y., Fan, Y., Zhu, Y., Du, C. & Xin, X. (2001). *Inorg. Chem. Commun.* **4**, 358–361.

Sheldrick, G. M. (2003). *TWINABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.

supporting information

Acta Cryst. (2014). E70, m198–m199 [doi:10.1107/S1600536814008745]

Poly[[diaqua[μ -1,4-bis(pyridin-4-ylmethyl)piperazine- κ^2 N:N']{ μ -2,2'-[(1,4-phenylene)bis(oxy)]diacetato- κ^2 O:O}]cobalt(II)] pentahydrate]

Alexander D. Sample and Robert L. LaDuca

S1. Chemical context

Some divalent metal terephthalate coordination polymers with bis(pyridin-4-ylmethyl)piperazine (4-bpmp) coligands show intriguing entangled topologies. (Farnum *et al.*, 2013). We hoped to expand the scope of these materials by using a *para* aromatic dicarboxylate with longer pendant arms, such as hydroquinone-*O,O'*-diacetic acid (H₂hqda). The title compound was obtained as pink crystals through the hydrothermal reaction of cobalt nitrate, H₂hqda, and 4-bpmp.

S2. Structural commentary

The asymmetric unit of the title compound contains a divalent cobalt atom on a crystallographic inversion centre, an aqua ligand, half of a hqda ligand whose centroid rests on another crystallographic inversion centre, and one half of a 4-bpmp ligand whose centroid rests on a third crystallographic inversion centre.

The cobalt atom is octahedrally coordinated (Fig. 1), with the equatorial plane containing *trans* pyridyl N atom donors from two 4-bpmp ligands and *trans* O atom donors from monodentate carboxylate groups belonging to two hqda ligands. The aqua ligands are located in the axial positions.

The Co atoms are connected by exobidentate, bis(monodentate) hqda ligands to form [Co(hqda)(H₂O)₂]_n coordination polymer chains that are oriented parallel to [0 1 0]. Each individual chain is linked to two others by tethering 4-bpmp ligands, to construct [Co(hqda)(4-bpmp)(H₂O)₂]_n coordination polymer layers parallel to (110) (Fig. 2). As each cobalt atom is connected to four others, the underlying topology of the layer is a (4,4) rectangular grid. The internuclear Co...Co through-space distances across the grid apertures are 13.17 Å and 25.14 Å.

S3. Supramolecular features

Individual [Co(hqda)(4-bpmp)(H₂O)₂]_n layers stack in a *AAA* pattern along the *a* crystal direction (Fig. 3). The supramolecular O—H...O hydrogen bonding between aqua ligands in one layer and ligated hqda O atoms in two others provides the impetus for the formation of the three-dimensional crystal structure of the title compound.

Disordered water molecules of crystallization occupy incipient channels along [1 0 0]. These could not be refined well, and thus their electron density was modeled using the *SQUEEZE* subroutine of *PLATON* (Spek, 2009). The resulting analysis indicated the presence of approximately five water molecules per unit cell, in a region comprising 20.6% of the total unit cell volume.

S4. Database survey

This compound was not previously reported in the CCDC.

S5. Synthesis and crystallization

Cobalt(II) nitrate hexahydrate and hydroquinone-*O,O'*-diacetic acid (H₂hqda) were obtained commercially. Bis(4-pyridymethyl)piperazine (4-bpmp) was prepared *via* a published procedure (Niu *et al.*, 2001). A mixture of cobalt(II) nitrate hexahydrate (68 mg, 0.23 mmol), H₂hqda (84 mg, 0.37 mmol), 4-bpmp (99 mg, 0.37 mmol), 0.25 ml of a 1.0 M NaOH solution and 10.0 g water (550 mmol) was placed into a 23 ml Teflon-lined Parr acid digestion bomb, which was then heated under autogenous pressure at 393 K for 24 h. Pink plates of the title compound were obtained in a multi-phase mixture.

S6. Refinement

All H atoms bound to C atoms were placed in calculated positions, with C—H = 0.95–0.99 Å, and refined in riding mode with $U_{\text{iso}} = 1.2U_{\text{eq}}(\text{C})$. The H atoms within the aqua ligand were found in a difference Fourier map, restrained with O—H = 0.85 Å and refined with $U_{\text{iso}} = 1.5U_{\text{eq}}(\text{O})$.

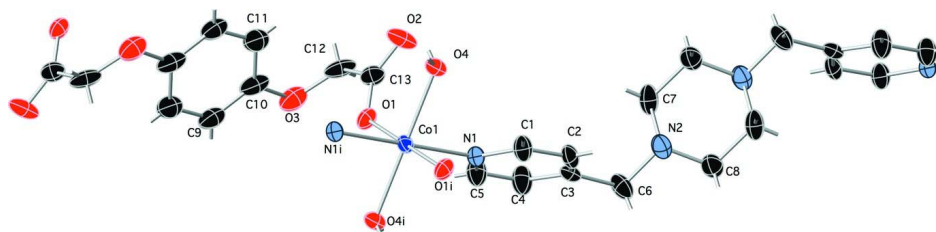


Figure 1

The octahedral coordination environment of the title compound, showing 50% probability ellipsoids and atom numbering scheme. Hydrogen atom positions are shown as gray sticks. Color codes: dark blue Co, red O, light blue N, black C. Symmetry code: (i) $-x + 1, -y, -z$.

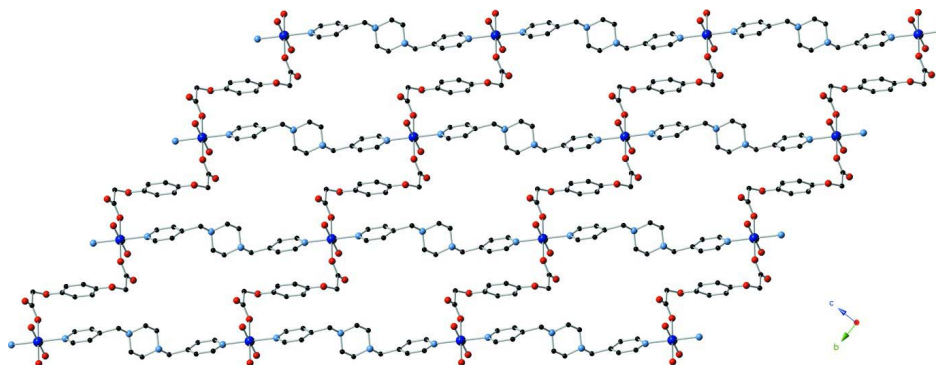


Figure 2

A single $[\text{Co}(\text{hqda})(4\text{-bpmp})(\text{H}_2\text{O})_2]_n$ coordination polymer layer.

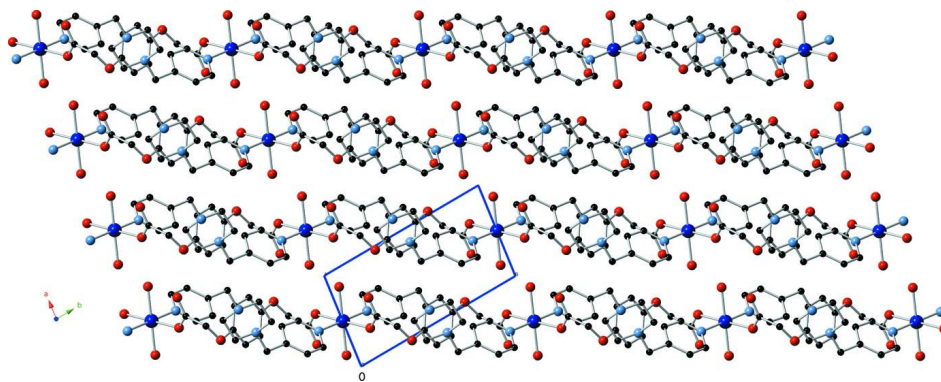


Figure 3

Stacking of coordination polymer layers within the title compound.

Poly[[diaqua[μ -1,4-bis(pyridin-4-ylmethyl)piperazine- κ^2 N:N']{ μ -2,2'-[(1,4-phenylene)bis(oxy)]diacetato- κ^2 O:O'}cobalt(II)] pentahydrate]

Crystal data

[Co(C₁₀H₈O₆)(C₁₆H₂₀N₄)(H₂O)₂]₂·5H₂O

$M_r = 677.57$

Triclinic, $P\bar{1}$

$a = 5.7727$ (8) Å

$b = 10.3421$ (15) Å

$c = 13.1675$ (19) Å

$\alpha = 87.175$ (2)°

$\beta = 78.856$ (2)°

$\gamma = 81.474$ (2)°

$V = 762.61$ (19) Å³

$Z = 1$

$F(000) = 357$

$D_x = 1.475$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 2300 reflections

$\theta = 2.5$ – 25.0 °

$\mu = 0.63$ mm⁻¹

$T = 173$ K

Plate, pink

$0.19 \times 0.17 \times 0.05$ mm

Data collection

Bruker APEXII CCD

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan

(TWINABS; Sheldrick, 2003)

$T_{\min} = 0.676$, $T_{\max} = 0.745$

13385 measured reflections

2786 independent reflections

2032 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.053$

$\theta_{\max} = 25.4$ °, $\theta_{\min} = 2.0$ °

$h = -6$ → 6

$k = -12$ → 12

$l = 0$ → 15

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.093$

$wR(F^2) = 0.228$

$S = 1.10$

2786 reflections

179 parameters

0 restraints

Primary atom site location: structure-invariant

direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0164P)^2 + 9.3P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.71$ e Å⁻³

$\Delta\rho_{\min} = -0.89$ e Å⁻³

Special details

Experimental. TWINABS-2012/1 (Bruker,2012) was used for absorption correction.

For component 1: wR2(int) was 0.0549 before and 0.0460 after correction. The Ratio of minimum to maximum transmission is 0.91. The $\lambda/2$ correction factor is Not present

For component 2: wR2(int) was 0.0664 before and 0.0469 after correction. The Ratio of minimum to maximum transmission not present. The $\lambda/2$ correction factor is Not present

Final HKLF 4 output contains 13385 reflections, Rint = 0.0533 (6718 with $I > 3\text{sig}(I)$), Rint = 0.0400)

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Co1	0.5000	0.0000	0.0000	0.0217 (4)
O4	0.8245 (8)	0.0527 (5)	-0.0875 (4)	0.0259 (11)
H4A	0.9116	0.0861	-0.0468	0.039*
H4B	0.7974	0.1148	-0.1372	0.039*
O1	0.2947 (8)	0.1293 (4)	-0.0851 (4)	0.0284 (12)
O2	0.5742 (10)	0.1838 (6)	-0.2164 (4)	0.0437 (15)
O3	-0.0339 (10)	0.3206 (5)	-0.1427 (5)	0.0442 (15)
N1	0.4646 (10)	0.1467 (5)	0.1144 (5)	0.0262 (14)
N2	0.1474 (11)	0.4777 (6)	0.4001 (5)	0.0335 (15)
C13	0.3664 (14)	0.1882 (7)	-0.1687 (6)	0.0318 (18)
C12	0.1723 (15)	0.2740 (8)	-0.2163 (7)	0.043 (2)
H12A	0.2401	0.3498	-0.2528	0.052*
H12B	0.1244	0.2227	-0.2684	0.052*
C10	-0.0057 (15)	0.4091 (8)	-0.0716 (8)	0.042 (2)
C9	-0.2010 (14)	0.4424 (8)	0.0052 (8)	0.042 (2)
H9	-0.3397	0.4018	0.0088	0.051*
C5	0.6335 (14)	0.1602 (8)	0.1688 (7)	0.038 (2)
H5	0.7780	0.1009	0.1571	0.046*
C1	0.2635 (13)	0.2314 (7)	0.1348 (6)	0.0335 (18)
H1	0.1390	0.2231	0.0988	0.040*
C6	0.3746 (14)	0.4588 (9)	0.3307 (6)	0.0380 (19)
H6A	0.5028	0.4440	0.3719	0.046*
H6B	0.3944	0.5396	0.2883	0.046*
C8	0.1075 (14)	0.6027 (8)	0.4482 (6)	0.0378 (19)
H8A	0.1156	0.6725	0.3939	0.045*
H8B	0.2350	0.6077	0.4878	0.045*
C7	0.1316 (15)	0.3748 (8)	0.4801 (6)	0.040 (2)
H7A	0.2605	0.3745	0.5200	0.048*
H7B	0.1524	0.2886	0.4475	0.048*
C11	0.1974 (15)	0.4660 (8)	-0.0770 (7)	0.042 (2)

H11	0.3332	0.4424	-0.1295	0.051*
C2	0.2273 (13)	0.3304 (7)	0.2053 (6)	0.0305 (17)
H2	0.0812	0.3883	0.2166	0.037*
C3	0.4032 (13)	0.3448 (7)	0.2590 (6)	0.0301 (17)
C4	0.6070 (14)	0.2550 (8)	0.2401 (7)	0.043 (2)
H4	0.7311	0.2593	0.2773	0.051*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Co1	0.0171 (7)	0.0232 (7)	0.0226 (8)	0.0000 (5)	-0.0008 (5)	-0.0004 (5)
O4	0.022 (3)	0.031 (3)	0.025 (3)	-0.006 (2)	-0.003 (2)	0.008 (2)
O1	0.027 (3)	0.022 (2)	0.036 (3)	0.002 (2)	-0.011 (2)	0.003 (2)
O2	0.036 (3)	0.059 (4)	0.035 (3)	-0.018 (3)	-0.001 (3)	0.022 (3)
O3	0.035 (3)	0.037 (3)	0.066 (4)	-0.006 (3)	-0.024 (3)	0.007 (3)
N1	0.021 (3)	0.023 (3)	0.031 (4)	0.001 (2)	0.000 (3)	-0.002 (3)
N2	0.036 (4)	0.035 (4)	0.026 (4)	0.001 (3)	-0.001 (3)	-0.004 (3)
C13	0.037 (5)	0.033 (4)	0.027 (4)	-0.010 (4)	-0.009 (4)	0.007 (3)
C12	0.046 (5)	0.043 (5)	0.046 (6)	-0.021 (4)	-0.017 (4)	0.028 (4)
C10	0.036 (5)	0.027 (4)	0.070 (7)	-0.010 (4)	-0.024 (4)	0.019 (4)
C9	0.023 (4)	0.028 (4)	0.078 (7)	-0.004 (3)	-0.019 (4)	0.012 (4)
C5	0.028 (4)	0.041 (5)	0.047 (5)	0.004 (4)	-0.012 (4)	-0.013 (4)
C1	0.027 (4)	0.034 (4)	0.037 (5)	0.009 (3)	-0.010 (3)	-0.003 (3)
C6	0.035 (4)	0.054 (5)	0.025 (4)	-0.010 (4)	0.000 (3)	-0.012 (4)
C8	0.035 (5)	0.040 (5)	0.034 (5)	0.001 (4)	-0.001 (4)	0.004 (4)
C7	0.038 (5)	0.040 (5)	0.039 (5)	0.014 (4)	-0.012 (4)	-0.009 (4)
C11	0.033 (5)	0.035 (5)	0.059 (6)	-0.004 (4)	-0.010 (4)	0.009 (4)
C2	0.024 (4)	0.033 (4)	0.030 (4)	0.008 (3)	-0.001 (3)	-0.002 (3)
C3	0.027 (4)	0.034 (4)	0.025 (4)	-0.008 (3)	0.008 (3)	0.000 (3)
C4	0.027 (4)	0.045 (5)	0.058 (6)	0.002 (4)	-0.012 (4)	-0.019 (4)

Geometric parameters (Å, °)

Co1—O4	2.131 (4)	C9—H9	0.9500
Co1—O4 ⁱ	2.131 (4)	C9—C11 ⁱⁱ	1.375 (12)
Co1—O1	2.084 (5)	C5—H5	0.9500
Co1—O1 ⁱ	2.084 (5)	C5—C4	1.363 (11)
Co1—N1	2.151 (6)	C1—H1	0.9500
Co1—N1 ⁱ	2.151 (6)	C1—C2	1.382 (10)
O4—H4A	0.9131	C6—H6A	0.9900
O4—H4B	0.9130	C6—H6B	0.9900
O1—C13	1.260 (9)	C6—C3	1.517 (10)
O2—C13	1.237 (9)	C8—H8A	0.9900
O3—C12	1.421 (11)	C8—H8B	0.9900
O3—C10	1.388 (10)	C8—C7 ⁱⁱⁱ	1.508 (11)
N1—C5	1.343 (10)	C7—C8 ⁱⁱⁱ	1.508 (11)
N1—C1	1.337 (9)	C7—H7A	0.9900
N2—C6	1.440 (10)	C7—H7B	0.9900

N2—C8	1.435 (10)	C11—C9 ⁱⁱ	1.375 (12)
N2—C7	1.461 (10)	C11—H11	0.9500
C13—C12	1.535 (11)	C2—H2	0.9500
C12—H12A	0.9900	C2—C3	1.373 (11)
C12—H12B	0.9900	C3—C4	1.376 (11)
C10—C9	1.376 (13)	C4—H4	0.9500
C10—C11	1.377 (11)		
O4—Co1—O4 ⁱ	180.0	C11 ⁱⁱ —C9—C10	120.6 (8)
O4—Co1—N1 ⁱ	85.7 (2)	C11 ⁱⁱ —C9—H9	119.7
O4 ⁱ —Co1—N1 ⁱ	94.3 (2)	N1—C5—H5	118.5
O4—Co1—N1	94.3 (2)	N1—C5—C4	123.0 (7)
O4 ⁱ —Co1—N1	85.7 (2)	C4—C5—H5	118.5
O1 ⁱ —Co1—O4	87.77 (19)	N1—C1—H1	118.2
O1—Co1—O4 ⁱ	87.77 (18)	N1—C1—C2	123.5 (7)
O1 ⁱ —Co1—O4 ⁱ	92.23 (19)	C2—C1—H1	118.2
O1—Co1—O4	92.23 (19)	N2—C6—H6A	108.9
O1 ⁱ —Co1—O1	180.0	N2—C6—H6B	108.9
O1 ⁱ —Co1—N1 ⁱ	90.1 (2)	N2—C6—C3	113.4 (7)
O1—Co1—N1	90.1 (2)	H6A—C6—H6B	107.7
O1 ⁱ —Co1—N1	89.9 (2)	C3—C6—H6A	108.9
O1—Co1—N1 ⁱ	89.9 (2)	C3—C6—H6B	108.9
N1—Co1—N1 ⁱ	180.0 (3)	N2—C8—H8A	109.3
Co1—O4—H4A	112.0	N2—C8—H8B	109.3
Co1—O4—H4B	111.7	N2—C8—C7 ⁱⁱⁱ	111.8 (7)
H4A—O4—H4B	106.8	H8A—C8—H8B	107.9
C13—O1—Co1	127.2 (5)	C7 ⁱⁱⁱ —C8—H8A	109.3
C10—O3—C12	116.7 (7)	C7 ⁱⁱⁱ —C8—H8B	109.3
C5—N1—Co1	124.3 (5)	N2—C7—C8 ⁱⁱⁱ	110.0 (6)
C1—N1—Co1	119.6 (5)	N2—C7—H7A	109.7
C1—N1—C5	116.2 (6)	N2—C7—H7B	109.7
C6—N2—C7	111.1 (6)	C8 ⁱⁱⁱ —C7—H7A	109.7
C8—N2—C6	110.9 (7)	C8 ⁱⁱⁱ —C7—H7B	109.7
C8—N2—C7	109.3 (6)	H7A—C7—H7B	108.2
O1—C13—C12	115.8 (7)	C10—C11—H11	120.5
O2—C13—O1	127.3 (7)	C9 ⁱⁱ —C11—C10	119.1 (9)
O2—C13—C12	116.9 (7)	C9 ⁱⁱ —C11—H11	120.5
O3—C12—C13	113.7 (7)	C1—C2—H2	120.1
O3—C12—H12A	108.8	C3—C2—C1	119.8 (7)
O3—C12—H12B	108.8	C3—C2—H2	120.1
C13—C12—H12A	108.8	C2—C3—C6	120.6 (7)
C13—C12—H12B	108.8	C2—C3—C4	116.6 (7)
H12A—C12—H12B	107.7	C4—C3—C6	122.8 (7)
C9—C10—O3	115.6 (7)	C5—C4—C3	121.0 (8)
C9—C10—C11	120.3 (9)	C5—C4—H4	119.5
C11—C10—O3	124.1 (9)	C3—C4—H4	119.5
C10—C9—H9	119.7		

Co1—O1—C13—O2	-1.8 (12)	N1—C1—C2—C3	-0.3 (12)
Co1—O1—C13—C12	177.7 (5)	N2—C6—C3—C2	48.1 (10)
Co1—N1—C5—C4	179.3 (7)	N2—C6—C3—C4	-135.2 (8)
Co1—N1—C1—C2	-178.6 (6)	C12—O3—C10—C9	-173.6 (7)
O4—Co1—O1—C13	12.3 (6)	C12—O3—C10—C11	8.8 (11)
O4 ⁱ —Co1—O1—C13	-167.7 (6)	C10—O3—C12—C13	66.9 (9)
O4 ⁱ —Co1—N1—C5	125.5 (6)	C9—C10—C11—C9 ⁱⁱ	-0.8 (13)
O4—Co1—N1—C5	-54.5 (6)	C5—N1—C1—C2	1.5 (12)
O4—Co1—N1—C1	125.6 (6)	C1—N1—C5—C4	-0.8 (12)
O4 ⁱ —Co1—N1—C1	-54.4 (6)	C1—C2—C3—C6	175.3 (7)
O1 ⁱ —Co1—N1—C5	33.2 (6)	C1—C2—C3—C4	-1.6 (12)
O1—Co1—N1—C5	-146.8 (6)	C6—N2—C8—C7 ⁱⁱⁱ	179.1 (7)
O1—Co1—N1—C1	33.3 (6)	C6—N2—C7—C8 ⁱⁱⁱ	179.8 (7)
O1 ⁱ —Co1—N1—C1	-146.7 (6)	C6—C3—C4—C5	-174.6 (8)
O1—C13—C12—O3	26.2 (10)	C8—N2—C6—C3	-166.4 (7)
O2—C13—C12—O3	-154.2 (7)	C8—N2—C7—C8 ⁱⁱⁱ	57.0 (10)
O3—C10—C9—C11 ⁱⁱ	-176.9 (7)	C7—N2—C6—C3	71.8 (9)
O3—C10—C11—C9 ⁱⁱ	176.7 (7)	C7—N2—C8—C7 ⁱⁱⁱ	-58.1 (9)
N1—Co1—O1—C13	106.6 (6)	C11—C10—C9—C11 ⁱⁱ	0.8 (13)
N1 ⁱ —Co1—O1—C13	-73.4 (6)	C2—C3—C4—C5	2.2 (13)
N1—C5—C4—C3	-1.1 (14)		

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

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O4—H4 <i>A</i> ...O1 ^{iv}	0.91	2.28	2.945 (7)	130
O4—H4 <i>B</i> ...O2	0.91	1.85	2.636 (7)	143

Symmetry code: (iv) $x+1, y, z$.