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catena-Poly[[diaquacobalt(II)]-bis(μ-3-carboxyadamantane-1-carboxylato-$\kappa^2O^1:O^3$)]

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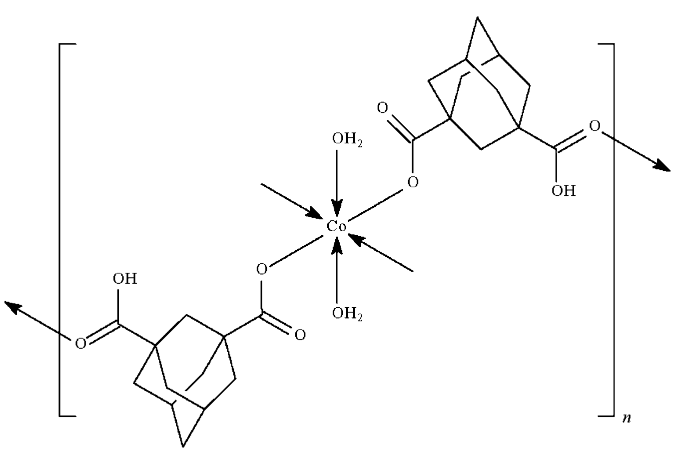
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.031; wR factor = 0.086; data-to-parameter ratio = 16.3.

In the title compound, $[Co(C_{12}H_{15}O_4)_2(H_2O)_2]_n$, adjacent Co^{II} atoms ($\overline{1}$ symmetry) are bridged by 3-carboxyadamantane-1-carboxylate anions, forming a chain running along [001]. Interchain O—H$\cdots$O hydrogen bonds link the chains into layers parallel to (100); the layers are further connected *via* interlayer hydrogen bonds interactions, forming a three-dimensional framework.

Related literature

For related compounds, see: Nielsen *et al.* (2008); Zhao *et al.* (2007); Zheng *et al.* (2008).



Experimental

Crystal data

$[Co(C_{12}H_{15}O_4)_2(H_2O)_2]$
M_r = 541.44
Orthorhombic, $Pccn$
a = 10.718 (2) Å
b = 23.638 (5) Å
c = 9.0726 (18) Å
V = 2298.6 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.81 mm^{-1}
T = 293 K
0.10 × 0.10 × 0.10 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.921, T_{max} = 0.925
20865 measured reflections
2622 independent reflections
2145 reflections with $I > 2\sigma(I)$
R_{int} = 0.033

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.086
S = 1.06
2622 reflections
161 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

Table 1
Selected bond lengths (Å).

Co—O1	2.0574 (12)	Co—O4^{i}	2.1061 (12)
Co—O5	2.0956 (14)		

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

Table 2
Hydrogen-bond geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O3—H1$\cdots$O1^{ii}	0.81	1.82	2.6058 (19)	166
O5—H2$\cdots$O2	0.80	2.07	2.7762 (18)	147
O5—H3$\cdots$O2^{iii}	0.81	2.02	2.8334 (18)	175

Symmetry codes: (ii) $x, y, z + 1$; (iii) $-x + \frac{3}{2}, y, z - \frac{1}{2}$.

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); 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: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2552).

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catena-Poly[[diaquacobalt(II)]-bis(μ-3-carboxyadamantane-1-carboxylato-κ^2O^1:O^3)]

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S1. Comment

The cambridge Structural Database (Version 5.30, February 2009) lists few examples of metal (II) adamantane-1,3-dicarboxylates (Nielsen *et al.*, 2008; Zhao *et al.*, 2007). The dicarboxylate group is rigid, much more different from the aliphatic dicarboxylic acids (Zheng *et al.*, 2008), effected severely by spacial steric hindrance. The asymmetric unit of the title compoud consists of one Co^{2+} cation, one aqua ligand and one $Hadc^-$ anion (H_2adc = adamantane-1,3-dicarboxylic acid) (Fig.1). The Co atoms at the Wckoff 4a sites are crystallographically imposed by iversion center and are each located in an elongated octahedral coordination sphere defined by two aqua ligands and four carboxylate oxygen atoms from four 3-carboxyadamantane-1-carboxylate anions. The axial Co—O bond distances averaged at 2.106 (1) Å are slightly longer than the equatorial ones of 2.078 (1) Å. The *trans*- and *cisoid* O—Co—O angles fall in the regions 88.49 (5)–91.51 (5)° and 180°, respectively, exhibiting slight diviation from the corresponding values for a regular geometry (Table 1). Each carboxylate anion monodentately coordinates one Co^{2+} ion in *syn* fashion. Interestingly, one of the two carboxylate anions from each ligand is protonated and coordinates one Co^{2+} ion by carbonyl oxygen atom, which is rare in former reports. The Co^{2+} ions are bridged by 3-carboxyadamantane-1-carboxylate anions to form one-dimensional chains running along the [001] direction. On the basis of the interchain O—H···O hydrogen bonds (Table 2),these chains are assembled into layers parallel to (100) (Fig.2). The layers are further connected to form a three-dimensional framework *via* interlayer hydrogen bonds interaction.

S2. Experimental

Adamantane-1,3-dicarboxylic acid (H_2adc) (0.0666 g, 0.3 mmol), 1 *M* NaOH (0.5 ml, 0.5 mmol) was consequently added to 15 mol aqueous solution, then the mixture was heated constantly at 90 °C and stirred for 30 min, yielding colorless solution, to which was added $CoCl_2.6H_2O$ (0.2485 g, 1.0 mmol) and continually stirred for 30 min, then the purple solution (pH = 5.12) was cooled to room temperature and laid undisturbed, purple block-like crystals was afforded after two weeks.

S3. Refinement

H atoms bonded to C atoms were palced in geometrically calculated position and were refined using a riding model, with U_{iso}(H) = 1.2 U_{eq}(C). H atoms attached to O atoms were found in a difference Fourier synthesis and were refined using a riding model, with the O—H distances fixed as initially found and with U_{iso}(H) values set at 1.2 Ueq(O).

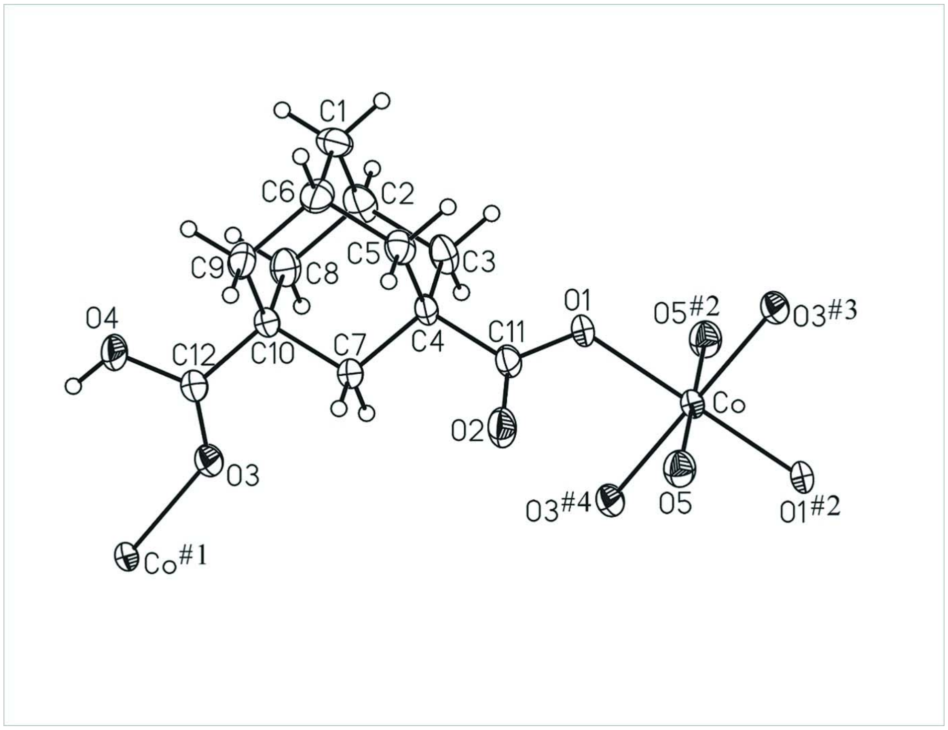

Figure 1

View of the molecular structure of the title compound, Displacement ellipsoids are drawn at the 45% probability level. [Symmetry codes: (i) *x*, *y*, *z* + 1; (ii) -*x* + 1, -*y* + 1, -*z*; (iii) *x*, *y*, *z* - 1; (iv) -*x* + 1, -*y* + 1, -*z* + 1.]

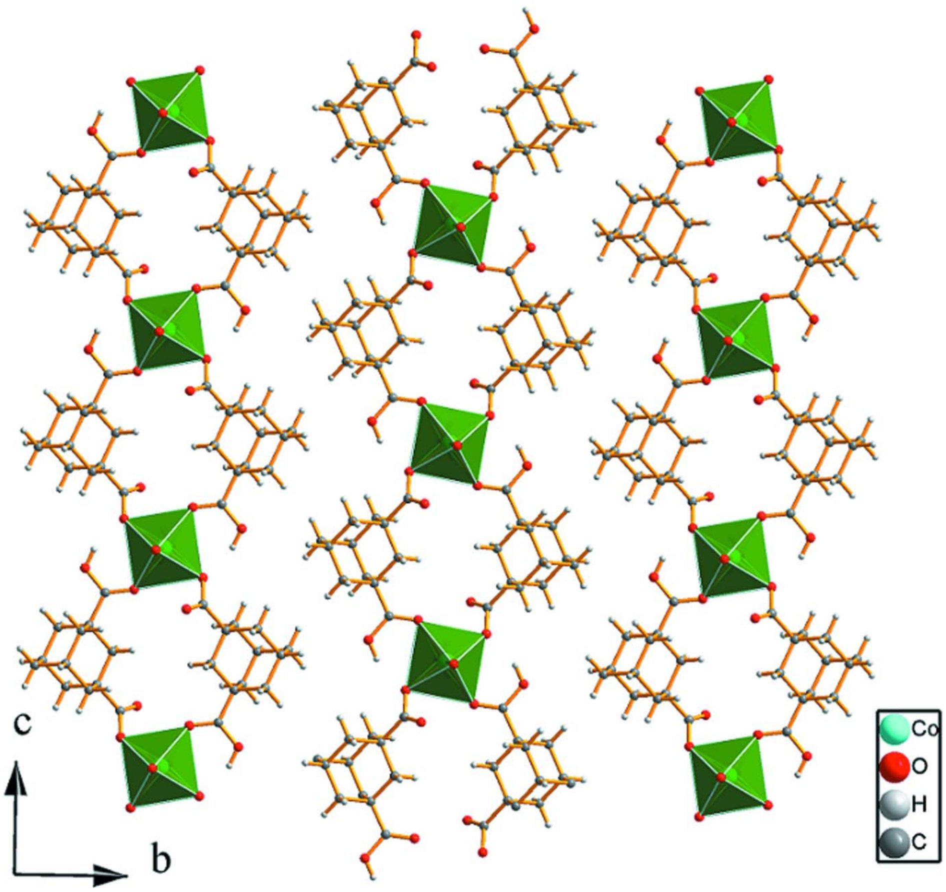

Figure 2

The two-dimensional layer structure constructed by one-dimensional chains through hydrogen bonds interaction (the hydrogen bonds are neglected)

catena-Poly[[diaquacobalt(II)]-bis(μ-3-carboxyadamantane-1-carboxylato- $\kappa^2O^1:O^3$)]

Crystal data

$[Co(C_{12}H_{15}O_4)_2(H_2O)_2]$
$M_r = 541.44$
Orthorhombic, *Pccn*
Hall symbol: -P 2ab 2ac
$a = 10.718$ (2) Å
$b = 23.638$ (5) Å
$c = 9.0726$ (18) Å
$V = 2298.6$ (8) Å^3
$Z = 4$
$F(000) = 1140$
$D_x = 1.565$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 20865 reflections
$\theta = 3.1–27.5°$
$\mu = 0.81$ mm^{-1}
$T = 293$ K
Block, purple
0.10 × 0.10 × 0.10 mm

Data collection

Rigaku R-AXIS RAPID
diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 0 pixels mm^{-1}
ω scans
Absorption correction: multi-scan
(*ABSCOR*; Higashi, 1995)
$T_{min} = 0.921$, $T_{max} = 0.925$
20865 measured reflections
2622 independent reflections
2145 reflections with $I > 2\sigma(I)$
$R_{int} = 0.033$

θ_{max} = 27.5°, θ_{min} = 3.1°
h = −13→13
k = −30→30
l = −11→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.086
S = 1.06
2622 reflections
161 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
$w = 1/[\sigma^2(F_o^2) + (0.0444P)^2 + 0.9177P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

Special details

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Co	0.5000	0.5000	0.0000	0.02148 (11)
C1	0.50924 (15)	0.62852 (7)	0.64285 (18)	0.0239 (3)
C2	0.37038 (16)	0.64210 (7)	0.61757 (18)	0.0300 (4)
H2A	0.3213	0.6077	0.6246	0.036*
H2B	0.3413	0.6683	0.6924	0.036*
C3	0.35442 (17)	0.66835 (9)	0.46523 (19)	0.0343 (4)
H3A	0.2660	0.6770	0.4490	0.041*
C4	0.39899 (16)	0.62682 (8)	0.34654 (18)	0.0310 (4)
H4A	0.3497	0.5924	0.3511	0.037*
H4B	0.3878	0.6435	0.2498	0.037*
C5	0.53696 (15)	0.61264 (7)	0.37055 (16)	0.0212 (3)
C6	0.55413 (17)	0.58683 (7)	0.52439 (16)	0.0256 (3)
H6A	0.6415	0.5780	0.5403	0.031*
H6B	0.5069	0.5519	0.5315	0.031*
C7	0.4296 (2)	0.72253 (8)	0.4553 (2)	0.0394 (5)
H7A	0.4012	0.7490	0.5297	0.047*
H7B	0.4176	0.7398	0.3593	0.047*
C8	0.56719 (19)	0.70952 (7)	0.47855 (19)	0.0327 (4)
H8A	0.6155	0.7446	0.4718	0.039*
C9	0.58580 (17)	0.68311 (7)	0.63123 (18)	0.0295 (4)
H9A	0.5594	0.7096	0.7068	0.035*
H9B	0.6735	0.6748	0.6465	0.035*
C10	0.61268 (16)	0.66778 (7)	0.36080 (18)	0.0278 (4)

H10A	0.7005	0.6597	0.3758	0.033*
H10B	0.6028	0.6844	0.2637	0.033*
C11	0.58484 (15)	0.57136 (7)	0.25371 (16)	0.0247 (3)
O1	0.52461 (12)	0.56998 (5)	0.13165 (12)	0.0296 (3)
O2	0.67854 (12)	0.54217 (6)	0.27703 (13)	0.0378 (3)
C12	0.51789 (16)	0.60130 (7)	0.79318 (18)	0.0280 (4)
O3	0.51926 (19)	0.63717 (6)	0.90404 (15)	0.0615 (5)
H1	0.5154	0.6209	0.9823	0.074*
O4	0.51779 (12)	0.55044 (5)	0.80980 (13)	0.0312 (3)
O5	0.69310 (12)	0.48644 (6)	0.00823 (12)	0.0314 (3)
H2	0.7197	0.5019	0.0807	0.038*
H3	0.7333	0.5010	−0.0575	0.038*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Co	0.02775 (18)	0.02296 (17)	0.01371 (17)	0.00182 (11)	0.00009 (11)	0.00032 (11)
C1	0.0317 (9)	0.0242 (7)	0.0158 (7)	0.0027 (6)	0.0009 (6)	0.0004 (6)
C2	0.0277 (9)	0.0389 (9)	0.0232 (8)	−0.0011 (7)	0.0058 (7)	−0.0017 (7)
C3	0.0239 (9)	0.0516 (11)	0.0274 (8)	0.0131 (8)	−0.0004 (7)	0.0010 (8)
C4	0.0250 (9)	0.0467 (10)	0.0213 (8)	0.0020 (7)	−0.0039 (6)	−0.0009 (7)
C5	0.0233 (7)	0.0267 (7)	0.0137 (6)	0.0007 (6)	−0.0009 (6)	−0.0011 (6)
C6	0.0361 (9)	0.0243 (7)	0.0164 (7)	0.0041 (7)	0.0003 (6)	−0.0009 (6)
C7	0.0554 (13)	0.0336 (9)	0.0292 (9)	0.0186 (9)	0.0036 (9)	0.0067 (8)
C8	0.0438 (11)	0.0246 (8)	0.0296 (9)	−0.0059 (7)	0.0065 (8)	0.0010 (7)
C9	0.0349 (9)	0.0297 (8)	0.0239 (8)	−0.0043 (7)	−0.0014 (7)	−0.0066 (7)
C10	0.0303 (9)	0.0308 (8)	0.0222 (8)	−0.0032 (7)	0.0047 (6)	0.0028 (7)
C11	0.0298 (8)	0.0283 (7)	0.0160 (7)	−0.0005 (6)	0.0006 (6)	0.0003 (6)
O1	0.0444 (7)	0.0296 (6)	0.0148 (5)	0.0054 (5)	−0.0070 (5)	−0.0027 (5)
O2	0.0362 (7)	0.0532 (8)	0.0239 (6)	0.0179 (6)	−0.0045 (5)	−0.0106 (6)
C12	0.0373 (10)	0.0288 (8)	0.0181 (8)	0.0048 (7)	0.0004 (6)	−0.0005 (7)
O3	0.1393 (17)	0.0298 (7)	0.0155 (6)	0.0092 (8)	−0.0021 (7)	−0.0003 (6)
O4	0.0484 (8)	0.0268 (6)	0.0185 (6)	−0.0005 (5)	0.0019 (5)	0.0024 (5)
O5	0.0289 (7)	0.0377 (6)	0.0274 (6)	0.0007 (5)	0.0016 (5)	−0.0031 (5)

Geometric parameters (Å, º)

Co—O1^{i}	2.0574 (12)	C5—C10	1.538 (2)
Co—O1	2.0574 (12)	C6—H6A	0.9700
Co—O5	2.0956 (14)	C6—H6B	0.9700
Co—O5^{i}	2.0956 (14)	C7—C8	1.522 (3)
Co—O4^{ii}	2.1061 (12)	C7—H7A	0.9700
Co—O4^{iii}	2.1061 (12)	C7—H7B	0.9700
C1—C12	1.511 (2)	C8—C9	1.532 (2)
C1—C9	1.533 (2)	C8—C10	1.534 (2)
C1—C6	1.535 (2)	C8—H8A	0.9800
C1—C2	1.540 (2)	C9—H9A	0.9700
C2—C3	1.525 (2)	C9—H9B	0.9700

C2—H2A	0.9700	C10—H10A	0.9700
C2—H2B	0.9700	C10—H10B	0.9700
C3—C7	1.516 (3)	C11—O2	1.237 (2)
C3—C4	1.533 (2)	C11—O1	1.2823 (19)
C3—H3A	0.9800	C12—O4	1.212 (2)
C4—C5	1.532 (2)	C12—O3	1.316 (2)
C4—H4A	0.9700	O3—H1	0.8083
C4—H4B	0.9700	O4—Co^{iv}	2.1061 (12)
C5—C11	1.530 (2)	O5—H2	0.8045
C5—C6	1.534 (2)	O5—H3	0.8128

O1^{i}—Co—O1	180.00 (6)	C6—C5—C10	109.03 (13)
O1^{i}—Co—O5	91.39 (5)	C5—C6—C1	110.11 (13)
O1—Co—O5	88.61 (5)	C5—C6—H6A	109.6
O1^{i}—Co—O5^{i}	88.61 (5)	C1—C6—H6A	109.6
O1—Co—O5^{i}	91.39 (5)	C5—C6—H6B	109.6
O5—Co—O5^{i}	180.00 (6)	C1—C6—H6B	109.6
O1^{i}—Co—O4^{ii}	90.51 (5)	H6A—C6—H6B	108.2
O1—Co—O4^{ii}	89.49 (5)	C3—C7—C8	109.63 (14)
O5—Co—O4^{ii}	88.49 (5)	C3—C7—H7A	109.7
O5^{i}—Co—O4^{ii}	91.51 (5)	C8—C7—H7A	109.7
O1^{i}—Co—O4^{iii}	89.49 (5)	C3—C7—H7B	109.7
O1—Co—O4^{iii}	90.51 (5)	C8—C7—H7B	109.7
O5—Co—O4^{iii}	91.51 (5)	H7A—C7—H7B	108.2
O5^{i}—Co—O4^{iii}	88.49 (5)	C7—C8—C9	109.50 (15)
O4^{ii}—Co—O4^{iii}	180.00 (5)	C7—C8—C10	109.98 (15)
C12—C1—C9	112.81 (14)	C9—C8—C10	109.04 (14)
C12—C1—C6	109.83 (13)	C7—C8—H8A	109.4
C9—C1—C6	108.93 (14)	C9—C8—H8A	109.4
C12—C1—C2	106.41 (13)	C10—C8—H8A	109.4
C9—C1—C2	109.37 (14)	C8—C9—C1	109.59 (13)
C6—C1—C2	109.42 (14)	C8—C9—H9A	109.8
C3—C2—C1	109.17 (13)	C1—C9—H9A	109.8
C3—C2—H2A	109.8	C8—C9—H9B	109.8
C1—C2—H2A	109.8	C1—C9—H9B	109.8
C3—C2—H2B	109.8	H9A—C9—H9B	108.2
C1—C2—H2B	109.8	C8—C10—C5	109.71 (13)
H2A—C2—H2B	108.3	C8—C10—H10A	109.7
C7—C3—C2	109.76 (15)	C5—C10—H10A	109.7
C7—C3—C4	109.50 (15)	C8—C10—H10B	109.7
C2—C3—C4	109.95 (15)	C5—C10—H10B	109.7
C7—C3—H3A	109.2	H10A—C10—H10B	108.2
C2—C3—H3A	109.2	O2—C11—O1	122.85 (15)
C4—C3—H3A	109.2	O2—C11—C5	120.66 (14)
C5—C4—C3	109.93 (13)	O1—C11—C5	116.47 (14)
C5—C4—H4A	109.7	C11—O1—Co	125.90 (11)
C3—C4—H4A	109.7	O4—C12—O3	122.98 (16)
C5—C4—H4B	109.7	O4—C12—C1	122.33 (15)

C3—C4—H4B	109.7	O3—C12—C1	114.61 (15)
H4A—C4—H4B	108.2	C12—O3—H1	111.4
C11—C5—C4	111.41 (13)	C12—O4—Co^{iv}	131.58 (11)
C11—C5—C6	109.66 (13)	Co—O5—H2	108.0
C4—C5—C6	109.40 (13)	Co—O5—H3	115.6
C11—C5—C10	108.90 (13)	H2—O5—H3	102.6
C4—C5—C10	108.42 (13)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H1···O1^{iv}	0.81	1.82	2.6058 (19)	166
O5—H2···O2	0.80	2.07	2.7762 (18)	147
O5—H3···O2^{v}	0.81	2.02	2.8334 (18)	175

Symmetry codes: (iv) $x, y, z+1$; (v) $-x+3/2, y, z-1/2$.