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Crystal structure of di- μ -hydroxido- κ^4 O:O-bis- [bis(acetylacetonato- κ^2 O,O')]cobalt(III)]

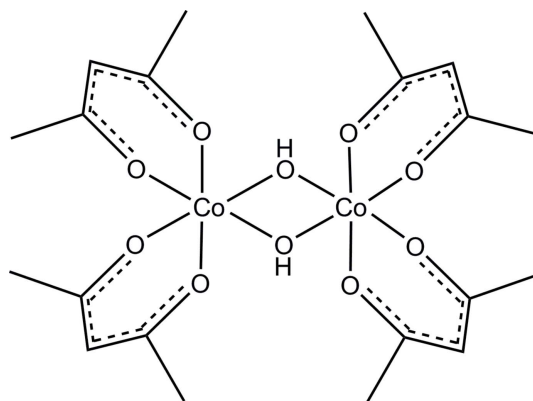
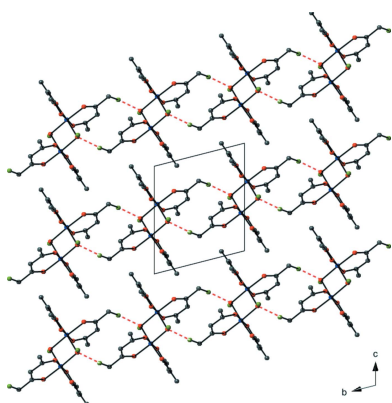
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The dinuclear title complex, $[\text{Co}_2(\text{C}_5\text{H}_7\text{O}_2)_4(\mu\text{-OH})_2]$ or $[\text{Co}(\text{acac})_2(\mu\text{-OH})]_2$, where acac is acetylacetonate, is centrosymmetric with half of the molecule per asymmetric unit. The molecular structure is a dimer of octahedrally coordinated Co^{III} atoms with four O atoms from two chelating acac ligands and two O atoms from bridging hydroxide ligands. The crystal packing features weak $\text{C}\cdots\text{H}\cdots\text{O}$ interactions between neighboring molecules, leading to the formation of chains normal to the *ac* plane. The hydroxide H atoms are not involved in hydrogen bonding because of the bulky acac ligands. This is the first crystal structure reported of a dimeric transition metal bis-acac complex with OH^- as the bridging group.

1. Chemical context

Well-defined cobalt(III) hydroxide complexes are relatively rare, especially in the absence of amine ligands (Bryndza & Tam, 1988). One of the earliest examples is $[\text{Co}(\text{acac})_2(\mu\text{-OH})]_2$ (acac is acetylacetonate, $\text{C}_5\text{H}_7\text{O}_2$), (I), which was prepared by oxidation of $\text{Co}(\text{II})(\text{acac})_2$ with hydrogen peroxide. The complex reacts with 2,4-pentanedione to form $\text{Co}^{\text{III}}(\text{acac})_3$ and may serve as a useful model for hydration and oxidation catalysts (Masłowska & Baranowski, 1978; Bergquist *et al.*, 2003; Zinn *et al.*, 2007; Wang *et al.*, 2009) Boucher and Herrington characterized the complex according to IR and ^1H NMR spectra (Boucher & Herrington, 1971). These data indicated a single diastereoisomer, the identity of which was not clear from the spectra. We now report its crystal structure, confirming that it is centrosymmetric.



2. Structural commentary

The structure of (I) contains one crystallographically independent Co^{III} atom with an approximately octahedral coord-

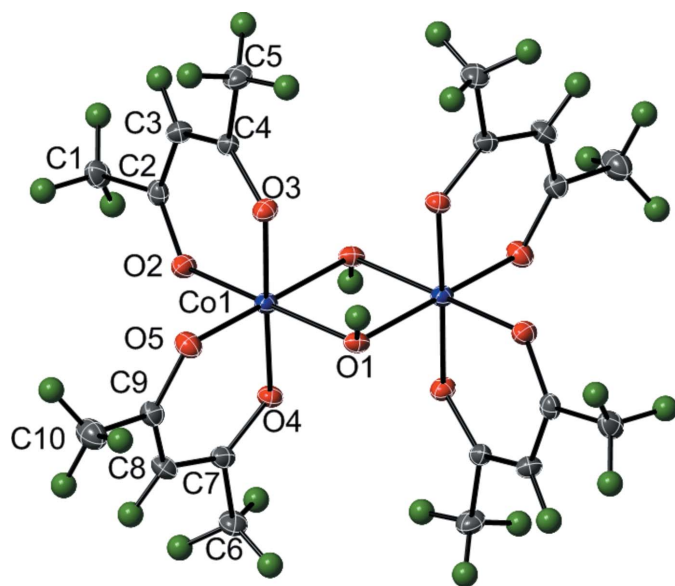


Figure 1
The molecular structure of (I), showing displacement ellipsoids at the 35% probability for non-H atoms and spheres of arbitrary size for H atoms. The unlabeled atoms are related by the symmetry operator $(-x, -y + 2, -z + 1)$.

ination environment. The coordination sphere of Co1 is filled by the oxygen atoms of two κ^2 - O,O' acac ligands [Co1—O2 = 1.8830 (16) Å, Co1—O3 = 1.8770 (16) Å, Co1—O4 = 1.8814 (16) Å, Co1—O5 = 1.8820 (17) Å] and two μ_2 -hydroxyl groups [Co1—O1 = 1.9131 (16) Å, Co1—O1ⁱ = 1.9087 (17) Å; symmetry code: (i) $-x, -y+2, -z+1$]. The angles around Co1 are distorted slightly from the ideal 90° and 180° of a perfect octahedron. The *cis* angles range from 82.07 (7) to 95.92 (7)° while the *trans* angles range from 173.53 (7) to 178.37 (6)°.

The molecular structure of (I) contains a $[\text{Co}_2(\mu_2\text{-OH})_2]$ motif with each metal coordinated by two acac ligands in a κ^2 - O,O' mode (Fig. 1). The two halves of the dimer are related *via* inversion symmetry. The Co1···Co1ⁱ distance is 2.8829 (7) Å. This distance falls within the range (2.696–3.355 Å) of all Co···Co distances reported in the Cambridge Crystallographic Database (Groom & Allen, 2014) for OH[−]-bridged Co complexes in which the metals are coordinated by six oxygen atoms. It is well below the average Co···Co distance of 3.108 Å.

3. Supramolecular features

There are no significant supramolecular features to discuss with the extended structure of (I). There are weak C—H···O intermolecular interactions (Table 1) between one methyl group of an acac ligand and the hydroxide oxygen atom. These interactions result in the formation of chains normal to the *ac* plane (Fig. 2). It should be noted that the hydroxyl H atom does not participate in hydrogen bonding. Examination of the packing diagram shows that the bulky acac ligands prevent any hydrogen-bonding interactions with neighboring molecules.

Table 1
Hydrogen-bond geometry (Å, °).

$D\text{—}H\cdots A$	$D\text{—}H$	$H\cdots A$	$D\cdots A$	$D\text{—}H\cdots A$
C1—H1B···O1 ⁱ	0.98	2.42	3.395 (3)	174

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

4. Database survey

One closely related crystal structure, $[\text{Co}(L)_2(\mu\text{-OH})_2]$; $L = 1$ -(dibenzylamino)-5,5-dimethyl-1,4-dioxohex-2-en-2-olate, has been reported previously (Wang *et al.*, 2009). The ligand in this complex is a modified acac with a *tert*-butyl group in place of one methyl and a $\{\text{CON}(\text{CH}_2\text{Ph})_2\}$ group in place of the other methyl group. The coordination environment of the Co^{III} atoms is the same as in (I). The average Co— O_L distance of 1.890 Å is similar to the average Co— O_{acac} distance in (I) of 1.881 Å. The average Co—OH distance of 1.907 Å is also comparable to that of (I) (1.911 Å).

A search of the Cambridge Crystallographic Database (Groom & Allen, 2014) returned 13 dimeric complexes with the general formula $[\text{TM}(\text{acac})_2(\mu\text{-X})_2]$; TM = transition metal, and $X = \text{O}, \text{OR}, \text{NO},$ or S (Bottomley *et al.*, 1982; Nakahanada *et al.*, 1992; Smith *et al.*, 1972; Sokolov *et al.*, 1999). Complex (I) is the first crystal structure reported that fits this general formula in which the bridging group is OH[−].

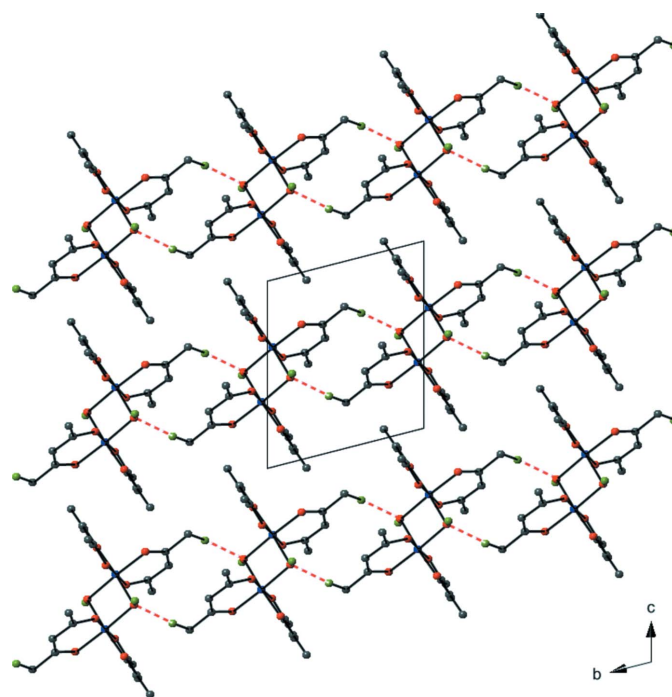


Figure 2
A view along the *a* axis of the crystal structure of (I), showing extended chains normal to the *ac* plane. The weak C—H···O interactions are shown as red dashed lines. All H atoms except the hydroxide H atom (H1) and the interacting H atoms (H1B) have been omitted for clarity. Color code: blue = Co, red = O, gray = C, green = H.

5. Synthesis and crystallization

The title complex was synthesized according to the procedures reported by Boucher & Herrington (1971). To a mixture of $\text{Co}(\text{acac})_2 \cdot 2\text{H}_2\text{O}$ (2 g, 7.27×10^{-3} mol, 1 equiv) and KOAc (3.2 g, 3.26×10^{-2} mol, 4.5 equiv) in methanol (125 ml) was added a solution of H_2O_2 in water (30%_{w/v}, 2 ml). The resulting solution changed color from pink to green. The reaction was stirred at room temperature for 1 h under an ambient atmosphere. The reaction was then concentrated to dryness on a rotary evaporator. The residual green solid was washed with water (3×20 ml) and then acetone (3×20 ml), and then dried in air, leaving the product (0.85 g, 1.55×10^{-3} mol, 43% yield). Crystals, suitable for X-ray diffraction, were grown by slow diffusion of pentane into chloroform solutions of the green product.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The hydroxyl H atom was located in a difference map and its position was allowed to refine freely. Methyl H atom positions, $R\text{-CH}_3$, were optimized by rotation about $R\text{-C}$ bonds with idealized C-H , $R\text{-H}$ and $\text{H} \cdots \text{H}$ distances. Remaining H atoms were included as riding idealized contributors. Methyl and hydroxide H atom U_{iso} 's were assigned as $1.5U_{\text{eq}}$ of the carrier atom; remaining H atom U_{iso} 's were assigned as $1.2U_{\text{eq}}$ of the carrier atom.

Acknowledgements

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Table 2
Experimental details.

Crystal data	
Chemical formula	$[\text{Co}_2(\text{C}_5\text{H}_7\text{O}_2)_4(\text{OH})_2]$
M_r	548.30
Crystal system, space group	Triclinic, $P\bar{1}$
Temperature (K)	173
a, b, c (Å)	7.8610 (11), 8.2481 (11), 9.8372 (13)
α, β, γ (°)	100.786 (8), 106.708 (8), 99.492 (9)
V (Å ³)	583.67 (14)
Z	1
Radiation type	Mo $K\alpha$
μ (mm ⁻¹)	1.47
Crystal size (mm)	0.23 × 0.19 × 0.04
Data collection	
Diffractometer	Bruker Kappa APEXII CCD
Absorption correction	Integration (<i>SADABS</i> ; Bruker, 2012)
$T_{\text{min}}, T_{\text{max}}$	0.776, 0.945
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	16164, 2617, 2228
R_{int}	0.083
$(\sin \theta/\lambda)_{\text{max}}$ (Å ⁻¹)	0.646
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.036, 0.093, 1.05
No. of reflections	2617
No. of parameters	152
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å ⁻³)	0.39, -0.53

Computer programs: *APEX2* and *SAINT* (Bruker, 2013), *SHELXS* and *SHELXTL* (Sheldrick, 2008), *SHELXL2013/4* (Sheldrick, 2015), *CrystalMaker* (CrystalMaker, 1994), *XCIF* (Bruker, 2013) and *pubCIF* (Westrip, 2010).

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supporting information

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Crystal structure of di- μ -hydroxido- κ^4 O:O-bis[bis(acetylacetonato- κ^2 O,O')cobalt(III)]

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Computing details

Data collection: *APEX2* (Bruker, 2013); cell refinement: *SAINTE* (Bruker, 2013); data reduction: *SAINTE* (Bruker, 2013); program(s) used to solve structure: *SHELXS* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2013/4* (Sheldrick, 2015); molecular graphics: *SHELXTL* (Sheldrick, 2008), *CrystalMaker* (*CrystalMaker*, 1994); software used to prepare material for publication: *XCIF* (Bruker, 2013) and *pubCIF* (Westrip, 2010).

Di- μ -hydroxido- κ^4 O:O-bis[bis(acetylacetonato- κ^2 O,O')cobalt(III)]

Crystal data

$[\text{Co}_2(\text{C}_5\text{H}_7\text{O}_2)_4(\text{OH})_2]$

$M_r = 548.30$

Triclinic, $P\bar{1}$

$a = 7.8610$ (11) Å

$b = 8.2481$ (11) Å

$c = 9.8372$ (13) Å

$\alpha = 100.786$ (8)°

$\beta = 106.708$ (8)°

$\gamma = 99.492$ (9)°

$V = 583.67$ (14) Å³

$Z = 1$

$F(000) = 284$

$D_x = 1.560$ Mg m⁻³

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

Cell parameters from 3791 reflections

$\theta = 2.6\text{--}25.5^\circ$

$\mu = 1.47$ mm⁻¹

$T = 173$ K

Plate, blue

$0.23 \times 0.19 \times 0.04$ mm

Data collection

Bruker Kappa APEXII CCD
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

profile data from φ and ω scans

Absorption correction: integration

(*SADABS*; Bruker, 2012)

$T_{\min} = 0.776$, $T_{\max} = 0.945$

16164 measured reflections

2617 independent reflections

2228 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.083$

$\theta_{\max} = 27.3^\circ$, $\theta_{\min} = 2.2^\circ$

$h = -10 \rightarrow 10$

$k = -10 \rightarrow 10$

$l = -12 \rightarrow 12$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.036$

$wR(F^2) = 0.093$

$S = 1.05$

2617 reflections

152 parameters

0 restraints

Hydrogen site location: mixed

H atoms treated by a mixture of independent
and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0366P)^2 + 0.1984P]$

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

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

$\Delta\rho_{\max} = 0.39$ e Å⁻³

$\Delta\rho_{\min} = -0.53$ e Å⁻³

Special details

Experimental. One distinct cell was identified using *APEX2* (Bruker, 2013). Fourteen frame series were integrated and filtered for statistical outliers using *SAINT* (Bruker, 2013) then corrected for absorption by integration using *SAINT/SADABS*, v2012/1 (Bruker, 2012) to sort, merge, and scale the combined data. No decay correction was applied.

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. Structure was phased by direct methods (Sheldrick, 2008). Systematic conditions suggested the ambiguous space group. The space group choice was confirmed by successful convergence of the full-matrix least-squares refinement on F^2 . The final map had no significant features. A final analysis of variance between observed and calculated structure factors showed little dependence on amplitude and resolution.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Co1	0.08356 (4)	0.96491 (4)	0.63894 (3)	0.01839 (12)
O1	0.0955 (2)	1.1492 (2)	0.54669 (18)	0.0197 (4)
H1	0.182 (4)	1.158 (4)	0.530 (3)	0.030*
O2	0.0442 (2)	0.7772 (2)	0.71880 (18)	0.0244 (4)
O3	0.2682 (2)	0.9024 (2)	0.56955 (18)	0.0220 (4)
C1	0.0467 (4)	0.5042 (3)	0.7559 (3)	0.0294 (6)
H1A	0.1238	0.5263	0.8584	0.044*
H1B	0.0576	0.3969	0.7002	0.044*
H1C	-0.0808	0.4967	0.7508	0.044*
C2	0.1074 (3)	0.6460 (3)	0.6920 (2)	0.0216 (5)
C3	0.2293 (3)	0.6301 (3)	0.6154 (3)	0.0239 (5)
H3	0.2605	0.5236	0.5962	0.029*
C4	0.3092 (3)	0.7594 (3)	0.5647 (2)	0.0204 (5)
C5	0.4588 (3)	0.7360 (3)	0.5010 (3)	0.0291 (6)
H5A	0.4703	0.8171	0.4411	0.044*
H5B	0.4290	0.6201	0.4398	0.044*
H5C	0.5745	0.7556	0.5805	0.044*
O4	-0.1041 (2)	1.0298 (2)	0.70365 (18)	0.0211 (3)
O5	0.2764 (2)	1.0871 (2)	0.81009 (18)	0.0258 (4)
C6	-0.2501 (3)	1.1417 (4)	0.8641 (3)	0.0302 (6)
H6A	-0.2682	1.2526	0.8490	0.045*
H6B	-0.2371	1.1397	0.9658	0.045*
H6C	-0.3556	1.0527	0.7980	0.045*
C7	-0.0812 (3)	1.1114 (3)	0.8328 (3)	0.0216 (5)
C8	0.0873 (3)	1.1769 (3)	0.9434 (3)	0.0265 (5)
H8	0.0877	1.2323	1.0373	0.032*
C9	0.2534 (3)	1.1666 (3)	0.9258 (3)	0.0245 (5)
C10	0.4268 (4)	1.2530 (4)	1.0500 (3)	0.0383 (7)
H10A	0.4992	1.1698	1.0733	0.057*
H10B	0.3972	1.3033	1.1363	0.057*
H10C	0.4973	1.3424	1.0215	0.057*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Co1	0.01960 (18)	0.01881 (19)	0.02005 (19)	0.00636 (13)	0.01007 (13)	0.00540 (13)
O1	0.0196 (8)	0.0184 (8)	0.0237 (9)	0.0052 (7)	0.0118 (7)	0.0033 (7)
O2	0.0309 (9)	0.0253 (9)	0.0253 (9)	0.0113 (7)	0.0164 (7)	0.0102 (7)
O3	0.0223 (8)	0.0223 (9)	0.0267 (9)	0.0086 (7)	0.0130 (7)	0.0083 (7)
C1	0.0407 (15)	0.0259 (13)	0.0245 (13)	0.0060 (11)	0.0144 (11)	0.0090 (10)
C2	0.0227 (12)	0.0217 (12)	0.0162 (11)	0.0041 (9)	0.0015 (9)	0.0035 (9)
C3	0.0276 (12)	0.0198 (12)	0.0253 (13)	0.0091 (10)	0.0091 (10)	0.0041 (10)
C4	0.0199 (11)	0.0214 (12)	0.0166 (11)	0.0057 (9)	0.0023 (9)	0.0018 (9)
C5	0.0292 (13)	0.0297 (14)	0.0366 (15)	0.0149 (11)	0.0175 (11)	0.0099 (11)
O4	0.0210 (8)	0.0233 (9)	0.0218 (8)	0.0066 (7)	0.0114 (7)	0.0042 (7)
O5	0.0216 (9)	0.0323 (10)	0.0231 (9)	0.0069 (7)	0.0088 (7)	0.0033 (7)
C6	0.0301 (13)	0.0399 (15)	0.0278 (14)	0.0147 (12)	0.0167 (11)	0.0085 (12)
C7	0.0292 (12)	0.0188 (12)	0.0231 (12)	0.0075 (10)	0.0155 (10)	0.0080 (9)
C8	0.0317 (13)	0.0303 (14)	0.0196 (12)	0.0094 (11)	0.0120 (10)	0.0034 (10)
C9	0.0282 (13)	0.0260 (13)	0.0214 (12)	0.0074 (10)	0.0098 (10)	0.0074 (10)
C10	0.0313 (14)	0.0479 (18)	0.0277 (15)	0.0049 (13)	0.0074 (12)	-0.0021 (13)

Geometric parameters (\AA , $^\circ$)

Co1—O3	1.8770 (16)	C4—C5	1.506 (3)
Co1—O4	1.8814 (16)	C5—H5A	0.9800
Co1—O5	1.8820 (17)	C5—H5B	0.9800
Co1—O2	1.8830 (16)	C5—H5C	0.9800
Co1—O1 ⁱ	1.9087 (17)	O4—C7	1.268 (3)
Co1—O1	1.9131 (16)	O5—C9	1.279 (3)
Co1—Co1 ⁱ	2.8829 (7)	C6—C7	1.495 (3)
O1—Co1 ⁱ	1.9087 (17)	C6—H6A	0.9800
O1—H1	0.74 (3)	C6—H6B	0.9800
O2—C2	1.278 (3)	C6—H6C	0.9800
O3—C4	1.269 (3)	C7—C8	1.395 (3)
C1—C2	1.502 (3)	C8—C9	1.380 (3)
C1—H1A	0.9800	C8—H8	0.9500
C1—H1B	0.9800	C9—C10	1.502 (3)
C1—H1C	0.9800	C10—H10A	0.9800
C2—C3	1.387 (3)	C10—H10B	0.9800
C3—C4	1.393 (3)	C10—H10C	0.9800
C3—H3	0.9500		
O3—Co1—O4	178.37 (6)	C2—C3—C4	124.5 (2)
O3—Co1—O5	85.06 (7)	C2—C3—H3	117.8
O4—Co1—O5	95.92 (7)	C4—C3—H3	117.8
O3—Co1—O2	95.73 (7)	O3—C4—C3	125.0 (2)
O4—Co1—O2	85.55 (7)	O3—C4—C5	115.1 (2)
O5—Co1—O2	91.96 (7)	C3—C4—C5	120.0 (2)
O3—Co1—O1 ⁱ	90.26 (7)	C4—C5—H5A	109.5

O4—Co1—O1 ⁱ	88.67 (7)	C4—C5—H5B	109.5
O5—Co1—O1 ⁱ	173.53 (7)	H5A—C5—H5B	109.5
O2—Co1—O1 ⁱ	92.95 (7)	C4—C5—H5C	109.5
O3—Co1—O1	88.10 (7)	H5A—C5—H5C	109.5
O4—Co1—O1	90.54 (7)	H5B—C5—H5C	109.5
O5—Co1—O1	93.29 (7)	C7—O4—Co1	124.45 (16)
O2—Co1—O1	173.75 (7)	C9—O5—Co1	123.82 (16)
O1 ⁱ —Co1—O1	82.07 (7)	C7—C6—H6A	109.5
O3—Co1—Co1 ⁱ	88.91 (5)	C7—C6—H6B	109.5
O4—Co1—Co1 ⁱ	89.47 (5)	H6A—C6—H6B	109.5
O5—Co1—Co1 ⁱ	134.10 (5)	C7—C6—H6C	109.5
O2—Co1—Co1 ⁱ	133.93 (6)	H6A—C6—H6C	109.5
O1 ⁱ —Co1—Co1 ⁱ	41.09 (5)	H6B—C6—H6C	109.5
O1—Co1—Co1 ⁱ	40.98 (5)	O4—C7—C8	125.1 (2)
Co1 ⁱ —O1—Co1	97.93 (7)	O4—C7—C6	116.0 (2)
Co1 ⁱ —O1—H1	103 (2)	C8—C7—C6	118.9 (2)
Co1—O1—H1	106 (2)	C9—C8—C7	124.4 (2)
C2—O2—Co1	123.65 (15)	C9—C8—H8	117.8
C4—O3—Co1	124.41 (15)	C7—C8—H8	117.8
C2—C1—H1A	109.5	O5—C9—C8	125.7 (2)
C2—C1—H1B	109.5	O5—C9—C10	114.7 (2)
H1A—C1—H1B	109.5	C8—C9—C10	119.6 (2)
C2—C1—H1C	109.5	C9—C10—H10A	109.5
H1A—C1—H1C	109.5	C9—C10—H10B	109.5
H1B—C1—H1C	109.5	H10A—C10—H10B	109.5
O2—C2—C3	125.1 (2)	C9—C10—H10C	109.5
O2—C2—C1	115.2 (2)	H10A—C10—H10C	109.5
C3—C2—C1	119.6 (2)	H10B—C10—H10C	109.5

Symmetry code: (i) $-x, -y+2, -z+1$.

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>
C1—H1B \cdots O1 ⁱⁱ	0.98	2.42	3.395 (3)	174

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