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Diaquabis(5-carboxy-2-methyl-1*H*-imidazole-4-carboxylato- κ^2 N³,O⁴)-cobalt(II) dimethylformamide disolvate

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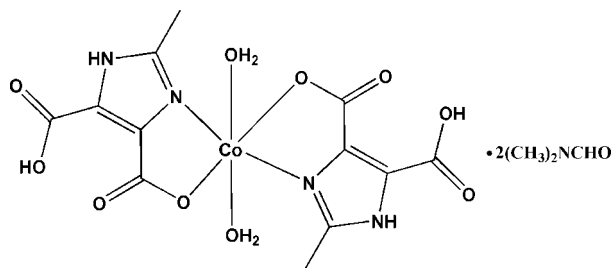
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Key indicators: single-crystal X-ray study; $T = 123$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.039; wR factor = 0.106; data-to-parameter ratio = 13.6.

In the title compound, $[\text{Co}(\text{C}_6\text{H}_5\text{N}_2\text{O}_4)_2(\text{H}_2\text{O})_2] \cdot 2\text{C}_3\text{H}_7\text{NO}$, the Co^{II} ion lies on an inversion center and adopts a slightly distorted CoN_2O_4 octahedral geometry binding two bidentate chelating 5-carboxy-2-methyl-1*H*-imidazole-4-carboxylate (H_2MIDA^-) monoanionic ligands and two axial aqua ligands. In the crystal structure, intermolecular $\text{O}-\text{H} \cdots \text{O}$ hydrogen bonds link neighboring molecules, generating a two-dimensional framework containing eight-membered H_4O_4 rings. In addition, the dimethylformamide solvent molecules are hydrogen bonded to the two-dimensional framework *via* the NH groups of the H_2MIDA^- ligands.

Related literature

For background to *N*-heterocyclic carboxylic acids as ligands in coordination complexes, see: Gao *et al.* (2004); Shimizu *et al.* (2004); Zhang *et al.* (2006). For related structures, see: Liu *et al.* (2007); Nie *et al.* (2007); Zeng *et al.* (2008).



Experimental

Crystal data

 $[\text{Co}(\text{C}_6\text{H}_5\text{N}_2\text{O}_4)_2(\text{H}_2\text{O})_2] \cdot 2\text{C}_3\text{H}_7\text{NO}$
 $M_r = 579.39$

 Triclinic, $P\bar{1}$
 $a = 7.1979$ (11) Å

 $b = 9.2180$ (15) Å
 $c = 10.8659$ (17) Å
 $\alpha = 65.173$ (2)°
 $\beta = 83.459$ (2)°
 $\gamma = 68.254$ (2)°
 $V = 607.02$ (17) Å³
 $Z = 1$
 Mo $K\alpha$ radiation
 $\mu = 0.78$ mm⁻¹
 $T = 123$ K
 $0.20 \times 0.15 \times 0.14$ mm

Data collection

 Bruker SMART APEX area-detector diffractometer
 Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
 $T_{\text{min}} = 0.859$, $T_{\text{max}} = 0.899$

 4588 measured reflections
 2335 independent reflections
 2176 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.026$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.106$
 $S = 1.06$
 2335 reflections
 172 parameters
 1 restraint

 H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.65$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.49$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> — <i>H</i> ··· <i>A</i>	<i>D</i> — <i>H</i>	<i>H</i> ··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> — <i>H</i> ··· <i>A</i>
N2—H2···O5 ⁱ	0.86	1.83	2.670 (2)	166
O2—H2A···O3	0.863 (10)	1.593 (11)	2.452 (2)	173 (3)
O1W—H1W2···O1 ⁱⁱ	0.85	1.91	2.7579 (19)	178
O1W—H1W1···O1 ⁱⁱⁱ	0.85	2.03	2.847 (2)	160

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

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; 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: *SHELXTL*.

We thank Hengyang Normal University for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2611).

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supporting information

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Diaquabis(5-carboxy-2-methyl-1*H*-imidazole-4-carboxylato- κ^2N^3,O^4)cobalt(II) dimethylformamide disolvate

Si-Ping Tang

S1. Comment

N-heterocyclic carboxylic acids have attracted considerable interests as ligands in metal complexes because of their structural diversity as multidentate chelating or bridging ligands (Gao *et al.*, 2004; Shimizu *et al.*, 2004; Zhang *et al.*, 2006). Recently, 2-methyl-1*H*-imidazole-4,5- dicarboxylic acid (H₃MIDA) has been used as a chelating ligand to generate mononuclear complexes with cadmium (II), cobalt (II) and manganese (II) ions (Liu *et al.*, 2007; Nie *et al.*, 2007; Zeng *et al.*, 2008). We report here the synthesis and structure of a new cobalt (II) complex incorporating H₃MIDA.

The title compound is composed of one Co (II) ion, two mono-deprotonated H₂MIDA ligands, two aqua ligands and two DMF solvent molecules, Fig 1. The Co (II) cation lies on a crystallographic inversion center and has a distorted octahedral geometry with the basal plane occupied by two carboxylate O atoms and two N atoms from two chelating H₂MIDA⁻ ligands. There are two axial aqua ligands. In the H₂MIDA ligand, the carboxyl and carboxylate groups form an intramolecular hydrogen bond with an O...O distance of 2.452 (2) Å.

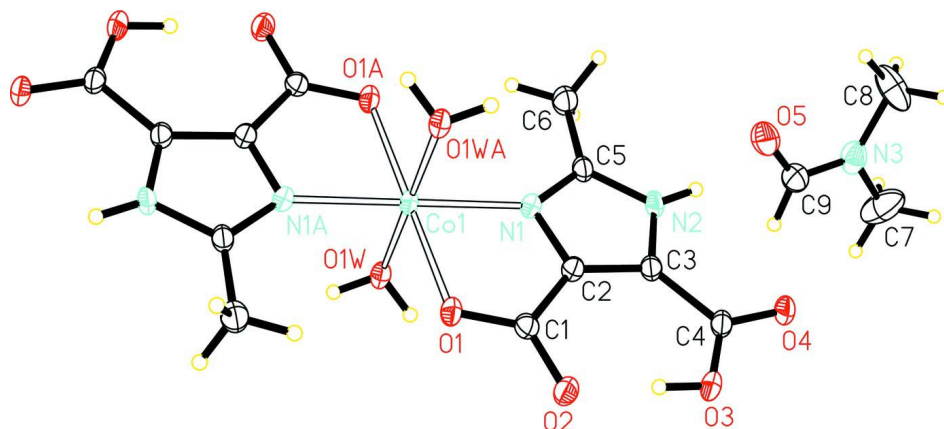
In the crystal packing, each aqua ligand is involved in two intermolecular O—H...O hydrogen bonds with two carboxyl O atoms from two neighboring molecules to generate a two-dimensional supramolecular structure (Fig. 2), in which two aqua ligands and two carboxyl O atoms form a H₄O₄ eight-membered ring. In addition, the DMF solvates are hydrogen-bonded to the two-dimensional framework *via* the —NH groups of the H₂MIDA⁻ ligands.

S2. Experimental

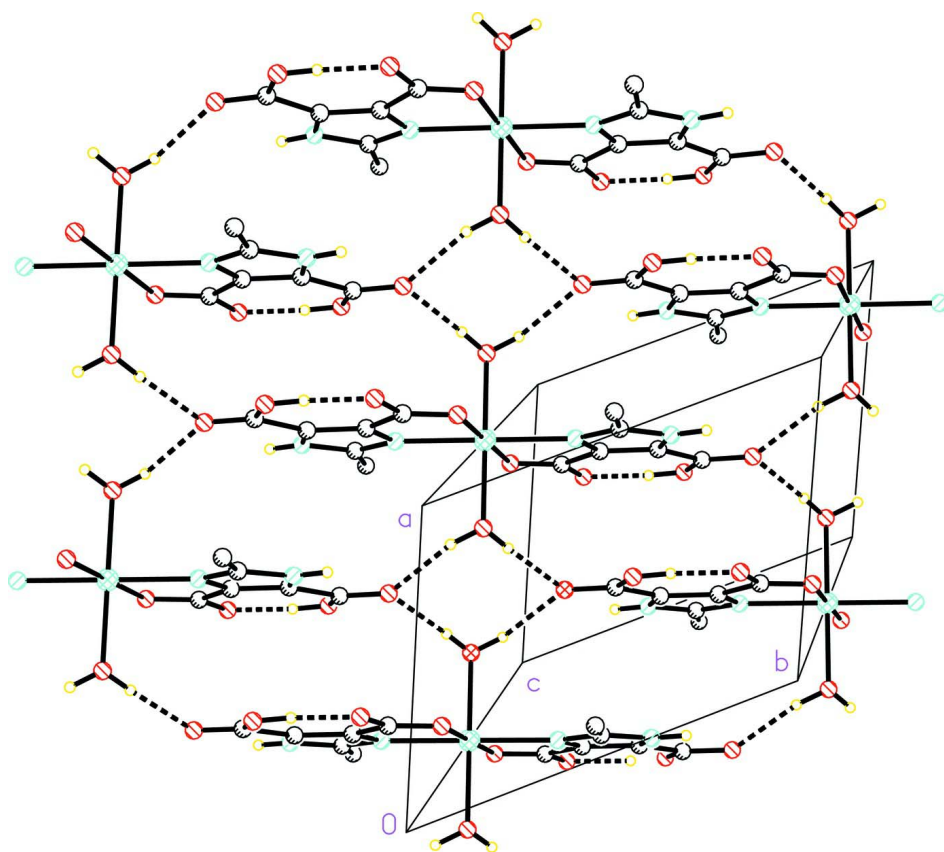
A solution of cobalt perchlorate hexahydrate (73.2 mg, 0.2 mmol) and H₃MIDA (15.8 mg, 0.1 mmol) in DMF (6 ml) and methanol (1 ml) was stirred for 5 h. After filtering, the filtrate was left for about two months and pink, block-like crystals of the title compound appeared. Yield: 11 mg (38%).

S3. Refinement

The carboxyl and water H atoms were located in a difference Fourier map and refined with $U_{\text{iso}}=1.5U_{\text{eq}}$ (O). The O—H distances of water were refined with idealized values of 0.85 Å, however, that of carboxyl is refined freely. All other H-atoms were positioned geometrically and refined using a riding model with C—H (methyl) = 0.96 Å, C—H (aldehyde) = 0.93 Å, N—H = 0.86 Å, and $U_{\text{iso}} = 1.2U_{\text{eq}}$ (C, N).

**Figure 1**

The title molecule with displacement ellipsoids drawn at the 50% probability level, and with the H atoms shown as spheres of arbitrary radii.

**Figure 2**

Packing diagram of the title compound showing the intra/intermolecular O—H...O hydrogen bonds as dashed lines. The H atoms not involved in hydrogen bonds and the DMF solvate molecules have been omitted for clarity.

Diaquabis(5-carboxy-2-methyl-1*H*-imidazole-4-carboxylato- κ^2N^3,O^4)cobalt(II) dimethylformamide disolvate

Crystal data

[Co(C₆H₅N₂O₄)₂(H₂O)₂] \cdot 2C₃H₇NO $M_r = 579.39$ Triclinic, $P\bar{1}$

Hall symbol: -P 1

 $a = 7.1979$ (11) Å $b = 9.2180$ (15) Å $c = 10.8659$ (17) Å $\alpha = 65.173$ (2)° $\beta = 83.459$ (2)° $\gamma = 68.254$ (2)° $V = 607.02$ (17) Å³ $Z = 1$ $F(000) = 301$ $D_x = 1.585$ Mg m⁻³Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 3423 reflections

 $\theta = 2.6$ – 27.8 ° $\mu = 0.78$ mm⁻¹ $T = 123$ K

Block, pink

 $0.20 \times 0.15 \times 0.14$ mm

Data collection

Bruker SMART APEX area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

 φ and ω scans

Absorption correction: multi-scan

(SADABS; Sheldrick, 1996)

 $T_{\min} = 0.859$, $T_{\max} = 0.899$

4588 measured reflections

2335 independent reflections

2176 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.026$ $\theta_{\text{max}} = 26.0$ °, $\theta_{\text{min}} = 2.6$ ° $h = -8 \rightarrow 7$ $k = -11 \rightarrow 11$ $l = -13 \rightarrow 13$

Refinement

Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.039$ $wR(F^2) = 0.106$ $S = 1.06$

2335 reflections

172 parameters

1 restraint

Primary atom site location: structure-invariant

direct methods

Secondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sitesH atoms treated by a mixture of independent
and constrained refinement $w = 1/[\sigma^2(F_o^2) + (0.0631P)^2 + 0.2477P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\text{max}} < 0.001$ $\Delta\rho_{\text{max}} = 0.65$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.49$ 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 (Å²)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Co1	0.0000	1.0000	0.5000	0.01688 (15)
O4	0.1583 (2)	0.97632 (18)	0.32480 (14)	0.0200 (3)
O1W	0.2666 (2)	0.96647 (18)	0.58550 (14)	0.0214 (3)

H1W2	0.3716	0.9017	0.5643	0.032*
H1W1	0.2857	1.0585	0.5685	0.032*
O3	0.3261 (2)	0.78687 (18)	0.23787 (14)	0.0220 (3)
O2	0.4205 (2)	0.47831 (19)	0.30617 (15)	0.0233 (3)
H2A	0.391 (4)	0.5850 (15)	0.288 (3)	0.035*
O1	0.3990 (2)	0.24920 (18)	0.48355 (16)	0.0241 (3)
O5	0.8297 (3)	-0.1528 (2)	0.20333 (17)	0.0343 (4)
N1	0.0922 (2)	0.7355 (2)	0.55599 (16)	0.0171 (4)
N2	0.1798 (2)	0.4572 (2)	0.62169 (17)	0.0181 (4)
H2	0.1932	0.3523	0.6713	0.022*
N3	0.6518 (3)	0.1293 (2)	0.08740 (18)	0.0296 (4)
C6	0.2260 (3)	0.8282 (3)	0.33158 (19)	0.0179 (4)
C5	0.1936 (3)	0.6909 (2)	0.4550 (2)	0.0174 (4)
C4	0.2493 (3)	0.5176 (2)	0.4945 (2)	0.0173 (4)
C3	0.3636 (3)	0.4049 (3)	0.4250 (2)	0.0194 (4)
C2	0.0862 (3)	0.5915 (2)	0.6556 (2)	0.0181 (4)
C1	-0.0105 (3)	0.5776 (3)	0.7860 (2)	0.0247 (5)
H1A	0.0882	0.5048	0.8597	0.037*
H1B	-0.1121	0.5297	0.7961	0.037*
H1C	-0.0703	0.6892	0.7866	0.037*
C7	0.4556 (5)	0.2630 (4)	0.0431 (3)	0.0559 (8)
H7A	0.3539	0.2136	0.0754	0.084*
H7B	0.4450	0.3176	-0.0543	0.084*
H7C	0.4384	0.3461	0.0788	0.084*
C8	0.8227 (5)	0.1820 (4)	0.0586 (3)	0.0480 (7)
H8A	0.8213	0.2500	-0.0369	0.072*
H8B	0.9434	0.0827	0.0854	0.072*
H8C	0.8175	0.2484	0.1081	0.072*
C9	0.6694 (4)	-0.0318 (3)	0.1589 (2)	0.0309 (5)
H9	0.5524	-0.0563	0.1773	0.037*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Co1	0.0189 (2)	0.0105 (2)	0.0213 (2)	-0.00437 (16)	0.00295 (15)	-0.00778 (16)
O4	0.0233 (8)	0.0124 (7)	0.0237 (7)	-0.0055 (6)	0.0032 (5)	-0.0082 (6)
O1W	0.0208 (7)	0.0153 (7)	0.0301 (8)	-0.0054 (6)	0.0030 (6)	-0.0126 (6)
O3	0.0273 (8)	0.0180 (7)	0.0221 (7)	-0.0078 (6)	0.0063 (6)	-0.0110 (6)
O2	0.0279 (8)	0.0168 (7)	0.0297 (8)	-0.0076 (6)	0.0065 (6)	-0.0151 (7)
O1	0.0227 (8)	0.0146 (8)	0.0375 (8)	-0.0059 (6)	0.0044 (6)	-0.0144 (6)
O5	0.0393 (10)	0.0179 (8)	0.0395 (9)	-0.0122 (7)	0.0049 (7)	-0.0051 (7)
N1	0.0172 (9)	0.0136 (8)	0.0209 (8)	-0.0043 (7)	0.0028 (6)	-0.0087 (7)
N2	0.0200 (8)	0.0098 (8)	0.0240 (8)	-0.0056 (7)	0.0010 (6)	-0.0063 (7)
N3	0.0419 (11)	0.0206 (9)	0.0236 (9)	-0.0106 (8)	0.0034 (8)	-0.0076 (7)
C6	0.0152 (9)	0.0167 (10)	0.0228 (10)	-0.0060 (8)	0.0005 (7)	-0.0086 (8)
C5	0.0161 (9)	0.0155 (10)	0.0218 (9)	-0.0049 (8)	0.0017 (7)	-0.0094 (8)
C4	0.0163 (9)	0.0147 (10)	0.0227 (9)	-0.0057 (8)	0.0003 (7)	-0.0091 (8)
C3	0.0152 (9)	0.0160 (10)	0.0303 (10)	-0.0045 (8)	-0.0002 (8)	-0.0130 (9)

C2	0.0172 (10)	0.0128 (10)	0.0243 (10)	-0.0054 (8)	-0.0001 (7)	-0.0074 (8)
C1	0.0299 (11)	0.0205 (11)	0.0245 (10)	-0.0105 (9)	0.0045 (8)	-0.0096 (9)
C7	0.0534 (15)	0.0351 (15)	0.0471 (15)	0.0034 (13)	0.0177 (13)	-0.0078 (12)
C8	0.0650 (16)	0.0353 (14)	0.0426 (14)	-0.0331 (13)	-0.0161 (13)	0.0028 (12)
C9	0.0366 (13)	0.0268 (12)	0.0336 (12)	-0.0164 (11)	0.0115 (10)	-0.0143 (10)

Geometric parameters (Å, °)

Co1—O1W ⁱ	2.0895 (14)	N2—H2	0.8600
Co1—O1W	2.0895 (14)	N3—C9	1.319 (3)
Co1—N1 ⁱ	2.0982 (16)	N3—C8	1.440 (3)
Co1—N1	2.0982 (16)	N3—C7	1.453 (3)
Co1—O4	2.1543 (14)	C6—C5	1.476 (3)
Co1—O4 ⁱ	2.1543 (14)	C5—C4	1.374 (3)
O4—C6	1.240 (2)	C4—C3	1.486 (3)
O1W—H1W2	0.8500	C2—C1	1.482 (3)
O1W—H1W1	0.8500	C1—H1A	0.9600
O3—C6	1.287 (2)	C1—H1B	0.9600
O2—C3	1.282 (3)	C1—H1C	0.9600
O2—H2A	0.863 (10)	C7—H7A	0.9600
O1—C3	1.237 (2)	C7—H7B	0.9600
O5—C9	1.237 (3)	C7—H7C	0.9600
N1—C2	1.327 (3)	C8—H8A	0.9600
N1—C5	1.371 (2)	C8—H8B	0.9600
N2—C2	1.353 (3)	C8—H8C	0.9600
N2—C4	1.368 (3)	C9—H9	0.9300
O1W ⁱ —Co1—O1W	180.00 (8)	N1—C5—C6	117.57 (17)
O1W ⁱ —Co1—N1 ⁱ	90.58 (6)	C4—C5—C6	132.78 (18)
O1W—Co1—N1 ⁱ	89.42 (6)	N2—C4—C5	105.63 (17)
O1W ⁱ —Co1—N1	89.42 (6)	N2—C4—C3	123.03 (18)
O1W—Co1—N1	90.58 (6)	C5—C4—C3	131.33 (19)
N1 ⁱ —Co1—N1	180.0	O1—C3—O2	124.18 (18)
O1W ⁱ —Co1—O4	90.84 (5)	O1—C3—C4	119.29 (19)
O1W—Co1—O4	89.16 (5)	O2—C3—C4	116.53 (17)
N1 ⁱ —Co1—O4	101.17 (6)	N1—C2—N2	110.61 (17)
N1—Co1—O4	78.83 (6)	N1—C2—C1	125.33 (18)
O1W ⁱ —Co1—O4 ⁱ	89.16 (5)	N2—C2—C1	124.06 (18)
O1W—Co1—O4 ⁱ	90.84 (5)	C2—C1—H1A	109.5
N1 ⁱ —Co1—O4 ⁱ	78.83 (6)	C2—C1—H1B	109.5
N1—Co1—O4 ⁱ	101.17 (6)	H1A—C1—H1B	109.5
O4—Co1—O4 ⁱ	180.0	C2—C1—H1C	109.5
C6—O4—Co1	113.98 (12)	H1A—C1—H1C	109.5
Co1—O1W—H1W2	114.5	H1B—C1—H1C	109.5
Co1—O1W—H1W1	114.9	N3—C7—H7A	109.5
H1W2—O1W—H1W1	106.7	N3—C7—H7B	109.5
C3—O2—H2A	108.9 (18)	H7A—C7—H7B	109.5
C2—N1—C5	106.13 (15)	N3—C7—H7C	109.5

C2—N1—Co1	142.57 (14)	H7A—C7—H7C	109.5
C5—N1—Co1	111.29 (12)	H7B—C7—H7C	109.5
C2—N2—C4	107.99 (16)	N3—C8—H8A	109.5
C2—N2—H2	126.0	N3—C8—H8B	109.5
C4—N2—H2	126.0	H8A—C8—H8B	109.5
C9—N3—C8	121.9 (2)	N3—C8—H8C	109.5
C9—N3—C7	120.7 (2)	H8A—C8—H8C	109.5
C8—N3—C7	117.2 (2)	H8B—C8—H8C	109.5
O4—C6—O3	123.66 (18)	O5—C9—N3	124.9 (2)
O4—C6—C5	118.29 (17)	O5—C9—H9	117.5
O3—C6—C5	118.04 (17)	N3—C9—H9	117.5
N1—C5—C4	109.64 (17)		
O1W ⁱ —Co1—O4—C6	-87.79 (14)	O4—C6—C5—C4	-179.21 (19)
O1W—Co1—O4—C6	92.21 (14)	O3—C6—C5—C4	-0.4 (3)
N1 ⁱ —Co1—O4—C6	-178.54 (13)	C2—N2—C4—C5	0.0 (2)
N1—Co1—O4—C6	1.46 (13)	C2—N2—C4—C3	178.91 (17)
O1W ⁱ —Co1—N1—C2	-89.6 (2)	N1—C5—C4—N2	0.0 (2)
O1W—Co1—N1—C2	90.4 (2)	C6—C5—C4—N2	178.5 (2)
O4—Co1—N1—C2	179.4 (2)	N1—C5—C4—C3	-178.80 (19)
O4 ⁱ —Co1—N1—C2	-0.6 (2)	C6—C5—C4—C3	-0.3 (4)
O1W ⁱ —Co1—N1—C5	89.19 (13)	N2—C4—C3—O1	-0.1 (3)
O1W—Co1—N1—C5	-90.81 (13)	C5—C4—C3—O1	178.5 (2)
O4—Co1—N1—C5	-1.78 (12)	N2—C4—C3—O2	-179.72 (17)
O4 ⁱ —Co1—N1—C5	178.22 (12)	C5—C4—C3—O2	-1.1 (3)
Co1—O4—C6—O3	-179.57 (15)	C5—N1—C2—N2	0.0 (2)
Co1—O4—C6—C5	-0.8 (2)	Co1—N1—C2—N2	178.85 (15)
C2—N1—C5—C4	0.0 (2)	C5—N1—C2—C1	-179.54 (18)
Co1—N1—C5—C4	-179.25 (13)	Co1—N1—C2—C1	-0.7 (4)
C2—N1—C5—C6	-178.74 (17)	C4—N2—C2—N1	0.0 (2)
Co1—N1—C5—C6	2.0 (2)	C4—N2—C2—C1	179.55 (18)
O4—C6—C5—N1	-0.8 (3)	C8—N3—C9—O5	-2.9 (4)
O3—C6—C5—N1	178.00 (16)	C7—N3—C9—O5	-178.2 (2)

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

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N2—H2 \cdots O5 ⁱⁱ	0.86	1.83	2.670 (2)	166
O2—H2A \cdots O3	0.86 (1)	1.59 (1)	2.452 (2)	173 (3)
O1W—H1W2 \cdots O1 ⁱⁱⁱ	0.85	1.91	2.7579 (19)	178
O1W—H1W1 \cdots O1 ^{iv}	0.85	2.03	2.847 (2)	160

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