

# Diaqua(isonicotinamide- $\kappa N^1$ )-(4-methoxybenzoato- $\kappa^2 O, O'$ )-(4-methoxybenzoato- $\kappa O$ )cobalt(II)

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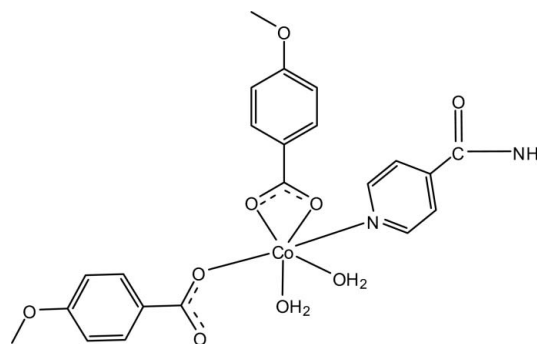
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Key indicators: single-crystal X-ray study;  $T = 100$  K; mean  $\sigma(C-C) = 0.002$  Å;  $R$  factor = 0.022;  $wR$  factor = 0.050; data-to-parameter ratio = 14.5.

In the title complex,  $[Co(C_8H_7O_3)_2(C_6H_6N_2O)(H_2O)_2]$ , the  $Co^{II}$  atom is coordinated by three O atoms from two 4-methoxybenzoate ligands, which act in different modes, *viz.* monodentate and bidentate, two water molecules and one N atom of the isonicotinamide ligand in a distorted octahedral geometry. The monodentate-coordinated carboxylate group is involved in an intramolecular  $O-H \cdots O$  hydrogen bond with the coordinated water molecule. In the crystal structure, intermolecular  $O-H \cdots O$  and  $N-H \cdots O$  hydrogen bonds link the molecules into layers parallel to the  $ab$  plane. The crystal packing is further stabilized by weak  $C-H \cdots O$  hydrogen bonds and  $\pi-\pi$  interactions indicated by the short distance of 3.6181 (8) Å between the centroids of the benzene and pyridine rings of neighbouring molecules.

## Related literature

For general background to niacin and the nicotinic acid derivative *N,N*-diethylnicotinamide, see: Krishnamachari (1974) and Bigoli *et al.* (1972), respectively. For related structures, see: Greenaway *et al.* (1984); Hökelek *et al.* (2009*a,b,c,d*); Necefoğlu *et al.* (2010).



## Experimental

### Crystal data

$[Co(C_8H_7O_3)_2(C_6H_6N_2O)(H_2O)_2]$

$M_r = 519.36$

Monoclinic,  $P2_1$

$a = 8.2666$  (2) Å

$b = 6.8055$  (2) Å

$c = 20.5415$  (4) Å

$\beta = 99.808$  (2)°

$V = 1138.74$  (5) Å<sup>3</sup>

$Z = 2$

Mo  $K\alpha$  radiation

$\mu = 0.81$  mm<sup>-1</sup>

$T = 100$  K

$0.39 \times 0.32 \times 0.28$  mm

### Data collection

Bruker Kappa APEXII CCD area-detector diffractometer

Absorption correction: multi-scan (*SADABS*; Bruker, 2005)

$T_{\min} = 0.739$ ,  $T_{\max} = 0.791$

11263 measured reflections

4838 independent reflections

4597 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.020$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.022$

$wR(F^2) = 0.050$

$S = 1.01$

4838 reflections

333 parameters

H atoms treated by a mixture of independent and constrained refinement

$\Delta\rho_{\text{max}} = 0.29$  e Å<sup>-3</sup>

$\Delta\rho_{\text{min}} = -0.24$  e Å<sup>-3</sup>

Absolute structure: Flack (1983),

1761 Friedel pairs

Flack parameter: 0.015 (7)

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$N2-H2A \cdots O2^i$	0.79 (3)	2.11 (3)	2.877 (2)	164.0 (17)
$N2-H2B \cdots O1^{ii}$	0.91 (3)	2.16 (3)	3.050 (2)	167 (2)
$O8-H81 \cdots O4$	0.83 (3)	1.84 (3)	2.6577 (17)	167 (3)
$O8-H82 \cdots O7^{iii}$	0.89 (2)	1.86 (3)	2.7427 (16)	172 (2)
$O9-H91 \cdots O6^{iv}$	0.786 (19)	2.078 (19)	2.8384 (16)	163 (2)
$O9-H92 \cdots O4^v$	0.91 (3)	1.72 (3)	2.6307 (18)	174.1 (15)
$C8-H8A \cdots O7^{vi}$	0.96	2.53	3.466 (2)	166
$C16-H16B \cdots O4^{vii}$	0.96	2.52	3.4752 (18)	171

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2009).

The authors are indebted to Anadolu University and the Medicinal Plants and Medicine Research Centre of Anadolu University, Eskişehir, Turkey, for the use of the diffractometer. This work was supported financially by Kafkas University Research Fund (grant No. 2009-FEF-03).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2727).

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**supplementary materials**

*Acta Cryst.* (2010). E66, m784-m785 [ doi:10.1107/S160053681002194X ]

## Diaqua(isonicotinamide- $\kappa N^1$ )(4-methoxybenzoato- $\kappa^2 O, O'$ )(4-methoxybenzoato- $\kappa O$ )cobalt(II)

T. Hökelek, Y. Süzen, B. Tercan, E. Tenlik and H. Necefoglu

### Comment

As a part of our ongoing investigation on transition metal complexes of nicotinamide (NA), one form of niacin (Krishnamachari, 1974), and/or the nicotinic acid derivative *N,N*-diethylnicotinamide (DENA), an important respiratory stimulant (Bigoli *et al.*, 1972), the title compound has been synthesized. Herein we report its crystal structure.

The title compound, (I), is a monomeric complex, where the Co<sup>II</sup> ion is surrounded by two methoxybenzoate (MB) anions, one isonicotinamide (INA) ligand and two coordinated water molecules. One of the MB anions acts as a bidentate ligand, while the other is monodentate. The structures of similar complexes, [Mn(C<sub>9</sub>H<sub>10</sub>NO<sub>2</sub>)<sub>2</sub>(C<sub>6</sub>H<sub>6</sub>N<sub>2</sub>O)(H<sub>2</sub>O)<sub>2</sub>] (II) (Hökelek *et al.*, 2009a), [Co(C<sub>9</sub>H<sub>10</sub>NO<sub>2</sub>)<sub>2</sub>(C<sub>6</sub>H<sub>6</sub>N<sub>2</sub>O)(H<sub>2</sub>O)<sub>2</sub>] (III) (Hökelek *et al.*, 2009b), [Cd(C<sub>8</sub>H<sub>7</sub>O<sub>2</sub>)<sub>2</sub>(C<sub>6</sub>H<sub>6</sub>N<sub>2</sub>O)<sub>2</sub>(H<sub>2</sub>O)].H<sub>2</sub>O (IV) (Necefoglu *et al.*, 2010), [Zn(C<sub>9</sub>H<sub>10</sub>NO<sub>2</sub>)<sub>2</sub>(C<sub>6</sub>H<sub>6</sub>N<sub>2</sub>O)(H<sub>2</sub>O)<sub>2</sub>] (V) (Hökelek *et al.*, 2009c) and [Zn(C<sub>8</sub>H<sub>8</sub>NO<sub>2</sub>)<sub>2</sub>(C<sub>6</sub>H<sub>6</sub>N<sub>2</sub>O)<sub>2</sub>].H<sub>2</sub>O (VI) (Hökelek *et al.*, 2009d) have also been determined.

In (I) (Fig. 1), the four O atoms (O1, O2, O5 and O9) in the equatorial plane around the Co1 form a highly distorted square-planar arrangement, while the distorted octahedral coordination geometry is completed by the N atom (N1) of INA ligand and the O atom (O8) of the second water molecule in the axial positions. The average Co—O bond length is 2.1171 (12) Å and the Co atom is displaced out of the least-squares planes of the carboxylate groups (O1/C1/O2) and (O4/C9/O5) by -0.0061 (2) Å and -0.5367 (2) Å, respectively. The dihedral angle between the planar carboxylate groups and the adjacent benzene rings A (C2—C7) and B (C9—C14) are 12.12 (12)° and 9.26 (13)°, respectively, while those between rings A, B and C (N1/C17—C21) are A/B = 78.18 (4), A/C = 74.20 (5) and B/C = 6.23 (5)°. The intramolecular O—H...O hydrogen bond (Table 1) between the monodentate-coordinated carboxyl group and a coordinated water molecule results in a six-membered ring D (Co1/O4/O5/O8/C9/H81) adopting envelope conformation, with atom Co1 displaced by -0.5481 (2) Å from the plane of the other ring atoms. In (I), the O1—Co1—O2 angle is 60.32 (4)°. The corresponding O—M—O (where *M* is a metal) angles are 54.71 (4)° in (IV), 60.03 (6)° in (V), 59.02 (8)° in (VI) and 55.2 (1)° in [Cu(Asp)<sub>2</sub>(py)<sub>2</sub>] (where Asp is acetylsalicylate and py is pyridine) [(VII); Greenaway *et al.*, 1984].

In the crystal structure, intermolecular O—H...O and N—H...O hydrogen bonds (Table 1) link the molecules into layers parallel to *ab* plane. The crystal packing is further stabilized by the weak C—H...O hydrogen bonds (Table 1). The  $\pi$ — $\pi$  contact between the benzene and pyridine rings, Cg2—Cg3<sup>i</sup> [symmetry code: (i) *x*, *y* + 1, *z*, where Cg2 and Cg3 are the centroids of the rings B (C9—C14) and C (N1/C17—C21), respectively] may also stabilize the structure, with centroid-centroid distance of 3.6181 (8) Å.

### Experimental

The title compound was prepared by the reaction of CoSO<sub>4</sub>·7H<sub>2</sub>O (2.81 g, 10 mmol) in H<sub>2</sub>O (50 ml) and INA (2.44 g, 20 mmol) in H<sub>2</sub>O (50 ml) with sodium 4-methoxybenzoate (3.48 g, 20 mmol) in H<sub>2</sub>O (100 ml). The mixture was filtered and set aside to crystallize at ambient temperature for one week, giving brown single crystals.

## Refinement

Atoms H81, H82, H91, H92 (for water molecules) and H2A, H2B (for NH<sub>2</sub>) were located in difference Fourier maps and refined isotropically. The remaining H atoms were positioned geometrically with C—H = 0.93 and 0.96 Å for aromatic and methyl H atoms, respectively, and constrained to ride on their parent atoms, with  $U_{\text{iso}}(\text{H}) = 1.2\text{--}1.5U_{\text{eq}}(\text{C})$ .

## Figures

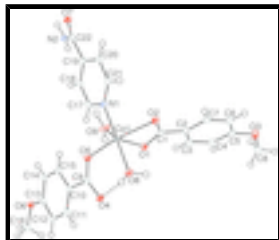


Fig. 1. The molecular structure of (I) with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Dashed line indicates the hydrogen-bonding.

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### Crystal data

[Co(C<sub>8</sub>H<sub>7</sub>O<sub>3</sub>)<sub>2</sub>(C<sub>6</sub>H<sub>6</sub>N<sub>2</sub>O)(H<sub>2</sub>O)<sub>2</sub>]

$M_r = 519.36$

Monoclinic,  $P2_1$

Hall symbol: P 2yb

$a = 8.2666$  (2) Å

$b = 6.8055$  (2) Å

$c = 20.5415$  (4) Å

$\beta = 99.808$  (2)°

$V = 1138.74$  (5) Å<sup>3</sup>

$Z = 2$

$F(000) = 538$

$D_x = 1.515$  Mg m<sup>-3</sup>

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

Cell parameters from 6882 reflections

$\theta = 2.5\text{--}28.4^\circ$

$\mu = 0.81$  mm<sup>-1</sup>

$T = 100$  K

Block, brown

$0.39 \times 0.32 \times 0.28$  mm

### Data collection

Bruker Kappa APEXII CCD area-detector diffractometer

Radiation source: fine-focus sealed tube graphite

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan (SADABS; Bruker, 2005)

$T_{\text{min}} = 0.739$ ,  $T_{\text{max}} = 0.791$

11263 measured reflections

4838 independent reflections

4597 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.020$

$\theta_{\text{max}} = 28.4^\circ$ ,  $\theta_{\text{min}} = 1.0^\circ$

$h = -11 \rightarrow 9$

$k = -8 \rightarrow 9$

$l = -27 \rightarrow 27$

*Refinement*

Refinement on $F^2$	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.022$	$w = 1/[\sigma^2(F_o^2) + (0.0215P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.050$	$(\Delta/\sigma)_{\max} = 0.003$
$S = 1.01$	$\Delta\rho_{\max} = 0.29 \text{ e } \text{\AA}^{-3}$
4838 reflections	$\Delta\rho_{\min} = -0.24 \text{ e } \text{\AA}^{-3}$
333 parameters	Absolute structure: Flack (1983), 1761 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.015 (7)
Secondary atom site location: difference Fourier map	

*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 ( $\text{\AA}^2$ )*

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
Co1	0.83963 (2)	0.92738 (3)	0.717117 (8)	0.01106 (5)
O1	0.87439 (13)	0.72789 (18)	0.79844 (5)	0.0132 (2)
O2	0.75818 (13)	1.01182 (18)	0.81134 (5)	0.0145 (2)
O3	0.68768 (15)	0.5746 (2)	1.08004 (5)	0.0212 (3)
O4	0.78237 (13)	0.51498 (18)	0.63163 (5)	0.0156 (2)
O5	0.95880 (13)	0.76378 (18)	0.65605 (5)	0.0146 (2)
O6	1.43741 (13)	0.13627 (18)	0.56391 (5)	0.0160 (2)
O7	1.44482 (14)	1.65456 (18)	0.77779 (5)	0.0160 (2)
O8	0.60930 (14)	0.78883 (19)	0.68139 (6)	0.0147 (2)
H81	0.652 (3)	0.692 (4)	0.6668 (10)	0.024 (6)*
H82	0.564 (3)	0.750 (4)	0.7153 (12)	0.052 (7)*
O9	0.72143 (15)	1.1511 (2)	0.66328 (6)	0.0177 (3)
H91	0.639 (2)	1.126 (4)	0.6396 (10)	0.024 (6)*
H92	0.749 (2)	1.277 (4)	0.6543 (9)	0.021 (5)*
N1	1.05301 (16)	1.0966 (2)	0.74674 (6)	0.0127 (3)
N2	1.62169 (17)	1.4016 (3)	0.79899 (7)	0.0175 (3)

## supplementary materials

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H2A	1.643 (2)	1.290 (4)	0.8055 (9)	0.015 (5)*
H2B	1.708 (3)	1.486 (4)	0.8026 (10)	0.033 (6)*
C1	0.80942 (19)	0.8473 (3)	0.83434 (7)	0.0130 (3)
C2	0.79173 (19)	0.7843 (3)	0.90221 (7)	0.0140 (3)
C3	0.86387 (19)	0.6109 (3)	0.92813 (7)	0.0172 (3)
H3	0.9323	0.5420	0.9046	0.021*
C4	0.8362 (2)	0.5378 (3)	0.98853 (7)	0.0190 (4)
H4	0.8862	0.4220	1.0056	0.023*
C5	0.7328 (2)	0.6403 (3)	1.02288 (7)	0.0172 (4)
C6	0.6650 (2)	0.8189 (3)	0.99909 (8)	0.0200 (4)
H6	0.6001	0.8901	1.0235	0.024*
C7	0.69427 (19)	0.8905 (3)	0.93914 (7)	0.0172 (4)
H7	0.6489	1.0099	0.9233	0.021*
C8	0.7330 (2)	0.3780 (3)	1.10016 (8)	0.0238 (4)
H8A	0.6763	0.3397	1.1352	0.036*
H8B	0.8493	0.3718	1.1154	0.036*
H8C	0.7038	0.2906	1.0633	0.036*
C9	0.92278 (19)	0.5941 (2)	0.63394 (7)	0.0125 (3)
C10	1.05374 (18)	0.4757 (2)	0.60954 (7)	0.0122 (4)
C11	1.0197 (2)	0.2936 (3)	0.57978 (7)	0.0160 (3)
H11	0.9117	0.2493	0.5716	0.019*
C12	1.14209 (19)	0.1759 (3)	0.56200 (7)	0.0160 (3)
H12	1.1168	0.0551	0.5416	0.019*
C13	1.30388 (19)	0.2427 (3)	0.57528 (7)	0.0135 (3)
C14	1.33919 (17)	0.4281 (3)	0.60267 (6)	0.0160 (3)
H14	1.4464	0.4749	0.6093	0.019*
C15	1.2158 (2)	0.5421 (3)	0.61986 (7)	0.0148 (3)
H15	1.2406	0.6651	0.6386	0.018*
C16	1.4086 (2)	-0.0503 (3)	0.53119 (7)	0.0191 (4)
H16A	1.5118	-0.1095	0.5271	0.029*
H16B	1.3448	-0.0312	0.4880	0.029*
H16C	1.3501	-0.1347	0.5566	0.029*
C17	1.20276 (19)	1.0299 (3)	0.74076 (7)	0.0156 (3)
H17	1.2125	0.9018	0.7261	0.019*
C18	1.34326 (19)	1.1433 (3)	0.75550 (7)	0.0143 (3)
H18	1.4452	1.0910	0.7518	0.017*
C19	1.32986 (18)	1.3358 (2)	0.77577 (7)	0.0119 (3)
C20	1.17472 (17)	1.4047 (3)	0.78314 (6)	0.0132 (3)
H20	1.1614	1.5323	0.7975	0.016*
C21	1.04258 (19)	1.2809 (3)	0.76878 (7)	0.0145 (3)
H21	0.9402	1.3273	0.7746	0.017*
C22	1.47067 (19)	1.4762 (2)	0.78517 (7)	0.0135 (3)

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Co1	0.00979 (9)	0.00942 (10)	0.01397 (8)	-0.00058 (10)	0.00201 (6)	-0.00009 (9)
O1	0.0127 (5)	0.0116 (6)	0.0158 (5)	0.0014 (5)	0.0036 (4)	-0.0003 (4)

O2	0.0120 (6)	0.0131 (6)	0.0190 (5)	0.0013 (5)	0.0042 (4)	0.0016 (5)
O3	0.0236 (7)	0.0243 (8)	0.0170 (5)	0.0001 (6)	0.0068 (5)	0.0039 (5)
O4	0.0115 (5)	0.0136 (6)	0.0223 (5)	-0.0021 (5)	0.0046 (4)	-0.0018 (5)
O5	0.0133 (5)	0.0123 (6)	0.0191 (5)	-0.0025 (5)	0.0051 (4)	-0.0027 (5)
O6	0.0133 (5)	0.0146 (7)	0.0201 (5)	0.0021 (5)	0.0033 (4)	-0.0031 (5)
O7	0.0144 (6)	0.0106 (6)	0.0237 (5)	0.0000 (5)	0.0057 (4)	-0.0008 (5)
O8	0.0124 (6)	0.0125 (7)	0.0192 (5)	-0.0017 (5)	0.0029 (4)	-0.0006 (5)
O9	0.0139 (6)	0.0125 (7)	0.0246 (6)	-0.0031 (5)	-0.0029 (5)	0.0033 (5)
N1	0.0124 (6)	0.0120 (7)	0.0137 (5)	-0.0005 (6)	0.0024 (5)	0.0007 (5)
N2	0.0111 (6)	0.0087 (9)	0.0324 (7)	-0.0007 (6)	0.0029 (5)	-0.0019 (7)
C1	0.0066 (7)	0.0138 (8)	0.0178 (7)	-0.0038 (6)	0.0000 (6)	-0.0006 (6)
C2	0.0121 (7)	0.0141 (9)	0.0153 (6)	-0.0031 (7)	0.0010 (5)	0.0003 (6)
C3	0.0148 (8)	0.0194 (10)	0.0174 (7)	0.0026 (7)	0.0031 (6)	-0.0007 (7)
C4	0.0178 (8)	0.0183 (10)	0.0202 (7)	0.0026 (7)	0.0015 (6)	0.0033 (7)
C5	0.0148 (8)	0.0226 (10)	0.0139 (6)	-0.0037 (7)	0.0019 (6)	0.0004 (6)
C6	0.0211 (9)	0.0204 (10)	0.0201 (7)	0.0019 (7)	0.0076 (6)	-0.0029 (7)
C7	0.0174 (7)	0.0140 (11)	0.0206 (7)	-0.0004 (7)	0.0042 (6)	-0.0006 (6)
C8	0.0236 (9)	0.0289 (13)	0.0188 (7)	-0.0002 (8)	0.0030 (6)	0.0080 (7)
C9	0.0140 (8)	0.0123 (9)	0.0112 (6)	0.0002 (6)	0.0017 (6)	0.0016 (6)
C10	0.0117 (7)	0.0137 (10)	0.0115 (6)	0.0007 (6)	0.0025 (5)	0.0023 (5)
C11	0.0116 (8)	0.0175 (9)	0.0189 (7)	-0.0026 (7)	0.0025 (6)	-0.0010 (7)
C12	0.0167 (8)	0.0138 (9)	0.0174 (7)	-0.0025 (7)	0.0029 (6)	-0.0037 (6)
C13	0.0150 (8)	0.0143 (9)	0.0116 (6)	0.0008 (7)	0.0031 (5)	0.0004 (6)
C14	0.0126 (6)	0.0170 (8)	0.0187 (6)	-0.0038 (9)	0.0034 (5)	-0.0018 (8)
C15	0.0175 (8)	0.0120 (9)	0.0151 (6)	-0.0023 (7)	0.0031 (6)	-0.0033 (6)
C16	0.0222 (8)	0.0158 (10)	0.0191 (6)	0.0021 (8)	0.0035 (6)	-0.0044 (7)
C17	0.0151 (8)	0.0119 (9)	0.0198 (7)	0.0013 (7)	0.0030 (6)	-0.0024 (6)
C18	0.0101 (7)	0.0135 (9)	0.0200 (7)	0.0021 (7)	0.0047 (6)	-0.0011 (6)
C19	0.0121 (7)	0.0126 (8)	0.0116 (6)	-0.0016 (6)	0.0031 (5)	0.0008 (6)
C20	0.0139 (7)	0.0089 (10)	0.0175 (6)	0.0032 (7)	0.0048 (5)	-0.0019 (6)
C21	0.0122 (8)	0.0142 (9)	0.0178 (7)	0.0008 (7)	0.0047 (6)	-0.0010 (6)
C22	0.0136 (7)	0.0136 (10)	0.0145 (6)	-0.0018 (6)	0.0052 (5)	-0.0022 (5)

*Geometric parameters (Å, °)*

Co1—O1	2.1338 (11)	C6—C5	1.392 (3)
Co1—O2	2.2301 (11)	C6—H6	0.9300
Co1—O5	2.0506 (11)	C7—C6	1.383 (2)
Co1—O8	2.1394 (12)	C7—H7	0.9300
Co1—O9	2.0317 (13)	C8—H8A	0.9600
Co1—N1	2.1077 (13)	C8—H8B	0.9600
O1—C1	1.276 (2)	C8—H8C	0.9600
O2—C1	1.260 (2)	C10—C9	1.502 (2)
O3—C5	1.3662 (19)	C10—C11	1.389 (2)
O3—C8	1.431 (2)	C10—C15	1.395 (2)
O4—C9	1.2729 (19)	C11—H11	0.9300
O5—C9	1.258 (2)	C12—C11	1.387 (2)
O6—C13	1.3730 (19)	C12—C13	1.395 (2)
O6—C16	1.437 (2)	C12—H12	0.9300



## supplementary materials

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O7—C22	1.237 (2)	C14—C13	1.392 (3)
O8—H81	0.83 (2)	C14—C15	1.375 (2)
O8—H82	0.89 (3)	C14—H14	0.9300
O9—H91	0.79 (2)	C15—H15	0.9300
O9—H92	0.91 (2)	C16—H16A	0.9600
N1—C17	1.344 (2)	C16—H16B	0.9600
N1—C21	1.342 (2)	C16—H16C	0.9600
N2—C22	1.333 (2)	C17—C18	1.385 (2)
N2—H2A	0.79 (2)	C17—H17	0.9300
N2—H2B	0.91 (2)	C18—H18	0.9300
C2—C1	1.489 (2)	C19—C18	1.385 (2)
C2—C3	1.387 (2)	C19—C20	1.398 (2)
C2—C7	1.398 (2)	C20—H20	0.9300
C3—C4	1.392 (2)	C21—C20	1.371 (2)
C3—H3	0.9300	C21—H21	0.9300
C4—H4	0.9300	C22—C19	1.493 (2)
C5—C4	1.386 (2)		
O1—Co1—O2	60.32 (4)	C6—C7—H7	119.8
O1—Co1—O8	88.94 (4)	O3—C8—H8A	109.5
O5—Co1—O1	96.83 (4)	O3—C8—H8B	109.5
O5—Co1—O2	156.69 (4)	O3—C8—H8C	109.5
O5—Co1—O8	92.47 (5)	H8A—C8—H8B	109.5
O5—Co1—N1	90.44 (5)	H8A—C8—H8C	109.5
O8—Co1—O2	91.64 (5)	H8B—C8—H8C	109.5
O9—Co1—O1	153.01 (5)	O4—C9—C10	117.72 (14)
O9—Co1—O2	95.22 (5)	O5—C9—O4	124.05 (14)
O9—Co1—O5	108.09 (5)	O5—C9—C10	118.23 (13)
O9—Co1—O8	79.99 (5)	C11—C10—C9	121.43 (14)
O9—Co1—N1	92.80 (5)	C11—C10—C15	118.24 (15)
N1—Co1—O1	97.30 (5)	C15—C10—C9	120.23 (14)
N1—Co1—O2	88.29 (5)	C10—C11—H11	119.1
N1—Co1—O8	172.76 (5)	C12—C11—C10	121.90 (15)
C1—O1—Co1	91.92 (10)	C12—C11—H11	119.1
C1—O2—Co1	87.97 (10)	C11—C12—C13	118.61 (16)
C5—O3—C8	117.25 (14)	C11—C12—H12	120.7
C9—O5—Co1	127.62 (10)	C13—C12—H12	120.7
C13—O6—C16	118.14 (12)	O6—C13—C14	115.31 (13)
Co1—O8—H81	93.8 (15)	O6—C13—C12	124.54 (15)
Co1—O8—H82	109.5 (15)	C14—C13—C12	120.15 (15)
H81—O8—H82	108 (2)	C13—C14—H14	119.9
Co1—O9—H91	117.3 (17)	C15—C14—C13	120.10 (14)
Co1—O9—H92	134.2 (12)	C15—C14—H14	119.9
H91—O9—H92	108 (2)	C10—C15—H15	119.6
C17—N1—Co1	121.84 (11)	C14—C15—C10	120.90 (16)
C21—N1—Co1	120.64 (11)	C14—C15—H15	119.6
C21—N1—C17	117.40 (14)	O6—C16—H16A	109.5
C22—N2—H2A	125.4 (14)	O6—C16—H16B	109.5
C22—N2—H2B	118.0 (14)	O6—C16—H16C	109.5
H2A—N2—H2B	117 (2)	H16A—C16—H16B	109.5

O1—C1—C2	118.41 (15)	H16A—C16—H16C	109.5
O2—C1—O1	119.79 (14)	H16B—C16—H16C	109.5
O2—C1—C2	121.77 (15)	N1—C17—C18	122.89 (16)
C3—C2—C7	118.77 (14)	N1—C17—H17	118.6
C3—C2—C1	120.01 (15)	C18—C17—H17	118.6
C7—C2—C1	121.09 (15)	C17—C18—H18	120.5
C2—C3—C4	121.42 (16)	C19—C18—C17	119.09 (15)
C2—C3—H3	119.3	C19—C18—H18	120.5
C4—C3—H3	119.3	C18—C19—C20	118.15 (15)
C3—C4—H4	120.6	C18—C19—C22	122.94 (15)
C5—C4—C3	118.90 (17)	C20—C19—C22	118.75 (15)
C5—C4—H4	120.6	C19—C20—H20	120.6
O3—C5—C4	123.71 (17)	C21—C20—C19	118.87 (17)
O3—C5—C6	115.79 (15)	C21—C20—H20	120.6
C4—C5—C6	120.49 (15)	N1—C21—C20	123.53 (15)
C7—C6—C5	119.92 (16)	N1—C21—H21	118.2
C7—C6—H6	120.0	C20—C21—H21	118.2
C5—C6—H6	120.0	O7—C22—N2	122.40 (15)
C2—C7—H7	119.8	O7—C22—C19	119.83 (14)
C6—C7—C2	120.36 (16)	N2—C22—C19	117.72 (15)
O2—Co1—O1—C1	0.09 (8)	C7—C2—C1—O2	10.6 (2)
O5—Co1—O1—C1	175.24 (9)	C3—C2—C1—O1	8.3 (2)
O8—Co1—O1—C1	-92.40 (9)	C7—C2—C1—O1	-167.51 (14)
O9—Co1—O1—C1	-27.20 (15)	C1—C2—C3—C4	-173.46 (15)
N1—Co1—O1—C1	83.91 (9)	C7—C2—C3—C4	2.4 (2)
O1—Co1—O2—C1	-0.10 (8)	C1—C2—C7—C6	173.09 (15)
O5—Co1—O2—C1	-12.36 (15)	C3—C2—C7—C6	-2.8 (2)
O8—Co1—O2—C1	87.75 (9)	C2—C3—C4—C5	0.7 (3)
O9—Co1—O2—C1	167.84 (9)	O3—C5—C4—C3	175.05 (15)
N1—Co1—O2—C1	-99.50 (9)	C6—C5—C4—C3	-3.5 (2)
O1—Co1—O5—C9	60.93 (12)	C7—C6—C5—O3	-175.47 (15)
O2—Co1—O5—C9	71.64 (17)	C7—C6—C5—C4	3.2 (2)
O8—Co1—O5—C9	-28.29 (12)	C2—C7—C6—C5	0.0 (2)
O9—Co1—O5—C9	-108.57 (12)	C11—C10—C9—O4	5.9 (2)
N1—Co1—O5—C9	158.34 (12)	C11—C10—C9—O5	-174.93 (13)
O1—Co1—N1—C17	75.29 (12)	C15—C10—C9—O4	-170.39 (13)
O1—Co1—N1—C21	-108.72 (11)	C15—C10—C9—O5	8.8 (2)
O2—Co1—N1—C17	135.07 (12)	C9—C10—C11—C12	-174.61 (14)
O2—Co1—N1—C21	-48.93 (11)	C15—C10—C11—C12	1.7 (2)
O5—Co1—N1—C17	-21.65 (12)	C9—C10—C15—C14	174.66 (14)
O5—Co1—N1—C21	154.35 (11)	C11—C10—C15—C14	-1.7 (2)
O9—Co1—N1—C17	-129.79 (12)	C13—C12—C11—C10	0.8 (2)
O9—Co1—N1—C21	46.21 (12)	C11—C12—C13—O6	175.92 (14)
Co1—O1—C1—O2	-0.17 (14)	C11—C12—C13—C14	-3.3 (2)
Co1—O1—C1—C2	177.95 (12)	C15—C14—C13—O6	-175.95 (13)
Co1—O2—C1—O1	0.16 (14)	C15—C14—C13—C12	3.3 (2)
Co1—O2—C1—C2	-177.89 (13)	C13—C14—C15—C10	-0.8 (2)
C8—O3—C5—C4	-8.5 (2)	N1—C17—C18—C19	-1.6 (2)
C8—O3—C5—C6	170.10 (14)	C20—C19—C18—C17	2.6 (2)

## supplementary materials

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Co1—O5—C9—O4	19.3 (2)	C22—C19—C18—C17	-172.69 (13)
Co1—O5—C9—C10	-159.84 (9)	C18—C19—C20—C21	-1.3 (2)
C16—O6—C13—C12	5.3 (2)	C22—C19—C20—C21	174.22 (13)
C16—O6—C13—C14	-175.44 (13)	N1—C21—C20—C19	-1.2 (2)
Co1—N1—C17—C18	175.31 (11)	O7—C22—C19—C18	151.80 (15)
C21—N1—C17—C18	-0.8 (2)	N2—C22—C19—C18	-25.7 (2)
Co1—N1—C21—C20	-173.91 (11)	O7—C22—C19—C20	-23.5 (2)
C17—N1—C21—C20	2.3 (2)	N2—C22—C19—C20	158.99 (13)
C3—C2—C1—O2	-173.63 (14)		

### 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>
N2—H2A $\cdots$ O2 <sup>i</sup>	0.79 (3)	2.11 (3)	2.877 (2)	164.0 (17)
N2—H2B $\cdots$ O1 <sup>ii</sup>	0.91 (3)	2.16 (3)	3.050 (2)	167 (2)
O8—H81 $\cdots$ O4	0.83 (3)	1.84 (3)	2.6577 (17)	167 (3)
O8—H82 $\cdots$ O7 <sup>iii</sup>	0.89 (2)	1.86 (3)	2.7427 (16)	172 (2)
O9—H91 $\cdots$ O6 <sup>iv</sup>	0.786 (19)	2.078 (19)	2.8384 (16)	163 (2)
O9—H92 $\cdots$ O4 <sup>v</sup>	0.91 (3)	1.72 (3)	2.6307 (18)	174.1 (15)
C8—H8A $\cdots$ O7 <sup>vi</sup>	0.96	2.53	3.466 (2)	166
C16—H16B $\cdots$ O4 <sup>vii</sup>	0.96	2.52	3.4752 (18)	171

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



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