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Bis(μ-4-amino-*N*-oxidobenzamide)bis[(4-amino-*N*-oxidobenzamide)aquacobalt] dihydrate

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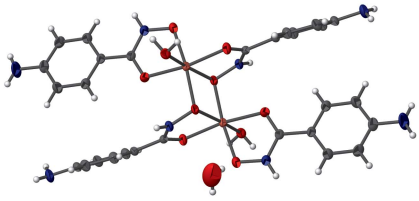
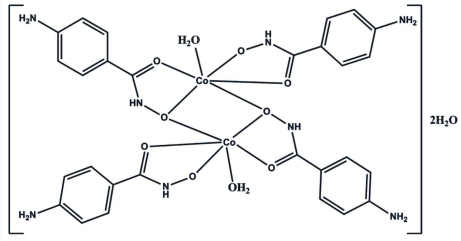
The structure of the title compound, $[Co_2(C_7H_7N_2O_2)_4(H_2O)_2]\cdot 2H_2O$, consists of a centrosymmetric binuclear $[Co_2(4\text{-Apha})_4(H_2O)_2]$ complex molecule (4-AphaH = 4-aminophenylhydroxamic acid), and two solvent water molecules. Each Co^{II} cation is six coordinate, binding five oxygen atoms from three 4-Apha$^-$ ligands and a water molecule in a slightly distorted octahedral geometry. Two of the 4-Apha$^-$ ligands bridge two neighbouring Co^{II} ions to form the binuclear complex. A three-dimensional network structure is generated by O—H···O, N—H···O, and N—H···N hydrogen bonds.

3D view



Chemical scheme



Structure description

The asymmetric unit of the title compound consists of a Co^{II} cation bound to two bidentate Apha$^-$ ligands and a water molecule. The Co1—O4*A* and Co1*A*—O4 bonds, [symmetry code: (*A*) $-x$, $-y$, $-z$], generate the centrosymmetric binuclear $[Co_2(4\text{-Apha})_4(H_2O)_2]$ complex, Fig. 1. The structure also has two solvent water molecules. Two of the 4-Apha$^-$ ligands bridge the adjacent Co^{II} nuclei in a μ^2 fashion while the two others are bidentate, each coordinating to a single Co^{II} atom. A water molecule also binds to the Co nucleus, completing the slightly distorted octahedral coordination geometry. The Co—O distances range from 2.0741 (15) to 2.1655 (15) Å, which agrees well with the values observed in related structures (Chen *et al.*, 2014, 2015). The Co1···Co1*A* distance is 3.1727 (5) Å with a Co1—O4—Co1*A* angle of 97.44 (6)°.

In the crystal, N—H···O hydrogen bonds form between the NH_2 groups of 4-Apha$^-$ ligands as donors and the O atoms of coordinated or solvate water molecules as acceptors, Table 1. N—H···N hydrogen bonds form between the NH groups of the 4-Apha$^-$ ligands and the NH_2 groups of adjacent ligands. The coordinated and solvent water molecules act as both hydrogen-bond donors and acceptors, Table 1, and this

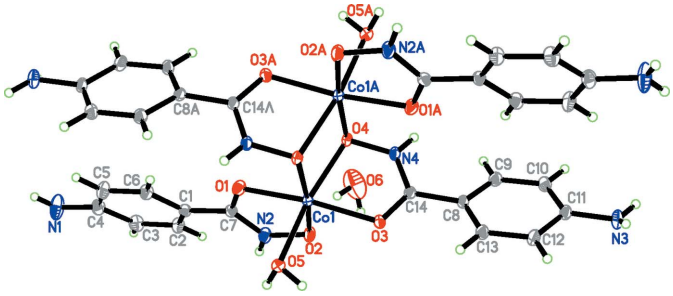


Figure 1
The structure of title complex with displacement ellipsoids drawn at the 30% probability level. [Symmetry code: (*A*) $-x, -y, -z$.]

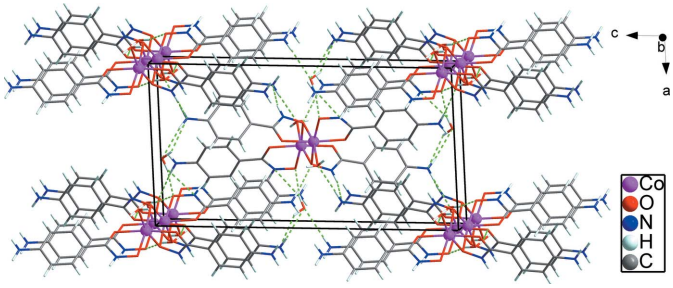


Figure 2
Crystal packing of title complex viewed along the *b* axis with hydrogen bonds drawn as dashed lines.

multitude of classical hydrogen bonds combines to generate a three-dimensional supramolecular network structure, Fig. 2.

Synthesis and crystallization

A mixture of 4-AphaH (0.0150 g, 0.1 mmol), $Co(CH_3COO)_2{\cdot}4H_2O$ (0.0249 g, 0.1 mmol) and H_2O/ethanol (v/v = 1:1, 1 ml) was sealed in a 6 ml Pyrex tube. The tube was heated at 60°C for 3 d under autogenous pressure. Slow cooling of the resulting solution to room temperature gave brown prism-like crystals. The yield was 0.0128 g (64%, based on 4-AphaH).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

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

D—H···A	D—H	H···A	D···A	D—H···A
O5—H5*A*···O2^{i}	0.85	1.85	2.681 (2)	164
N1—H1*B*···O6^{ii}	0.81	2.41	3.106 (3)	144
N1—H1*A*···O4^{iii}	0.86	2.20	3.060 (3)	174
O5—H5*B*···O3^{i}	0.85	2.05	2.817 (2)	150
N3—H3*A*···O6^{iv}	0.87	2.20	3.047 (3)	163
N3—H3*B*···O5^{v}	0.86	2.24	3.103 (2)	178
O6—H6*A*···O2	0.85	1.99	2.822 (3)	164
O6—H6*A*···N2	0.85	2.58	3.392 (3)	160
N4—H4···N3^{vi}	0.86	2.28	3.063 (3)	152

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$[Co_2(C_7H_7N_2O_2)_4(H_2O)_2]{\cdot}2H_2O$
M_r	794.51
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	296
a, b, c (Å)	10.6382 (7), 7.6075 (5), 19.8693 (13)
β (°)	93.219 (1)
V (Å^3)	1605.49 (18)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	1.11
Crystal size (mm)	0.28 × 0.25 × 0.23
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2005)
T_{min}, T_{max}	0.746, 0.784
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	10770, 3984, 3123
R_{int}	0.033
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.669
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.033, 0.086, 0.95
No. of reflections	3984
No. of parameters	226
No. of restraints	2
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.59, −0.48

Computer programs: *APEX2* and *SAINT* (Bruker, 2005), *SHELXS97* and *SHELXTL* (Sheldrick, 2008), *SHELXL2014/7* (Sheldrick, 2015), and *DIAMOND* (Brandenburg, 1999).

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full crystallographic data

IUCrData (2016). **1**, x161640 [https://doi.org/10.1107/S2414314616016400]

Bis(*μ*-4-amino-*N*-oxidobenzamide)bis[(4-amino-*N*-oxidobenzamide)aquacobalt] dihydrate

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Bis(*μ*-4-amino-*N*-oxidobenzamide)bis[(4-amino-*N*-oxidobenzamide)aquacobalt] dihydrate

Crystal data

$[Co_2(C_7H_7N_2O_2)_4(H_2O)_2]\cdot 2H_2O$
M_r = 794.51
Monoclinic, $P2_1/n$
a = 10.6382 (7) Å
b = 7.6075 (5) Å
c = 19.8693 (13) Å
β = 93.219 (1)°
V = 1605.49 (18) Å^3
Z = 2
$F(000)$ = 820
D_x = 1.643 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2780 reflections
θ = 2.9–27.8°
μ = 1.11 mm^{-1}
T = 296 K
Prism, brown
0.28 × 0.25 × 0.23 mm

Data collection

Bruker APEXII CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2005)
T_{min} = 0.746, T_{max} = 0.784
10770 measured reflections
3984 independent reflections
3123 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
θ_{max} = 28.4°, θ_{min} = 2.1°
h = −13→14
k = −10→9
l = −26→20

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.086
S = 0.95
3984 reflections
226 parameters
2 restraints
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0398P)^2 + 1.2061P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.59 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}

Special details

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Co1	0.02073 (3)	0.19515 (4)	0.02648 (2)	0.01957 (9)
O1	0.03470 (15)	0.1624 (2)	0.13027 (7)	0.0276 (3)
O3	0.02485 (14)	0.26232 (19)	−0.07576 (7)	0.0243 (3)
O2	0.17079 (15)	0.3594 (2)	0.05349 (7)	0.0271 (3)
N2	0.17829 (18)	0.3738 (3)	0.12290 (9)	0.0280 (4)
H2	0.2269	0.4511	0.1423	0.034*
C8	0.12639 (19)	0.2018 (3)	−0.17786 (10)	0.0203 (4)
O5	−0.11627 (15)	0.39954 (19)	0.04215 (7)	0.0263 (3)
H5A	−0.1234	0.4649	0.0074	0.039*
H5B	−0.0824	0.4815	0.0659	0.032*
C14	0.10440 (19)	0.1713 (3)	−0.10601 (10)	0.0194 (4)
C7	0.1105 (2)	0.2684 (3)	0.15950 (10)	0.0218 (4)
C4	0.1405 (2)	0.2830 (3)	0.37565 (11)	0.0301 (5)
C12	0.0396 (2)	0.2982 (3)	−0.28674 (10)	0.0267 (5)
H12	−0.0247	0.3530	−0.3125	0.032*
C11	0.1431 (2)	0.2297 (3)	−0.31795 (10)	0.0239 (4)
C1	0.12380 (19)	0.2772 (3)	0.23381 (10)	0.0210 (4)
C13	0.0319 (2)	0.2851 (3)	−0.21764 (10)	0.0242 (4)
H13	−0.0372	0.3325	−0.1974	0.029*
N1	0.1472 (2)	0.2922 (4)	0.44496 (10)	0.0537 (7)
H1A	0.2143	0.3404	0.4631	0.064*
H1B	0.0937	0.2416	0.4655	0.064*
C6	0.0353 (2)	0.1913 (3)	0.27101 (10)	0.0268 (5)
H6	−0.0302	0.1307	0.2484	0.032*
C5	0.0426 (2)	0.1937 (3)	0.34055 (11)	0.0326 (5)
H5	−0.0199	0.1340	0.3650	0.039*
N3	0.1540 (2)	0.2500 (3)	−0.38711 (9)	0.0324 (4)
H3A	0.0772	0.2451	−0.4051	0.039*
H3B	0.2181	0.2073	−0.4059	0.039*
C10	0.2395 (2)	0.1511 (3)	−0.27810 (11)	0.0272 (5)
H10	0.3100	0.1073	−0.2981	0.033*
C9	0.2312 (2)	0.1375 (3)	−0.20921 (11)	0.0255 (4)
H9	0.2964	0.0849	−0.1833	0.031*
C2	0.2233 (2)	0.3625 (3)	0.26928 (11)	0.0280 (5)
H2A	0.2847	0.4187	0.2456	0.034*
C3	0.2321 (2)	0.3649 (3)	0.33879 (11)	0.0314 (5)
H3	0.2996	0.4215	0.3613	0.038*
O6	0.4080 (2)	0.2162 (4)	0.02767 (13)	0.0874 (9)
H6A	0.3381	0.2481	0.0425	0.105*
H6B	0.4146	0.3256	0.0198	0.105*
N4	0.16524 (16)	0.0421 (2)	−0.07437 (8)	0.0211 (4)
H4	0.2273	−0.0089	−0.0923	0.025*
O4	0.12774 (13)	−0.01100 (19)	−0.01141 (6)	0.0209 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Co1	0.02660 (16)	0.01796 (14)	0.01431 (13)	−0.00035 (11)	0.00260 (10)	−0.00054 (10)
O1	0.0365 (9)	0.0277 (8)	0.0183 (7)	−0.0102 (7)	−0.0017 (6)	−0.0001 (6)
O3	0.0322 (8)	0.0240 (7)	0.0171 (7)	0.0064 (6)	0.0044 (6)	0.0008 (6)
O2	0.0339 (9)	0.0311 (8)	0.0165 (7)	−0.0052 (7)	0.0030 (6)	0.0001 (6)
N2	0.0336 (10)	0.0317 (10)	0.0188 (9)	−0.0110 (8)	0.0008 (7)	−0.0033 (7)
C8	0.0241 (10)	0.0198 (10)	0.0172 (9)	−0.0028 (8)	0.0035 (7)	0.0008 (8)
O5	0.0352 (9)	0.0211 (7)	0.0231 (8)	0.0015 (6)	0.0055 (6)	0.0016 (6)
C14	0.0226 (10)	0.0187 (10)	0.0171 (9)	−0.0031 (8)	0.0016 (7)	−0.0011 (7)
C7	0.0248 (10)	0.0210 (10)	0.0196 (10)	0.0012 (8)	0.0006 (8)	−0.0010 (8)
C4	0.0310 (12)	0.0380 (13)	0.0206 (10)	0.0021 (10)	−0.0029 (9)	−0.0011 (9)
C12	0.0271 (11)	0.0321 (12)	0.0210 (10)	0.0018 (9)	0.0024 (8)	0.0057 (9)
C11	0.0287 (11)	0.0253 (11)	0.0183 (10)	−0.0047 (9)	0.0058 (8)	0.0010 (8)
C1	0.0231 (10)	0.0221 (11)	0.0177 (9)	0.0004 (8)	−0.0012 (7)	−0.0014 (8)
C13	0.0233 (11)	0.0280 (11)	0.0220 (10)	0.0030 (9)	0.0059 (8)	0.0013 (8)
N1	0.0495 (14)	0.093 (2)	0.0175 (10)	−0.0225 (14)	−0.0037 (9)	−0.0022 (11)
C6	0.0274 (11)	0.0310 (11)	0.0214 (10)	−0.0051 (9)	−0.0030 (8)	−0.0029 (9)
C5	0.0322 (12)	0.0444 (14)	0.0212 (10)	−0.0109 (11)	0.0009 (9)	0.0019 (10)
N3	0.0341 (11)	0.0459 (12)	0.0177 (9)	−0.0006 (9)	0.0065 (8)	0.0020 (8)
C10	0.0262 (11)	0.0305 (12)	0.0258 (11)	0.0030 (9)	0.0087 (9)	0.0005 (9)
C9	0.0261 (11)	0.0276 (11)	0.0230 (10)	0.0050 (9)	0.0025 (8)	0.0030 (9)
C2	0.0258 (11)	0.0341 (12)	0.0241 (11)	−0.0061 (9)	0.0018 (8)	−0.0017 (9)
C3	0.0264 (12)	0.0399 (13)	0.0270 (11)	−0.0069 (10)	−0.0061 (9)	−0.0062 (10)
O6	0.0394 (13)	0.137 (3)	0.0859 (18)	−0.0009 (15)	0.0076 (12)	−0.0290 (18)
N4	0.0234 (9)	0.0241 (9)	0.0163 (8)	0.0018 (7)	0.0046 (6)	0.0012 (7)
O4	0.0279 (8)	0.0221 (7)	0.0129 (6)	0.0004 (6)	0.0023 (5)	0.0021 (5)

Geometric parameters (Å, º)

Co1—O2	2.0741 (15)	C11—N3	1.394 (3)
Co1—O1	2.0742 (14)	C11—C10	1.395 (3)
Co1—O3	2.0975 (14)	C1—C6	1.392 (3)
Co1—O4	2.1021 (14)	C1—C2	1.398 (3)
Co1—O4^{i}	2.1198 (15)	C13—H13	0.9300
Co1—O5	2.1655 (15)	N1—H1A	0.8628
O1—C7	1.259 (2)	N1—H1B	0.8150
O3—C14	1.270 (2)	C6—C5	1.380 (3)
O2—N2	1.381 (2)	C6—H6	0.9300
N2—C7	1.323 (3)	C5—H5	0.9600
N2—H2	0.8600	N3—H3A	0.8745
C8—C9	1.396 (3)	N3—H3B	0.8598
C8—C13	1.396 (3)	C10—C9	1.380 (3)
C8—C14	1.478 (3)	C10—H10	0.9300
O5—H5A	0.8500	C9—H9	0.9300
O5—H5B	0.8500	C2—C3	1.379 (3)
C14—N4	1.317 (3)	C2—H2A	0.9300

C7—C1	1.477 (3)	C3—H3	0.9300
C4—N1	1.377 (3)	O6—H6A	0.8499
C4—C5	1.397 (3)	O6—H6B	0.8501
C4—C3	1.398 (3)	N4—O4	1.394 (2)
C12—C13	1.384 (3)	N4—H4	0.8600
C12—C11	1.395 (3)	O4—Co1^{i}	2.1199 (15)
C12—H12	0.9300		

O2—Co1—O1	78.69 (6)	N3—C11—C12	120.6 (2)
O2—Co1—O3	92.65 (6)	C10—C11—C12	118.77 (19)
O1—Co1—O3	171.07 (6)	C6—C1—C2	117.76 (18)
O2—Co1—O4	96.76 (6)	C6—C1—C7	118.68 (18)
O1—Co1—O4	104.87 (6)	C2—C1—C7	123.52 (19)
O3—Co1—O4	78.10 (5)	C12—C13—C8	120.90 (19)
O2—Co1—O4^{i}	172.41 (5)	C12—C13—H13	119.5
O1—Co1—O4^{i}	94.15 (5)	C8—C13—H13	119.5
O3—Co1—O4^{i}	94.60 (6)	C4—N1—H1A	115.7
O4—Co1—O4^{i}	82.56 (6)	C4—N1—H1B	118.6
O2—Co1—O5	92.56 (6)	H1A—N1—H1B	125.2
O1—Co1—O5	87.39 (6)	C5—C6—C1	121.6 (2)
O3—Co1—O5	90.87 (5)	C5—C6—H6	119.2
O4—Co1—O5	165.83 (5)	C1—C6—H6	119.2
O4^{i}—Co1—O5	89.58 (6)	C6—C5—C4	120.3 (2)
C7—O1—Co1	112.97 (13)	C6—C5—H5	120.0
C14—O3—Co1	112.10 (12)	C4—C5—H5	119.7
N2—O2—Co1	107.85 (11)	C11—N3—H3A	105.8
C7—N2—O2	119.88 (17)	C11—N3—H3B	119.9
C7—N2—H2	120.1	H3A—N3—H3B	123.5
O2—N2—H2	120.1	C9—C10—C11	120.6 (2)
C9—C8—C13	118.33 (18)	C9—C10—H10	119.7
C9—C8—C14	123.36 (18)	C11—C10—H10	119.7
C13—C8—C14	118.04 (18)	C10—C9—C8	120.9 (2)
Co1—O5—H5A	109.5	C10—C9—H9	119.6
Co1—O5—H5B	109.8	C8—C9—H9	119.6
H5A—O5—H5B	92.2	C3—C2—C1	121.2 (2)
O3—C14—N4	120.23 (18)	C3—C2—H2A	119.4
O3—C14—C8	121.16 (18)	C1—C2—H2A	119.4
N4—C14—C8	118.54 (18)	C2—C3—C4	120.6 (2)
O1—C7—N2	119.28 (18)	C2—C3—H3	119.7
O1—C7—C1	120.88 (18)	C4—C3—H3	119.7
N2—C7—C1	119.83 (18)	H6A—O6—H6B	82.3
N1—C4—C5	121.3 (2)	C14—N4—O4	119.12 (17)
N1—C4—C3	120.2 (2)	C14—N4—H4	120.4
C5—C4—C3	118.5 (2)	O4—N4—H4	120.4
C13—C12—C11	120.4 (2)	N4—O4—Co1	107.06 (11)
C13—C12—H12	119.8	N4—O4—Co1^{i}	108.24 (10)
C11—C12—H12	119.8	Co1—O4—Co1^{i}	97.44 (6)
N3—C11—C10	120.5 (2)		

Co1—O2—N2—C7	10.8 (2)	C2—C1—C6—C5	1.9 (3)
Co1—O3—C14—N4	5.4 (2)	C7—C1—C6—C5	179.7 (2)
Co1—O3—C14—C8	−177.58 (14)	C1—C6—C5—C4	−0.3 (4)
C9—C8—C14—O3	164.1 (2)	N1—C4—C5—C6	177.8 (2)
C13—C8—C14—O3	−21.9 (3)	C3—C4—C5—C6	−1.7 (4)
C9—C8—C14—N4	−18.8 (3)	N3—C11—C10—C9	177.3 (2)
C13—C8—C14—N4	155.16 (19)	C12—C11—C10—C9	1.5 (3)
Co1—O1—C7—N2	−5.6 (3)	C11—C10—C9—C8	0.1 (3)
Co1—O1—C7—C1	173.90 (15)	C13—C8—C9—C10	−2.0 (3)
O2—N2—C7—O1	−3.8 (3)	C14—C8—C9—C10	171.9 (2)
O2—N2—C7—C1	176.66 (18)	C6—C1—C2—C3	−1.4 (3)
C13—C12—C11—N3	−177.0 (2)	C7—C1—C2—C3	−179.1 (2)
C13—C12—C11—C10	−1.2 (3)	C1—C2—C3—C4	−0.6 (4)
O1—C7—C1—C6	−11.9 (3)	N1—C4—C3—C2	−177.3 (2)
N2—C7—C1—C6	167.6 (2)	C5—C4—C3—C2	2.2 (4)
O1—C7—C1—C2	165.8 (2)	O3—C14—N4—O4	9.8 (3)
N2—C7—C1—C2	−14.7 (3)	C8—C14—N4—O4	−167.29 (17)
C11—C12—C13—C8	−0.7 (3)	C14—N4—O4—Co1	−18.85 (19)
C9—C8—C13—C12	2.3 (3)	C14—N4—O4—Co1^{i}	85.25 (17)
C14—C8—C13—C12	−172.0 (2)		

Symmetry code: (i) −*x*, −*y*, −*z*.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H5*A*···O2^{ii}	0.85	1.85	2.681 (2)	164
N1—H1*B*···O6^{iii}	0.81	2.41	3.106 (3)	144
N1—H1*A*···O4^{iv}	0.86	2.20	3.060 (3)	174
O5—H5*B*···O3^{ii}	0.85	2.05	2.817 (2)	150
N3—H3*A*···O6^{v}	0.87	2.20	3.047 (3)	163
N3—H3*B*···O5^{vi}	0.86	2.24	3.103 (2)	178
O6—H6*A*···O2	0.85	1.99	2.822 (3)	164
O6—H6*A*···N2	0.85	2.58	3.392 (3)	160
N4—H4···N3^{vii}	0.86	2.28	3.063 (3)	152

Symmetry codes: (ii) −*x*, −*y*+1, −*z*; (iii) *x*−1/2, −*y*+1/2, *z*+1/2; (iv) −*x*+1/2, *y*+1/2, −*z*+1/2; (v) *x*−1/2, −*y*+1/2, *z*−1/2; (vi) *x*+1/2, −*y*+1/2, *z*−1/2; (vii) −*x*+1/2, *y*−1/2, −*z*−1/2.