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Bis(2,2′,2″-nitrilotriacetamide-κ^3O,N,O')cobalt(II) dinitrate tetrahydrate

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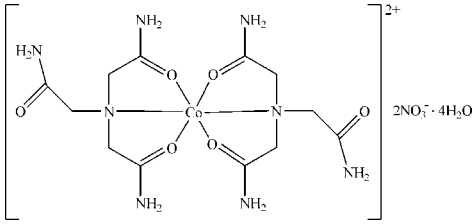
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.028; wR factor = 0.080; data-to-parameter ratio = 12.0.

In the centrosymmetric title compound, $[Co(C_6H_{12}N_4O_3)_2](NO_3)_2\cdot4H_2O$, the Co^{II} ion, lying on an inversion center, is O,N,O'-chelated by two nitrilotriacetamide molecules, forming a distorted octahedral geometry. In the crystal, extensive O—H···O and N—H···O hydrogen bonds link the complex cations, nitrate anions and lattice water molecules into a three-dimensional network.

Related literature

For related structures, see: Kumari *et al.* (2012). For the synthesis of the ligand, see: Smith *et al.* (1995).



Experimental

Crystal data

$[Co(C_6H_{12}N_4O_3)_2](NO_3)_2\cdot4H_2O$
M_r = 631.41
Triclinic, $P\overline{1}$
a = 8.4910 (17) Å
b = 9.1410 (18) Å
c = 9.2580 (19) Å
α = 91.55 (3)°
β = 96.03 (3)°
γ = 110.68 (3)°
V = 667.0 (2) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.73 mm^{-1}
T = 293 K
0.36 × 0.32 × 0.25 mm

Data collection

Siemens P4 diffractometer
Absorption correction: ψ scan (*XSCANS*; Siemens, 1994)
T_{min} = 0.779, T_{max} = 0.838
3692 measured reflections
2301 independent reflections
2185 reflections with $I > 2\sigma(I)$
R_{int} = 0.016
2 standard reflections every 150 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.080
S = 1.05
2301 reflections
191 parameters
6 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O6^{i}	0.86	2.26	3.026 (3)	149
N2—H2B···O3^{ii}	0.86	1.96	2.816 (2)	173
N3—H3C···O8^{iii}	0.86	2.11	2.958 (3)	168
N3—H3D···O5^{ii}	0.86	2.16	3.001 (3)	166
N4—H4A···O7^{iv}	0.86	2.20	2.992 (3)	152
N4—H4B···O7	0.86	2.30	3.045 (3)	145
O7—H7A···O6	0.88 (2)	2.05 (2)	2.877 (3)	156 (3)
O7—H7B···O8^{v}	0.87 (2)	1.96 (2)	2.826 (3)	176 (3)
O8—H8A···O1^{i}	0.85 (2)	2.14 (2)	2.976 (2)	166 (3)
O8—H8B···O6	0.85 (2)	2.16 (2)	2.980 (3)	161 (3)
O8—H8B···O5	0.85 (2)	2.42 (3)	3.078 (3)	134 (3)

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

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *XSCANS*; 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*.

This research was supported by the Huangzhou Scholar Fund (2012).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HY2611).

References

Kumari, N., Ward, B. D., Kar, S. & Mishra, L. (2012). *Polyhedron*, **33**, 425–434.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
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supporting information

Acta Cryst. (2013). E**69**, m325 [doi:10.1107/S1600536813012968]

Bis(2,2′,2′′-nitrilotriacetamide-κ^3O,N,O')cobalt(II) dinitrate tetrahydrate

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S1. Comment

Transition metal compounds have been of great interest for many years. They are very important in the development of coordination chemistry. As an extension of work on the structural characterization of Co compounds, we report here the crystal structure of a new mononuclear cobalt(II) compound.

The title compound consists of a $[Co(NTA)_2]^{2+}$ cation (NTA = nitrilotriacetamide), two nitrate anions and four solvent water molecules. The Co^{II} atom has a distorted octahedral coordination environment (Fig. 1), which is centrosymmetric as the Co^{II} atom occupies an inversion center. In the equatorial plane, the Co—N1 distance is 2.1696 (16) Å and the Co—O1 distance is 2.1057 (14) Å. The axial Co—O2 bond is appreciably shortened, which is 2.0329 (14) Å. In the crystal, extensive O—H···O and N—H···O hydrogen bonds (Table 1) link the complex cations, nitrate anions and lattice water molecules into a three-dimensional network (Fig. 2).

S2. Experimental

The ligand was prepared according to the literature method (Smith *et al.*, 1995). The title compound was synthesized by adding water solution of $Co(NO_3)_2.6H_2O$ (291 mg, 2 mmol) to a solution of the ligand (752 mg, 4 mmol) in methanol/water (v/v 3:1, 20 ml). The mixture was stirred for 30 min at room temperature. The solution was filtered and the filtrate was allowed to stand in air for 1 week, and pink crystals were formed at the bottom of the vessel on slow evaporation of the solvent at room temperature (yield: 30%).

S3. Refinement

H atoms on C and N atoms were positioned geometrically and refined as riding atoms, with C—H = 0.97, N—H = 0.86 Å, and with $U_{iso}(H) = 1.2U_{eq}(C,N)$. The water H atoms were located from a difference Fourier map and refined with restraints of O—H = 0.86 (1) Å and $U_{iso}(H) = 1.5U_{eq}(O)$.

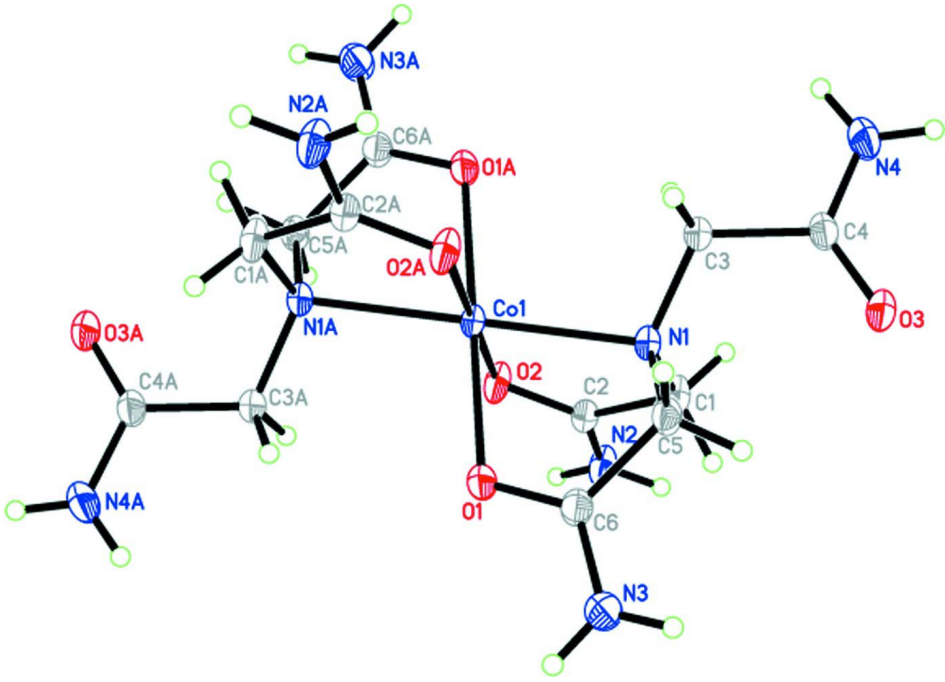

Figure 1

The molecular structure of the complex cation in the title compound. Displacement ellipsoids are drawn at the 30% probability level. [Symmetry code: (A) 2-x, 1-y, 2-z.]

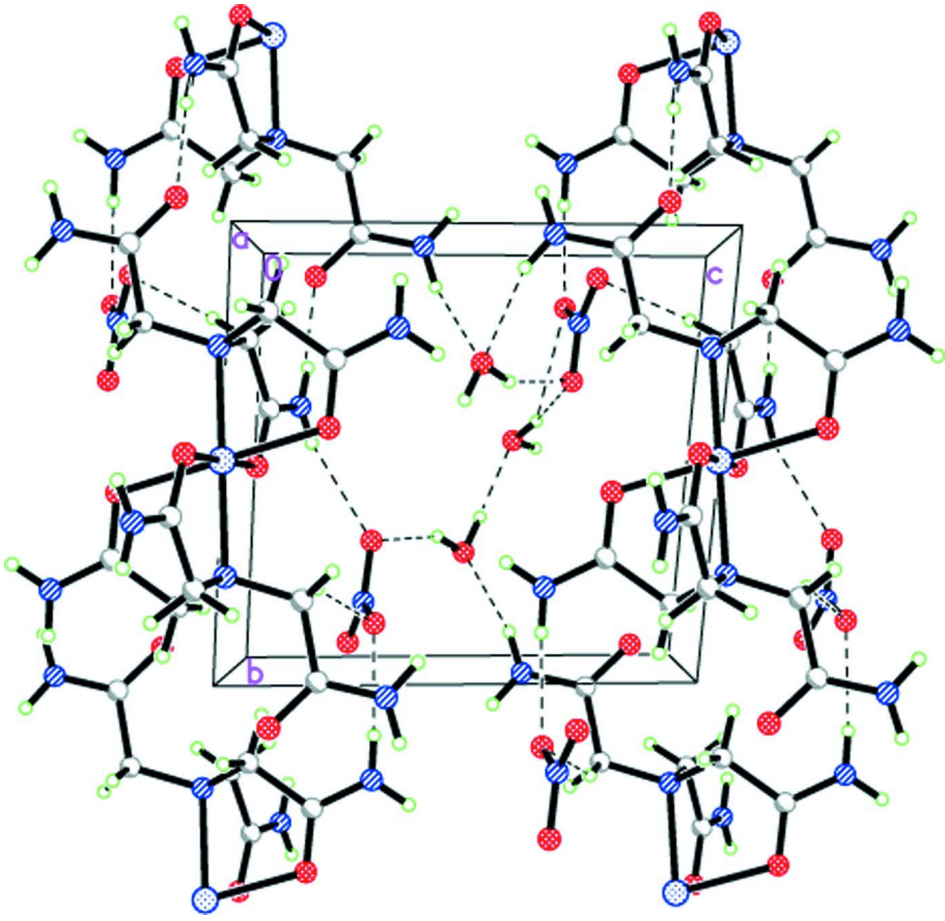

Figure 2

The packing diagram for the title compound, viewed down the *a* axis, with hydrogen bonds drawn as dashed lines.

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Crystal data

$[Co(C_6H_{12}N_4O_3)_2](NO_3)_2{\cdot}4H_2O$
$M_r = 631.41$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 8.4910\ (17)$ Å
$b = 9.1410\ (18)$ Å
$c = 9.2580\ (19)$ Å
$\alpha = 91.55\ (3)°$
$\beta = 96.03\ (3)°$
$\gamma = 110.68\ (3)°$
$V = 667.0\ (2)$ Å^3
$Z = 1$
$F(000) = 329$
$D_x = 1.572$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4804 reflections
$\theta = 2.4–28.3°$
$\mu = 0.73$ mm^{-1}
$T = 293$ K
Block, pink
0.36 × 0.32 × 0.25 mm

Data collection

Siemens P4
diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: ψ scan
(*XSCANS*; Siemens, 1994)
$T_{min} = 0.779$, $T_{max} = 0.838$
3692 measured reflections
2301 independent reflections
2185 reflections with $I > 2\sigma(I)$

R_{int} = 0.016
θ_{max} = 25.0°, θ_{min} = 2.2°
h = −8→10
k = −10→6
l = −11→11
2 standard reflections every 150 reflections
intensity decay: none

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.080
S = 1.05
2301 reflections
191 parameters
6 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0402P)^2 + 0.3421P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.036 (4)

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_{iso}*/U_{eq}
Co1	1.0000	0.5000	1.0000	0.02488 (14)
O1	1.04636 (18)	0.42993 (15)	1.21011 (14)	0.0357 (3)
N1	0.84838 (18)	0.25174 (17)	0.97367 (17)	0.0255 (3)
O2	0.77965 (17)	0.51316 (15)	1.05517 (16)	0.0358 (3)
N2	0.5284 (2)	0.37589 (19)	1.1249 (2)	0.0385 (4)
H2A	0.5140	0.4602	1.1531	0.046*
H2B	0.4520	0.2860	1.1335	0.046*
C2	0.6662 (2)	0.3843 (2)	1.0691 (2)	0.0279 (4)
C4	0.7310 (2)	0.0285 (2)	0.7776 (2)	0.0309 (4)
C1	0.6803 (2)	0.2307 (2)	1.0199 (2)	0.0344 (5)
H1A	0.6607	0.1616	1.0990	0.041*
H1B	0.5929	0.1810	0.9393	0.041*
C5	0.9455 (3)	0.1821 (2)	1.0737 (2)	0.0311 (4)
H5A	1.0417	0.1752	1.0298	0.037*
H5B	0.8741	0.0772	1.0931	0.037*
C6	1.0065 (2)	0.2848 (2)	1.2144 (2)	0.0320 (4)
C3	0.8369 (3)	0.2003 (2)	0.8196 (2)	0.0319 (4)
H3A	0.9508	0.2204	0.7957	0.038*
H3B	0.7894	0.2642	0.7604	0.038*

O3	0.7098 (2)	−0.07085 (16)	0.86712 (17)	0.0436 (4)
N3	1.0164 (3)	0.2180 (2)	1.3353 (2)	0.0498 (5)
H3C	1.0514	0.2739	1.4165	0.060*
H3D	0.9878	0.1178	1.3342	0.060*
N4	0.6714 (3)	−0.0027 (2)	0.6381 (2)	0.0481 (5)
H4A	0.6138	−0.0976	0.6053	0.058*
H4B	0.6903	0.0720	0.5802	0.058*
O4	0.3650 (3)	0.0756 (2)	0.7431 (3)	0.0729 (6)
N5	0.3013 (3)	0.1744 (2)	0.7124 (2)	0.0490 (5)
O6	0.3922 (3)	0.3140 (2)	0.7051 (3)	0.0790 (7)
O5	0.1443 (3)	0.1328 (2)	0.6828 (3)	0.0753 (6)
O8	0.1387 (2)	0.4513 (2)	0.58788 (17)	0.0494 (4)
O7	0.6172 (3)	0.2799 (2)	0.5059 (2)	0.0606 (5)
H8A	0.072 (4)	0.470 (4)	0.642 (3)	0.091*
H8B	0.195 (4)	0.405 (4)	0.637 (3)	0.091*
H7A	0.566 (4)	0.319 (4)	0.566 (3)	0.091*
H7B	0.689 (4)	0.364 (3)	0.475 (4)	0.091*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Co1	0.0248 (2)	0.0160 (2)	0.0289 (2)	0.00148 (14)	0.00284 (14)	0.00066 (13)
O1	0.0466 (8)	0.0217 (7)	0.0305 (7)	0.0042 (6)	−0.0021 (6)	0.0001 (5)
N1	0.0247 (8)	0.0181 (7)	0.0304 (8)	0.0039 (6)	0.0024 (6)	−0.0002 (6)
O2	0.0316 (7)	0.0186 (7)	0.0550 (9)	0.0044 (6)	0.0126 (6)	0.0028 (6)
N2	0.0346 (9)	0.0217 (8)	0.0584 (11)	0.0066 (7)	0.0156 (8)	0.0010 (8)
C2	0.0262 (9)	0.0220 (9)	0.0330 (10)	0.0064 (8)	0.0018 (7)	0.0018 (7)
C4	0.0296 (10)	0.0236 (9)	0.0357 (11)	0.0052 (8)	0.0043 (8)	−0.0038 (8)
C1	0.0269 (10)	0.0204 (9)	0.0521 (12)	0.0030 (8)	0.0085 (9)	−0.0014 (8)
C5	0.0367 (10)	0.0199 (9)	0.0347 (10)	0.0091 (8)	−0.0002 (8)	0.0008 (7)
C6	0.0331 (10)	0.0254 (10)	0.0334 (10)	0.0069 (8)	−0.0013 (8)	0.0026 (8)
C3	0.0374 (11)	0.0220 (9)	0.0291 (10)	0.0025 (8)	0.0027 (8)	0.0003 (7)
O3	0.0528 (9)	0.0226 (7)	0.0426 (9)	−0.0009 (6)	0.0011 (7)	0.0011 (6)
N3	0.0765 (14)	0.0299 (9)	0.0343 (10)	0.0119 (9)	−0.0060 (9)	0.0041 (8)
N4	0.0604 (12)	0.0313 (9)	0.0387 (10)	0.0034 (9)	−0.0061 (9)	−0.0075 (8)
O4	0.0603 (12)	0.0588 (12)	0.1021 (17)	0.0247 (10)	0.0046 (11)	0.0217 (11)
N5	0.0592 (13)	0.0371 (11)	0.0523 (12)	0.0154 (10)	0.0204 (10)	0.0052 (9)
O6	0.0978 (16)	0.0309 (9)	0.0995 (16)	0.0039 (10)	0.0445 (13)	−0.0050 (10)
O5	0.0557 (12)	0.0608 (12)	0.1194 (19)	0.0294 (10)	0.0200 (12)	0.0227 (12)
O8	0.0611 (11)	0.0471 (10)	0.0394 (9)	0.0186 (8)	0.0064 (8)	0.0022 (7)
O7	0.0692 (13)	0.0430 (10)	0.0559 (11)	0.0022 (9)	0.0120 (9)	0.0010 (8)

Geometric parameters (Å, º)

Co1—O2	2.0329 (14)	C5—C6	1.516 (3)
Co1—O1	2.1057 (14)	C5—H5A	0.9700
Co1—N1	2.1696 (16)	C5—H5B	0.9700
O1—C6	1.250 (2)	C6—N3	1.299 (3)

N1—C3	1.472 (2)	C3—H3A	0.9700
N1—C5	1.478 (2)	C3—H3B	0.9700
N1—C1	1.482 (2)	N3—H3C	0.8600
O2—C2	1.251 (2)	N3—H3D	0.8600
N2—C2	1.307 (3)	N4—H4A	0.8600
N2—H2A	0.8600	N4—H4B	0.8600
N2—H2B	0.8600	O4—N5	1.231 (3)
C2—C1	1.513 (3)	N5—O6	1.245 (3)
C4—O3	1.225 (3)	N5—O5	1.247 (3)
C4—N4	1.321 (3)	O8—H8A	0.85 (2)
C4—C3	1.524 (3)	O8—H8B	0.85 (2)
C1—H1A	0.9700	O7—H7A	0.88 (2)
C1—H1B	0.9700	O7—H7B	0.87 (2)

O2^{i}—Co1—O2	180.0	N1—C1—C2	112.43 (15)
O2^{i}—Co1—O1	91.39 (7)	N1—C1—H1A	109.1
O2—Co1—O1	88.61 (7)	C2—C1—H1A	109.1
O2^{i}—Co1—O1^{i}	88.61 (7)	N1—C1—H1B	109.1
O2—Co1—O1^{i}	91.39 (7)	C2—C1—H1B	109.1
O1—Co1—O1^{i}	180.0	H1A—C1—H1B	107.9
O2^{i}—Co1—N1	98.08 (6)	N1—C5—C6	108.58 (15)
O2—Co1—N1	81.92 (6)	N1—C5—H5A	110.0
O1—Co1—N1	78.83 (6)	C6—C5—H5A	110.0
O1^{i}—Co1—N1	101.17 (6)	N1—C5—H5B	110.0
O2^{i}—Co1—N1^{i}	81.92 (6)	C6—C5—H5B	110.0
O2—Co1—N1^{i}	98.08 (6)	H5A—C5—H5B	108.4
O1—Co1—N1^{i}	101.17 (6)	O1—C6—N3	122.53 (19)
O1^{i}—Co1—N1^{i}	78.83 (6)	O1—C6—C5	119.19 (17)
N1—Co1—N1^{i}	180.00 (8)	N3—C6—C5	118.28 (17)
C6—O1—Co1	113.26 (12)	N1—C3—C4	115.74 (16)
C3—N1—C5	113.47 (15)	N1—C3—H3A	108.3
C3—N1—C1	112.51 (15)	C4—C3—H3A	108.3
C5—N1—C1	111.69 (16)	N1—C3—H3B	108.3
C3—N1—Co1	107.54 (11)	C4—C3—H3B	108.3
C5—N1—Co1	103.12 (11)	H3A—C3—H3B	107.4
C1—N1—Co1	107.83 (11)	C6—N3—H3C	120.0
C2—O2—Co1	115.18 (12)	C6—N3—H3D	120.0
C2—N2—H2A	120.0	H3C—N3—H3D	120.0
C2—N2—H2B	120.0	C4—N4—H4A	120.0
H2A—N2—H2B	120.0	C4—N4—H4B	120.0
O2—C2—N2	121.59 (17)	H4A—N4—H4B	120.0
O2—C2—C1	121.70 (17)	O4—N5—O6	120.7 (2)
N2—C2—C1	116.70 (16)	O4—N5—O5	119.5 (2)
O3—C4—N4	124.06 (18)	O6—N5—O5	119.7 (2)
O3—C4—C3	121.43 (17)	H8A—O8—H8B	108 (2)
N4—C4—C3	114.46 (18)	H7A—O7—H7B	102 (2)

O2^{i}—Co1—O1—C6	−81.44 (15)	Co1—O2—C2—N2	169.65 (15)

O2—Co1—O1—C6	98.56 (15)	Co1—O2—C2—C1	−11.4 (2)
N1—Co1—O1—C6	16.52 (14)	C3—N1—C1—C2	−118.18 (18)
N1^{i}—Co1—O1—C6	−163.48 (14)	C5—N1—C1—C2	112.86 (18)
O2^{i}—Co1—N1—C3	−62.62 (13)	Co1—N1—C1—C2	0.2 (2)
O2—Co1—N1—C3	117.38 (13)	O2—C2—C1—N1	7.4 (3)
O1—Co1—N1—C3	−152.44 (13)	N2—C2—C1—N1	−173.64 (17)
O1^{i}—Co1—N1—C3	27.56 (13)	C3—N1—C5—C6	158.56 (16)
O2^{i}—Co1—N1—C5	57.55 (12)	C1—N1—C5—C6	−72.98 (19)
O2—Co1—N1—C5	−122.45 (12)	Co1—N1—C5—C6	42.55 (17)
O1—Co1—N1—C5	−32.27 (12)	Co1—O1—C6—N3	−175.96 (18)
O1^{i}—Co1—N1—C5	147.73 (12)	Co1—O1—C6—C5	4.6 (2)
O2^{i}—Co1—N1—C1	175.82 (12)	N1—C5—C6—O1	−34.3 (3)
O2—Co1—N1—C1	−4.18 (12)	N1—C5—C6—N3	146.2 (2)
O1—Co1—N1—C1	86.00 (13)	C5—N1—C3—C4	68.0 (2)
O1^{i}—Co1—N1—C1	−94.00 (13)	C1—N1—C3—C4	−60.0 (2)
O1—Co1—O2—C2	−70.37 (14)	Co1—N1—C3—C4	−178.61 (13)
O1^{i}—Co1—O2—C2	109.63 (14)	O3—C4—C3—N1	−25.6 (3)
N1—Co1—O2—C2	8.54 (14)	N4—C4—C3—N1	156.74 (18)
N1^{i}—Co1—O2—C2	−171.46 (14)		

Symmetry code: (i) −*x*+2, −*y*+1, −*z*+2.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O6^{ii}	0.86	2.26	3.026 (3)	149
N2—H2*B*···O3^{iii}	0.86	1.96	2.816 (2)	173
N3—H3*C*···O8^{iv}	0.86	2.11	2.958 (3)	168
N3—H3*D*···O5^{iii}	0.86	2.16	3.001 (3)	166
N4—H4*A*···O7^{v}	0.86	2.20	2.992 (3)	152
N4—H4*B*···O7	0.86	2.30	3.045 (3)	145
O7—H7*A*···O6	0.88 (2)	2.05 (2)	2.877 (3)	156 (3)
O7—H7*B*···O8^{vi}	0.87 (2)	1.96 (2)	2.826 (3)	176 (3)
O8—H8*A*···O1^{ii}	0.85 (2)	2.14 (2)	2.976 (2)	166 (3)
O8—H8*B*···O6	0.85 (2)	2.16 (2)	2.980 (3)	161 (3)
O8—H8*B*···O5	0.85 (2)	2.42 (3)	3.078 (3)	134 (3)

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