metal-organic papers

Redetermination of bis(2-amino-3-hydroxy-1-phenyl-propanolato-κ^2N,O^1)(ethylenediamine-κ^2N,N')-cobalt(III) iodide monohydrate

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New data for the title complex, $[\text{Co}(\text{C}_9\text{H}_{12}\text{NO}_2)_2(\text{C}_2\text{H}_8\text{N}_2)]\text{I}\cdot\text{H}_2\text{O}$, allow the modelling of previously unresolved disorder [Wardeska *et al.* (1979). *Inorg. Chem.* **18**, 1641–1648] in the ethylenediamine ligand coordinated to the octahedral cation.

Comment

The title complex, (I), was synthesized and crystallized in about 1978, and its structure published the following year as part of a synthetic and spectroscopic project (Wardeska *et al.*, 1979). Crystals of this obviously robust material were recently rediscovered in perfect condition after 28 years in a glass vial and its structure has been redetermined in order to resolve some disorder in the earlier determination.

(I)
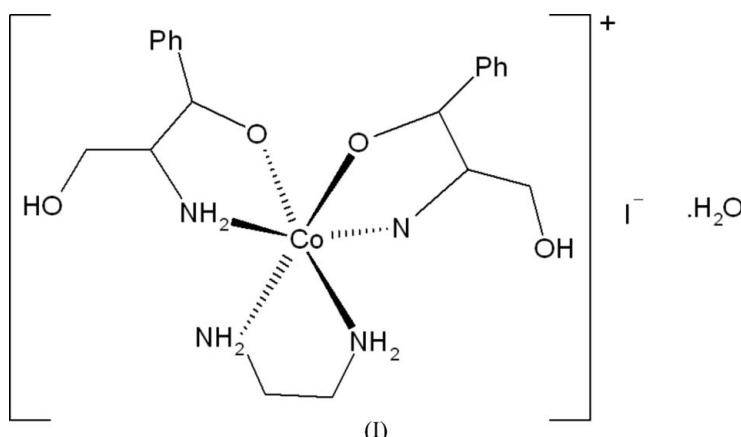
While the structure in the original report gave very good residuals, the disorder in the ethylenediamine ligand was not resolved and the determination of the absolute structure was based only on a comparison of the R values given by the correct *versus* the inverted structure. We also take this opportunity to present the first published structure from data collected on a new type of single-crystal diffraction instrument, the Rigaku SCXmini Benchtop Crystallography System. This structure was used as a test of the efficacy of this new paradigm for crystallographic instrumentation.

As shown in Fig. 1, there are two distinct conformations of the ethylenediamine ligand. The occupancy of the major orientation (specified by the letter *A*) refined to 0.66 (1).

The molecular structure of the cation (Table 1) and the hydrogen-bonding scheme (Table 2) involving all components of the unit-cell contents, are, of course, similar to those originally described in detail by Wardeska *et al.* (1979), although in the present determination the positions of the O-bound H atoms were fully refined.
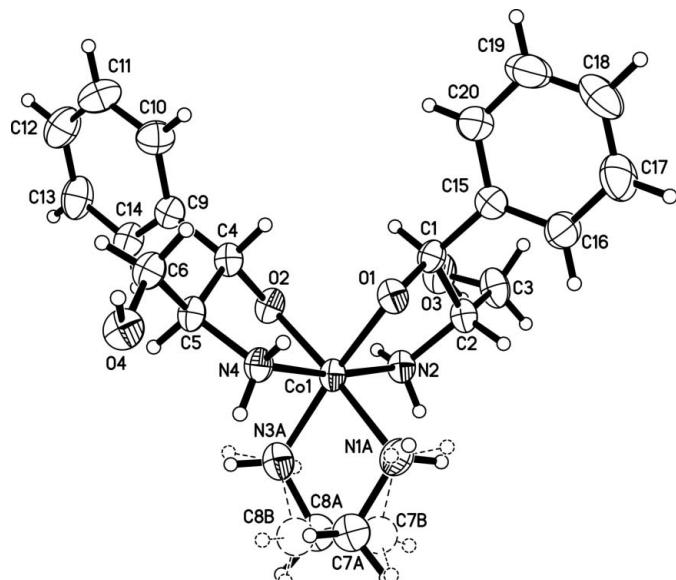
Figure 1
A view of the cation in (I). The atoms and bonds in the minor orientation of the disordered ethylenediamine ligand are shown with dashed lines.

Experimental

The title compound was synthesized from a methanol–water solution of (1S,2S)-(+)-1-phenyl-2-amino-1,3-dihydroxypropane to which was added sodium hexanitrocobaltate(III). The resulting solids were dissolved in a 2:1 methanol–water solution and treated with ethylenediamine, warmed, filtered, and then converted to the iodide salt by recrystallizing twice from potassium iodide solution. The complete experimental preparation is described by Wardeska *et al.* (1979).

Crystal data
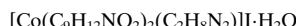
[Co(C$_9$H$_{12}$NO$_2$)$_2$(C$_2$H$_8$N$_2$)]I·H$_2$O
$M_r = 596.34$
Orthorhombic, $P2_12_12_1$
$a = 6.7895$ (2) Å
$b = 14.5013$ (4) Å
$c = 24.8565$ (8) Å
$V = 2447.29$ (13) Å^3
$Z = 4$
$D_x = 1.619$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 19448 reflections
$\theta = 3.0–27.5°$
$\mu = 2.00$ mm^{-1}
$T = 294$ (2) K
Prism, translucent pale-brown
$0.42 \times 0.39 \times 0.28$ mm

Data collection

Rigaku SCXmini diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min} = 0.466$, $T_{max} = 0.57$
19627 measured reflections
5482 independent reflections

5172 reflections with $I > 2\sigma(I)$
$R_{int} = 0.029$
$\theta_{max} = 27.5°$
$h = -8 \rightarrow 8$
$k = -13 \rightarrow 18$
$l = -30 \rightarrow 32$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.027$
$wR(F^2) = 0.062$
$S = 1.10$
5482 reflections
291 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.022P)^2 + P]$
   where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.003$
$\Delta\rho_{max} = 0.58$ e Å^{-3}
$\Delta\rho_{min} = -0.69$ e Å^{-3}
Absolute structure: Flack (1983), 2272 Friedel pairs
Flack parameter: 0.007 (14)

Table 1
Selected geometric parameters (Å, °).

Co1—O1	1.8793 (17)	Co1—N4	1.955 (2)
Co1—O2	1.9002 (18)	Co1—N1A	1.973 (2)
Co1—N2	1.9456 (19)	Co1—N3A	1.987 (2)
O1—Co1—O2	93.18 (8)	O2—Co1—N3A	91.74 (9)
O1—Co1—N2	85.45 (8)	N2—Co1—N4	170.41 (9)
O1—Co1—N4	87.21 (8)	N2—Co1—N1A	92.23 (10)
O1—Co1—N1A	90.85 (8)	N2—Co1—N3A	93.98 (9)
O1—Co1—N3A	175.03 (9)	N4—Co1—N1A	94.03 (9)
O2—Co1—N2	88.43 (8)	N4—Co1—N3A	93.87 (9)
O2—Co1—N4	85.83 (8)	N1A—Co1—N3A	84.23 (9)
O2—Co1—N1A	175.95 (9)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O4—H4$\cdots$O2^i	0.899 (17)	1.73 (2)	2.594 (2)	159 (3)
O5—H2W$\cdots$O4	0.906 (19)	1.99 (3)	2.792 (3)	146 (5)
O5—H1W$\cdots$I1	0.90 (3)	2.91 (4)	3.639 (3)	140 (5)
O3—H3$\cdots$O1^{ii}	0.919 (18)	1.83 (2)	2.714 (3)	160 (4)
N1A—H1A1$\cdots$I1^{iii}	0.90	3.26	4.078 (2)	152
N1A—H1A2$\cdots$I1^{iv}	0.90	2.92	3.709 (2)	147
N3A—H3A1$\cdots$I1	0.90	2.90	3.697 (2)	149
N3A—H3A2$\cdots$O4^{ii}	0.90	2.35	3.094 (3)	140
N3A—H3A2$\cdots$O5^{ii}	0.90	2.57	3.368 (4)	148
N1B—H1B2$\cdots$I1^{iv}	0.90	2.91	3.709 (2)	148
N3B—H3B1$\cdots$I1	0.90	2.87	3.697 (2)	154
N3B—H3B2$\cdots$O5^{ii}	0.90	2.61	3.368 (4)	142
N3B—H3B2$\cdots$O4^{ii}	0.90	2.66	3.094 (3)	111
N2—H2A$\cdots$I1^{iii}	0.90	2.80	3.632 (2)	155
N4—H4A$\cdots$O5	0.90	2.34	3.139 (4)	149
N4—H4B$\cdots$O3^i	0.90	2.41	3.219 (3)	149

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

The positions of the H atoms bonded to O atoms were fully refined. All other H atoms were placed in idealized positions, with C—H = 0.98 (methine), 0.93 (phenyl) or 0.97 Å (methylene), and N—H = 0.90 Å. U_{iso}(H) values were set to $1.2U_{eq}$(C,N) or $1.5U_{eq}$(O). For the disordered group, only the positions of the two C atoms were split; the N positions were not distinct enough to allow modelling over two positions. The isotropic displacement parameters for disordered atoms C7A and C7B were constrained to be equal, as were those for C8A and C8B. The error in the C—C distance introduced by the disorder is greatly reduced in this resolved model compared with that in the earlier report. When these disordered C atoms are not resolved and they are allowed to refine anisotropically, the resulting apparent C—C distance is 1.395 (10) Å (Wardeska *et al.*, 1979). The present refinement allows for two separate positions for this C—C group, and the distances refine to 1.506 (6) Å for the *A* group and 1.497 (10) Å for the lower-occupancy *B* group (*i.e.* statistically indistinguishable at the 2σ level).

Data collection: *SCXmini* (Rigaku, 2006); cell refinement: *PROCESS-AUTO* (Rigaku, 1998); data reduction: *PROCESS-AUTO*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *CrystalStructure* (Rigaku, 2005); software used to prepare material for publication: *SHELXL97*.

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development, and production of the Rigaku SCXmini benchtop crystallography system.

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supporting information

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bis(2-amino-3-hydroxy-1-phenylpropanolato-κ^2N,O^1)(ethylenediamine- κ^2N,N')cobalt(III) iodide monohydrate

Crystal data

[Co(C$_9$H$_{12}$NO$_2$)$_2$(C$_2$H$_8$N$_2$)]I·H$_2$O	$F(000) = 1208$
$M_r = 596.34$	$D_x = 1.619$ Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: P 21 21 21	Cell parameters from 19448 reflections
$a = 6.7895$ (2) Å	$\theta = 3.0–27.5°$
$b = 14.5013$ (4) Å	$\mu = 2.00$ mm^{-1}
$c = 24.8565$ (8) Å	$T = 294$ K
$V = 2447.29$ (13) Å^3	Prism, translucent pale-brown
$Z = 4$	$0.42 \times 0.39 \times 0.28$ mm

Data collection

Rigaku SCXmini diffractometer	19627 measured reflections
Radiation source: long-fine-focus sealed tube	5482 independent reflections
Graphite monochromator	5172 reflections with $I > 2\sigma(I)$
Detector resolution: 6.85 pixels mm^{-1}	$R_{int} = 0.029$
ω scans	$\theta_{max} = 27.5°$, $\theta_{min} = 3.1°$
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	$h = -8 \rightarrow 8$
	$k = -13 \rightarrow 18$
$T_{min} = 0.466$, $T_{max} = 0.57$	$l = -30 \rightarrow 32$

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
$R[F^2 > 2\sigma(F^2)] = 0.027$	
$wR(F^2) = 0.062$	$w = 1/[\sigma^2(F_o^2) + (0.022P)^2 + P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.10$	
5482 reflections	$(\Delta/\sigma)_{max} = 0.003$
291 parameters	$\Delta\rho_{max} = 0.58$ e Å^{-3}
215 restraints	$\Delta\rho_{min} = -0.69$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), with how many Friedel pairs
Secondary atom site location: difference Fourier map	Absolute structure parameter: 0.007 (14)

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}	Occ. (<1)
I1	0.24806 (3)	1.289623 (13)	0.701876 (8)	0.05308 (7)	
Co1	0.21430 (5)	0.93696 (2)	0.673829 (13)	0.02590 (7)	
O4	0.8157 (3)	1.08284 (14)	0.62835 (8)	0.0383 (4)	
H4	0.930 (3)	1.062 (2)	0.6145 (12)	0.042 (8)*	
O5	0.6808 (5)	1.1481 (2)	0.72736 (12)	0.0804 (9)	
H2W	0.739 (7)	1.151 (4)	0.6947 (11)	0.121*	
H1W	0.571 (5)	1.175 (3)	0.740 (2)	0.121*	
O1	0.3046 (2)	0.83092 (11)	0.63805 (7)	0.0294 (4)	
O3	−0.3187 (3)	0.81759 (15)	0.60232 (9)	0.0474 (5)	
H3	−0.441 (3)	0.809 (3)	0.6175 (14)	0.071*	
O2	0.1432 (2)	0.99710 (12)	0.60874 (7)	0.0301 (4)	
N1A	0.2831 (3)	0.88261 (14)	0.74411 (8)	0.0357 (5)	0.657 (6)
H1A1	0.1910	0.8413	0.7540	0.043*	0.657 (6)
H1A2	0.3999	0.8535	0.7419	0.043*	0.657 (6)
N3A	0.1217 (3)	1.04309 (15)	0.71760 (9)	0.0369 (5)	0.657 (6)
H3A1	0.2014	1.0917	0.7119	0.044*	0.657 (6)
H3A2	−0.0006	1.0589	0.7072	0.044*	0.657 (6)
N1B	0.2831 (3)	0.88261 (14)	0.74411 (8)	0.0357 (5)	0.343 (6)
H1B1	0.2280	0.8263	0.7463	0.043*	0.343 (6)
H1B2	0.4146	0.8751	0.7455	0.043*	0.343 (6)
N3B	0.1217 (3)	1.04309 (15)	0.71760 (9)	0.0369 (5)	0.343 (6)
H3B1	0.1555	1.0966	0.7017	0.044*	0.343 (6)
H3B2	−0.0101	1.0415	0.7211	0.044*	0.343 (6)
N2	−0.0417 (3)	0.87671 (13)	0.67324 (9)	0.0286 (4)	
H2A	−0.0911	0.8754	0.7068	0.034*	
H2B	−0.1254	0.9089	0.6523	0.034*	
N4	0.4763 (3)	0.98935 (14)	0.66224 (8)	0.0282 (4)	
H4A	0.5135	1.0216	0.6915	0.034*	
H4B	0.5641	0.9438	0.6570	0.034*	
C1	0.1466 (3)	0.78521 (18)	0.61191 (10)	0.0288 (5)	
H1	0.1054	0.8216	0.5806	0.035*	
C2	−0.0230 (3)	0.78085 (17)	0.65230 (11)	0.0293 (5)	
H2	0.0169	0.7408	0.6821	0.035*	
C15	0.2218 (4)	0.69206 (16)	0.59293 (10)	0.0312 (5)	
C16	0.2528 (5)	0.61960 (17)	0.62874 (11)	0.0431 (6)	

H16	0.2211	0.6266	0.6649	0.052*	
C17	0.3307 (5)	0.5371 (2)	0.61045 (15)	0.0540 (9)	
H17	0.3467	0.4882	0.6343	0.065*	
C18	0.3844 (5)	0.5268 (2)	0.55766 (15)	0.0560 (9)	
H18	0.4380	0.4713	0.5458	0.067*	
C19	0.3591 (6)	0.5977 (2)	0.52268 (15)	0.0568 (9)	
H19	0.3981	0.5909	0.4870	0.068*	
C20	0.2757 (5)	0.67989 (19)	0.53957 (11)	0.0453 (7)	
H20	0.2558	0.7273	0.5150	0.054*	
C5	0.4717 (3)	1.05122 (18)	0.61421 (10)	0.0271 (5)	
H5	0.4302	1.1130	0.6255	0.033*	
C3	−0.2219 (4)	0.74665 (18)	0.63105 (12)	0.0403 (6)	
H3A	−0.2014	0.6941	0.6076	0.048*	
H3B	−0.3036	0.7270	0.6609	0.048*	
C9	0.2787 (4)	1.07077 (16)	0.52822 (9)	0.0294 (5)	
C14	0.1607 (4)	1.14859 (19)	0.52991 (12)	0.0371 (6)	
H14	0.0962	1.1642	0.5616	0.045*	
C13	0.1378 (4)	1.2035 (2)	0.48459 (14)	0.0465 (7)	
H13	0.0554	1.2547	0.4859	0.056*	
C12	0.2360 (5)	1.1829 (2)	0.43779 (12)	0.0512 (8)	
H12	0.2208	1.2201	0.4076	0.061*	
C11	0.3566 (5)	1.1070 (2)	0.43583 (12)	0.0500 (8)	
H11	0.4257	1.0937	0.4045	0.060*	
C10	0.3762 (4)	1.0500 (2)	0.48032 (11)	0.0404 (6)	
H10	0.4548	0.9976	0.4782	0.048*	
C6	0.6744 (3)	1.05765 (19)	0.58866 (10)	0.0317 (5)	
H6A	0.7098	0.9987	0.5730	0.038*	
H6B	0.6729	1.1034	0.5602	0.038*	
C4	0.3141 (3)	1.01045 (16)	0.57716 (10)	0.0279 (5)	
H4C	0.3600	0.9500	0.5647	0.034*	
C7A	0.2942 (8)	0.9589 (3)	0.78466 (19)	0.0429 (10)*	0.657 (6)
H7A1	0.4162	0.9929	0.7806	0.051*	0.657 (6)
H7A2	0.2886	0.9340	0.8209	0.051*	0.657 (6)
C8A	0.1202 (9)	1.0209 (4)	0.77437 (19)	0.0448 (10)*	0.657 (6)
H8A1	−0.0013	0.9897	0.7839	0.054*	0.657 (6)
H8A2	0.1307	1.0766	0.7957	0.054*	0.657 (6)
C7B	0.2222 (16)	0.9357 (6)	0.7904 (3)	0.0429 (10)*	0.343 (6)
H7B1	0.3118	0.9252	0.8201	0.051*	0.343 (6)
H7B2	0.0909	0.9175	0.8015	0.051*	0.343 (6)
C8B	0.2241 (19)	1.0347 (5)	0.7749 (3)	0.0448 (10)*	0.343 (6)
H8B1	0.3585	1.0573	0.7731	0.054*	0.343 (6)
H8B2	0.1528	1.0711	0.8012	0.054*	0.343 (6)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.05905 (14)	0.04394 (11)	0.05623 (12)	−0.00603 (12)	0.01004 (12)	−0.00782 (8)
Co1	0.02083 (15)	0.02368 (14)	0.03319 (15)	0.00013 (13)	0.00077 (13)	0.00319 (12)

O4	0.0212 (9)	0.0493 (11)	0.0442 (11)	−0.0008 (8)	−0.0001 (8)	0.0009 (9)
O5	0.071 (2)	0.102 (2)	0.0673 (18)	0.0044 (17)	0.0020 (15)	−0.0235 (17)
O1	0.0201 (9)	0.0284 (8)	0.0397 (9)	0.0000 (7)	0.0017 (7)	−0.0011 (7)
O3	0.0312 (11)	0.0498 (12)	0.0613 (13)	−0.0049 (9)	−0.0030 (10)	0.0091 (10)
O2	0.0182 (8)	0.0349 (10)	0.0374 (10)	−0.0007 (7)	−0.0008 (7)	0.0081 (8)
N1A	0.0300 (12)	0.0387 (11)	0.0383 (11)	−0.0002 (11)	0.0025 (10)	0.0069 (9)
N3A	0.0307 (12)	0.0318 (12)	0.0481 (14)	0.0000 (9)	0.0028 (10)	0.0005 (10)
N1B	0.0300 (12)	0.0387 (11)	0.0383 (11)	−0.0002 (11)	0.0025 (10)	0.0069 (9)
N3B	0.0307 (12)	0.0318 (12)	0.0481 (14)	0.0000 (9)	0.0028 (10)	0.0005 (10)
N2	0.0212 (10)	0.0273 (10)	0.0375 (11)	0.0016 (8)	0.0039 (9)	0.0019 (9)
N4	0.0236 (10)	0.0272 (10)	0.0337 (11)	−0.0013 (8)	−0.0006 (8)	0.0036 (9)
C1	0.0241 (12)	0.0294 (12)	0.0328 (13)	−0.0006 (11)	0.0012 (10)	0.0054 (11)
C2	0.0235 (12)	0.0246 (11)	0.0397 (14)	0.0001 (10)	0.0006 (10)	0.0022 (11)
C15	0.0246 (12)	0.0334 (11)	0.0357 (12)	−0.0017 (11)	0.0001 (11)	−0.0017 (9)
C16	0.0481 (17)	0.0389 (13)	0.0422 (14)	0.0119 (16)	0.0066 (16)	0.0038 (11)
C17	0.056 (2)	0.0372 (16)	0.069 (2)	0.0143 (14)	0.0014 (16)	0.0037 (15)
C18	0.0459 (19)	0.0424 (17)	0.080 (3)	0.0035 (15)	0.0101 (17)	−0.0226 (18)
C19	0.063 (2)	0.059 (2)	0.0489 (19)	−0.0081 (17)	0.0134 (16)	−0.0207 (16)
C20	0.0544 (19)	0.0433 (14)	0.0380 (14)	−0.0080 (16)	0.0038 (15)	−0.0040 (11)
C5	0.0192 (11)	0.0264 (12)	0.0358 (13)	0.0003 (10)	0.0016 (9)	0.0054 (11)
C3	0.0271 (14)	0.0319 (12)	0.0619 (17)	−0.0030 (12)	0.0009 (14)	0.0002 (12)
C9	0.0228 (11)	0.0331 (11)	0.0325 (11)	−0.0017 (12)	−0.0034 (10)	0.0023 (9)
C14	0.0344 (14)	0.0350 (14)	0.0420 (15)	0.0013 (11)	0.0035 (12)	0.0076 (12)
C13	0.0398 (16)	0.0389 (16)	0.061 (2)	0.0048 (14)	−0.0013 (14)	0.0165 (15)
C12	0.053 (2)	0.0556 (17)	0.0450 (16)	0.0007 (18)	−0.0038 (16)	0.0191 (13)
C11	0.0536 (19)	0.065 (2)	0.0309 (15)	0.0033 (17)	0.0015 (13)	0.0038 (14)
C10	0.0395 (15)	0.0459 (16)	0.0356 (14)	0.0052 (13)	−0.0034 (12)	−0.0006 (13)
C6	0.0227 (12)	0.0356 (13)	0.0367 (14)	−0.0001 (11)	0.0016 (10)	0.0055 (11)
C4	0.0204 (12)	0.0270 (11)	0.0364 (13)	0.0007 (10)	0.0007 (10)	0.0022 (10)

Geometric parameters (Å, º)

Co1—O1	1.8793 (17)	C17—H17	0.9300
Co1—O2	1.9002 (18)	C18—C19	1.357 (5)
Co1—N2	1.9456 (19)	C18—H18	0.9300
Co1—N4	1.955 (2)	C19—C20	1.385 (4)
Co1—N1A	1.973 (2)	C19—H19	0.9300
Co1—N3A	1.987 (2)	C20—H20	0.9300
O4—C6	1.424 (3)	C5—C6	1.519 (3)
O4—H4	0.899 (17)	C5—C4	1.531 (3)
O5—H2W	0.91 (2)	C5—H5	0.9800
O5—H1W	0.90 (3)	C3—H3A	0.9700
O1—C1	1.419 (3)	C3—H3B	0.9700
O3—C3	1.414 (3)	C9—C14	1.384 (4)
O3—H3	0.919 (18)	C9—C10	1.395 (4)
O2—C4	1.415 (3)	C9—C4	1.517 (3)
N1A—C7A	1.498 (5)	C14—C13	1.389 (4)
N1A—H1A1	0.9000	C14—H14	0.9300

N1A—H1A2	0.9000	C13—C12	1.374 (4)
N3A—C8A	1.448 (5)	C13—H13	0.9300
N3A—H3A1	0.9000	C12—C11	1.373 (4)
N3A—H3A2	0.9000	C12—H12	0.9300
N2—C2	1.490 (3)	C11—C10	1.387 (4)
N2—H2A	0.9000	C11—H11	0.9300
N2—H2B	0.9000	C10—H10	0.9300
N4—C5	1.494 (3)	C6—H6A	0.9700
N4—H4A	0.9000	C6—H6B	0.9700
N4—H4B	0.9000	C4—H4C	0.9800
C1—C15	1.519 (3)	C7A—C8A	1.506 (6)
C1—C2	1.529 (3)	C7A—H7A1	0.9700
C1—H1	0.9800	C7A—H7A2	0.9700
C2—C3	1.532 (4)	C8A—H8A1	0.9700
C2—H2	0.9800	C8A—H8A2	0.9700
C15—C20	1.387 (4)	C7B—C8B	1.487 (10)
C15—C16	1.393 (3)	C7B—H7B1	0.9700
C16—C17	1.385 (4)	C7B—H7B2	0.9700
C16—H16	0.9300	C8B—H8B1	0.9700
C17—C18	1.370 (5)	C8B—H8B2	0.9700
O1—Co1—O2	93.18 (8)	C17—C18—H18	120.1
O1—Co1—N2	85.45 (8)	C18—C19—C20	120.6 (3)
O1—Co1—N4	87.21 (8)	C18—C19—H19	119.7
O1—Co1—N1A	90.85 (8)	C20—C19—H19	119.7
O1—Co1—N3A	175.03 (9)	C19—C20—C15	120.5 (3)
O2—Co1—N2	88.43 (8)	C19—C20—H20	119.8
O2—Co1—N4	85.83 (8)	C15—C20—H20	119.8
O2—Co1—N1A	175.95 (9)	N4—C5—C6	110.6 (2)
O2—Co1—N3A	91.74 (9)	N4—C5—C4	105.27 (19)
N2—Co1—N4	170.41 (9)	C6—C5—C4	113.9 (2)
N2—Co1—N1A	92.23 (10)	N4—C5—H5	109.0
N2—Co1—N3A	93.98 (9)	C6—C5—H5	109.0
N4—Co1—N1A	94.03 (9)	C4—C5—H5	109.0
N4—Co1—N3A	93.87 (9)	O3—C3—C2	110.4 (2)
N1A—Co1—N3A	84.23 (9)	O3—C3—H3A	109.6
C6—O4—H4	103 (2)	C2—C3—H3A	109.6
H2W—O5—H1W	131 (5)	O3—C3—H3B	109.6
C1—O1—Co1	110.63 (14)	C2—C3—H3B	109.6
C3—O3—H3	96 (2)	H3A—C3—H3B	108.1
C4—O2—Co1	109.08 (14)	C14—C9—C10	118.4 (2)
C7A—N1A—Co1	108.2 (2)	C14—C9—C4	122.5 (2)
C7A—N1A—H1A1	110.1	C10—C9—C4	119.0 (2)
Co1—N1A—H1A1	110.1	C9—C14—C13	120.5 (3)
C7A—N1A—H1A2	110.1	C9—C14—H14	119.7
Co1—N1A—H1A2	110.1	C13—C14—H14	119.7
H1A1—N1A—H1A2	108.4	C12—C13—C14	120.5 (3)
C8A—N3A—Co1	111.3 (2)	C12—C13—H13	119.7

C8A—N3A—H3A1	109.4	C14—C13—H13	119.7
Co1—N3A—H3A1	109.4	C11—C12—C13	119.6 (3)
C8A—N3A—H3A2	109.4	C11—C12—H12	120.2
Co1—N3A—H3A2	109.4	C13—C12—H12	120.2
H3A1—N3A—H3A2	108.0	C12—C11—C10	120.4 (3)
C2—N2—Co1	110.22 (14)	C12—C11—H11	119.8
C2—N2—H2A	109.6	C10—C11—H11	119.8
Co1—N2—H2A	109.6	C11—C10—C9	120.4 (3)
C2—N2—H2B	109.6	C11—C10—H10	119.8
Co1—N2—H2B	109.6	C9—C10—H10	119.8
H2A—N2—H2B	108.1	O4—C6—C5	109.7 (2)
C5—N4—Co1	109.41 (14)	O4—C6—H6A	109.7
C5—N4—H4A	109.8	C5—C6—H6A	109.7
Co1—N4—H4A	109.8	O4—C6—H6B	109.7
C5—N4—H4B	109.8	C5—C6—H6B	109.7
Co1—N4—H4B	109.8	H6A—C6—H6B	108.2
H4A—N4—H4B	108.2	O2—C4—C9	113.18 (19)
O1—C1—C15	107.69 (19)	O2—C4—C5	107.0 (2)
O1—C1—C2	106.8 (2)	C9—C4—C5	111.8 (2)
C15—C1—C2	114.8 (2)	O2—C4—H4C	108.3
O1—C1—H1	109.1	C9—C4—H4C	108.3
C15—C1—H1	109.1	C5—C4—H4C	108.3
C2—C1—H1	109.1	N1A—C7A—C8A	106.7 (4)
N2—C2—C1	104.8 (2)	N1A—C7A—H7A1	110.4
N2—C2—C3	110.3 (2)	C8A—C7A—H7A1	110.4
C1—C2—C3	116.8 (2)	N1A—C7A—H7A2	110.4
N2—C2—H2	108.2	C8A—C7A—H7A2	110.4
C1—C2—H2	108.2	H7A1—C7A—H7A2	108.6
C3—C2—H2	108.2	N3A—C8A—C7A	107.0 (4)
C20—C15—C16	118.4 (2)	N3A—C8A—H8A1	110.3
C20—C15—C1	119.9 (2)	C7A—C8A—H8A1	110.3
C16—C15—C1	121.5 (2)	N3A—C8A—H8A2	110.3
C17—C16—C15	120.0 (3)	C7A—C8A—H8A2	110.3
C17—C16—H16	120.0	H8A1—C8A—H8A2	108.6
C15—C16—H16	120.0	C8B—C7B—H7B1	110.1
C18—C17—C16	120.7 (3)	C8B—C7B—H7B2	110.1
C18—C17—H17	119.6	H7B1—C7B—H7B2	108.5
C16—C17—H17	119.6	C7B—C8B—H8B1	110.2
C19—C18—C17	119.8 (3)	C7B—C8B—H8B2	110.2
C19—C18—H18	120.1	H8B1—C8B—H8B2	108.5

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4···O2[i]	0.90 (2)	1.73 (2)	2.594 (2)	159 (3)
O5—H2W···O4	0.91 (2)	1.99 (3)	2.792 (3)	146 (5)
O5—H1W···I1	0.90 (3)	2.91 (4)	3.639 (3)	140 (5)
O3—H3···O1[ii]	0.92 (2)	1.83 (2)	2.714 (3)	160 (4)

N1*A*—H1*A*1···I1ⁱⁱⁱ	0.90	3.26	4.078 (2)	152
N1*A*—H1*A*2···I1ⁱᵛ	0.90	2.92	3.709 (2)	147
N3*A*—H3*A*1···I1	0.90	2.90	3.697 (2)	149
N3*A*—H3*A*2···O4ⁱⁱ	0.90	2.35	3.094 (3)	140
N3*A*—H3*A*2···O5ⁱⁱ	0.90	2.57	3.368 (4)	148
N1*B*—H1*B*2···I1ⁱᵛ	0.90	2.91	3.709 (2)	148
N3*B*—H3*B*1···I1	0.90	2.87	3.697 (2)	154
N3*B*—H3*B*2···O5ⁱⁱ	0.90	2.61	3.368 (4)	142
N3*B*—H3*B*2···O4ⁱⁱ	0.90	2.66	3.094 (3)	111
N2—H2*A*···I1ⁱⁱⁱ	0.90	2.80	3.632 (2)	155
N4—H4*A*···O5	0.90	2.34	3.139 (4)	149
N4—H4*B*···O3ⁱ	0.90	2.41	3.219 (3)	149

Symmetry codes: (i) *x*+1, *y*, *z*; (ii) *x*−1, *y*, *z*; (iii) −*x*, *y*−1/2, −*z*+3/2; (iv) −*x*+1, *y*−1/2, −*z*+3/2.