

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

catena-Poly[[tetraaquacobalt(II)]- μ -2,2'-dihydroxy-5,5'-diazenediyl]dibenzoato]Yu-Hui Tan,^{a*} Yin-bao Li,^a Fa-Ming Ji,^b Ting-Ting Xiong^b and Li-Bin Xia^b

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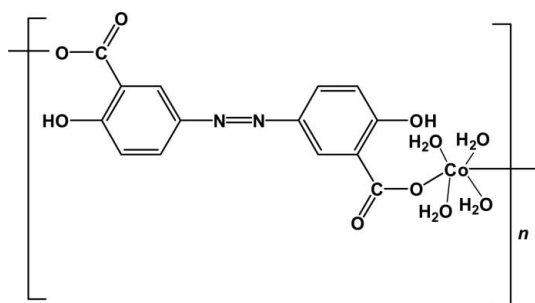
Received 19 November 2007; accepted 19 December 2007

Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.007$ Å; R factor = 0.050; wR factor = 0.133; data-to-parameter ratio = 13.9.

In the title compound, $[\text{Co}(\text{C}_{14}\text{H}_8\text{N}_2\text{O}_6)(\text{H}_2\text{O})_4]_n$, each 5,5'-diazenediylbis(2-hydroxybenzoato) ligand acts as a dicarboxylate bridge, leading to the formation of polymeric chains running in the $[\bar{1}10]$ direction. The Co atom is hexacoordinated in a distorted octahedral geometry by six O atoms [$\text{Co}-\text{O} = 2.039(4)-2.115(4)$ Å] from two ligands and four water molecules. Intermolecular $\text{O}-\text{H}\cdots\text{O}$ and $\text{O}-\text{H}\cdots\text{N}$ hydrogen bonds build up a three-dimensional supramolecular structure.

Related literature

For related literature, see: Klotz (2005); Tang, Tan & Cao (2007); Tang, Tan, Chen *et al.* (2007); Tang, Yang *et al.* (2007); Riordan & Blair (1979).



Experimental

Crystal data

$[\text{Co}(\text{C}_{14}\text{H}_8\text{N}_2\text{O}_6)(\text{H}_2\text{O})_4]$
 $M_r = 431.22$

Monoclinic, $P2_1/c$
 $a = 9.5152(14)$ Å

$b = 11.2452(17)$ Å
 $c = 16.194(2)$ Å
 $\beta = 106.687(2)^\circ$
 $V = 1659.8(4)$ Å³
 $Z = 4$

Mo $K\alpha$ radiation
 $\mu = 1.09$ mm⁻¹
 $T = 296(2)$ K
 $0.10 \times 0.08 \times 0.08$ mm

Data collection

Bruker SMART APEX CCD
area-detector diffractometer
Absorption correction: multi-scan
(*SADABS*; Bruker, 2000)
 $T_{\min} = 0.898$, $T_{\max} = 0.917$

11094 measured reflections
3427 independent reflections
1976 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.062$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.049$
 $wR(F^2) = 0.133$
 $S = 0.94$
3427 reflections

246 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.50$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.44$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O1W}-\text{H1WA}\cdots\text{O6}^i$	0.82	1.89	2.667 (5)	157
$\text{O1W}-\text{H1WB}\cdots\text{N1}^{ii}$	0.82	2.08	2.871 (5)	161
$\text{O2W}-\text{H2WA}\cdots\text{O2}$	0.82	1.84	2.629 (5)	161
$\text{O3W}-\text{H3WA}\cdots\text{O2}^i$	0.82	1.88	2.682 (4)	167
$\text{O3W}-\text{H3WB}\cdots\text{O4}^{iii}$	0.81	2.23	2.899 (5)	141
$\text{O4W}-\text{H4WA}\cdots\text{N2}^i$	0.82	2.35	3.074 (5)	148
$\text{O4W}-\text{H4WB}\cdots\text{O6}^{iv}$	0.82	1.88	2.665 (4)	159
$\text{O3}-\text{H3A}\cdots\text{O1}$	0.82	1.80	2.521 (5)	147
$\text{O4}-\text{H4A}\cdots\text{O5}$	0.82	1.82	2.541 (4)	147

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

This work was supported by the Gannan Medical University Masters Development Foundation.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2293).

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Tang, Y. Z., Yang, S. P., Tan, Y. H., Chen, S. H., Cao, Y. W. & Wang, P. (2007). *Chin. J. Inorg. Chem.* **23**, 70–74.

supporting information

Acta Cryst. (2008). E64, m274 [doi:10.1107/S1600536807067827]

catena-Poly[[tetraaquacobalt(II)]- μ -2,2'-dihydroxy-5,5'-diazenediyldibenzoato]

Yu-Hui Tan, Yin-bao Li, Fa-Ming Ji, Ting-Ting Xiong and Li-Bin Xia

S1. Comment

Olsalazine - 3,3-azobis(6-hydroxybenzoic acid) - has been widely used to prevent and treat the inflammatory bowel diseases, such as ulcerative colitis (Klotz, 2005). In previous work, we have synthesized a serial of Zn (Tang, Tan, Chen & Cao, 2007), Cd and Co (Tang, Yang *et al.*, 2007) complexes with phenanthroline as auxiliary ligand. Also we have reported a Mn complex of olsalazine (Tang, Tan & Cao, 2007), however the cobalt complex with single olsalazine as building block have not been reported yet, Here we reported the crystal structure of the title compound, (I) - a new cobalt complex of olsalazine.

In (I), the Co atom is hexacoordinated (Fig. 1) by two O atoms from two *L* ligands ($H_2L=3,3$ -azo-bis(6-hydroxybenzoic acid)) *cis* to each other and four water molecules in a distorted octahedral geometry. Each ligand *L* acts as a carboxylate bridge, that leads to the formation of polymeric chain running in the [-110] direction. Intermolecular O—H \cdots O and O—H \cdots N hydrogen bonds build up a three dimensional supramolecular structure (Table 1).

S2. Refinement

All H atoms attached to C atoms and O(hydroxyl) atom were fixed geometrically and treated as riding with C—H = 0.93 Å and O—H = 0.86 Å with $U_{iso}(H) = 1.2U_{eq}(C)$ or $U_{iso}(H) = 1.5U_{eq}(O)$. H atoms of water molecule were located in difference Fourier maps and included in the subsequent refinement using restraints (O—H = 0.82 (1) Å and H \cdots H = 1.34 (2) Å) with $U_{iso}(H) = 1.5U_{eq}(O)$. In the last stage of refinement, the H atoms were treated as riding on their parent O atoms.

The crystal used was twinned with two domains in the ratio 0.076/0.924. The twin law is [1.00 0.00 0.00 0.00 - 1.00 0.00 - 1.00 0.00 - 1.00].

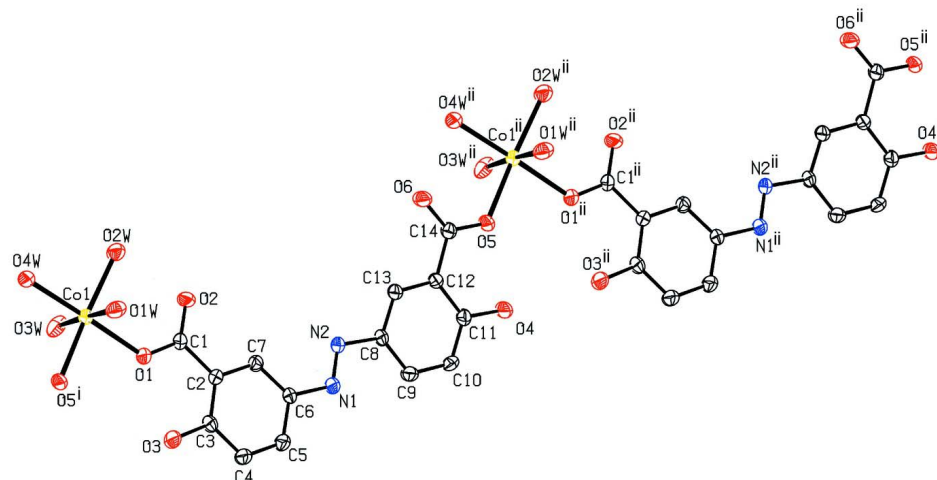


Figure 1

Partial view of the polymeric chain in (I), showing the atom-labelling-scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity. [Symmetry codes: (i) $x + 1, y - 1, z$; (ii) $x - 1, y + 1, z$]

catena-Poly[[tetraaquacobalt(II)]- μ -2,2'-dihydroxy-5,5'- diazenediylidibenzoato]

Crystal data

[Co(C₁₄H₈N₂O₆)(H₂O)₄]

$M_r = 431.22$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 9.5152 (14) \text{ \AA}$

$b = 11.2452 (17) \text{ \AA}$

$c = 16.194 (2) \text{ \AA}$

$\beta = 106.687 (2)^\circ$

$V = 1659.8 (4) \text{ \AA}^3$

$Z = 4$

$F(000) = 884$

$D_x = 1.726 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 935 reflections

$\theta = 2.2\text{--}26^\circ$

$\mu = 1.10 \text{ mm}^{-1}$

$T = 296 \text{ K}$

Block, red

$0.10 \times 0.08 \times 0.08 \text{ mm}$

Data collection

Bruker SMART APEX CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2000)

$T_{\min} = 0.898, T_{\max} = 0.918$

11094 measured reflections

3427 independent reflections

1976 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.062$

$\theta_{\max} = 26.4^\circ, \theta_{\min} = 1.8^\circ$

$h = -11 \rightarrow 11$

$k = -14 \rightarrow 14$

$l = -19 \rightarrow 20$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.049$

$wR(F^2) = 0.133$

$S = 0.94$

3427 reflections

246 parameters

0 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0717P)^2]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} = 0.001$$

$$\Delta\rho_{\max} = 0.50 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.44 \text{ e } \text{\AA}^{-3}$$

Extinction correction: *SHELXL97* (Sheldrick, 1997), $F_c^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0050 (11)

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Co1	0.71433 (7)	0.14001 (5)	0.21254 (4)	0.0333 (2)
O1W	0.8824 (4)	0.2527 (3)	0.2134 (2)	0.0515 (9)
H1WA	0.9251	0.3002	0.2506	0.077*
H1WB	0.8799	0.2780	0.1654	0.077*
O2W	0.6621 (4)	0.2503 (3)	0.3053 (2)	0.0537 (10)
H2WA	0.5912	0.2842	0.2726	0.080*
H2WB	0.6352	0.2127	0.3415	0.080*
O3W	0.5405 (4)	0.0277 (3)	0.2068 (2)	0.0575 (10)
H3WA	0.5494	-0.0263	0.2418	0.086*
H3WB	0.5144	0.0028	0.1579	0.086*
O4W	0.8418 (4)	0.0398 (3)	0.31539 (19)	0.0455 (9)
H4WA	0.8933	0.0663	0.3610	0.068*
H4WB	0.8900	-0.0006	0.2912	0.068*
N2	0.0915 (4)	0.6415 (3)	0.0012 (2)	0.0366 (9)
N1	0.1224 (4)	0.6059 (3)	-0.0651 (2)	0.0374 (10)
O1	0.5775 (4)	0.2345 (3)	0.10743 (19)	0.0402 (8)
O2	0.4732 (4)	0.3607 (3)	0.1776 (2)	0.0542 (10)
O3	0.5168 (4)	0.2395 (3)	-0.0547 (2)	0.0609 (11)
H3A	0.5587	0.2174	-0.0055	0.091*
O4	-0.3150 (4)	1.0087 (3)	-0.0450 (2)	0.0533 (10)
H4A	-0.3126	1.0395	0.0012	0.080*
O5	-0.2290 (4)	1.0400 (3)	0.1171 (2)	0.0455 (9)
O6	-0.0537 (4)	0.9267 (3)	0.2009 (2)	0.0637 (12)
C1	0.4906 (5)	0.3199 (4)	0.1097 (3)	0.0358 (11)
C2	0.4027 (5)	0.3698 (4)	0.0249 (3)	0.0326 (10)
C3	0.4191 (6)	0.3262 (4)	-0.0520 (3)	0.0399 (12)
C4	0.3349 (6)	0.3724 (4)	-0.1303 (3)	0.0523 (15)
H4	0.3427	0.3408	-0.1819	0.063*
C5	0.2420 (5)	0.4632 (4)	-0.1309 (3)	0.0441 (13)

H5	0.1885	0.4954	-0.1835	0.053*
C6	0.2240 (5)	0.5101 (4)	-0.0547 (3)	0.0346 (11)
C7	0.3054 (5)	0.4617 (4)	0.0228 (3)	0.0356 (11)
H7	0.2944	0.4914	0.0742	0.043*
C8	-0.0120 (5)	0.7371 (4)	-0.0142 (3)	0.0348 (11)
C9	-0.1058 (5)	0.7662 (4)	-0.0956 (3)	0.0414 (12)
H9	-0.1024	0.7229	-0.1439	0.050*
C10	-0.2027 (6)	0.8587 (4)	-0.1037 (3)	0.0446 (12)
H10	-0.2621	0.8798	-0.1580	0.054*
C11	-0.2134 (5)	0.9212 (4)	-0.0320 (3)	0.0386 (12)
C12	-0.1231 (5)	0.8923 (4)	0.0500 (3)	0.0351 (11)
C13	-0.0236 (6)	0.8005 (4)	0.0566 (3)	0.0419 (12)
H13	0.0377	0.7808	0.1108	0.050*
C14	-0.1338 (5)	0.9566 (4)	0.1286 (3)	0.0403 (12)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Co1	0.0379 (4)	0.0295 (3)	0.0312 (3)	0.0035 (3)	0.0078 (3)	0.0014 (3)
O1W	0.060 (2)	0.055 (2)	0.0364 (19)	-0.0171 (19)	0.0088 (17)	-0.0010 (17)
O2W	0.061 (3)	0.060 (2)	0.040 (2)	0.023 (2)	0.0160 (18)	0.0057 (17)
O3W	0.054 (2)	0.061 (2)	0.047 (2)	-0.015 (2)	-0.0007 (17)	0.0173 (19)
O4W	0.054 (2)	0.044 (2)	0.0359 (18)	0.0113 (17)	0.0085 (16)	-0.0011 (15)
N2	0.042 (2)	0.0304 (19)	0.035 (2)	0.0096 (19)	0.0072 (18)	0.0031 (19)
N1	0.039 (3)	0.036 (2)	0.038 (2)	0.0101 (18)	0.0122 (18)	0.0017 (17)
O1	0.048 (2)	0.0362 (18)	0.0361 (18)	0.0122 (16)	0.0121 (16)	0.0027 (15)
O2	0.070 (3)	0.055 (2)	0.0340 (18)	0.028 (2)	0.0094 (18)	-0.0057 (17)
O3	0.080 (3)	0.062 (2)	0.042 (2)	0.045 (2)	0.019 (2)	0.0083 (18)
O4	0.061 (2)	0.052 (2)	0.0403 (19)	0.0296 (19)	0.0029 (17)	-0.0053 (17)
O5	0.054 (2)	0.0407 (19)	0.0385 (19)	0.0189 (18)	0.0075 (16)	-0.0045 (16)
O6	0.079 (3)	0.067 (3)	0.035 (2)	0.043 (2)	0.0014 (19)	-0.0079 (19)
C1	0.039 (3)	0.030 (2)	0.037 (3)	0.004 (2)	0.008 (2)	0.004 (2)
C2	0.036 (3)	0.031 (2)	0.030 (2)	0.002 (2)	0.009 (2)	0.003 (2)
C3	0.043 (3)	0.035 (3)	0.045 (3)	0.014 (2)	0.018 (2)	0.000 (2)
C4	0.071 (4)	0.052 (3)	0.033 (3)	0.025 (3)	0.014 (3)	0.003 (3)
C5	0.050 (3)	0.045 (3)	0.035 (3)	0.017 (3)	0.008 (2)	0.006 (2)
C6	0.041 (3)	0.028 (2)	0.035 (3)	0.007 (2)	0.012 (2)	0.001 (2)
C7	0.043 (3)	0.031 (2)	0.037 (3)	0.003 (2)	0.017 (2)	0.004 (2)
C8	0.036 (3)	0.030 (2)	0.039 (3)	0.010 (2)	0.012 (2)	0.001 (2)
C9	0.045 (3)	0.047 (3)	0.031 (3)	0.010 (2)	0.009 (2)	-0.005 (2)
C10	0.055 (3)	0.048 (3)	0.026 (2)	0.015 (3)	0.004 (2)	0.004 (2)
C11	0.041 (3)	0.033 (2)	0.039 (3)	0.006 (2)	0.006 (2)	0.001 (2)
C12	0.037 (3)	0.032 (3)	0.033 (2)	0.005 (2)	0.004 (2)	-0.0027 (19)
C13	0.050 (3)	0.037 (3)	0.034 (3)	0.014 (2)	0.003 (2)	-0.002 (2)
C14	0.038 (3)	0.039 (3)	0.038 (3)	0.012 (2)	0.003 (2)	-0.002 (2)

Geometric parameters (Å, °)

Co1—O1W	2.037 (3)	O5—C14	1.280 (5)
Co1—O3W	2.062 (3)	O5—Co1 ⁱⁱ	2.103 (3)
Co1—O4W	2.087 (3)	O6—C14	1.245 (5)
Co1—O5 ⁱ	2.103 (3)	C1—C2	1.497 (6)
Co1—O1	2.110 (3)	C2—C7	1.381 (6)
Co1—O2W	2.115 (3)	C2—C3	1.388 (6)
O1W—H1WA	0.8195	C3—C4	1.391 (7)
O1W—H1WB	0.8219	C4—C5	1.349 (6)
O2W—H2WA	0.8220	C4—H4	0.9300
O2W—H2WB	0.8212	C5—C6	1.397 (6)
O3W—H3WA	0.8183	C5—H5	0.9300
O3W—H3WB	0.8089	C6—C7	1.383 (6)
O4W—H4WA	0.8158	C7—H7	0.9300
O4W—H4WB	0.8205	C8—C13	1.383 (6)
N2—N1	1.257 (5)	C8—C9	1.401 (6)
N2—C8	1.430 (6)	C9—C10	1.371 (6)
N1—C6	1.426 (6)	C9—H9	0.9300
O1—C1	1.275 (5)	C10—C11	1.385 (6)
O2—C1	1.245 (5)	C10—H10	0.9300
O3—C3	1.357 (5)	C11—C12	1.398 (6)
O3—H3A	0.8200	C12—C13	1.383 (6)
O4—C11	1.353 (5)	C12—C14	1.493 (6)
O4—H4A	0.8200	C13—H13	0.9300
O1W—Co1—O3W	177.81 (14)	C3—C2—C1	120.9 (4)
O1W—Co1—O4W	93.15 (13)	O3—C3—C2	122.5 (4)
O3W—Co1—O4W	88.88 (13)	O3—C3—C4	117.4 (4)
O1W—Co1—O5 ⁱ	88.37 (14)	C2—C3—C4	120.2 (4)
O3W—Co1—O5 ⁱ	90.63 (15)	C5—C4—C3	119.6 (4)
O4W—Co1—O5 ⁱ	94.86 (12)	C5—C4—H4	120.2
O1W—Co1—O1	89.64 (14)	C3—C4—H4	120.2
O3W—Co1—O1	88.33 (13)	C4—C5—C6	121.7 (4)
O4W—Co1—O1	177.14 (13)	C4—C5—H5	119.2
O5 ⁱ —Co1—O1	84.59 (12)	C6—C5—H5	119.2
O1W—Co1—O2W	88.52 (15)	C7—C6—C5	118.3 (4)
O3W—Co1—O2W	92.42 (15)	C7—C6—N1	126.0 (4)
O4W—Co1—O2W	87.20 (13)	C5—C6—N1	115.6 (4)
O5 ⁱ —Co1—O2W	176.36 (14)	C2—C7—C6	120.9 (4)
O1—Co1—O2W	93.49 (13)	C2—C7—H7	119.6
Co1—O1W—H1WA	129.4	C6—C7—H7	119.6
Co1—O1W—H1WB	113.6	C13—C8—C9	118.6 (4)
H1WA—O1W—H1WB	110.0	C13—C8—N2	117.2 (4)
Co1—O2W—H2WA	97.5	C9—C8—N2	124.1 (4)
Co1—O2W—H2WB	112.9	C10—C9—C8	119.8 (4)
H2WA—O2W—H2WB	109.5	C10—C9—H9	120.1
Co1—O3W—H3WA	120.1	C8—C9—H9	120.1

Co1—O3W—H3WB	105.9	C9—C10—C11	120.9 (4)
H3WA—O3W—H3WB	111.2	C9—C10—H10	119.5
Co1—O4W—H4WA	125.8	C11—C10—H10	119.5
Co1—O4W—H4WB	101.2	O4—C11—C10	117.4 (4)
H4WA—O4W—H4WB	111.2	O4—C11—C12	122.3 (4)
N1—N2—C8	114.1 (3)	C10—C11—C12	120.3 (4)
N2—N1—C6	117.2 (4)	C13—C12—C11	117.9 (4)
C1—O1—Co1	127.7 (3)	C13—C12—C14	120.5 (4)
C3—O3—H3A	109.5	C11—C12—C14	121.6 (4)
C11—O4—H4A	109.5	C8—C13—C12	122.5 (4)
C14—O5—Co1 ⁱⁱ	127.3 (3)	C8—C13—H13	118.8
O2—C1—O1	123.7 (4)	C12—C13—H13	118.8
O2—C1—C2	119.4 (4)	O6—C14—O5	123.4 (4)
O1—C1—C2	116.9 (4)	O6—C14—C12	119.9 (4)
C7—C2—C3	119.3 (4)	O5—C14—C12	116.7 (4)
C7—C2—C1	119.8 (4)		

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

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1W—H1WA \cdots O6 ⁱⁱⁱ	0.82	1.89	2.667 (5)	157
O1W—H1WB \cdots N1 ^{iv}	0.82	2.08	2.871 (5)	161
O2W—H2WA \cdots O2	0.82	1.84	2.629 (5)	161
O3W—H3WA \cdots O2 ⁱⁱⁱ	0.82	1.88	2.682 (4)	167
O3W—H3WB \cdots O4 ^v	0.81	2.23	2.899 (5)	141
O4W—H4WA \cdots N2 ⁱⁱⁱ	0.82	2.35	3.074 (5)	148
O4W—H4WB \cdots O6 ⁱ	0.82	1.88	2.665 (4)	159
O3—H3A \cdots O1	0.82	1.80	2.521 (5)	147
O4—H4A \cdots O5	0.82	1.82	2.541 (4)	147

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