

# Tris[hexaamminecobalt(III)] bis[tri-oxalatocobaltate(II)] chloride dodecahydrate

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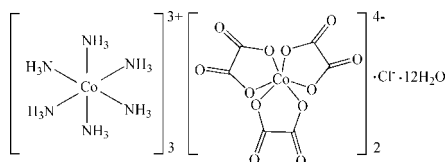
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Key indicators: single-crystal X-ray study;  $T = 296$  K; mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å; disorder in solvent or counterion;  $R$  factor = 0.036;  $wR$  factor = 0.108; data-to-parameter ratio = 16.7.

The title compound,  $[\text{Co}^{\text{III}}(\text{NH}_3)_6]_3[\text{Co}^{\text{II}}(\text{C}_2\text{O}_4)_3]_2\text{Cl}\cdot 12\text{H}_2\text{O}$ , was synthesized under hydrothermal conditions. The asymmetric unit comprises two  $[\text{Co}(\text{NH}_3)_6]^{3+}$  cations, one located on a threefold axis and the other on a site of symmetry  $\bar{3}$ , a  $[\text{Co}(\text{C}_2\text{O}_4)_3]^{4-}$  anion, located on a threefold axis, one sixth of a chloride anion [disordered over two sites, one threefold (site occupancy = 0.5) and the other  $\bar{3}$  (site occupancy (0.25))] and two water molecules. Both  $\text{Co}^{\text{III}}$  centers are six-coordinated by  $\text{NH}_3$  molecules, forming  $[\text{Co}(\text{NH}_3)_6]^{3+}$  octahedra, with  $\text{Co}-\text{N}$  distances in the range 1.958 (2)–1.977 (3) Å. The title structure gives the first example of the  $[\text{Co}(\text{C}_2\text{O}_4)_3]^{4-}$  anion, with the distorted octahedral environment of  $\text{Co}^{\text{II}}$  center formed by six O atoms from three oxalate residues. The  $\text{Co}-\text{O}$  bond lengths are 2.0817 (18) to 2.0979 (18) Å. Multiple  $\text{N}-\text{H}\cdots\text{O}$ ,  $\text{N}-\text{H}\cdots\text{Cl}$  and  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonds link the cations, anions and water molecules into a three-dimensional network.

## Related literature

For metal phosphates and germanates templated by metal complexes, see: Wang *et al.* (2003, 2006); Pan *et al.* (2005, 2008). For our continued research interest, see: Pan *et al.* (2010*a,b*, 2011). For a compound containing the  $[\text{Co}^{\text{III}}(\text{NH}_3)_6]^{3+}$  cation, see: Wu *et al.* (2012).



## Experimental

### Crystal data

$[\text{Co}(\text{NH}_3)_6][\text{Co}(\text{C}_2\text{O}_4)_3]\text{Cl}\cdot 12\text{H}_2\text{O}$   
 $M_r = 1381.02$   
 Trigonal,  $P\bar{3}$   
 $a = 12.2138$  (4) Å  
 $c = 9.9090$  (8) Å  
 $V = 1280.15$  (12) Å<sup>3</sup>  
 $Z = 1$   
 Mo  $K\alpha$  radiation  
 $\mu = 1.75$  mm<sup>-1</sup>  
 $T = 296$  K  
 $0.30 \times 0.15 \times 0.15$  mm

### Data collection

Bruker APEXII CCD area-detector diffractometer  
 Absorption correction: multi-scan (*SADABS*; Bruker, 2005)  
 $T_{\text{min}} = 0.737$ ,  $T_{\text{max}} = 0.769$   
 7736 measured reflections  
 1923 independent reflections  
 1514 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.030$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.036$   
 $wR(F^2) = 0.108$   
 $S = 1.16$   
 1923 reflections  
 115 parameters  
 1 restraint  
 H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.61$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -1.09$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> — <i>H</i> ⋯ <i>A</i>	<i>D</i> — <i>H</i>	<i>H</i> ⋯ <i>A</i>	<i>D</i> ⋯ <i>A</i>	<i>D</i> — <i>H</i> ⋯ <i>A</i>
N1—H1A⋯Cl1 <sup>i</sup>	0.89	2.62	3.176 (15)	121
N1—H1A⋯O1 <sup>i</sup>	0.89	2.27	3.055 (4)	147
N1—H1B⋯O2 <sup>iii</sup>	0.89	2.40	2.950 (4)	120
N1—H1B⋯O2 <sup>iii</sup>	0.89	2.52	3.151 (4)	128
N2—H2⋯O4 <sup>iv</sup>	0.91	2.09	2.993 (3)	171
N2—H2A⋯O1W	0.91	2.18	3.047 (4)	160
N2—H2B⋯O2W	0.91	2.15	2.958 (3)	147
N3—H3⋯O3 <sup>v</sup>	0.91	2.09	2.988 (3)	172
N3—H3A⋯O2W	0.91	2.19	3.051 (3)	157
N3—H3B⋯O1W	0.91	2.36	3.120 (4)	141
O1W—H1W⋯O1	0.87	2.13	2.971 (4)	162
O1W—H1WA⋯O1 <sup>vi</sup>	0.87	2.32	2.973 (4)	132
O2W—H2W⋯O2 <sup>vii</sup>	0.87	1.97	2.830 (3)	171
O2W—H2WA⋯O4 <sup>viii</sup>	0.87	2.06	2.868 (3)	154

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT* 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: YK2060).

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## supplementary materials

*Acta Cryst.* (2012). E68, m914–m915 [doi:10.1107/S1600536812026414]

**Tris[hexaamminecobalt(III)] bis[trioxalatocobaltate(II)] chloride dodecahydrate****Ruijing Tian, Yan Yan, Cailing Zhang, Liyan Wang and Qinhe Pan****Comment**

Recently, more attention has been paid to employ transition metal complexes as templates for supramolecular structures, because they are versatile and can be made with a wide variety of shapes and charges. Up to now, transition metal cTris[hexaamminecobalt(III)] bis[trioxalatocobaltate(II)] chloride dodecahydrate complexes have been introduced into the synthesis of various open-framework materials, including metal phosphates (Wang *et al.*, 2003,2006), germanates (Pan *et al.*, 2005,2008). Our continued interest has been focused on the synthesis of microporous open-framework metal-organic structures by introducing transition metal complexes as templates (Pan *et al.*, 2010*a,b*,2011). Unexpectedly, in the reaction of  $\text{Co}(\text{OAc})_2 \cdot 4\text{H}_2\text{O}$ ,  $\text{Co}(\text{NH}_3)_6\text{Cl}_3$ , and  $\text{K}_2\text{C}_2\text{O}_4$  the title compound,  $[\text{Co}^{\text{III}}(\text{NH}_3)_6]_3[\text{Co}^{\text{II}}(\text{C}_2\text{O}_4)_3]_2 \cdot \text{Cl} \cdot 12\text{H}_2\text{O}$ , was obtained.

The title compound is composed of  $[\text{Co}(\text{NH}_3)_6]^{3+}$  cations, the counterions  $[\text{Co}(\text{C}_2\text{O}_4)_3]^{4-}$  and  $\text{Cl}^-$  anions, and two water molecules, as shown in Figure 1. The crystal structure contains two  $\text{Co}^{\text{III}}$  centers, one is located on a threefold rotation axis, and the other is located at a  $\bar{3}$  position. Each  $\text{Co}^{\text{III}}$  center is six-coordinated by  $\text{NH}_3$  molecules to form octahedral  $[\text{Co}(\text{NH}_3)_6]^{3+}$  cations, similar to that observed in  $[\text{Co}(\text{NH}_3)_6]_2(\text{NO}_3)\text{Cl}_5$  (Wu *et al.*, 2012). The  $\text{Co}-\text{N}$  distances are in the range 1.958 (2)–1.977 (3) Å. The crystal structure also contains one  $\text{Co}^{\text{II}}$  center, which is located on a threefold rotation axis. It is coordinated by six O atoms from three different oxalate residues to form  $[\text{Co}(\text{C}_2\text{O}_4)_3]^{4-}$  anion having a slightly distorted octahedral geometry, with the distances  $\text{Co}-\text{O}$  ranging from 2.0804 (17) to 2.0968 (17) Å. The  $\text{Cl}^-$  anion is splitted into two positions, Cl1 and Cl1'. The  $[\text{Co}(\text{III})(\text{NH}_3)_6]^{3+}$  cations,  $[\text{Co}(\text{II})(\text{C}_2\text{O}_4)_3]^{4-}$  anions,  $\text{Cl}^-$  anions, and water molecules form an extensive hydrogen-bonding network, with the distance of  $\text{N}-\text{H}\cdots\text{N}$  hydrogen bonds of 2.751 (4) Å, the distance of  $\text{N}-\text{H}\cdots\text{Cl}$  hydrogen bonds of 3.169 (9) Å, the distance of  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds in the range of 2.959 (3)–3.147 (3) Å, and the distance of  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonds in the range of 2.829 (3)–2.973 (4) Å (Table 1).

**Experimental**

In a typical synthesis, a mixture of  $\text{Co}(\text{OAc})_2 \cdot 4\text{H}_2\text{O}$  (0.250 g),  $\text{Co}(\text{NH}_3)_6\text{Cl}_3$  (0.1 g),  $\text{K}_2\text{C}_2\text{O}_4$  (0.276 g), and  $\text{H}_2\text{O}$  (5 ml), was placed into a 20 ml Teflon-lined reactor and heated to 100 °C for 3 days under autogenous pressure. Orange rod-like crystals were obtained.

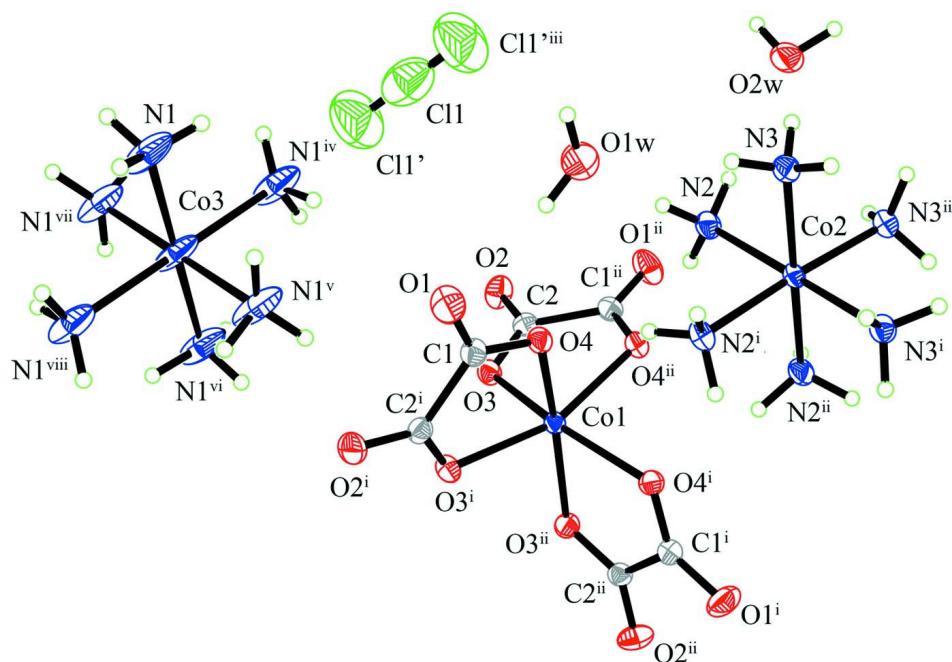
**Refinement**

All H atoms were positioned geometrically ( $\text{N}-\text{H} = 0.91$  Å,  $\text{O}-\text{H} = 0.87$  Å) and allowed to ride on their parent atoms, with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{parent atom})$ .

**Computing details**

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT* (Bruker, 2005); 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 (Sheldrick, 2008).



**Figure 1**

A view of the asymmetric unit of title compound. Ellipsoids are drawn at the 50% probability level. [Symmetry codes: (i)  $1-y, x-y, z$ ; (ii)  $1-x+y, 1-x, z$ ; (iii)  $-x, -y, 1-z$ ; (iv)  $-x+y, -x, z$ ; (v)  $-y, x-y, z$ ; (vi)  $-x, -y, -z$ ; (vii)  $y, -x+y, -z$ ; (viii)  $x-y, x, -z$ .

**Tris[hexaamminecobalt(III)] bis[trioxalatocobaltate(II)] chloride dodecahydrate**

*Crystal data*

$[\text{Co}(\text{NH}_3)_6][\text{Co}(\text{C}_2\text{O}_4)_3]\text{Cl}\cdot 12\text{H}_2\text{O}$

$M_r = 1381.02$

Trigonal,  $P\bar{3}$

Hall symbol:  $-P\ 3$

$a = 12.2138\ (4)\ \text{\AA}$

$c = 9.9090\ (8)\ \text{\AA}$

$V = 1280.15\ (12)\ \text{\AA}^3$

$Z = 1$

$F(000) = 716$

$D_x = 1.791\ \text{Mg m}^{-3}$

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

Cell parameters from 7736 reflections

$\theta = 2.1\text{--}27.2^\circ$

$\mu = 1.75\ \text{mm}^{-1}$

$T = 296\ \text{K}$

Rod, orange

$0.30 \times 0.15 \times 0.15\ \text{mm}$

*Data collection*

Bruker APEXII CCD area-detector  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution:  $5.00\ \text{pixels mm}^{-1}$

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2005)

$T_{\min} = 0.737, T_{\max} = 0.769$

7736 measured reflections

1923 independent reflections

1514 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.030$

$\theta_{\max} = 27.2^\circ, \theta_{\min} = 2.1^\circ$

$h = -8 \rightarrow 15$

$k = -15 \rightarrow 14$

$l = -12 \rightarrow 12$

Refinement

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.036$	H-atom parameters constrained
$wR(F^2) = 0.108$	$w = 1/[\sigma^2(F_o^2) + (0.0482P)^2 + 0.6892P]$
$S = 1.16$	where $P = (F_o^2 + 2F_c^2)/3$
1923 reflections	$(\Delta/\sigma)_{\max} = 0.011$
115 parameters	$\Delta\rho_{\max} = 0.61 \text{ e } \text{\AA}^{-3}$
1 restraint	$\Delta\rho_{\min} = -1.09 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	

Special details

**Geometry.** All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 ( $\text{\AA}^2$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Co1	0.6667	0.3333	0.14192 (6)	0.02287 (18)	
Co2	0.6667	0.3333	0.64732 (5)	0.02393 (18)	
Co3	0.0000	0.0000	0.0000	0.0646 (5)	
Cl1	0.0000	0.0000	0.5000	0.085 (5)	0.498 (19)
Cl1'	0.0000	0.0000	0.3949 (17)	0.118 (4)	0.251 (9)
O1	0.3041 (2)	0.0971 (2)	0.2657 (2)	0.0530 (6)	
O2	0.6796 (2)	0.65788 (19)	0.0164 (2)	0.0454 (6)	
O3	0.64285 (18)	0.45991 (18)	0.02214 (18)	0.0311 (4)	
O4	0.50524 (17)	0.24996 (18)	0.26488 (18)	0.0291 (4)	
N1	-0.1492 (2)	-0.0618 (3)	0.1172 (4)	0.0725 (11)	
H1A	-0.1436	-0.1075	0.1840	0.087*	
H1B	-0.2187	-0.1096	0.0692	0.087*	
H1C	-0.1528	0.0037	0.1514	0.087*	
N2	0.6170 (2)	0.4327 (2)	0.5344 (2)	0.0328 (5)	
H2B	0.6232	0.4999	0.5808	0.039*	
H2A	0.5333	0.3879	0.5132	0.039*	
H2	0.6626	0.4599	0.4568	0.039*	
N3	0.5188 (2)	0.2924 (2)	0.7604 (2)	0.0307 (5)	
H3B	0.4436	0.2449	0.7175	0.037*	
H3A	0.5208	0.3659	0.7815	0.037*	
H3	0.5185	0.2514	0.8372	0.037*	
C1	0.4097 (3)	0.1551 (3)	0.2134 (3)	0.0310 (6)	
C2	0.6835 (2)	0.5688 (2)	0.0718 (3)	0.0288 (6)	
O1W	0.3354 (2)	0.2406 (3)	0.5187 (3)	0.0747 (9)	
H1WA	0.2644	0.2152	0.5612	0.090*	
H1W	0.3234	0.1853	0.4567	0.090*	

O2W	0.5721 (2)	0.5645 (2)	0.7595 (2)	0.0523 (6)
H2WA	0.5434	0.6113	0.7265	0.063*
H2W	0.5970	0.5901	0.8416	0.063*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Co1	0.0244 (2)	0.0244 (2)	0.0198 (3)	0.01220 (11)	0.000	0.000
Co2	0.0275 (2)	0.0275 (2)	0.0167 (3)	0.01377 (12)	0.000	0.000
Co3	0.0176 (3)	0.0176 (3)	0.1585 (15)	0.00881 (16)	0.000	0.000
Cl1	0.062 (2)	0.062 (2)	0.131 (13)	0.0310 (12)	0.000	0.000
Cl1'	0.148 (7)	0.148 (7)	0.060 (8)	0.074 (4)	0.000	0.000
O1	0.0294 (11)	0.0621 (15)	0.0463 (13)	0.0069 (10)	0.0097 (10)	-0.0024 (11)
O2	0.0507 (13)	0.0307 (11)	0.0548 (14)	0.0203 (10)	-0.0105 (11)	0.0070 (9)
O3	0.0394 (11)	0.0309 (10)	0.0263 (10)	0.0201 (9)	-0.0026 (8)	0.0002 (8)
O4	0.0269 (9)	0.0335 (10)	0.0246 (9)	0.0133 (8)	0.0009 (7)	-0.0008 (7)
N1	0.0262 (14)	0.0270 (14)	0.164 (4)	0.0127 (12)	0.0017 (18)	0.0026 (18)
N2	0.0411 (13)	0.0394 (13)	0.0220 (11)	0.0232 (11)	0.0011 (10)	0.0031 (9)
N3	0.0322 (12)	0.0354 (12)	0.0251 (11)	0.0172 (10)	0.0016 (9)	0.0010 (9)
C1	0.0286 (14)	0.0331 (14)	0.0308 (15)	0.0150 (12)	0.0000 (11)	0.0026 (11)
C2	0.0260 (13)	0.0287 (13)	0.0307 (14)	0.0129 (11)	0.0032 (11)	0.0031 (11)
O1W	0.0478 (15)	0.107 (2)	0.0662 (18)	0.0367 (16)	-0.0013 (13)	-0.0213 (17)
O2W	0.0805 (18)	0.0623 (15)	0.0407 (13)	0.0555 (15)	-0.0056 (12)	-0.0047 (11)

Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

Co1—O3 <sup>i</sup>	2.0817 (18)	Cl1'—Cl1 <sup>viii</sup>	2.08 (3)
Co1—O3 <sup>ii</sup>	2.0817 (18)	O1—C1	1.233 (3)
Co1—O3	2.0817 (18)	O2—C2	1.240 (3)
Co1—O4 <sup>ii</sup>	2.0979 (18)	O3—C2	1.264 (3)
Co1—O4 <sup>i</sup>	2.0979 (18)	O4—C1	1.270 (3)
Co1—O4	2.0979 (18)	N1—H1A	0.8900
Co2—N2 <sup>i</sup>	1.957 (2)	N1—H1B	0.8900
Co2—N2 <sup>ii</sup>	1.957 (2)	N1—H1C	0.8900
Co2—N2	1.957 (2)	N2—H2B	0.9101
Co2—N3 <sup>ii</sup>	1.966 (2)	N2—H2A	0.9100
Co2—N3 <sup>i</sup>	1.966 (2)	N2—H2	0.9100
Co2—N3	1.966 (2)	N3—H3B	0.9100
Co3—N1 <sup>iii</sup>	1.965 (3)	N3—H3A	0.9099
Co3—N1 <sup>iv</sup>	1.965 (3)	N3—H3	0.9100
Co3—N1 <sup>v</sup>	1.965 (3)	C1—C2 <sup>ii</sup>	1.553 (4)
Co3—N1	1.965 (3)	C2—C1 <sup>i</sup>	1.553 (4)
Co3—N1 <sup>vi</sup>	1.965 (3)	O1W—H1WA	0.8700
Co3—N1 <sup>vii</sup>	1.965 (3)	O1W—H1W	0.8700
Cl1—Cl1 <sup>viii</sup>	1.041 (17)	O2W—H2WA	0.8699
Cl1—Cl1'	1.041 (17)	O2W—H2W	0.8700
O3 <sup>i</sup> —Co1—O3 <sup>ii</sup>	90.71 (7)	N1 <sup>iv</sup> —Co3—N1 <sup>vi</sup>	91.36 (15)
O3 <sup>i</sup> —Co1—O3	90.71 (7)	N1 <sup>v</sup> —Co3—N1 <sup>vi</sup>	180.0 (2)
O3 <sup>ii</sup> —Co1—O3	90.71 (7)	N1—Co3—N1 <sup>vi</sup>	88.64 (15)

O3 <sup>i</sup> —Co1—O4 <sup>ii</sup>	78.45 (7)	N1 <sup>iii</sup> —Co3—N1 <sup>vii</sup>	91.36 (15)
O3 <sup>ii</sup> —Co1—O4 <sup>ii</sup>	104.20 (7)	N1 <sup>iv</sup> —Co3—N1 <sup>vii</sup>	88.64 (15)
O3—Co1—O4 <sup>ii</sup>	161.53 (7)	N1 <sup>v</sup> —Co3—N1 <sup>vii</sup>	88.64 (15)
O3 <sup>i</sup> —Co1—O4 <sup>i</sup>	104.20 (7)	N1—Co3—N1 <sup>vii</sup>	180.0 (2)
O3 <sup>ii</sup> —Co1—O4 <sup>i</sup>	161.53 (7)	N1 <sup>vi</sup> —Co3—N1 <sup>vii</sup>	91.36 (15)
O3—Co1—O4 <sup>i</sup>	78.45 (7)	C11 <sup>viii</sup> —C11—C11'	180.000 (2)
O4 <sup>ii</sup> —Co1—O4 <sup>i</sup>	89.66 (7)	C2—O3—Co1	115.64 (17)
O3 <sup>i</sup> —Co1—O4	161.53 (7)	C1—O4—Co1	114.93 (16)
O3 <sup>ii</sup> —Co1—O4	78.45 (7)	Co3—N1—H1A	109.5
O3—Co1—O4	104.20 (7)	Co3—N1—H1B	109.5
O4 <sup>ii</sup> —Co1—O4	89.66 (7)	H1A—N1—H1B	109.5
O4 <sup>i</sup> —Co1—O4	89.66 (7)	Co3—N1—H1C	109.5
N2 <sup>i</sup> —Co2—N2 <sup>ii</sup>	90.56 (10)	H1A—N1—H1C	109.5
N2 <sup>i</sup> —Co2—N2	90.56 (10)	H1B—N1—H1C	109.5
N2 <sup>ii</sup> —Co2—N2	90.56 (10)	Co2—N2—H2B	111.0
N2 <sup>i</sup> —Co2—N3 <sup>iii</sup>	91.47 (10)	Co2—N2—H2A	111.2
N2 <sup>ii</sup> —Co2—N3 <sup>iii</sup>	87.31 (10)	H2B—N2—H2A	102.8
N2—Co2—N3 <sup>iii</sup>	177.07 (9)	Co2—N2—H2	112.7
N2 <sup>i</sup> —Co2—N3 <sup>i</sup>	87.31 (10)	H2B—N2—H2	109.7
N2 <sup>ii</sup> —Co2—N3 <sup>i</sup>	177.07 (9)	H2A—N2—H2	108.8
N2—Co2—N3 <sup>i</sup>	91.47 (10)	Co2—N3—H3B	113.7
N3 <sup>ii</sup> —Co2—N3 <sup>i</sup>	90.74 (9)	Co2—N3—H3A	108.2
N2 <sup>i</sup> —Co2—N3	177.07 (9)	H3B—N3—H3A	104.8
N2 <sup>ii</sup> —Co2—N3	91.47 (10)	Co2—N3—H3	111.6
N2—Co2—N3	87.31 (10)	H3B—N3—H3	108.4
N3 <sup>ii</sup> —Co2—N3	90.74 (9)	H3A—N3—H3	109.9
N3 <sup>i</sup> —Co2—N3	90.74 (9)	O1—C1—O4	125.1 (3)
N1 <sup>iii</sup> —Co3—N1 <sup>iv</sup>	180.0 (2)	O1—C1—C2 <sup>ii</sup>	119.5 (2)
N1 <sup>iii</sup> —Co3—N1 <sup>v</sup>	91.36 (15)	O4—C1—C2 <sup>ii</sup>	115.4 (2)
N1 <sup>iv</sup> —Co3—N1 <sup>v</sup>	88.64 (15)	O2—C2—O3	125.7 (3)
N1 <sup>iii</sup> —Co3—N1	88.64 (15)	O2—C2—C1 <sup>i</sup>	118.7 (2)
N1 <sup>iv</sup> —Co3—N1	91.36 (15)	O3—C2—C1 <sup>i</sup>	115.5 (2)
N1 <sup>v</sup> —Co3—N1	91.36 (15)	H1WA—O1W—H1W	108.3
N1 <sup>iii</sup> —Co3—N1 <sup>vi</sup>	88.64 (15)	H2WA—O2W—H2W	107.4

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

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>
N1—H1A $\cdots$ C11'	0.89	2.62	3.176 (15)	121
N1—H1A $\cdots$ O1 <sup>iii</sup>	0.89	2.27	3.055 (4)	147
N1—H1B $\cdots$ O2 <sup>ix</sup>	0.89	2.40	2.950 (4)	120
N1—H1B $\cdots$ O2 <sup>x</sup>	0.89	2.52	3.151 (4)	128
N2—H2 $\cdots$ O4 <sup>i</sup>	0.91	2.09	2.993 (3)	171
N2—H2A $\cdots$ O1W	0.91	2.18	3.047 (4)	160
N2—H2B $\cdots$ O2W	0.91	2.15	2.958 (3)	147
N3—H3 $\cdots$ O3 <sup>xi</sup>	0.91	2.09	2.988 (3)	172
N3—H3A $\cdots$ O2W	0.91	2.19	3.051 (3)	157
N3—H3B $\cdots$ O1W	0.91	2.36	3.120 (4)	141

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O1 <i>W</i> —H1 <i>W</i> ···O1	0.87	2.13	2.971 (4)	162
O1 <i>W</i> —H1 <i>WA</i> ···O1 <sup>xiii</sup>	0.87	2.32	2.973 (4)	132
O2 <i>W</i> —H2 <i>W</i> ···O2 <sup>xiii</sup>	0.87	1.97	2.830 (3)	171
O2 <i>W</i> —H2 <i>WA</i> ···O4 <sup>xiv</sup>	0.87	2.06	2.868 (3)	154

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Symmetry codes: (i)  $-x+y+1, -x+1, z$ ; (iii)  $-x+y, -x, z$ ; (ix)  $y-1, -x+y, -z$ ; (x)  $x-1, y-1, z$ ; (xi)  $-y+1, x-y, z+1$ ; (xii)  $x-y, x, -z+1$ ; (xiii)  $x, y, z+1$ ; (xiv)  $-x+1, -y+1, -z+1$ .