research communications

Crystal structure of tris[4-(dimethylamino)-pyridinium] tris(oxalato-$\kappa^2 O,O'$)chromate(III) tetrahydrate

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In the title hybrid salt, $(C_7H_{11}N_2)_3[Cr(C_2O_4)_3]\cdot 4H_2O$, the central Cr^{III} ion of the complex anion (point group symmetry 2) is coordinated by six O atoms from three chelating oxalate(2−) ligands in a slightly distorted octahedral coordination sphere. The Cr—O bond lengths vary from 1.9577 (11) to 1.9804 (11) Å, while the chelate O—Cr—O angles range from 82.11 (6) to 93.41 (5)°. The 4-(dimethylamino)pyridinium cations (one situated in a general position and one on a twofold rotation axis) are protonated at the pyridine N atoms. In the crystal, N—H···O and O—H···O hydrogen bonds link the cations and anions into a three-dimensional network. π–π interactions between the pyridine rings of adjacent cations provide additional stabilization of the crystal packing, with the closest centroid-to-centroid distances being 3.541 (1) and 3.575 (1) Å.

1. Chemical context

The coordination chemistry of oxalate $(C_2O_4^{2-})$ continues to receive considerable attention because of the ability of this ion to act as a remarkably flexible ligand system in complexations with a wide range of metal ions (Martin *et al.*, 2007). Over the last decade, Bélombé and coworkers (Bélombé *et al.*, 2003) prepared a novel barium-oxalatochromate(III), $\{Ba_6(H_2O)_{17}$-$[Cr(ox)_3]_4\}\cdot 7H_2O$, and demonstrated the use of this complex as a suitable precursor for the synthesis of multi-functional crystalline materials (Bélombé *et al.*, 2009*a*,*b*; Mbiangué *et al.*, 2012). Moreover, this complex has received much attention in the field of materials science for its use as a convenient route for the preparation of technologically important metallic composite oxides (Neo *et al.*, 2006). As part of our ongoing research program, we have now combined this versatile barium-oxalatochromate(III) complex with 4-(dimethyl-amino)pyridinium oxalate to isolate the organic–inorganic hybrid salt, $(C_7H_{11}N_2)_3[Cr(C_2O_4)_3]\cdot 4H_2O$.
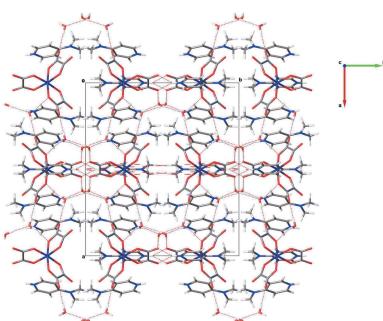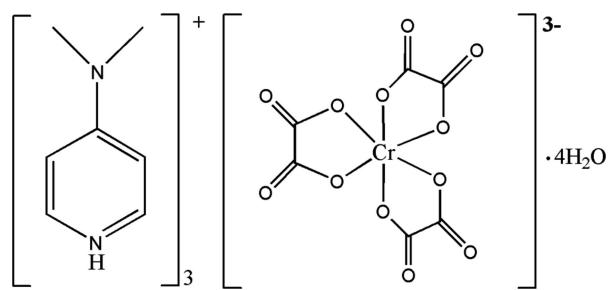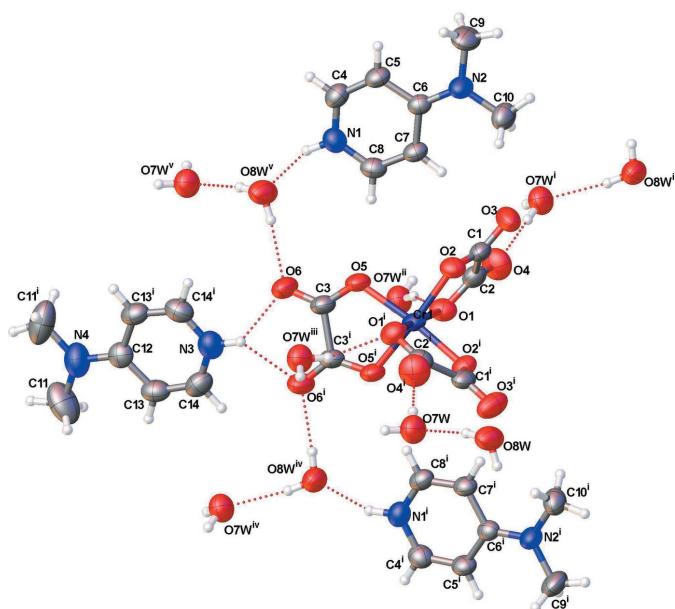
Figure 1
The molecular components of the title compound. Displacement ellipsoids are drawn at the 50% probability level. Hydrogen bonds are shown as dashed lines. [Symmetry codes: (i) $1 - x, y, \frac{1}{2} - z$; (ii) $-\frac{1}{2} + x, \frac{1}{2} - y, -\frac{1}{2} + z$; (iii) $\frac{3}{2} - x, \frac{1}{2} - y, 1 - z$; (iv) $\frac{3}{2} - x, -\frac{1}{2} + y, \frac{1}{2} - z$; (v) $-\frac{1}{2} + x, -\frac{1}{2} + y, z$.]

2. Structural commentary

The molecular components of the title compound are shown in Fig. 1. The asymmetric unit contains one and a half 4-(dimethylamino)pyridinium cations, one half of the tris-(oxalato)chromate(III) complex anion and two lattice water molecules. The entities are completed by application of twofold rotation symmetry. The central Cr^{III} ion of the complex anion is coordinated by six O atoms from three chelating oxalato(2−) ligands in a slightly distorted (2 + 2 + 2)

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N1—H1$\cdots$O8W^i	0.91 (2)	1.84 (2)	2.702 (2)	157.8 (19)
N3—H3$\cdots$O6^{ii}	0.92 (4)	2.12 (3)	2.879 (3)	139 (1)
N3—H3$\cdots$O6^{iii}	0.92 (4)	2.12 (3)	2.879 (3)	139 (1)
O7W—H7$WA\cdots$O4^{iv}	0.83 (1)	1.99 (1)	2.819 (2)	178 (3)
O7W—H7$WB\cdots$O1^v	0.82 (1)	2.12 (1)	2.9079 (19)	161 (3)
O8W—H8$WA\cdots$O7W	0.81 (1)	1.95 (1)	2.7578 (19)	172 (3)
O8W—H8$WB\cdots$O6^{vi}	0.82 (1)	1.99 (1)	2.8007 (19)	175 (3)

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

octahedral coordination sphere. The chelate O—Cr—O angles range from 82.11 (6) to 93.41 (5)°. The Cr—O bond lengths vary from 1.9577 (11) to 1.9804 (11) Å and are similar to those found in the guanidinium tris(oxalato)chromate(III) salt (Golič & Bulc, 1988). Bond lengths and angles in the organic cations, [C$_7$H$_{11}$N$_2$]$^+$, are in agreement with those found in salts with the same cationic entity (Nenwa *et al.*, 2010; Ghouili *et al.*, 2010; Benslimane *et al.*, 2012; Ben Nasr *et al.*, 2015).

3. Supramolecular features

In the title compound, the crystal packing is stabilized by a network of intermolecular N—H$\cdots$O and O—H$\cdots$O hydrogen bonds linking the coordination octahedra, 4-(dimethylamino)pyridinium cations and lattice water molecules (Table 1, Fig. 2). In addition, π–π stacking interactions [centroid-to-centroid distances of 3.541 (1) and 3.575 (1) Å] between the pyridine rings contribute to the stabilization of the three-dimensional network (Fig. 3).

Figure 2
Projection on the *ab* plane of the crystal structure of the title compound. Hydrogen bonds are shown as dashed lines.
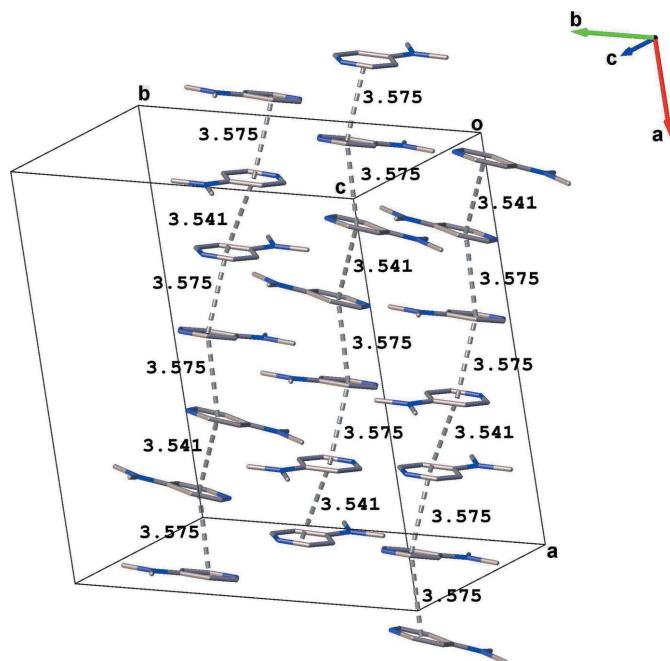
Figure 3
π–π stacking interactions (dashed lines) between adjacent organic cations in the title compound.

Table 2
Experimental details.

Crystal data	
Chemical formula	$(C_7H_{11}N_2)_3[Cr(C_3O_4)_3]\cdot 4H_2O$
M_r	757.66
Crystal system, space group	Monoclinic, $C2/c$
Temperature (K)	296
a, b, c (Å)	19.1141 (5), 16.7537 (4), 11.0053 (2)
β (°)	98.803 (1)
V (Å^3)	3482.73 (14)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.41
Crystal size (mm)	$0.58 \times 0.21 \times 0.14$
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.708, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	56955, 5322, 3757
R_{int}	0.038
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.714
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.038, 0.120, 1.03
No. of reflections	5322
No. of parameters	249
No. of restraints	6
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å^{-3})	0.23, −0.42

Computer programs: *SAINT* and *APEX2* (Bruker, 2014), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *OLEX2* (Dolomanov *et al.*, 2009), *publCIF* (Westrip, 2010) and *PLATON* (Spek, 2009).

4. Synthesis and crystallization

The title compound was obtained by reaction of an aqueous solution of the freshly prepared barium-oxalatochromate(III) salt $\{Ba_6(H_2O)_{17}[Cr(C_2O_4)_3]_4\}\cdot 7H_2O$ (1 mmol, 2.536 g), with an aqueous solution of 4-(dimethylamino)pyridine (12 mmol, 1.464 g) and oxalic acid (6 mmol, 0.756 g). The mixture was stirred at 333 K for about 30 minutes and then cooled to room temperature and filtered. The title compound crystallized by slow evaporation of the solvent at room temperature in form of light-violet crystals with dimensions up to 3 mm within a few weeks.

5. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. H atoms bonded to C atoms were positioned geometrically and allowed to ride on their parent atoms with C—H = 0.93 Å and $U_{iso}(H)$ = $1.2U_{eq}(C)$ for aromatic and 0.96 Å and $U_{iso}(H)$ = $1.5U_{eq}(C)$ for methyl H atoms. H atoms of water molecules as well as those bonded to N atoms were located from a difference Fourier map. Water H atoms were refined with soft restraints on O—H and H···H distances [O—H = 0.82 (1) Å and H···H = 1.30 (2) Å] and $U_{iso}(H)$ = $1.5U_{eq}(O)$ whereas H atoms bonded to N atoms were refined freely.

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Crystal structure of tris[4-(dimethylamino)pyridinium] tris(oxalato-κ^2O,O')chromate(III) tetrahydrate

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Computing details

Data collection: *SAINT* (Bruker, 2014); cell refinement: *APEX2* (Bruker, 2014); data reduction: *SAINT* (Bruker, 2014); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009), *publCIF* (Westrip, 2010) and *PLATON* (Spek, 2009).

Tris[4-(dimethylamino)pyridinium] tris(oxalato-κ^2O,O')chromate(III) tetrahydrate

Crystal data
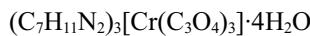
$(C_7H_{11}N_2)_3[Cr(C_3O_4)_3]\cdot4H_2O$	$F(000) = 1588$
$M_r = 757.66$	$D_x = 1.445$ Mg m^{-3}
Monoclinic, $C2/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 19.1141$ (5) Å	Cell parameters from 9987 reflections
$b = 16.7537$ (4) Å	$\theta = 2.4$–27.8°
$c = 11.0053$ (2) Å	$\mu = 0.41$ mm^{-1}
$\beta = 98.803$ (1)°	$T = 296$ K
$V = 3482.73$ (14) Å^3	Prism, violet
$Z = 4$	$0.58 \times 0.21 \times 0.14$ mm

Data collection

Bruker APEXII CCD diffractometer	5322 independent reflections
Radiation source: sealed X-ray tube	3757 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.038$
Absorption correction: multi-scan (*SADABS*; Krause *et al.*, 2015)	$\theta_{max} = 30.5°$, $\theta_{min} = 2.6°$
	$h = -27 \rightarrow 27$
	$k = -23 \rightarrow 23$
$T_{min} = 0.708$, $T_{max} = 0.746$	$l = -15 \rightarrow 15$
56955 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.038$	Hydrogen site location: mixed
$wR(F^2) = 0.120$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.03$	
5322 reflections	$w = 1/[\sigma^2(F_o^2) + (0.056P)^2 + 1.554P]$ where $P = (F_o^2 + 2F_c^2)/3$
249 parameters	
6 restraints	$(\Delta/\sigma)_{max} = 0.001$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max} = 0.23$ e Å^{-3}
	$\Delta\rho_{min} = -0.42$ e Å^{-3}

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	y	z	$U_{iso}*/U_{eq}$
Cr1	0.5000	0.25122 (2)	0.2500	0.04246 (11)
O2	0.44865 (6)	0.33279 (7)	0.32829 (9)	0.0502 (3)
O1	0.41659 (6)	0.25871 (7)	0.12056 (10)	0.0521 (3)
O5	0.45866 (6)	0.16208 (7)	0.33354 (10)	0.0522 (3)
O7W	0.82663 (8)	0.36686 (8)	0.49953 (15)	0.0698 (4)
O6	0.45686 (8)	0.02935 (8)	0.34038 (13)	0.0695 (4)
O3	0.35256 (8)	0.40942 (8)	0.29279 (12)	0.0714 (4)
N2	0.28924 (8)	0.40160 (8)	0.65828 (12)	0.0507 (3)
O8W	0.87475 (9)	0.52183 (8)	0.53015 (16)	0.0762 (4)
N1	0.33363 (7)	0.16521 (9)	0.60672 (13)	0.0499 (3)
O4	0.31735 (7)	0.32756 (9)	0.06984 (14)	0.0767 (4)
N3	0.5000	0.87873 (15)	0.2500	0.0615 (5)
C6	0.30440 (7)	0.32468 (9)	0.64377 (12)	0.0405 (3)
N4	0.5000	0.63400 (15)	0.2500	0.0730 (7)
C1	0.39125 (9)	0.35760 (9)	0.26335 (14)	0.0475 (3)
C7	0.34073 (8)	0.29953 (10)	0.54716 (12)	0.0442 (3)
H7	0.3556	0.3370	0.4943	0.053*
C2	0.37147 (9)	0.31285 (10)	0.13931 (15)	0.0496 (4)
C5	0.28590 (9)	0.26379 (10)	0.72219 (14)	0.0483 (4)
H5	0.2636	0.2770	0.7888	0.058*
C3	0.47543 (8)	0.09271 (10)	0.30033 (14)	0.0486 (4)
C4	0.30043 (9)	0.18699 (11)	0.70089 (14)	0.0521 (4)
H4	0.2872	0.1478	0.7527	0.063*
C12	0.5000	0.71391 (15)	0.2500	0.0505 (5)
C8	0.35377 (8)	0.22127 (10)	0.53192 (14)	0.0476 (3)
H8	0.3773	0.2057	0.4679	0.057*
C10	0.30788 (12)	0.46256 (11)	0.57456 (17)	0.0626 (5)
H10A	0.3583	0.4633	0.5769	0.094*
H10B	0.2924	0.5138	0.5991	0.094*
H10C	0.2853	0.4508	0.4925	0.094*
C13	0.47586 (10)	0.75888 (12)	0.34435 (16)	0.0563 (4)
H13	0.4593	0.7330	0.4091	0.068*
C14	0.47685 (9)	0.83913 (12)	0.34033 (17)	0.0608 (4)
H14	0.4608	0.8678	0.4030	0.073*
C9	0.25007 (13)	0.42746 (13)	0.75514 (19)	0.0741 (6)
H9A	0.2037	0.4041	0.7419	0.111*
H9B	0.2460	0.4846	0.7537	0.111*
H9C	0.2748	0.4108	0.8336	0.111*
C11	0.47408 (17)	0.58935 (16)	0.3474 (3)	0.1074 (10)

H11A	0.5028	0.6012	0.4247	0.161*
H11B	0.4764	0.5332	0.3310	0.161*
H11C	0.4259	0.6041	0.3509	0.161*
H3	0.5000	0.934 (2)	0.2500	0.091 (11)*
H7WA	0.7843 (7)	0.3544 (19)	0.481 (3)	0.136*
H8WA	0.8595 (16)	0.4767 (9)	0.528 (3)	0.136*
H8WB	0.8988 (14)	0.5268 (17)	0.475 (2)	0.136*
H7WB	0.8432 (14)	0.3280 (13)	0.540 (3)	0.136*
H1	0.3406 (11)	0.1122 (13)	0.5955 (18)	0.068 (6)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cr1	0.04232 (19)	0.0513 (2)	0.03681 (16)	0.000	0.01578 (13)	0.000
O2	0.0557 (6)	0.0565 (7)	0.0411 (5)	0.0033 (5)	0.0164 (5)	−0.0054 (4)
O1	0.0505 (6)	0.0617 (7)	0.0445 (6)	−0.0005 (5)	0.0087 (5)	−0.0094 (5)
O5	0.0572 (7)	0.0538 (6)	0.0530 (6)	−0.0008 (5)	0.0318 (5)	−0.0011 (5)
O7W	0.0775 (9)	0.0497 (7)	0.0849 (10)	−0.0032 (7)	0.0211 (8)	−0.0005 (7)
O6	0.0851 (9)	0.0556 (7)	0.0795 (9)	−0.0022 (7)	0.0499 (8)	0.0054 (6)
O3	0.0916 (10)	0.0639 (8)	0.0653 (8)	0.0297 (7)	0.0330 (7)	0.0100 (6)
N2	0.0646 (8)	0.0478 (7)	0.0431 (6)	−0.0014 (6)	0.0191 (6)	−0.0055 (5)
O8W	0.0914 (11)	0.0531 (8)	0.0954 (11)	−0.0067 (7)	0.0501 (9)	−0.0132 (7)
N1	0.0509 (7)	0.0481 (8)	0.0498 (7)	0.0025 (6)	0.0041 (6)	−0.0044 (6)
O4	0.0598 (8)	0.0853 (10)	0.0794 (9)	0.0068 (7)	−0.0076 (7)	0.0037 (8)
N3	0.0568 (12)	0.0558 (13)	0.0701 (14)	0.000	0.0036 (10)	0.000
C6	0.0399 (7)	0.0492 (8)	0.0323 (6)	−0.0036 (6)	0.0057 (5)	−0.0039 (5)
N4	0.0845 (17)	0.0570 (13)	0.0703 (14)	0.000	−0.0111 (12)	0.000
C1	0.0565 (9)	0.0447 (8)	0.0467 (8)	0.0018 (7)	0.0250 (7)	0.0078 (6)
C7	0.0462 (8)	0.0530 (8)	0.0351 (6)	−0.0040 (6)	0.0123 (6)	−0.0008 (6)
C2	0.0469 (8)	0.0526 (9)	0.0512 (8)	−0.0051 (7)	0.0132 (7)	0.0065 (7)
C5	0.0542 (9)	0.0568 (9)	0.0363 (7)	−0.0040 (7)	0.0150 (6)	−0.0003 (6)
C3	0.0462 (8)	0.0569 (9)	0.0469 (8)	−0.0014 (7)	0.0205 (6)	0.0010 (7)
C4	0.0598 (10)	0.0540 (9)	0.0431 (8)	−0.0056 (7)	0.0098 (7)	0.0052 (7)
C12	0.0461 (12)	0.0573 (14)	0.0448 (11)	0.000	−0.0033 (9)	0.000
C8	0.0435 (8)	0.0603 (9)	0.0401 (7)	0.0017 (7)	0.0097 (6)	−0.0075 (6)
C10	0.0877 (14)	0.0462 (9)	0.0567 (10)	−0.0061 (9)	0.0197 (9)	−0.0016 (7)
C13	0.0509 (9)	0.0763 (13)	0.0430 (8)	−0.0002 (8)	0.0113 (7)	0.0062 (7)
C14	0.0546 (10)	0.0712 (12)	0.0572 (10)	0.0095 (9)	0.0107 (8)	−0.0094 (8)
C9	0.1006 (16)	0.0638 (12)	0.0665 (11)	0.0046 (11)	0.0406 (11)	−0.0154 (9)
C11	0.127 (2)	0.0759 (16)	0.108 (2)	−0.0233 (15)	−0.0179 (17)	0.0327 (14)

Geometric parameters (Å, º)

Cr1—O2^i	1.9577 (11)	C6—C5	1.416 (2)
Cr1—O2	1.9577 (11)	N4—C12	1.339 (3)
Cr1—O1^i	1.9728 (12)	N4—C11	1.455 (3)
Cr1—O1	1.9728 (12)	N4—C11^i	1.455 (3)
Cr1—O5^i	1.9804 (11)	C1—C2	1.553 (2)

Cr1—O5	1.9804 (11)	C7—H7	0.9300
O2—C1	1.2834 (19)	C7—C8	1.350 (2)
O1—C2	1.290 (2)	C5—H5	0.9300
O5—C3	1.274 (2)	C5—C4	1.344 (2)
O7W—H7WA	0.830 (10)	C3—C3ⁱ	1.558 (3)
O7W—H7WB	0.822 (10)	C4—H4	0.9300
O6—C3	1.223 (2)	C12—C13ⁱ	1.416 (2)
O3—C1	1.2162 (19)	C12—C13	1.416 (2)
N2—C6	1.336 (2)	C8—H8	0.9300
N2—C10	1.456 (2)	C10—H10A	0.9600
N2—C9	1.459 (2)	C10—H10B	0.9600
O8W—H8WA	0.810 (10)	C10—H10C	0.9600
O8W—H8WB	0.818 (10)	C13—H13	0.9300
N1—C4	1.346 (2)	C13—C14	1.345 (3)
N1—C8	1.343 (2)	C14—H14	0.9300
N1—H1	0.91 (2)	C9—H9A	0.9600
O4—C2	1.214 (2)	C9—H9B	0.9600
N3—C14ⁱ	1.326 (2)	C9—H9C	0.9600
N3—C14	1.326 (2)	C11—H11A	0.9600
N3—H3	0.92 (4)	C11—H11B	0.9600
C6—C7	1.4200 (19)	C11—H11C	0.9600
O2ⁱ—Cr1—O2	91.45 (7)	O4—C2—O1	124.63 (17)
O2—Cr1—O1	82.48 (5)	O4—C2—C1	121.68 (16)
O2—Cr1—O1ⁱ	92.41 (5)	C6—C5—H5	119.8
O2ⁱ—Cr1—O1	92.41 (5)	C4—C5—C6	120.39 (15)
O2ⁱ—Cr1—O1ⁱ	82.47 (5)	C4—C5—H5	119.8
O2—Cr1—O5ⁱ	173.35 (5)	O5—C3—C3ⁱ	114.17 (8)
O2—Cr1—O5	93.41 (5)	O6—C3—O5	126.08 (14)
O2ⁱ—Cr1—O5	173.35 (5)	O6—C3—C3ⁱ	119.75 (9)
O2ⁱ—Cr1—O5ⁱ	93.41 (5)	N1—C4—H4	119.1
O1—Cr1—O1ⁱ	172.70 (7)	C5—C4—N1	121.88 (15)
O1—Cr1—O5ⁱ	92.78 (5)	C5—C4—H4	119.1
O1ⁱ—Cr1—O5	92.79 (5)	N4—C12—C13	122.15 (11)
O1—Cr1—O5	92.72 (5)	N4—C12—C13ⁱ	122.15 (11)
O1ⁱ—Cr1—O5ⁱ	92.72 (5)	C13—C12—C13ⁱ	115.7 (2)
O5ⁱ—Cr1—O5	82.11 (6)	N1—C8—C7	121.82 (14)
C1—O2—Cr1	115.12 (10)	N1—C8—H8	119.1
C2—O1—Cr1	114.55 (10)	C7—C8—H8	119.1
C3—O5—Cr1	114.77 (9)	N2—C10—H10A	109.5
H7WA—O7W—H7WB	102 (2)	N2—C10—H10B	109.5
C6—N2—C10	121.50 (13)	N2—C10—H10C	109.5
C6—N2—C9	121.25 (15)	H10A—C10—H10B	109.5
C10—N2—C9	117.18 (15)	H10A—C10—H10C	109.5
H8WA—O8W—H8WB	108 (2)	H10B—C10—H10C	109.5
C4—N1—H1	117.7 (13)	C12—C13—H13	120.0
C8—N1—C4	119.70 (15)	C14—C13—C12	119.98 (17)
C8—N1—H1	122.5 (13)	C14—C13—H13	120.0

C14—N3—C14[i]	120.0 (3)	N3—C14—C13	122.18 (19)
C14[i]—N3—H3	120.02 (13)	N3—C14—H14	118.9
C14—N3—H3	120.01 (13)	C13—C14—H14	118.9
N2—C6—C7	121.05 (14)	N2—C9—H9A	109.5
N2—C6—C5	122.92 (13)	N2—C9—H9B	109.5
C5—C6—C7	116.04 (14)	N2—C9—H9C	109.5
C12—N4—C11[i]	120.94 (15)	H9A—C9—H9B	109.5
C12—N4—C11	120.93 (15)	H9A—C9—H9C	109.5
C11[i]—N4—C11	118.1 (3)	H9B—C9—H9C	109.5
O2—C1—C2	113.93 (13)	N4—C11—H11A	109.5
O3—C1—O2	125.85 (16)	N4—C11—H11B	109.5
O3—C1—C2	120.19 (15)	N4—C11—H11C	109.5
C6—C7—H7	119.9	H11A—C11—H11B	109.5
C8—C7—C6	120.14 (14)	H11A—C11—H11C	109.5
C8—C7—H7	119.9	H11B—C11—H11C	109.5
O1—C2—C1	113.67 (13)		
Cr1—O2—C1—O3	−177.75 (14)	C7—C6—C5—C4	−2.3 (2)
Cr1—O2—C1—C2	4.37 (16)	C5—C6—C7—C8	2.0 (2)
Cr1—O1—C2—O4	178.94 (14)	C4—N1—C8—C7	−1.0 (2)
Cr1—O1—C2—C1	−2.76 (16)	C12—C13—C14—N3	−0.1 (3)
Cr1—O5—C3—O6	179.09 (15)	C8—N1—C4—C5	0.7 (2)
Cr1—O5—C3—C3[i]	−0.6 (2)	C10—N2—C6—C7	1.3 (2)
O2—C1—C2—O1	−1.05 (19)	C10—N2—C6—C5	−178.85 (16)
O2—C1—C2—O4	177.30 (15)	C13[i]—C12—C13—C14	0.02 (12)
O3—C1—C2—O1	−179.06 (15)	C14[i]—N3—C14—C13	0.03 (13)
O3—C1—C2—O4	−0.7 (2)	C9—N2—C6—C7	178.16 (16)
N2—C6—C7—C8	−178.10 (14)	C9—N2—C6—C5	−2.0 (3)
N2—C6—C5—C4	177.81 (15)	C11[i]—N4—C12—C13	−179.27 (16)
C6—C7—C8—N1	−0.4 (2)	C11[i]—N4—C12—C13[i]	0.74 (16)
C6—C5—C4—N1	1.0 (3)	C11—N4—C12—C13[i]	−179.26 (16)
N4—C12—C13—C14	−179.98 (12)	C11—N4—C12—C13	0.74 (16)

Symmetry code: (i) −x+1, y, −z+1/2.

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O8W[vii]	0.91 (2)	1.84 (2)	2.702 (2)	157.8 (19)
N3—H3···O6[iii]	0.92 (4)	2.12 (3)	2.879 (3)	139 (1)
N3—H3···O6[iv]	0.92 (4)	2.12 (3)	2.879 (3)	139 (1)
O7W—H7WA···O4[i]	0.83 (1)	1.99 (1)	2.819 (2)	178 (3)
O7W—H7WB···O1[v]	0.82 (1)	2.12 (1)	2.9079 (19)	161 (3)
O8W—H8WA···O7W	0.81 (1)	1.95 (1)	2.7578 (19)	172 (3)
O8W—H8WB···O6[vi]	0.82 (1)	1.99 (1)	2.8007 (19)	175 (3)

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