

Crystal structure of bis[(oxalato- κ^2O^1,O^2)(1,4,8,11-tetraazacyclotetradecane- κ^4N)chromium(III)] dichromate octahydrate from synchrotron X-ray data

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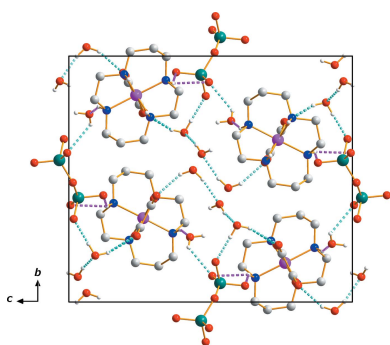
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Keywords: crystal structure; cyclam; chromium(III) complex; bidentate oxalato ligand; *cis*-V conformation; dichromate anion; hydrogen bonding; synchrotron radiation.**CCDC reference:** 1532785**Supporting information:** this article has supporting information at journals.iucr.org/e

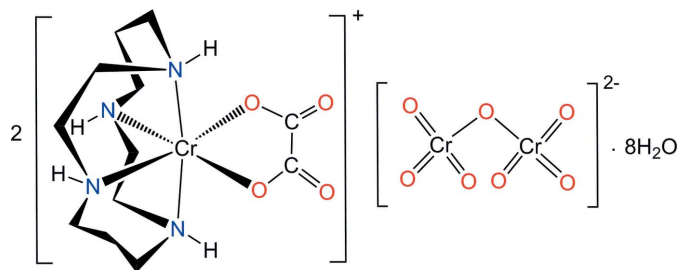
The asymmetric unit of the title compound, $[\text{Cr}(\text{C}_2\text{O}_4)(\text{C}_{10}\text{H}_{24}\text{N}_4)]_2[\text{Cr}_2\text{O}_7] \cdot 8\text{H}_2\text{O}$ ($\text{C}_{10}\text{H}_{24}\text{N}_4$ = 1,4,8,11-tetraazacyclotetradecane, cyclam; C_2O_4 = oxalate, ox) contains one $[\text{Cr}(\text{ox})(\text{cyclam})]^+$ cation, one half of a dichromate anion that lies about an inversion centre so that the bridging O atom is equally disordered over two positions, and four water molecules. The terminal O atoms of the dichromate anion are also disordered over two positions with a refined occupancy ratio 0.586 (6):0.414 (6). The Cr^{III} ion is coordinated by the four N atoms of the cyclam ligand and one bidentate oxalato ligand in a *cis* arrangement, resulting in a distorted octahedral geometry. The Cr–N(cyclam) bond lengths are in the range 2.069 (2)–2.086 (2) Å, while the average Cr–O(ox) bond length is 1.936 Å. The macrocyclic cyclam moiety adopts the *cis*-V conformation. The dichromate anion has a staggered conformation. The crystal structure is stabilized by intermolecular hydrogen bonds involving the cyclam N–H groups and water O–H groups as donors, and the O atoms of oxalate ligand, water molecules and the $\text{Cr}_2\text{O}_7^{2-}$ anion as acceptors, giving rise to a three-dimensional network.

1. Chemical context

Chromium (Cr) is considered a trace element essential for the proper functioning of living organisms and is also a highly toxic material (Yusof & Malek, 2009). Cr can exist in all oxidation states from 0 to VI, the most common oxidation states in water being Cr^{III} and Cr^{VI} . In an aqueous environment, the toxicity of Cr^{VI} has been shown to be greater than that of Cr^{III} (Guzel *et al.*, 2016). Transition metal complexes of the cyclam (1,4,8,11-tetraazacyclotetradecane, $\text{C}_{10}\text{H}_{24}\text{N}_4$) ligand have been the subject of numerous investigations because of their particular conformational stereochemistry (Poon & Pun, 1980; Choi, 2009; Subhan *et al.*, 2011). Recently, it has been found that cyclam derivatives and their metal complexes exhibit anti-HIV activity (Ronconi & Sadler, 2007; De Clercq, 2010; Ross *et al.*, 2012). The conformation of the macrocyclic ligand is a very important factor for co-receptor recognition. Therefore, knowledge of the conformation and hydrogen-bonding interactions in Cr^{III} – Cr^{VI} complex systems containing the cyclam ligand has become important in the development of new anti-HIV drugs (De Clercq, 2010). The use of such complexes for the more effective removal of toxic metals is also important (Guzel *et al.*, 2016). As part of a study of the conformation and structure of (cyclam)chromium(III) complexes with auxiliary ligand(s) and various anions, we



report here the structural characterization of the new complex salt, $[\text{Cr}(\text{C}_2\text{O}_4)(\text{C}_{10}\text{H}_{24}\text{N}_4)]_2[\text{Cr}_2\text{O}_7]\cdot 8\text{H}_2\text{O}$, (I).



2. Structural commentary

An ellipsoid plot of the molecular components in (I) is shown in Fig. 1 along with the atom-numbering scheme. The structure is another example of a $[\text{Cr}(\text{ox})(\text{cyclam})]^+$ cation (Choi *et al.*, 2004*b*; Moon & Choi, 2016*b*), but with a different counteranion. The asymmetric unit contains one $[\text{Cr}(\text{ox})(\text{cyclam})]^+$ cation, one half of a $\text{Cr}_2\text{O}_7^{2-}$ anion (completed by inversion symmetry with the bridging O atom disordered about the inversion centre) and four non-coordinating water molecules. The three terminal O atoms of the dichromate anion are also disordered over two positions with occupancy ratio of the atom pairs O2B1/O2B2, O3B1/O3B2 and O4B1/O4B2 converging at 0.586 (6):0.414 (6). The conformation of the cyclam ligand can be described as *cis*-V (*anti-anti*) (Subhan *et al.*, 2011). In the complex cation, the Cr^{III} ion is coordinated by the four nitrogen atoms of the cyclam ligand in a folded conformation. Two oxygen atoms of the oxalato ligand complete the distorted octahedral coordination sphere. The Cr–N bond lengths from the donor atoms of cyclam ligand lie in the range 2.069 (2) to 2.086 (2) Å, in good agreement with those determined in *cis*- $[\text{Cr}(\text{N}_3)_2(\text{cyclam})]\text{ClO}_4$ [2.069 (3)–2.103 (3) Å] (Meyer *et al.*, 1998), *cis*- $[\text{Cr}(\text{ONO})_2(\text{cyclam})]\text{NO}_2$ [2.0874 (16)–2.0916 (15) Å] (Choi *et al.*, 2004*a*), $[\text{Cr}(\text{acac})(\text{cyclam})](\text{ClO}_4)_2\cdot 0.5\text{H}_2\text{O}$ [2.070 (5)–2.089 (5) Å] (acac = acetylacetonate; Subhan *et al.*, 2011), *cis*- $[\text{Cr}(\text{NCS})_2(\text{cyclam})]\text{NCS}$ [2.0851 (14)–2.0897 (14) Å] (Moon *et al.*, 2013) and $[\text{CrCl}_2(\text{cyclam})][\text{Cr}(\text{ox})(\text{cyclam})](\text{ClO}_4)_2$ [2.075 (5)–2.096 (5) Å] (Moon & Choi, 2016*b*). However, the Cr–N bond lengths of the cyclam ligand with a *cis* geometry are slightly longer than those found in *trans*- $[\text{Cr}(\text{NCS})_2(\text{cyclam})]\text{ClO}_4$ [2.046 (2)–2.060 (2) Å] (Friesen *et al.*, 1997), *trans*- $[\text{Cr}(\text{ONO})_2(\text{cyclam})]\text{BF}_4$ [2.064 (4)–2.073 (4) Å] (De Leo *et al.*, 2000), *trans*- $[\text{Cr}(\text{NH}_3)_2(\text{cyclam})][\text{ZnCl}_4]\text{Cl}\cdot\text{H}_2\text{O}$ [2.0501 (15)–2.0615 (15) Å] (Moon & Choi, 2016*a*) and *trans*- $[\text{Cr}(\text{nic-O})_2(\text{cyclam})]\text{ClO}_4$ [2.058 (4)–2.064 (4) Å] (nic-O = O-coordinated nicotinate; Choi, 2009).

The Cr1A–O1A distance [1.9665 (16) Å] in the oxalate ligand is very slightly longer than the Cr1A–O3A [1.9600 (16) Å] bond length. This elongation may be attributed to the weak hydrogen bond formed by O1A ($x, -y + \frac{1}{2}, z - \frac{1}{2}$) with the O3S–H2O3 atoms of a water molecule. The mean Cr–O bond length is comparable to the mean values of 1.959, 1.956 and 1.969 Å observed in $[\text{Cr}(\text{ox})(\text{cyclam})]\text{ClO}_4$

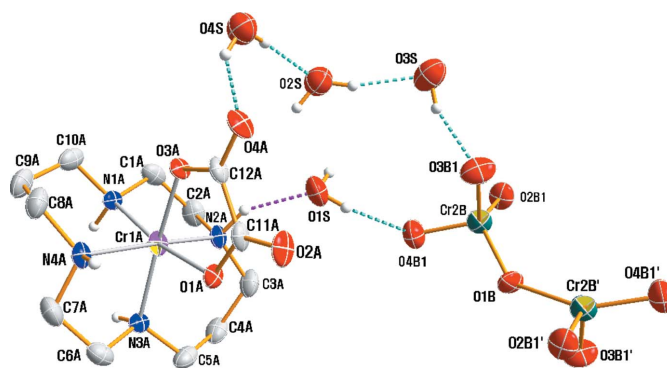


Figure 1

A perspective view of the asymmetric unit of the title compound, (I), with the dichromate anion, which lies about an inversion centre, drawn in full. Displacement ellipsoids are drawn at the 30% probability level and primed atoms are related by the symmetry operation $(2 - x, -y, 1 - z)$. For clarity, only the major disorder components are shown for the disordered dichromate anion.

(Choi *et al.*, 2004*b*), $[\text{CrCl}_2(\text{cyclam})][\text{Cr}(\text{ox})(\text{cyclam})](\text{ClO}_4)_2$ (Moon & Choi, 2016*b*) and $\text{K}_3[\text{Cr}(\text{ox})_3]\cdot 3\text{H}_2\text{O}$ (Taylor, 1978), respectively. The five- and six-membered chelate rings of the cyclam ligand adopt *gauche* and stable chair conformations, respectively. As expected for a bidentate ox ligand, the O1A–Cr1A–O3A bite angle $82.34(7)^\circ$ is considerably less than 90° , while the folding angle of the cyclam in the $[\text{Cr}(\text{ox})(\text{cyclam})]^+$ cation is $98.97(8)^\circ$. The significant distortion of the octahedron and the larger folding angle in the $[\text{Cr}(\text{ox})(\text{cyclam})]^+$ cation seem to arise from the small bite angle of the bidentate oxalato ligand.

It is of interest to compare the conformation of the $\text{Cr}_2\text{O}_7^{2-}$ anion with that found in other ionic crystals. In (I), the $\text{Cr}_2\text{O}_7^{2-}$ anion exhibits a staggered conformation whereas a nearly eclipsed conformation is observed for $(\text{C}_9\text{H}_{14}\text{N})_2[\text{Cr}_2\text{O}_7]$ and $(\text{C}_{10}\text{H}_{22}\text{N}_2)[\text{Cr}_2\text{O}_7]$, when viewed along the backbone of the dichromate anion (Trabelsi *et al.*, 2015; Chebbi *et al.*, 2016). This structural conformation of dichromate seems to depend on the size of the associated counteranion (Moon *et al.*, 2015, 2017). The O1B–Cr2B–O bond angles range from $107.1(3)$ to $117.0(3)^\circ$; while the terminal Cr2B–O bond lengths vary from 1.572 (12) to 1.673 (5) Å, with a mean terminal Cr2B–O bond length of 1.627 Å. The bridging Cr2B–O1B bond is 1.684 (4) Å long, with the Cr2B–O1B–Cr2B($-x + 2, -y, -z + 1$) bond angle of $136.0(3)^\circ$. These values are similar to those reported for the anions in the structures of $[\text{Cr}(\text{urea})_6][\text{Cr}_2\text{O}_7]\text{Br}\cdot\text{H}_2\text{O}$ (Moon *et al.*, 2015) and $[\text{Cr}(\text{NCS})_2(\text{cyclam})]_2[\text{Cr}_2\text{O}_7]\cdot\text{H}_2\text{O}$ (Moon *et al.*, 2017). A further distortion of the anion undoubtedly results from its involvement in hydrogen-bonding interactions with the solvent water molecules (see *Supramolecular features*).

3. Supramolecular features

In the asymmetric unit, O–H \cdots O and N–H \cdots O hydrogen bonds link the water molecules to the $\text{Cr}_2\text{O}_7^{2-}$ anion,

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1A—H1A \cdots O4A ⁱ	0.98	1.99	2.804 (3)	139
N2A—H2A \cdots O1S	0.98	2.00	2.894 (3)	150
N3A—H3A \cdots O2A ⁱ	0.98	1.89	2.842 (3)	163
N4A—H4A \cdots O2B1 ⁱⁱⁱ	0.98	2.25	3.100 (16)	144
N4A—H4A \cdots O4B1 ⁱⁱⁱ	0.98	2.37	3.108 (6)	132
N4A—H4A \cdots O4B2 ⁱⁱⁱ	0.98	2.15	3.05 (2)	151
O1S—H1O1 \cdots O4B1	0.85 (1)	2.33 (5)	2.876 (6)	123 (5)
O1S—H1O1 \cdots O3B2	0.85 (1)	2.20 (4)	2.903 (10)	141 (5)
O2S—H2O2 \cdots O3S	0.85 (1)	1.91 (2)	2.729 (6)	164 (6)
O3S—H1O3 \cdots O3B1	0.85 (1)	1.93 (3)	2.725 (6)	156 (7)
O3S—H1O3 \cdots O3B2	0.85 (1)	2.28 (2)	3.113 (10)	167 (6)
O3S—H2O3 \cdots O1A ⁱⁱⁱ	0.85 (1)	2.52 (3)	3.301 (4)	154 (6)
O3S—H2O3 \cdots O2A ⁱⁱⁱ	0.85 (1)	2.21 (5)	2.911 (5)	140 (6)
O4S—H1O4 \cdots O4A	0.84 (1)	2.11 (4)	2.834 (4)	144 (6)
O4S—H2O4 \cdots O2S	0.85 (1)	1.89 (2)	2.723 (5)	166 (6)

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

[Cr(ox)(cyclam)]⁺ cation and other water molecules, while N—H \cdots O hydrogen bonds involving the cyclam N—H groups and the O atoms of oxalate interconnect two [Cr(ox)(cyclam)]⁺ cations (Table 1, Figs. 1 and 2). An extensive array of these contacts generate a three-dimensional network of molecules (Fig. 2), and these hydrogen-bonding interactions help to stabilize the crystal structure.

4. Database survey

A search of the Cambridge Structural Database (Version 5.37, Feb 2016 with two updates; Groom *et al.*, 2016) gave just one hit for a [Cr(C₂O₄)(C₁₀H₂₄N₄)]⁺ unit, namely the complex [Cr(ox)(cyclam)]ClO₄ (Choi *et al.*, 2004b). However, the

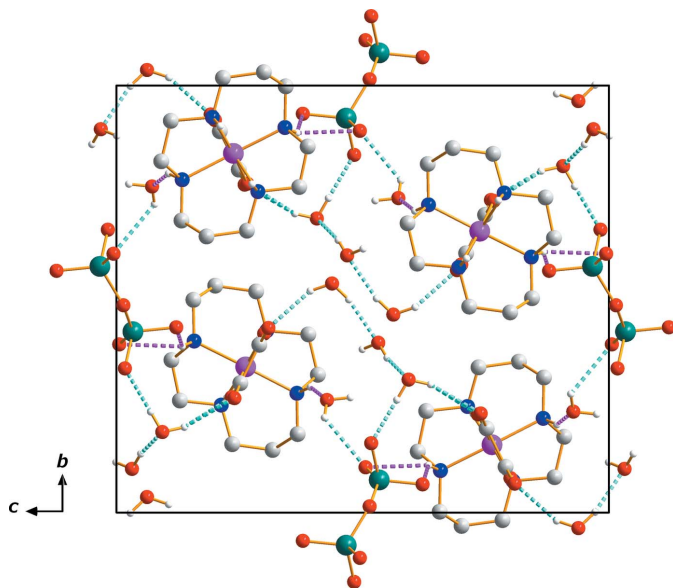


Figure 2
 The crystal packing in compound (I), viewed perpendicular to the bc plane. Dashed lines represent N—H \cdots O (pink) and O—H \cdots O (cyan) hydrogen-bonding interactions, respectively. C-bound H atoms have been omitted.

Table 2
 Experimental details.

Crystal data	
Chemical formula	[Cr(C ₂ O ₄)(C ₁₀ H ₂₄ N ₄) ₂][Cr ₂ O ₇] \cdot 8H ₂ O
M_r	1040.83
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	298
a, b, c (Å)	7.8270 (16), 15.407 (3), 18.086 (4)
β (°)	100.86 (3)
V (Å ³)	2141.9 (8)
Z	2
Radiation type	Synchrotron, $\lambda = 0.610$ Å
μ (mm ⁻¹)	0.71
Crystal size (mm)	0.15 \times 0.09 \times 0.08
Data collection	
Diffractometer	ADSC Q210 CCD area detector
Absorption correction	Empirical (using intensity measurements) (HKL3000sm SCALEPACK; Otwinowski & Minor, 1997)
T_{\min}, T_{\max}	0.889, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	21270, 5775, 4844
R_{int}	0.027
($\sin \theta/\lambda$) _{max} (Å ⁻¹)	0.693
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.048, 0.145, 1.04
No. of reflections	5775
No. of parameters	324
No. of restraints	24
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å ⁻³)	1.62, -0.69

Computer programs: PAL BL2D-SMDC (Shin *et al.*, 2016), HKL3000sm (Otwinowski & Minor, 1997), SHELXT2014 (Sheldrick, 2015a), SHELXL2016 (Sheldrick, 2015b), DIAMOND 4 (Putz & Brandenburg, 2014) and publCIF (Westrip, 2010).

structure of [CrCl₂(cyclam)][Cr(ox)(cyclam)](ClO₄)₂ (Moon & Choi, 2016b) has also been reported recently. Until now, no structure of the [Cr(ox)(cyclam)]⁺ cation with a dichromate counter-anion has been deposited.

5. Synthesis and crystallization

The free ligand cyclam (98%) was purchased from Sigma-Aldrich and used without further purification. All chemicals were reagent grade materials, and were used as received. The starting material, [Cr(ox)(cyclam)]ClO₄ was prepared according to the literature method (House & McKee, 1984). The perchlorate salt of the complex (0.03 g) was dissolved in 10 mL of distilled water at 347 K. The solution was filtered and the filtrate was added to 5 mL of water containing solid K₂Cr₂O₇ (0.02 g). Orange block-like crystals of (I) suitable for X-ray structural analysis were obtained after one week of slow evaporation at room temperature.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All H atoms were placed in geometrically idealized positions and constrained to ride on

their parent atoms, with C—H = 0.97 Å and N—H = 0.98 Å, and with $U_{\text{iso}}(\text{H})$ values of $1.2U_{\text{eq}}$ of the parent atoms. The hydrogen atoms of the solvent water molecules were assigned based on a difference-Fourier map, and the O—H distance and the H—O—H angle were restrained using DFIX and DANG constraints. The terminal O atoms of the dichromate anion are positionally disordered over two sets of sites. The occupancies of the respective pairs, O2B1/O2B2, O3B1/O3B2 and O4B1/O4B2, were refined freely and, for the O2B2 and O3B2 atoms, ISOR restraints were applied. The occupancy ratio refined to 0.586 (6):0.414 (6). The bridging O1B atom of the dichromate anion is also disordered, in this case about the inversion centre. Consequently the components were refined at half-occupancy. The bridging atoms O1B/O1B ($-x + 2, -y, -z + 1$) sites were refined using EXYZ/EADP restraints.

Acknowledgements

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supporting information

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Crystal structure of bis[(oxalato- κ^2O^1,O^2)(1,4,8,11-tetraazacyclotetradecane- κ^4N)chromium(III)] dichromate octahydrate from synchrotron X-ray data

Dohyun Moon and Jong-Ha Choi

Computing details

Data collection: *PAL BL2D-SMDC* (Shin *et al.*, 2016); cell refinement: *HKL3000sm* (Otwinowski & Minor, 1997); data reduction: *HKL3000sm* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXT2014* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2016* (Sheldrick, 2015b); molecular graphics: *DIAMOND 4* (Putz & Brandenburg, 2014); software used to prepare material for publication: *pubCIF* (Westrip, 2010).

Bis[(oxalato- κ^2O^1,O^2)(1,4,8,11-tetraazacyclotetradecane- κ^4N)chromium(III)] dichromate octahydrate

Crystal data

$[\text{Cr}(\text{C}_2\text{O}_4)(\text{C}_{10}\text{H}_{24}\text{N}_4)]_2[\text{Cr}_2\text{O}_7] \cdot 8\text{H}_2\text{O}$

$M_r = 1040.83$

Monoclinic, $P2_1/c$

$a = 7.8270$ (16) Å

$b = 15.407$ (3) Å

$c = 18.086$ (4) Å

$\beta = 100.86$ (3)°

$V = 2141.9$ (8) Å³

$Z = 2$

$F(000) = 1088$

$D_x = 1.614$ Mg m⁻³

Synchrotron radiation, $\lambda = 0.610$ Å

Cell parameters from 63673 reflections

$\theta = 0.4$ – 33.7 °

$\mu = 0.71$ mm⁻¹

$T = 298$ K

Block, orange

$0.15 \times 0.09 \times 0.08$ mm

Data collection

ADSC Q210 CCD area detector
diffractometer

Radiation source: PLSII 2D bending magnet
 ω scan

Absorption correction: empirical (using
intensity measurements)

(*HKL3000sm SCALEPACK*; Otwinowski &
Minor, 1997)

$T_{\min} = 0.889$, $T_{\max} = 1.000$

21270 measured reflections

5775 independent reflections

4844 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.027$

$\theta_{\max} = 25.0$ °, $\theta_{\min} = 2.3$ °

$h = -10 \rightarrow 10$

$k = -20 \rightarrow 20$

$l = -25 \rightarrow 25$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.048$

$wR(F^2) = 0.145$

$S = 1.04$

5775 reflections

324 parameters

24 restraints

Hydrogen site location: mixed

H atoms treated by a mixture of independent
and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0902P)^2 + 1.1358P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.002$

$\Delta\rho_{\max} = 1.62$ e Å⁻³

$\Delta\rho_{\min} = -0.69$ e Å⁻³

Extinction correction: SHELXL2016
 (Sheldrick, 2015b),
 $F_c^* = kF_c [1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
 Extinction coefficient: 0.037 (3)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Cr1A	0.38205 (4)	0.34112 (2)	0.73752 (2)	0.02641 (13)	
O1A	0.59297 (19)	0.27010 (12)	0.76591 (10)	0.0396 (4)	
O2A	0.8739 (2)	0.26797 (16)	0.76043 (14)	0.0626 (6)	
O3A	0.54391 (19)	0.41929 (11)	0.69995 (10)	0.0396 (4)	
O4A	0.8222 (2)	0.43225 (16)	0.69140 (14)	0.0598 (6)	
N1A	0.1816 (2)	0.42579 (13)	0.69489 (12)	0.0378 (4)	
H1A	0.075547	0.405210	0.710638	0.045*	
N2A	0.3113 (2)	0.27935 (15)	0.63360 (12)	0.0409 (4)	
H2A	0.403674	0.293806	0.605848	0.049*	
N3A	0.2422 (2)	0.25158 (13)	0.78721 (12)	0.0390 (4)	
H3A	0.119637	0.269026	0.775430	0.047*	
N4A	0.4158 (3)	0.40170 (14)	0.84214 (11)	0.0406 (4)	
H4A	0.535961	0.389115	0.866550	0.049*	
C1A	0.1568 (4)	0.4172 (2)	0.61046 (18)	0.0582 (8)	
H1AA	0.049076	0.445014	0.586857	0.070*	
H1AB	0.252088	0.445169	0.592350	0.070*	
C2A	0.1514 (4)	0.3226 (2)	0.59104 (18)	0.0612 (8)	
H2AA	0.048592	0.296162	0.604142	0.073*	
H2AB	0.145782	0.315414	0.537358	0.073*	
C3A	0.3008 (4)	0.1838 (2)	0.6321 (2)	0.0609 (8)	
H3AA	0.417078	0.160207	0.647097	0.073*	
H3AB	0.256590	0.165122	0.580818	0.073*	
C4A	0.1857 (4)	0.1466 (2)	0.6829 (2)	0.0638 (9)	
H4AA	0.071236	0.172674	0.669584	0.077*	
H4AB	0.172624	0.084736	0.673435	0.077*	
C5A	0.2513 (4)	0.16009 (18)	0.7649 (2)	0.0586 (8)	
H5AA	0.183071	0.125105	0.793222	0.070*	
H5AB	0.371013	0.140530	0.777515	0.070*	
C6A	0.3031 (4)	0.2623 (2)	0.87113 (18)	0.0597 (8)	
H6AA	0.224823	0.232351	0.898189	0.072*	
H6AB	0.418710	0.238033	0.886411	0.072*	
C7A	0.3053 (5)	0.3571 (3)	0.88827 (18)	0.0640 (8)	
H7AA	0.187953	0.380198	0.876854	0.077*	
H7AB	0.351179	0.366653	0.941312	0.077*	
C8A	0.3993 (4)	0.4980 (2)	0.8437 (2)	0.0605 (8)	

H8AA	0.493986	0.523712	0.823521	0.073*	
H8AB	0.410781	0.516864	0.895598	0.073*	
C9A	0.2285 (4)	0.5313 (2)	0.7993 (2)	0.0657 (9)	
H9AA	0.133959	0.502373	0.817229	0.079*	
H9AB	0.219591	0.592861	0.809118	0.079*	
C10A	0.2063 (4)	0.51784 (19)	0.7163 (2)	0.0595 (8)	
H10A	0.106358	0.550800	0.691276	0.071*	
H10B	0.308132	0.539863	0.699087	0.071*	
C11A	0.7305 (3)	0.30305 (18)	0.74841 (14)	0.0415 (5)	
C12A	0.7022 (3)	0.39281 (18)	0.70995 (14)	0.0412 (5)	
Cr2B	0.86158 (6)	0.07641 (3)	0.46646 (2)	0.04561 (15)	
O1B	0.9327 (6)	-0.0145 (4)	0.5141 (3)	0.0780 (16)	0.5
O2B1	0.814 (2)	0.0661 (9)	0.3785 (7)	0.068 (3)	0.586 (6)
O3B1	0.9854 (7)	0.1624 (3)	0.4827 (3)	0.0797 (17)	0.586 (6)
O4B1	0.6779 (6)	0.1061 (4)	0.4942 (3)	0.0811 (16)	0.586 (6)
O2B2	0.7152 (9)	0.0097 (6)	0.4864 (4)	0.093 (3)	0.414 (6)
O3B2	0.8779 (13)	0.1556 (6)	0.5165 (5)	0.097 (3)	0.414 (6)
O4B2	0.812 (3)	0.0895 (14)	0.3789 (11)	0.075 (5)	0.414 (6)
O1S	0.6259 (5)	0.2636 (2)	0.57243 (19)	0.0909 (10)	
H1O1	0.698 (7)	0.222 (3)	0.577 (3)	0.136*	
H2O1	0.596 (8)	0.269 (4)	0.5252 (8)	0.136*	
O2S	0.6340 (5)	0.3977 (2)	0.4684 (2)	0.0935 (9)	
H1O2	0.576 (7)	0.382 (4)	0.501 (3)	0.140*	
H2O2	0.698 (7)	0.362 (3)	0.450 (3)	0.140*	
O3S	0.8777 (6)	0.3113 (2)	0.4068 (2)	0.1014 (11)	
H1O3	0.880 (9)	0.264 (2)	0.431 (3)	0.152*	
H2O3	0.827 (8)	0.300 (4)	0.3622 (14)	0.152*	
O4S	0.7101 (5)	0.5364 (2)	0.56219 (18)	0.0920 (9)	
H1O4	0.708 (9)	0.519 (3)	0.6063 (12)	0.138*	
H2O4	0.675 (8)	0.499 (3)	0.528 (2)	0.138*	

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cr1A	0.01035 (16)	0.0353 (2)	0.03406 (19)	0.00068 (10)	0.00548 (11)	-0.00334 (12)
O1A	0.0188 (7)	0.0472 (10)	0.0521 (9)	0.0076 (6)	0.0050 (6)	-0.0005 (7)
O2A	0.0197 (8)	0.0833 (16)	0.0835 (15)	0.0165 (8)	0.0066 (8)	-0.0129 (12)
O3A	0.0177 (7)	0.0490 (10)	0.0546 (10)	-0.0027 (6)	0.0133 (6)	0.0023 (7)
O4A	0.0229 (8)	0.0871 (16)	0.0737 (14)	-0.0124 (9)	0.0203 (8)	-0.0010 (11)
N1A	0.0173 (8)	0.0424 (11)	0.0544 (11)	0.0062 (7)	0.0089 (7)	0.0063 (8)
N2A	0.0258 (9)	0.0561 (12)	0.0409 (10)	-0.0004 (8)	0.0066 (7)	-0.0124 (9)
N3A	0.0216 (8)	0.0435 (11)	0.0537 (11)	-0.0007 (7)	0.0115 (8)	0.0059 (8)
N4A	0.0273 (9)	0.0533 (12)	0.0408 (10)	-0.0009 (8)	0.0058 (7)	-0.0127 (8)
C1A	0.0373 (13)	0.080 (2)	0.0565 (16)	0.0114 (13)	0.0069 (11)	0.0225 (15)
C2A	0.0434 (15)	0.087 (2)	0.0473 (15)	0.0015 (15)	-0.0069 (12)	-0.0098 (14)
C3A	0.0495 (16)	0.0600 (18)	0.073 (2)	-0.0032 (13)	0.0114 (14)	-0.0286 (15)
C4A	0.0488 (17)	0.0501 (17)	0.093 (2)	-0.0108 (12)	0.0130 (16)	-0.0183 (15)
C5A	0.0442 (15)	0.0407 (15)	0.092 (2)	-0.0025 (10)	0.0169 (15)	0.0052 (13)

C6A	0.0497 (16)	0.077 (2)	0.0546 (16)	0.0013 (14)	0.0162 (13)	0.0168 (14)
C7A	0.067 (2)	0.086 (2)	0.0446 (15)	-0.0042 (17)	0.0243 (14)	-0.0113 (14)
C8A	0.0517 (17)	0.0561 (18)	0.074 (2)	-0.0037 (13)	0.0125 (14)	-0.0275 (15)
C9A	0.0498 (17)	0.0528 (18)	0.097 (3)	0.0099 (13)	0.0195 (16)	-0.0179 (16)
C10A	0.0376 (14)	0.0447 (15)	0.098 (2)	0.0084 (11)	0.0189 (14)	0.0074 (14)
C11A	0.0160 (9)	0.0608 (15)	0.0470 (12)	0.0055 (9)	0.0041 (8)	-0.0140 (10)
C12A	0.0177 (9)	0.0612 (15)	0.0472 (12)	-0.0071 (9)	0.0120 (8)	-0.0119 (10)
Cr2B	0.0406 (2)	0.0553 (3)	0.0379 (2)	0.00143 (17)	-0.00027 (16)	0.00325 (16)
O1B	0.058 (3)	0.083 (3)	0.094 (4)	0.032 (3)	0.016 (3)	0.051 (3)
O2B1	0.074 (5)	0.082 (6)	0.042 (4)	0.010 (4)	-0.002 (3)	-0.009 (4)
O3B1	0.076 (3)	0.065 (3)	0.086 (3)	-0.025 (2)	-0.016 (3)	-0.006 (2)
O4B1	0.066 (3)	0.095 (4)	0.087 (3)	0.011 (2)	0.027 (2)	-0.005 (3)
O2B2	0.065 (4)	0.126 (6)	0.094 (5)	-0.007 (4)	0.027 (3)	0.045 (4)
O3B2	0.096 (3)	0.095 (3)	0.097 (3)	0.0059 (19)	0.0135 (19)	-0.0124 (18)
O4B2	0.050 (5)	0.113 (13)	0.060 (7)	0.023 (7)	0.009 (5)	0.047 (7)
O1S	0.097 (2)	0.102 (2)	0.089 (2)	0.0078 (17)	0.0558 (19)	-0.0074 (17)
O2S	0.110 (3)	0.082 (2)	0.089 (2)	0.0060 (18)	0.0202 (18)	-0.0030 (16)
O3S	0.131 (3)	0.084 (2)	0.082 (2)	0.013 (2)	0.003 (2)	-0.0045 (16)
O4S	0.111 (3)	0.091 (2)	0.0739 (18)	0.0025 (19)	0.0187 (19)	-0.0037 (15)

Geometric parameters (Å, °)

Cr1A—O3A	1.9600 (16)	C5A—H5AB	0.9700
Cr1A—O1A	1.9665 (16)	C6A—C7A	1.493 (5)
Cr1A—N3A	2.069 (2)	C6A—H6AA	0.9700
Cr1A—N1A	2.0739 (19)	C6A—H6AB	0.9700
Cr1A—N4A	2.081 (2)	C7A—H7AA	0.9700
Cr1A—N2A	2.086 (2)	C7A—H7AB	0.9700
O1A—C11A	1.283 (3)	C8A—C9A	1.513 (5)
O2A—C11A	1.228 (3)	C8A—H8AA	0.9700
O3A—C12A	1.284 (3)	C8A—H8AB	0.9700
O4A—C12A	1.218 (3)	C9A—C10A	1.493 (5)
N1A—C10A	1.473 (4)	C9A—H9AA	0.9700
N1A—C1A	1.508 (4)	C9A—H9AB	0.9700
N1A—H1A	0.9800	C10A—H10A	0.9700
N2A—C3A	1.475 (4)	C10A—H10B	0.9700
N2A—C2A	1.497 (4)	C11A—C12A	1.545 (4)
N2A—H2A	0.9800	Cr2B—O3B2	1.511 (8)
N3A—C5A	1.472 (4)	Cr2B—O4B2	1.571 (19)
N3A—C6A	1.512 (4)	Cr2B—O2B1	1.572 (12)
N3A—H3A	0.9800	Cr2B—O2B2	1.630 (7)
N4A—C7A	1.479 (4)	Cr2B—O3B1	1.635 (4)
N4A—C8A	1.491 (4)	Cr2B—O4B1	1.673 (5)
N4A—H4A	0.9800	Cr2B—O1B	1.684 (4)
C1A—C2A	1.498 (5)	Cr2B—O1B ⁱ	1.847 (4)
C1A—H1AA	0.9700	O1B—O1B ⁱ	1.332 (10)
C1A—H1AB	0.9700	O1B—O2B2	1.723 (9)
C2A—H2AA	0.9700	O1S—H1O1	0.845 (10)

C2A—H2AB	0.9700	O1S—H2O1	0.847 (10)
C3A—C4A	1.516 (5)	O2S—H1O2	0.841 (10)
C3A—H3AA	0.9700	O2S—H2O2	0.845 (10)
C3A—H3AB	0.9700	O3S—H1O3	0.845 (10)
C4A—C5A	1.489 (5)	O3S—H2O3	0.848 (10)
C4A—H4AA	0.9700	O4S—H1O4	0.843 (10)
C4A—H4AB	0.9700	O4S—H2O4	0.851 (10)
C5A—H5AA	0.9700		
O3A—Cr1A—O1A	82.34 (7)	N3A—C5A—H5AB	109.1
O3A—Cr1A—N3A	171.83 (8)	C4A—C5A—H5AB	109.1
O1A—Cr1A—N3A	90.14 (8)	H5AA—C5A—H5AB	107.8
O3A—Cr1A—N1A	88.72 (7)	C7A—C6A—N3A	107.7 (2)
O1A—Cr1A—N1A	170.42 (8)	C7A—C6A—H6AA	110.2
N3A—Cr1A—N1A	98.97 (8)	N3A—C6A—H6AA	110.2
O3A—Cr1A—N4A	93.44 (8)	C7A—C6A—H6AB	110.2
O1A—Cr1A—N4A	93.19 (8)	N3A—C6A—H6AB	110.2
N3A—Cr1A—N4A	83.74 (9)	H6AA—C6A—H6AB	108.5
N1A—Cr1A—N4A	90.77 (9)	N4A—C7A—C6A	108.8 (2)
O3A—Cr1A—N2A	92.69 (8)	N4A—C7A—H7AA	109.9
O1A—Cr1A—N2A	92.78 (8)	C6A—C7A—H7AA	109.9
N3A—Cr1A—N2A	90.86 (9)	N4A—C7A—H7AB	109.9
N1A—Cr1A—N2A	84.17 (9)	C6A—C7A—H7AB	109.9
N4A—Cr1A—N2A	171.96 (8)	H7AA—C7A—H7AB	108.3
C11A—O1A—Cr1A	114.69 (16)	N4A—C8A—C9A	113.4 (2)
C12A—O3A—Cr1A	115.17 (17)	N4A—C8A—H8AA	108.9
C10A—N1A—C1A	109.6 (2)	C9A—C8A—H8AA	108.9
C10A—N1A—Cr1A	117.10 (17)	N4A—C8A—H8AB	108.9
C1A—N1A—Cr1A	105.39 (15)	C9A—C8A—H8AB	108.9
C10A—N1A—H1A	108.1	H8AA—C8A—H8AB	107.7
C1A—N1A—H1A	108.1	C10A—C9A—C8A	114.2 (3)
Cr1A—N1A—H1A	108.1	C10A—C9A—H9AA	108.7
C3A—N2A—C2A	113.4 (2)	C8A—C9A—H9AA	108.7
C3A—N2A—Cr1A	118.47 (19)	C10A—C9A—H9AB	108.7
C2A—N2A—Cr1A	108.32 (17)	C8A—C9A—H9AB	108.7
C3A—N2A—H2A	105.2	H9AA—C9A—H9AB	107.6
C2A—N2A—H2A	105.2	N1A—C10A—C9A	112.5 (3)
Cr1A—N2A—H2A	105.2	N1A—C10A—H10A	109.1
C5A—N3A—C6A	111.0 (2)	C9A—C10A—H10A	109.1
C5A—N3A—Cr1A	117.56 (18)	N1A—C10A—H10B	109.1
C6A—N3A—Cr1A	105.70 (17)	C9A—C10A—H10B	109.1
C5A—N3A—H3A	107.4	H10A—C10A—H10B	107.8
C6A—N3A—H3A	107.4	O2A—C11A—O1A	124.3 (3)
Cr1A—N3A—H3A	107.4	O2A—C11A—C12A	121.5 (2)
C7A—N4A—C8A	112.9 (2)	O1A—C11A—C12A	114.15 (18)
C7A—N4A—Cr1A	108.49 (17)	O4A—C12A—O3A	125.2 (3)
C8A—N4A—Cr1A	117.86 (19)	O4A—C12A—C11A	121.2 (2)
C7A—N4A—H4A	105.5	O3A—C12A—C11A	113.63 (19)

C8A—N4A—H4A	105.5	O3B2—Cr2B—O4B2	118.5 (8)
Cr1A—N4A—H4A	105.5	O3B2—Cr2B—O2B2	111.1 (5)
C2A—C1A—N1A	108.4 (2)	O4B2—Cr2B—O2B2	104.7 (8)
C2A—C1A—H1AA	110.0	O2B1—Cr2B—O3B1	106.5 (6)
N1A—C1A—H1AA	110.0	O2B1—Cr2B—O4B1	106.1 (6)
C2A—C1A—H1AB	110.0	O3B1—Cr2B—O4B1	103.8 (3)
N1A—C1A—H1AB	110.0	O3B2—Cr2B—O1B	112.5 (4)
H1AA—C1A—H1AB	108.4	O4B2—Cr2B—O1B	128.2 (8)
N2A—C2A—C1A	109.2 (2)	O2B1—Cr2B—O1B	115.3 (6)
N2A—C2A—H2AA	109.8	O2B2—Cr2B—O1B	62.6 (3)
C1A—C2A—H2AA	109.8	O3B1—Cr2B—O1B	117.0 (3)
N2A—C2A—H2AB	109.8	O4B1—Cr2B—O1B	107.1 (3)
C1A—C2A—H2AB	109.8	O3B2—Cr2B—O1B ⁱ	109.3 (4)
H2AA—C2A—H2AB	108.3	O4B2—Cr2B—O1B ⁱ	107.5 (8)
N2A—C3A—C4A	113.8 (2)	O2B1—Cr2B—O1B ⁱ	100.0 (6)
N2A—C3A—H3AA	108.8	O2B2—Cr2B—O1B ⁱ	104.8 (3)
C4A—C3A—H3AA	108.8	O3B1—Cr2B—O1B ⁱ	85.3 (3)
N2A—C3A—H3AB	108.8	O4B1—Cr2B—O1B ⁱ	148.4 (2)
C4A—C3A—H3AB	108.8	O1B—Cr2B—O1B ⁱ	44.0 (3)
H3AA—C3A—H3AB	107.7	O1B ⁱ —O1B—Cr2B	74.5 (3)
C5A—C4A—C3A	114.7 (3)	O1B ⁱ —O1B—O2B2	128.7 (5)
C5A—C4A—H4AA	108.6	Cr2B—O1B—O2B2	57.1 (3)
C3A—C4A—H4AA	108.6	O1B ⁱ —O1B—Cr2B ⁱ	61.5 (3)
C5A—C4A—H4AB	108.6	Cr2B—O1B—Cr2B ⁱ	136.0 (3)
C3A—C4A—H4AB	108.6	Cr2B—O2B2—O1B	60.2 (3)
H4AA—C4A—H4AB	107.6	H1O1—O1S—H2O1	104 (2)
N3A—C5A—C4A	112.4 (3)	H1O2—O2S—H2O2	122 (3)
N3A—C5A—H5AA	109.1	H1O3—O3S—H2O3	105 (2)
C4A—C5A—H5AA	109.1	H1O4—O4S—H2O4	114 (3)
C10A—N1A—C1A—C2A	174.0 (2)	O2A—C11A—C12A—O4A	-1.2 (4)
Cr1A—N1A—C1A—C2A	47.1 (2)	O1A—C11A—C12A—O4A	178.8 (2)
C3A—N2A—C2A—C1A	166.9 (3)	O2A—C11A—C12A—O3A	178.4 (2)
Cr1A—N2A—C2A—C1A	33.3 (3)	O1A—C11A—C12A—O3A	-1.6 (3)
N1A—C1A—C2A—N2A	-54.5 (3)	O3B2—Cr2B—O1B—O1B ⁱ	-95.0 (7)
C2A—N2A—C3A—C4A	-75.2 (3)	O4B2—Cr2B—O1B—O1B ⁱ	74.6 (12)
Cr1A—N2A—C3A—C4A	53.4 (3)	O2B1—Cr2B—O1B—O1B ⁱ	77.7 (9)
N2A—C3A—C4A—C5A	-66.3 (4)	O2B2—Cr2B—O1B—O1B ⁱ	162.1 (8)
C6A—N3A—C5A—C4A	177.8 (2)	O3B1—Cr2B—O1B—O1B ⁱ	-48.7 (7)
Cr1A—N3A—C5A—C4A	-60.4 (3)	O4B1—Cr2B—O1B—O1B ⁱ	-164.6 (5)
C3A—C4A—C5A—N3A	69.8 (4)	O3B2—Cr2B—O1B—O2B2	103.0 (6)
C5A—N3A—C6A—C7A	175.0 (2)	O4B2—Cr2B—O1B—O2B2	-87.4 (11)
Cr1A—N3A—C6A—C7A	46.5 (3)	O1B ⁱ —Cr2B—O1B—O2B2	-162.1 (8)
C8A—N4A—C7A—C6A	168.8 (3)	O3B2—Cr2B—O1B—Cr2B ⁱ	-95.0 (7)
Cr1A—N4A—C7A—C6A	36.2 (3)	O4B2—Cr2B—O1B—Cr2B ⁱ	74.6 (12)
N3A—C6A—C7A—N4A	-55.8 (3)	O2B1—Cr2B—O1B—Cr2B ⁱ	77.7 (9)
C7A—N4A—C8A—C9A	-72.7 (4)	O2B2—Cr2B—O1B—Cr2B ⁱ	162.1 (8)
Cr1A—N4A—C8A—C9A	55.1 (3)	O3B1—Cr2B—O1B—Cr2B ⁱ	-48.7 (7)

N4A—C8A—C9A—C10A	-67.0 (4)	O4B1—Cr2B—O1B—Cr2B ⁱ	-164.6 (5)
C1A—N1A—C10A—C9A	179.0 (2)	O1B ⁱ —Cr2B—O1B—Cr2B ⁱ	0.005 (1)
Cr1A—N1A—C10A—C9A	-61.1 (3)	O3B2—Cr2B—O2B2—O1B	-105.2 (5)
C8A—C9A—C10A—N1A	70.3 (4)	O4B2—Cr2B—O2B2—O1B	125.8 (8)
Cr1A—O1A—C11A—O2A	-178.9 (2)	O1B ⁱ —Cr2B—O2B2—O1B	12.8 (5)
Cr1A—O1A—C11A—C12A	1.1 (3)	O1B ⁱ —O1B—O2B2—Cr2B	-22.3 (10)
Cr1A—O3A—C12A—O4A	-179.2 (2)	Cr2B ⁱ —O1B—O2B2—Cr2B	-138.9 (13)
Cr1A—O3A—C12A—C11A	1.2 (3)		

Symmetry code: (i) $-x+2, -y, -z+1$.

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>
N1A—H1A \cdots O4A ⁱⁱ	0.98	1.99	2.804 (3)	139
N2A—H2A \cdots O1S	0.98	2.00	2.894 (3)	150
N3A—H3A \cdots O2A ⁱⁱ	0.98	1.89	2.842 (3)	163
N4A—H4A \cdots O2B1 ⁱⁱⁱ	0.98	2.25	3.100 (16)	144
N4A—H4A \cdots O4B1 ⁱⁱⁱ	0.98	2.37	3.108 (6)	132
N4A—H4A \cdots O4B2 ⁱⁱⁱ	0.98	2.15	3.05 (2)	151
O1S—H1O1 \cdots O4B1	0.85 (1)	2.33 (5)	2.876 (6)	123 (5)
O1S—H1O1 \cdots O3B2	0.85 (1)	2.20 (4)	2.903 (10)	141 (5)
O2S—H2O2 \cdots O3S	0.85 (1)	1.91 (2)	2.729 (6)	164 (6)
O3S—H1O3 \cdots O3B1	0.85 (1)	1.93 (3)	2.725 (6)	156 (7)
O3S—H1O3 \cdots O3B2	0.85 (1)	2.28 (2)	3.113 (10)	167 (6)
O3S—H2O3 \cdots O1A ^{iv}	0.85 (1)	2.52 (3)	3.301 (4)	154 (6)
O3S—H2O3 \cdots O2A ^{iv}	0.85 (1)	2.21 (5)	2.911 (5)	140 (6)
O4S—H1O4 \cdots O4A	0.84 (1)	2.11 (4)	2.834 (4)	144 (6)
O4S—H2O4 \cdots O2S	0.85 (1)	1.89 (2)	2.723 (5)	166 (6)

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