research communications

Crystal structure of bis[*cis*-(1,4,8,11-tetraazacyclotetradecane-$\kappa^4 N$)bis(thiocyanato-κN)chromium(III)] dichromate monohydrate from synchrotron X-ray diffraction data

Dohyun Moon,[a] Masahiro Takase,[b] Takashiro Akitsu[b] and Jong-Ha Choi[c]*

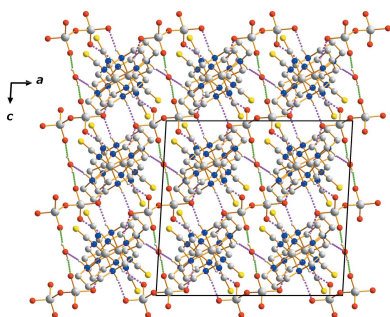[a]Pohang Accelerator Laboratory, POSTECH, Pohang 37673, Republic of Korea, [b]Department of Chemistry, Tokyo University of Science, 1-3 Kagurazaka, Shinjuku-ku, Tokyo 162-8601, Japan, and [c]Department of Chemistry, Andong National University, Andong 36729, Republic of Korea. *Correspondence e-mail: jhchoi@anu.ac.kr

The structure of the complex salt, *cis*-[Cr(NCS)$_2$(cyclam)]$_2$[Cr$_2$O$_7$]·H$_2$O (cyclam = 1,4,8,11-tetraazacyclotetradecane, C$_{10}$H$_{24}$N$_4$), has been determined from synchrotron data. The asymmetric unit comprises of one [Cr(NCS)$_2$(cyclam)]$^+$ cation, one half of a Cr$_2$O$_7^{2-}$ anion (completed by inversion symmetry) and one half of a water molecule (completed by twofold rotation symmetry). The Cr^{III} ion is coordinated by the four cyclam N atoms and by two N atoms of *cis*-arranged thiocyanate anions, displaying a distorted octahedral coordination sphere. The Cr—N(cyclam) bond lengths are in the range 2.080 (2) to 2.097 (2) Å while the average Cr—N(NCS) bond length is 1.985 (4) Å. The macrocyclic cyclam moiety adopts the *cis*-V conformation. The bridging O atom of the dichromate anion is disordered around an inversion centre, leading to a bending of the Cr—O—Cr bridging angle [157.7 (3)°]; the 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 donor groups, and the O atoms of the Cr$_2$O$_7^{2-}$ anion and water molecules as acceptor groups, giving rise to a three-dimensional network.

1. Chemical context

Recently, it has been established that cyclam (1,4,8,11-tetraazacyclotetradecane, C$_{10}$H$_{24}$N$_4$) derivatives and their complexes can exhibit anti-HIV effects or stimulate the activity of stem cells from bone marrow (Ronconi & Sadler, 2007; De Clercq, 2010; Ross *et al.*, 2012). Cyclam has a moderately flexible structure and can adopt both planar (*trans*) and folded (*cis*) conformations (Poon & Pun, 1980). There are five configurational *trans* isomers for the macrocycle, which differ in the chirality of the *sec*-NH sites (Choi, 2009). The *trans*-I, *trans*-II and *trans*-V configurations also can fold to form *cis*-I, *cis*-II and *cis*-V isomers, respectively (Subhan *et al.*, 2011). The configuration of the macrocyclic ligand and the influence of the counter-anion are important factors in developing new highly effective anti-HIV drugs.

The dichromate anion is environmentally important due to its high toxicity (Yusof & Malek, 2009), and its use in industrial processing (Goyal *et al.*, 2003). Since counter-anionic species play an important role in coordination chemistry (Martínez-Máñez & Sancenón, 2003; Fabbrizzi & Poggi, 2013), it may be possible that the [Cr(NCS)$_2$(cyclam)]$^+$ cation is suitable to bind specifically to an oxoanion. In this context, we report here on the synthesis of a new chromium(III)–dichro-

mate salt, [Cr(NCS)$_2$(cyclam)]$_2$(Cr$_2$O$_7$)·H$_2$O, (I), and its structural characterization by synchrotron single-crystal X-ray diffraction.
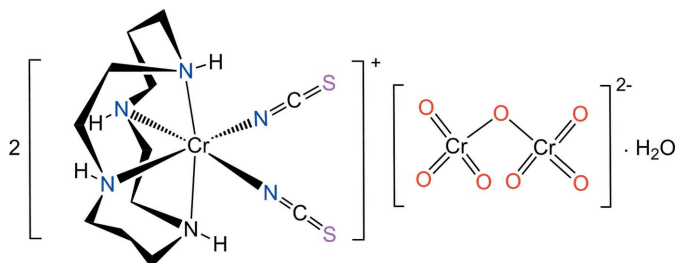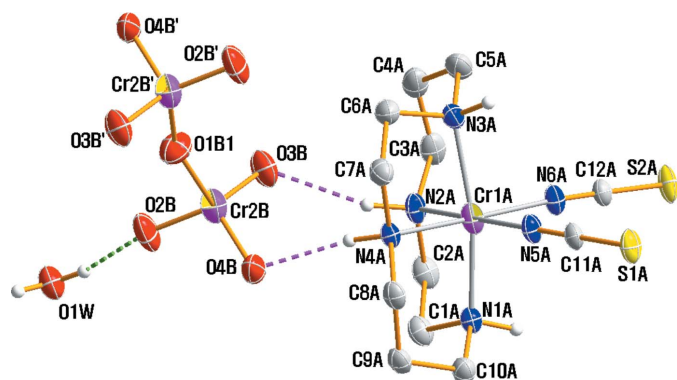

2. Structural commentary

Fig. 1 displays the molecular components of (I). The structure is another example of a [Cr(NCS)$_2$(cyclam)]$^+$ cation (Friesen *et al.*, 1997; Moon *et al.*, 2013) but with a different counter-anion. The asymmetric unit comprises of one [Cr(NCS)$_2$(cyclam)]$^+$ cation, one half of a Cr$_2$O$_7^{2-}$ anion (completed by inversion symmetry) and one half of a water molecule (completed by twofold rotation symmetry). In the complex cation, the Cr^{III} ion is coordinated by the N atoms of the cyclam ligand in the folded conformation. The nitrogen atoms of two NCS$^-$ ligands coordinate to the chromium atoms in a *cis* arrangement. The cyclam moiety adopts the *cis*-V (*anti–anti*) conformation (Subhan *et al.*, 2011). The Cr—N(cyclam) bond lengths are in the range 2.080 (2) to 2.097 (2) Å, in good agreement with those determined in related structures, namely *cis*-[Cr(NCS)$_2$(cyclam)]SCN [2.0851 (14)– 2.0897 (14) Å; Moon *et al.*, 2013], *cis*-[Cr(N$_3$)$_2$(cyclam)]ClO$_4$ [2.069 (3)–2.103 (3) Å; Meyer *et al.*, 1998], *cis*-[Cr(ONO)$_2$(cyclam)]NO$_2$ [2.0874 (16)–2.0916 (15) Å; Choi *et al.*, 2004a], [Cr(acac)(cyclam)](ClO$_4$)$_2$·0.5H$_2$O [2.070(5–2.089 (5) Å, acac = acetylacetonate; Subhan *et al.*, 2011] or *cis*-[CrCl$_2$(cyclam)][Cr(ox)(cyclam)](ClO$_4$)$_2$ [2.075 (5)–2.096 (5) Å; Moon

& Choi, 2016a]. The Cr—N(cyclam) bond lengths with co-ligands in *cis* orientations are slightly longer than those found in *trans*-[Cr(NCS)$_2$(cyclam)]ClO$_4$ [2.046 (2)–2.060 (2) Å; Friesen *et al.*, 1997], *trans*-[Cr(ONO)$_2$(cyclam)]BF$_4$ [2.064 (4)– 2.073 (4) Å; De Leo *et al.*, 2000], *trans*-[Cr(NH$_3$)$_2$(cyclam)][ZnCl$_4$]Cl·H$_2$O [2.0501 (15)–2.0615 (15) Å; Moon & Choi, 2016b] or *trans*-[Cr(nic-O)$_2$(cyclam)]ClO$_4$ [2.058 (4) – 2.064 (4) Å, nic-O = O-coordinating nicotinate; Choi, 2009]. The two Cr—N(NCS) bond lengths in (I) average to 1.985 (4) Å and are close to the values found in *cis*-[Cr(NCS)$_2$(cyclam)]NCS [1.996 (15) Å; Moon *et al.*, 2013], *cis*-[Cr(NCS)$_2$(cyclam)]ClO$_4$ [1.981 (4)–1.998 (4) Å; Friesen *et al.*, 1997], *trans*-[Cr(NCS)$_2$(cyclam)]$_2$[ZnCl$_4$] [1.995 (6) Å; Moon *et al.*, 2015a] or *trans*-[Cr(NCS)$_2$(Me$_2$tn)$_2$]SCN·0.5H$_2$O [1.983 (2)–1.990 (2) Å; Choi & Lee, 2009]. The five- and six-membered chelate rings of the cyclam ligand adopt *gauche* and stable chair conformations, respectively. The folded angle [96.05 (8)°] of cyclam is comparable to the values of 98.55 (2), 97.17 (5), 97.03 (2), 95.09 (9), 94.51 (2) and 92.8 (2)° in [Cr(ox)(cyclam)]ClO$_4$, *cis*-[Cr(NCS)$_2$(cyclam)]SCN, [Cr(acac)(cyclam)](ClO$_4$)$_2$·0.5H$_2$O, *cis*-[Cr(ONO)$_2$(cyclam)]-NO$_2$, *cis*-[Cr(N$_3$)$_2$(cyclam)]ClO$_4$ and *cis*-[Cr(cyclam)Cl$_2$]Cl, respectively (Choi *et al.*, 2004b; Moon *et al.*, 2013; Subhan *et al.*, 2011; Choi *et al.*, 2004a; Meyer *et al.*, 1998; Forsellini *et al.*, 1986, respectively).

The two N-bound thiocyanate anions are almost linear, with N—C—S angles of 178.8 (2) and 179.0 (3)°. The bridging O atom of the Cr$_2$O$_7^{2-}$ anion is positionally disordered over an inversion centre, giving rise to a bending of the Cr2*B*—O1*B*—Cr2*B*(−x + 1, −y + 1, −z + 1) angle [157.7 (3)°]. The Cr$_2$O$_7^{2-}$ anion in (I) has a staggered conformation while a nearly eclipsed conformation is observed in ionic compounds K$_2$Cr$_2$O$_7$, Rb$_2$Cr$_2$O$_7$ and (C$_3$H$_5$N$_2$)(NH$_4$)[Cr$_2$O$_7$] (Brandon & Brown, 1968; Löfgren, 1971; Zhu, 2012). The conformation of the dichromate anion is influenced by the charge and size of the counter-cation (Moon *et al.*, 2015b; Moon & Choi, 2016). The O—Cr2*B*—O bond angles range from 102.3 (3) to 119.5 (2)°; the terminal Cr2*B*—O bond lengths vary from 1.596 (2) to 1.612 (2) Å, with a mean terminal Cr2*B*—O bond length of 1.604 (12) Å. The bridging Cr2*B*—O1*B* bond has a length of 1.746 (9) Å. These values are comparable to those reported for the anions in the structures of [Cr(urea)$_6$]-(Cr$_2$O$_7$)Br·H$_2$O (Moon *et al.*, 2015b) or [CrCl$_2$(tn)$_2$]$_2$(Cr$_2$O$_7$) (tn = propane-1,3-diamine; Moon & Choi, 2016). A further distortion of the anion is due to its involvement in hydrogen-bonding interactions with water molecule and complex cation (see *Supramolecular features*).

Figure 1
The molecular components in the structure of (I) with displacement ellipsoids drawn at the 30% probability level. Only one orientation of the disordered anion is shown; primed atoms are related by symmetry code (−x, −y + 1, −z − $\frac{1}{2}$). Dashed lines represent hydrogen bonds.

3. Supramolecular features

Two O—H···O hydrogen bonds link the water molecule to neighboring Cr$_2$O$_7^{2-}$ anions while N—H···O hydrogen bonds interconnect [Cr(NCS)$_2$(cyclam)]$^+$ cations with both the anions and water molecules (Table 1; Figs. 1 and 2) . An extensive array of these contacts generates a three-dimensional network of molecules stacked along the *c*-axis.

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N1A-H1A\cdots O1W^{i}$	0.99	2.15	3.089 (3)	157
$N2A-H2A\cdots O3B$	0.99	2.17	3.127 (3)	163
$N3A-H3A\cdots O4B^{ii}$	0.99	2.10	2.953 (3)	143
$N4A-H4A\cdots O4B$	0.99	1.99	2.904 (3)	152
$O1W-H1OW\cdots O2B$	0.84 (1)	2.24 (1)	3.052 (3)	164 (2)

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

4. Database survey

A search of the Cambridge Structural Database (Version 5.37, Feb 2016 with two updates; Groom *et al.*, 2016) gave 17 hits for a *cis*-$[CrL_2(C_{10}H_{24}N_4)]^+$ unit.

5. Synthesis and crystallization

Cyclam was purchased from Stream Chemicals and used as provided. All chemicals were reagent-grade materials and used without further purification. The starting material, *cis*-$[Cr(NCS)_2(cyclam)]SCN$ was prepared according to a literature protocol (Ferguson & Tobe, 1970). The thiocyanate salt (0.513 g) was dissolved in 15 mL water at 347 K. The filtrate was added to 5 mL of water containing solid $K_2Cr_2O_7$ (0.02 g).

The resulting solution was evaporated slowly at room temperature until formation of crystals. The obtained block-like orange crystals of the dichromate salt were washed with small amounts of 2-propanol and dried in air before collecting the synchrotron data. Elemental analysis calculated for $[Cr(NCS)_2(C_{10}H_{24}N_4)]_2(Cr_2O_7)\cdot H_2O$: C, 29.69; H, 5.19; N, 17.31%; found C, 29.84; H, 4.90; N, 17.28%.

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 = 0.98 Å and N—H = 0.99 Å, and with $U_{iso}(H)$ values of $1.2U_{eq}$ of the parent atoms. The hydrogen atom of the solvent water molecule was assigned based on a difference Fourier map, and the O—H distance and the H—O—H angle were restrained [0.84 (1) Å, 136 (2)°]. The bridging oxygen atom of the dichromate anion is positionally disordered around an inversion centre and consequently was refined with half-occupancy.
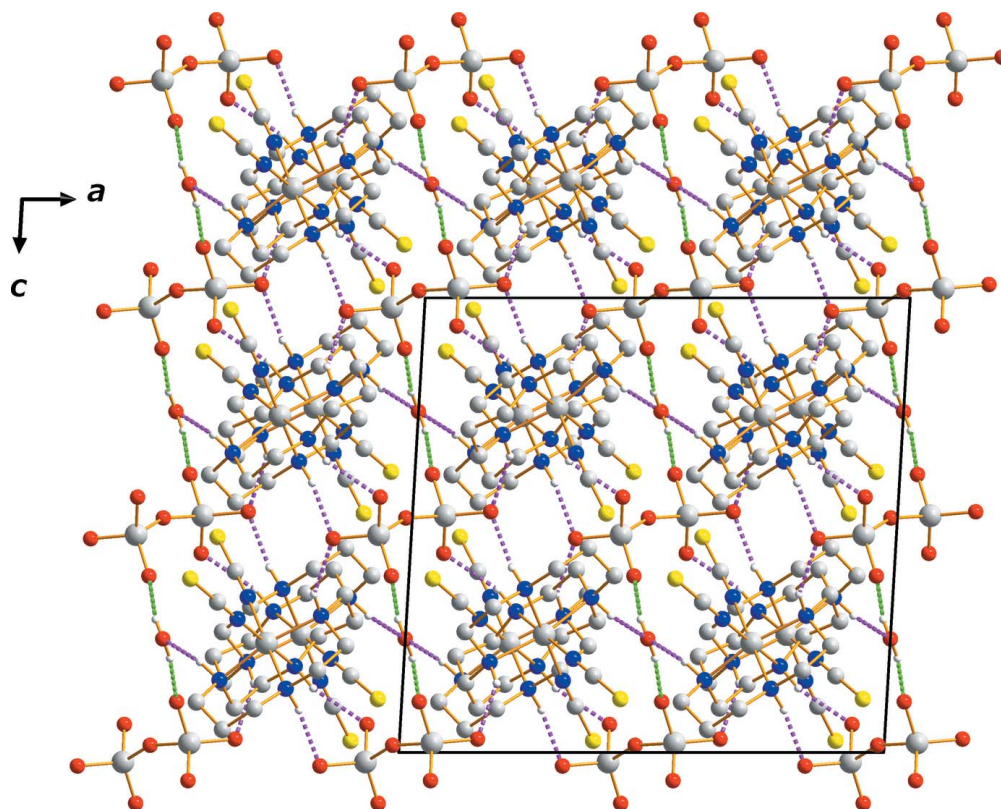
Figure 2
The crystal packing in compound (I), viewed perpendicular to the *ac* plane. Dashed lines represent O—H⋯O (green) and N—-H⋯O (pink) hydrogen-bonding interactions.

Table 2
Experimental details.

Crystal data	
Chemical formula	[Cr(NCS)$_2$(C$_{10}$H$_{24}$N$_4$)]$_2$[Cr$_2$O$_7$]$\cdot$H$_2$O
M_r	971.00
Crystal system, space group	Monoclinic, $C2/c$
Temperature (K)	243
a, b, c (Å)	16.044 (2), 16.221 (2), 15.041 (2)
β (°)	93.335 (3)
V (Å^3)	3907.8 (9)
Z	4
Radiation type	Synchrotron, $\lambda = 0.620$ Å
μ (mm^{-1})	0.92
Crystal size (mm)	0.04 × 0.03 × 0.02
Data collection	
Diffractometer	ADSC Q210 CCD area detector
Absorption correction	Empirical (using intensity measurements) (*HKL3000sm SCALEPACK*; Otwinowski & Minor, 1997)
T_{min}, T_{max}	0.799, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	11326, 5767, 4156
R_{int}	0.018
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.707
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.046, 0.148, 1.06
No. of reflections	5767
No. of parameters	244
No. of restraints	3
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å^{-3})	1.07, −0.73

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

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supporting information

Crystal structure of bis[*cis*-(1,4,8,11-tetraazacyclotetradecane-*κ⁴N*)bis(thio-cyanato-*κN*)chromium(III)] dichromate monohydrate from synchrotron X-ray diffraction data

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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: *publCIF* (Westrip,2010).

Bis[*cis*-(1,4,8,11-tetraazacyclotetradecane-*κ⁴N*)bis(thiocyanato-*κN*)chromium(III)] dichromate monohydrate

Crystal data

[Cr(NCS)$_2$(C$_{10}$H$_{24}$N$_4$)]$_2$[Cr$_2$O$_7$]·H$_2$O	$F(000) = 2008$
$M_r = 971.00$	$D_x = 1.650$ Mg m^{-3}
Monoclinic, $C2/c$	Synchrotron radiation, $\lambda = 0.620$ Å
$a = 16.044$ (2) Å	Cell parameters from 51334 reflections
$b = 16.221$ (2) Å	$\theta = 0.4–33.6°$
$c = 15.041$ (2) Å	$\mu = 0.92$ mm^{-1}
$\beta = 93.335$ (3)°	$T = 243$ K
$V = 3907.8$ (9) Å^3	Block, orange
$Z = 4$	$0.04 \times 0.03 \times 0.02$ mm

Data collection

ADSC Q210 CCD area detector diffractometer	11326 measured reflections
	5767 independent reflections
Radiation source: PLSII 2D bending magnet	4156 reflections with $I > 2\sigma(I)$
ω scan	$R_{int} = 0.018$
Absorption correction: empirical (using intensity measurements) (*HKL3000sm Scalepack*; Otwinowski & Minor, 1997)	$\theta_{max} = 26.0°$, $\theta_{min} = 1.6°$
	$h = -22 \rightarrow 22$
	$k = -22 \rightarrow 22$
	$l = -21 \rightarrow 21$
$T_{min} = 0.799$, $T_{max} = 1.000$	

Refinement

Refinement on F^2	3 restraints
Least-squares matrix: full	Hydrogen site location: mixed
$R[F^2 > 2\sigma(F^2)] = 0.046$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.148$	
$S = 1.06$	$w = 1/[\sigma^2(F_o^2) + (0.0961P)^2]$
5767 reflections	where $P = (F_o^2 + 2F_c^2)/3$
244 parameters	$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max}$ = 1.07 e Å⁻³

$\Delta\rho_{min}$ = −0.73 e Å⁻³

Extinction correction: SHELXL-2016/6
(Sheldrick 2015),
$F_c^* = kF_c[1+0.001xF_c^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0074 (7)

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Cr1A	0.21438 (2)	0.57578 (2)	0.25925 (2)	0.03091 (13)	
S1A	0.04950 (5)	0.36485 (4)	0.11771 (4)	0.04880 (19)	
S2A	0.10063 (7)	0.74359 (5)	0.02581 (6)	0.0745 (3)	
N1A	0.12183 (13)	0.61482 (13)	0.34086 (12)	0.0382 (4)	
H1A	0.079016	0.642089	0.300936	0.046*	
N2A	0.27533 (14)	0.68147 (13)	0.31024 (13)	0.0434 (5)	
H2A	0.316020	0.664275	0.358709	0.052*	
N3A	0.32354 (14)	0.54328 (15)	0.19902 (14)	0.0446 (5)	
H3A	0.306385	0.502794	0.152215	0.054*	
N4A	0.25948 (13)	0.49636 (12)	0.35970 (12)	0.0346 (4)	
H4A	0.288918	0.530211	0.406434	0.041*	
N5A	0.15160 (14)	0.48029 (13)	0.20628 (13)	0.0413 (5)	
N6A	0.17022 (15)	0.64396 (14)	0.15798 (14)	0.0438 (5)	
C1A	0.15574 (19)	0.68028 (17)	0.40197 (18)	0.0494 (6)	
H1A1	0.188173	0.655601	0.452403	0.059*	
H1A2	0.110059	0.712466	0.425097	0.059*	
C2A	0.2102 (2)	0.73442 (17)	0.34991 (19)	0.0529 (7)	
H2A1	0.237031	0.776231	0.388958	0.063*	
H2A2	0.176737	0.762617	0.302565	0.063*	
C3A	0.3207 (2)	0.73028 (18)	0.24429 (19)	0.0538 (7)	
H3A1	0.341165	0.781628	0.272119	0.065*	
H3A2	0.282160	0.744693	0.193779	0.065*	
C4A	0.39357 (19)	0.6825 (2)	0.2110 (2)	0.0590 (8)	
H4A1	0.428513	0.663492	0.262585	0.071*	
H4A2	0.427404	0.720062	0.176952	0.071*	
C5A	0.3701 (2)	0.6088 (2)	0.15325 (18)	0.0562 (7)	
H5A1	0.335888	0.627816	0.101176	0.067*	
H5A2	0.421255	0.584763	0.131733	0.067*	
C6A	0.37777 (18)	0.49700 (19)	0.26556 (18)	0.0504 (6)	
H6A1	0.409094	0.535645	0.304869	0.061*	
H6A2	0.417792	0.463030	0.235079	0.061*	
C7A	0.32269 (18)	0.44260 (17)	0.31975 (18)	0.0461 (6)	
H7A1	0.294855	0.400899	0.281353	0.055*	
H7A2	0.356505	0.414318	0.366772	0.055*	
C8A	0.19634 (16)	0.44486 (15)	0.40364 (16)	0.0410 (5)	

H8A1	0.225168	0.408506	0.447454	0.049*
H8A2	0.166940	0.410082	0.358709	0.049*
C9A	0.13381 (18)	0.49627 (17)	0.44937 (16)	0.0446 (6)
H9A1	0.164157	0.532275	0.492398	0.053*
H9A2	0.098970	0.459365	0.483051	0.053*
C10A	0.07670 (17)	0.54960 (17)	0.38902 (17)	0.0441 (6)
H10A	0.046651	0.514150	0.345211	0.053*
H10B	0.035225	0.575948	0.425015	0.053*
C11A	0.10820 (16)	0.43251 (14)	0.16879 (14)	0.0354 (5)
C12A	0.14135 (15)	0.68562 (15)	0.10176 (15)	0.0376 (5)
Cr2B	0.43256 (3)	0.58043 (3)	0.52084 (3)	0.04068 (14)
O1B1	0.5133 (5)	0.5087 (6)	0.5160 (6)	0.0703 (17)	0.25
O1B2	0.5133 (5)	0.5087 (6)	0.5160 (6)	0.0703 (17)	0.25
O2B	0.46377 (19)	0.62450 (18)	0.61164 (14)	0.0817 (8)
O3B	0.43108 (16)	0.64288 (14)	0.43819 (14)	0.0686 (6)
O4B	0.33924 (14)	0.54580 (18)	0.52960 (14)	0.0737 (7)
O1W	0.500000	0.75939 (19)	0.750000	0.0587 (8)
H1OW	0.483 (2)	0.7293 (10)	0.7074 (11)	0.088*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cr1A	0.0380 (2)	0.0291 (2)	0.02458 (18)	−0.00533 (14)	−0.00667 (13)	0.00188 (12)
S1A	0.0635 (4)	0.0365 (3)	0.0439 (3)	−0.0122 (3)	−0.0183 (3)	−0.0014 (3)
S2A	0.1159 (8)	0.0473 (4)	0.0554 (5)	0.0173 (5)	−0.0380 (5)	0.0057 (4)
N1A	0.0448 (11)	0.0365 (11)	0.0325 (9)	−0.0010 (9)	−0.0040 (8)	0.0010 (8)
N2A	0.0535 (12)	0.0377 (11)	0.0372 (10)	−0.0140 (9)	−0.0116 (9)	0.0014 (9)
N3A	0.0447 (12)	0.0561 (13)	0.0328 (10)	−0.0070 (10)	0.0005 (8)	0.0015 (9)
N4A	0.0430 (10)	0.0318 (9)	0.0282 (8)	−0.0009 (8)	−0.0035 (7)	0.0029 (7)
N5A	0.0519 (12)	0.0368 (11)	0.0337 (9)	−0.0103 (9)	−0.0092 (8)	−0.0011 (8)
N6A	0.0552 (13)	0.0402 (12)	0.0343 (10)	−0.0057 (9)	−0.0104 (9)	0.0065 (8)
C1A	0.0675 (18)	0.0390 (13)	0.0409 (13)	−0.0006 (12)	−0.0021 (12)	−0.0068 (11)
C2A	0.075 (2)	0.0344 (13)	0.0481 (14)	−0.0057 (13)	−0.0049 (13)	−0.0066 (11)
C3A	0.0632 (18)	0.0456 (15)	0.0511 (15)	−0.0241 (13)	−0.0085 (13)	0.0059 (12)
C4A	0.0543 (17)	0.071 (2)	0.0515 (15)	−0.0223 (15)	−0.0041 (13)	0.0133 (14)
C5A	0.0542 (16)	0.072 (2)	0.0432 (14)	−0.0119 (15)	0.0063 (12)	0.0119 (14)
C6A	0.0456 (14)	0.0634 (17)	0.0422 (13)	0.0067 (13)	0.0018 (11)	0.0010 (13)
C7A	0.0494 (14)	0.0466 (14)	0.0418 (13)	0.0099 (12)	−0.0011 (11)	0.0019 (11)
C8A	0.0526 (14)	0.0347 (12)	0.0351 (11)	−0.0056 (10)	−0.0029 (10)	0.0099 (9)
C9A	0.0524 (15)	0.0485 (14)	0.0329 (11)	−0.0050 (12)	0.0027 (10)	0.0081 (10)
C10A	0.0446 (13)	0.0479 (14)	0.0397 (12)	−0.0025 (11)	0.0004 (10)	0.0054 (11)
C11A	0.0472 (13)	0.0308 (11)	0.0273 (10)	−0.0001 (9)	−0.0068 (9)	0.0033 (8)
C12A	0.0463 (13)	0.0354 (12)	0.0300 (10)	−0.0024 (10)	−0.0068 (9)	−0.0036 (9)
Cr2B	0.0457 (2)	0.0434 (3)	0.0319 (2)	0.00024 (17)	−0.00628 (16)	0.00294 (15)
O1B1	0.060 (5)	0.063 (4)	0.087 (6)	0.014 (3)	0.000 (3)	−0.009 (4)
O1B2	0.060 (5)	0.063 (4)	0.087 (6)	0.014 (3)	0.000 (3)	−0.009 (4)
O2B	0.107 (2)	0.0956 (19)	0.0407 (11)	−0.0337 (16)	−0.0147 (12)	−0.0019 (12)
O3B	0.0960 (18)	0.0621 (14)	0.0461 (11)	0.0021 (13)	−0.0096 (11)	0.0156 (10)

O4B	0.0511 (12)	0.122 (2)	0.0466 (11)	−0.0224 (13)	−0.0071 (9)	−0.0004 (13)
O1W	0.076 (2)	0.0548 (17)	0.0435 (15)	0.000	−0.0078 (14)	0.000

Geometric parameters (Å, º)

Cr1A—N6A	1.980 (2)	C3A—H3A2	0.9800
Cr1A—N5A	1.989 (2)	C4A—C5A	1.512 (4)
Cr1A—N1A	2.080 (2)	C4A—H4A1	0.9800
Cr1A—N4A	2.0829 (19)	C4A—H4A2	0.9800
Cr1A—N3A	2.086 (2)	C5A—H5A1	0.9800
Cr1A—N2A	2.097 (2)	C5A—H5A2	0.9800
S1A—C11A	1.612 (2)	C6A—C7A	1.519 (4)
S2A—C12A	1.590 (2)	C6A—H6A1	0.9800
N1A—C1A	1.487 (3)	C6A—H6A2	0.9800
N1A—C10A	1.493 (3)	C7A—H7A1	0.9800
N1A—H1A	0.9900	C7A—H7A2	0.9800
N2A—C3A	1.492 (3)	C8A—C9A	1.502 (4)
N2A—C2A	1.502 (4)	C8A—H8A1	0.9800
N2A—H2A	0.9900	C8A—H8A2	0.9800
N3A—C6A	1.490 (3)	C9A—C10A	1.522 (4)
N3A—C5A	1.491 (4)	C9A—H9A1	0.9800
N3A—H3A	0.9900	C9A—H9A2	0.9800
N4A—C7A	1.490 (3)	C10A—H10A	0.9800
N4A—C8A	1.496 (3)	C10A—H10B	0.9800
N4A—H4A	0.9900	Cr2B—O2B	1.596 (2)
N5A—C11A	1.165 (3)	Cr2B—O3B	1.603 (2)
N6A—C12A	1.158 (3)	Cr2B—O4B	1.612 (2)
C1A—C2A	1.493 (4)	Cr2B—O1B1	1.746 (9)
C1A—H1A1	0.9800	Cr2B—O1B2	1.746 (9)
C1A—H1A2	0.9800	Cr2B—O1B1[i]	1.791 (9)
C2A—H2A1	0.9800	O1B1—O1B1[i]	0.686 (9)
C2A—H2A2	0.9800	O1W—H1OW	0.839 (7)
C3A—C4A	1.511 (5)	O1W—H1OW[ii]	0.839 (7)
C3A—H3A1	0.9800		
N6A—Cr1A—N5A	88.66 (9)	C5A—C4A—H4A1	108.5
N6A—Cr1A—N1A	92.76 (9)	C3A—C4A—H4A2	108.5
N5A—Cr1A—N1A	96.39 (9)	C5A—C4A—H4A2	108.5
N6A—Cr1A—N4A	175.72 (8)	H4A1—C4A—H4A2	107.5
N5A—Cr1A—N4A	87.44 (8)	N3A—C5A—C4A	114.4 (2)
N1A—Cr1A—N4A	89.43 (8)	N3A—C5A—H5A1	108.7
N6A—Cr1A—N3A	94.53 (9)	C4A—C5A—H5A1	108.7
N5A—Cr1A—N3A	92.73 (9)	N3A—C5A—H5A2	108.7
N1A—Cr1A—N3A	168.45 (8)	C4A—C5A—H5A2	108.7
N4A—Cr1A—N3A	83.89 (8)	H5A1—C5A—H5A2	107.6
N6A—Cr1A—N2A	87.88 (8)	N3A—C6A—C7A	108.5 (2)
N5A—Cr1A—N2A	176.30 (9)	N3A—C6A—H6A1	110.0
N1A—Cr1A—N2A	82.47 (8)	C7A—C6A—H6A1	110.0

N4A—Cr1A—N2A	96.05 (8)	N3A—C6A—H6A2	110.0
N3A—Cr1A—N2A	88.86 (9)	C7A—C6A—H6A2	110.0
C1A—N1A—C10A	112.12 (19)	H6A1—C6A—H6A2	108.4
C1A—N1A—Cr1A	109.52 (16)	N4A—C7A—C6A	107.9 (2)
C10A—N1A—Cr1A	116.98 (17)	N4A—C7A—H7A1	110.1
C1A—N1A—H1A	105.8	C6A—C7A—H7A1	110.1
C10A—N1A—H1A	105.8	N4A—C7A—H7A2	110.1
Cr1A—N1A—H1A	105.8	C6A—C7A—H7A2	110.1
C3A—N2A—C2A	109.8 (2)	H7A1—C7A—H7A2	108.4
C3A—N2A—Cr1A	115.22 (16)	N4A—C8A—C9A	112.3 (2)
C2A—N2A—Cr1A	107.01 (16)	N4A—C8A—H8A1	109.1
C3A—N2A—H2A	108.2	C9A—C8A—H8A1	109.1
C2A—N2A—H2A	108.2	N4A—C8A—H8A2	109.1
Cr1A—N2A—H2A	108.2	C9A—C8A—H8A2	109.1
C6A—N3A—C5A	112.4 (2)	H8A1—C8A—H8A2	107.9
C6A—N3A—Cr1A	107.93 (15)	C8A—C9A—C10A	116.0 (2)
C5A—N3A—Cr1A	118.5 (2)	C8A—C9A—H9A1	108.3
C6A—N3A—H3A	105.7	C10A—C9A—H9A1	108.3
C5A—N3A—H3A	105.7	C8A—C9A—H9A2	108.3
Cr1A—N3A—H3A	105.7	C10A—C9A—H9A2	108.3
C7A—N4A—C8A	110.21 (19)	H9A1—C9A—H9A2	107.4
C7A—N4A—Cr1A	106.58 (14)	N1A—C10A—C9A	113.6 (2)
C8A—N4A—Cr1A	116.75 (15)	N1A—C10A—H10A	108.8
C7A—N4A—H4A	107.7	C9A—C10A—H10A	108.8
C8A—N4A—H4A	107.7	N1A—C10A—H10B	108.8
Cr1A—N4A—H4A	107.7	C9A—C10A—H10B	108.8
C11A—N5A—Cr1A	170.5 (2)	H10A—C10A—H10B	107.7
C12A—N6A—Cr1A	176.3 (2)	N5A—C11A—S1A	178.8 (2)
N1A—C1A—C2A	107.5 (2)	N6A—C12A—S2A	179.0 (3)
N1A—C1A—H1A1	110.2	O2B—Cr2B—O3B	111.73 (13)
C2A—C1A—H1A1	110.2	O2B—Cr2B—O4B	109.44 (13)
N1A—C1A—H1A2	110.2	O3B—Cr2B—O4B	108.17 (13)
C2A—C1A—H1A2	110.2	O2B—Cr2B—O1B1	97.9 (2)
H1A1—C1A—H1A2	108.5	O3B—Cr2B—O1B1	111.5 (4)
C1A—C2A—N2A	108.3 (2)	O4B—Cr2B—O1B1	117.8 (3)
C1A—C2A—H2A1	110.0	O2B—Cr2B—O1B2	97.9 (2)
N2A—C2A—H2A1	110.0	O3B—Cr2B—O1B2	111.5 (4)
C1A—C2A—H2A2	110.0	O4B—Cr2B—O1B2	117.8 (3)
N2A—C2A—H2A2	110.0	O2B—Cr2B—O1B1[i]	119.5 (2)
H2A1—C2A—H2A2	108.4	O3B—Cr2B—O1B1[i]	104.8 (4)
N2A—C3A—C4A	111.4 (2)	O4B—Cr2B—O1B1[i]	102.3 (3)
N2A—C3A—H3A1	109.3	O1B1—Cr2B—O1B1[i]	22.3 (3)
C4A—C3A—H3A1	109.3	O1B2—Cr2B—O1B1[i]	22.3 (3)
N2A—C3A—H3A2	109.3	O1B1[i]—O1B1—Cr2B	82.5 (15)
C4A—C3A—H3A2	109.3	O1B1[i]—O1B1—Cr2B[i]	75.2 (15)
H3A1—C3A—H3A2	108.0	Cr2B—O1B1—Cr2B[i]	157.7 (3)
C3A—C4A—C5A	115.1 (2)	Cr2B—O1B2—Cr2B[i]	157.7 (3)
C3A—C4A—H4A1	108.5	H1OW—O1W—H1OW[ii]	109 (2)

C10A—N1A—C1A—C2A	171.4 (2)	C7A—N4A—C8A—C9A	176.9 (2)
Cr1A—N1A—C1A—C2A	39.8 (3)	Cr1A—N4A—C8A—C9A	−61.4 (2)
N1A—C1A—C2A—N2A	−55.7 (3)	N4A—C8A—C9A—C10A	65.3 (3)
C3A—N2A—C2A—C1A	169.5 (2)	C1A—N1A—C10A—C9A	−69.4 (3)
Cr1A—N2A—C2A—C1A	43.8 (2)	Cr1A—N1A—C10A—C9A	58.4 (3)
C2A—N2A—C3A—C4A	173.1 (2)	C8A—C9A—C10A—N1A	−64.1 (3)
Cr1A—N2A—C3A—C4A	−66.0 (3)	O2B—Cr2B—O1B1—O1B1^{i}	166.3 (18)
N2A—C3A—C4A—C5A	68.6 (3)	O3B—Cr2B—O1B1—O1B1^{i}	−76.5 (19)
C6A—N3A—C5A—C4A	−72.0 (3)	O4B—Cr2B—O1B1—O1B1^{i}	49 (2)
Cr1A—N3A—C5A—C4A	55.0 (3)	O2B—Cr2B—O1B1—Cr2B^{i}	166.3 (18)
C3A—C4A—C5A—N3A	−62.5 (4)	O3B—Cr2B—O1B1—Cr2B^{i}	−76.5 (19)
C5A—N3A—C6A—C7A	170.2 (2)	O4B—Cr2B—O1B1—Cr2B^{i}	49 (2)
Cr1A—N3A—C6A—C7A	37.7 (3)	O1B1^{i}—Cr2B—O1B1—Cr2B^{i}	0.004 (6)
C8A—N4A—C7A—C6A	172.4 (2)	O2B—Cr2B—O1B2—Cr2B^{i}	166.3 (18)
Cr1A—N4A—C7A—C6A	44.8 (2)	O3B—Cr2B—O1B2—Cr2B^{i}	−76.5 (19)
N3A—C6A—C7A—N4A	−55.9 (3)	O4B—Cr2B—O1B2—Cr2B^{i}	49 (2)

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

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
N1A—H1A···O1W^{iii}	0.99	2.15	3.089 (3)	157
N2A—H2A···O3B	0.99	2.17	3.127 (3)	163
N3A—H3A···O4B^{iv}	0.99	2.10	2.953 (3)	143
N4A—H4A···O4B	0.99	1.99	2.904 (3)	152
O1W—H1OW···O2B	0.84 (1)	2.24 (1)	3.052 (3)	164 (2)

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