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Crystal structure of ammonium/potassium *trans*-bis(*N*-methyliminodiacetato-κ^3O,N,O')chromate(III) from synchrotron data

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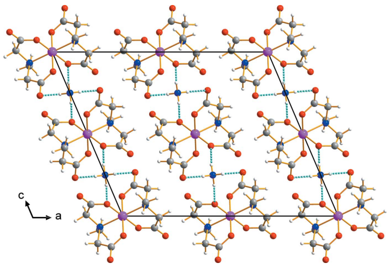
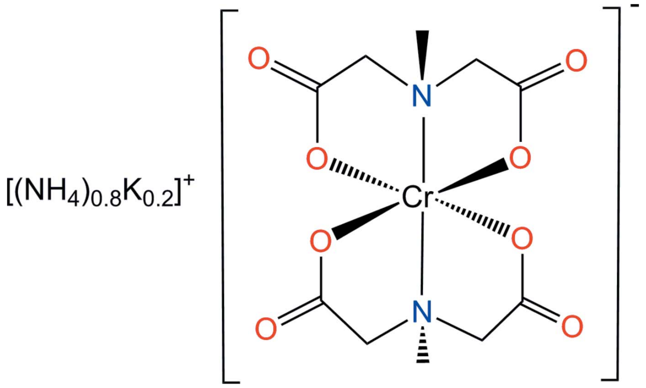
The structure of the title compound, $[(NH_4)_{0.8}K_{0.2}][Cr(C_5H_7NO_4)_2]$ ($C_5H_7NO_4$ is methyliminodiacetate; mida), has been determined from synchrotron data. The Cr^{III} atom is located on a centre of symmetry and is coordinated by two N atoms and four O atoms of two facially arranged tridentate mida ligands, displaying a slightly distorted octahedral coordination environment. The Cr—N and mean Cr—O bond lengths are 2.0792 (14) and 1.958 (14) Å, respectively. The cation site is located on a twofold rotation axis and shows occupational disorder, being occupied by ammonium and potassium cations in a 0.8:0.2 ratio. In the crystal, intermolecular hydrogen bonds involving the N—H groups of the ammonium cation as donor and the two non-coordinating O atoms of the carboxylate group as acceptor groups consolidate the three-dimensional packing.

1. Chemical context

Methyliminodiacetate (abbreviated here as mida; $C_5H_7NO_4$) can coordinate to a central metal ion as a tridentate ligand through one N atom and two O atoms. The mida ligand differs from iminodiacetate (ida) in the substitution of the imino hydrogen with a methyl group. This change has significant consequences with respect to the configuration of the bis-chromate(III) complexes with these ligands. Two facial configurations in *cis* or *trans* mode relative to the two N atoms have been observed: for example $K[Cr(ida)_2]\cdot 3H_2O$ (Mootz & Wunderlich, 1980) and $Na[Cr(ida)_2]\cdot 1.5H_2O$ (Li *et al.*, 2003) are *cis-fac* structures whereas $Na[Cr(mida)_2]$ is a *trans-fac* structure (Suh *et al.*, 1997). However, the *trans* meridional isomer of octahedrally coordinated chromium(III) with ida or mida ligands has not yet been identified. In order to confirm the bonding mode of the methyliminodiacetato ligand and its structural arrangement, we report herein on the crystal structure of the title salt, $[(NH_4)_{0.8}K_{0.2}][Cr(C_5H_7NO_4)_2]$, (I).





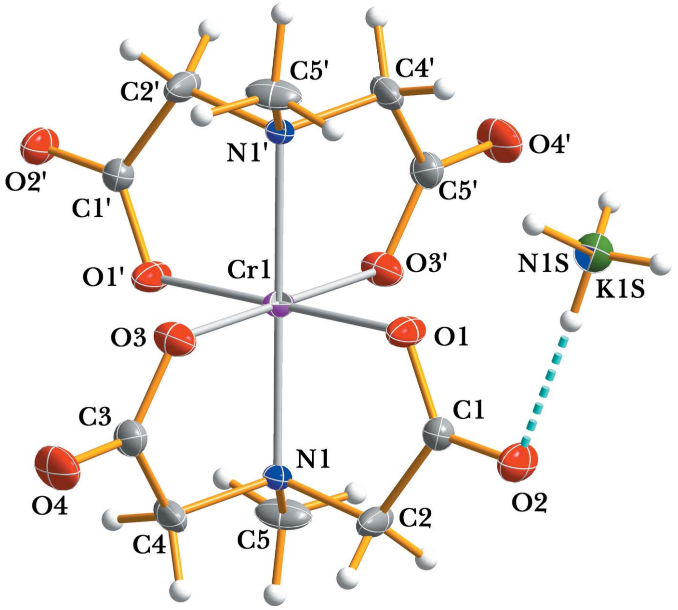


Figure 1
The structures of the molecular entities of (I), showing the atom-numbering scheme. Non-H atoms are shown as displacement ellipsoids at the 50% probability level. The primed atoms are related by symmetry code $(-x + 1, -y + 1, -z + 1)$. Dashed lines represent hydrogen-bonding interactions.

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1S—H1NS⋯O3^{i}	0.92 (1)	2.07 (1)	2.9658 (16)	166 (3)
N1S—H2NS⋯O2	0.90 (1)	1.95 (1)	2.8485 (17)	175 (3)

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

2. Structural commentary

Counter-ionic species play important roles in crystal packings and hydrogen-bonding patterns. The structure reported here is another example of a $[Cr(mida)_2]^-$ salt but with a different cation (Suh *et al.*, 1996, 1997). The structural analysis shows that the two tridentate mida dianions octahedrally coordinate to the Cr^{III} metal atom through one N atom and two carboxylate O atoms in a facial configuration. The coordinating N atoms are mutually *trans* due to point group $\overline{1}$ for the entire anionic complex. The asymmetric unit of (I) comprises one half of the Cr^{III} complex anion and one occupationally disordered ammonium/potassium cation (situated on a twofold rotation axis), respectively. An ellipsoid plot of title compound together with the atomic numbering is illustrated in Fig. 1.

The facial configuration of the complex anion in (I) can be compared with that of $NH_4[Cr(pydc)_2]$ (pydc = pyridine-2,6-dicarboxylate; Moon & Choi, 2015) where it displays a *trans* meridional configuration. The Cr—N and mean Cr—O bond lengths involving the mida ligands are 2.0792 (14) and 1.958 (14) Å, respectively, in good agreement with the values observed for $Na[Cr(mida)_2]$ (Suh *et al.*, 1997). Bond angles about the central chromium atom are 90.23 (6) for O1—Cr1—O3, 84.66 (6) for O1—Cr1—N1 and 82.62 (5)° for N1—Cr1—O3 indicating a distorted octahedral coordination environment. The C–O bond lengths within the carboxylate group of the mida ligand range from 1.219 (2) to 1.296 (2) Å and can be compared with values of 1.225 (15) and 1.294 (15) Å for $NH_4[Cr(pydc)_2]$ (Moon & Choi, 2015). The slightly longer C—

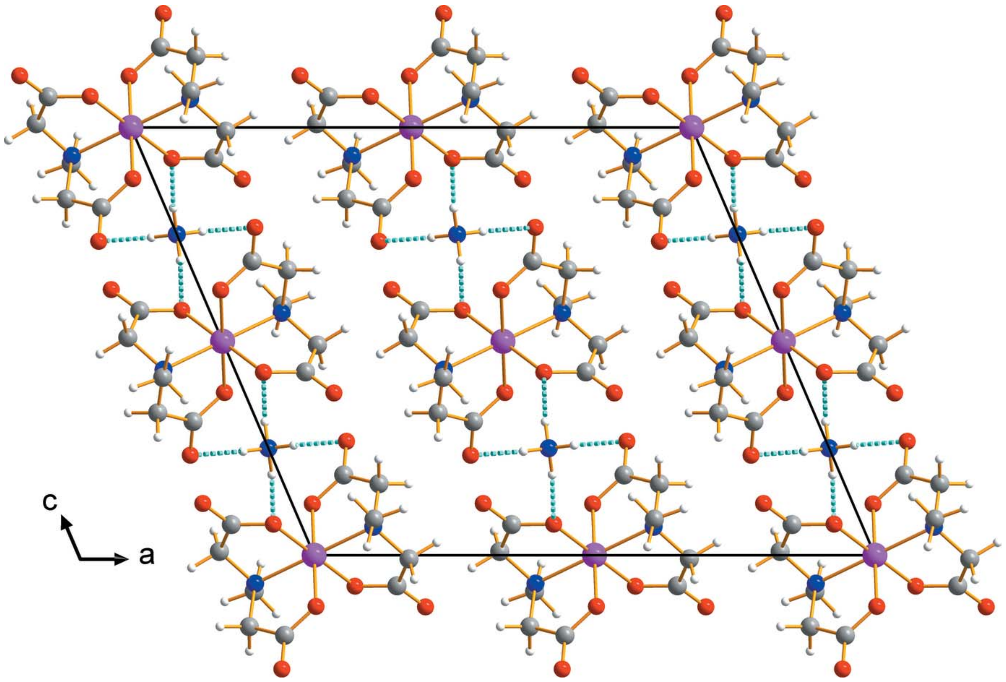


Figure 2
A packing diagram of (I), viewed perpendicular to the *ac* plane. Dashed lines represent hydrogen-bonding interactions N—H⋯O (cyan).

O bond length (C1—O2 and C3—O4) and smaller O—C—O bond angles of the carboxylate groups in the mida ligand of (I) compared to the ligand in Na[Cr(mida)$_2$] (Suh *et al.*, 1997) may be attributed to the involvement of the two non-coordinating O atoms in hydrogen bonds with the N—H groups of the ammonium cation. The N—C and C—C distances in the mida moieties are close to those found in the free H$_2$mida molecule (Shkol'nikova *et al.*, 1986) and are equal to 1.479 (2)–1.494 (2) and 1.508 (3)–1.512 (2) Å, respectively.

3. Supramolecular features

The pattern of hydrogen bonding around the cation is different from the crystal packing network in the related sodium salt (Suh *et al.*, 1996, 1997). The cation is linked to four non-coordinating O atoms of carboxylate groups from four neighboring mida ligands through classical N—H···O hydrogen bonds (Table 1). An array of these interactions generate a three-dimensional network of molecules whereby individual molecules are stacked along the *b*-axis direction (Fig. 2).

4. Database survey

A search of the Cambridge Structural Database (Version 5.37, Feb. 2016 with two updates; Groom *et al.*, 2016) gave just two hits for a complex anion [Cr(C$_5$H$_7$NO$_4$)$_2$]$^-$ unit. The crystal structures of Na[Cr(mida)$_2$] with three different space groups have been reported and compared previously (Suh *et al.*, 1996, 1997).

5. Synthesis and crystallization

All chemicals were reagent-grade materials and were used without further purification. The starting material, K[Cr(mida)$_2$] was prepared by a method similar to that outlined previously (Wernicke *et al.*, 1977; Uehara *et al.*, 1970). The potassium salt (0.25 g) was dissolved in 15 mL of water at 343 K and added to 5 mL of water containing 0.50 g of NH$_4$Cl. The resulting solution was filtered to remove any impurities and allowed to stand at room temperature for several days to give pale pink plate-like crystals of the mixed-occupancy ammonium/potassium salt, (I), suitable for X-ray diffraction analysis.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All H atoms of the complex were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances of 0.97–0.98 Å and with $U_{\rm iso}$(H) values of 1.5 (methyl) and 1.2 times $U_{\rm eq}$ (all others) of the parent atoms. The H atoms of the cation were located from difference Fourier maps and refined with DFIX and DANG restraints and fixed N—H distances of 0.855 (9) and 0.869 (9) Å, with $U_{\rm iso}$(H) values of 1.2$U_{\rm eq}$(N). The occupancy of mixed-occupied (NH$_4$/K) first was refined and then fixed at a ratio of 0.8:0.2. The corresponding (NH$_4$/K) sites was refined using EXYZ/EADP commands for the two atom types.

Table 2
Experimental details.

Crystal data	
Chemical formula	[(NH$_4$)$_{0.8}$K$_{0.2}$][Cr(C$_5$H$_7$NO$_4$)$_2$]
M_r	364.48
Crystal system, space group	Monoclinic, *C*2/*c*
Temperature (K)	243
a, *b*, *c* (Å)	16.786 (3), 6.5240 (13), 13.925 (3)
β (°)	113.19 (3)
V (Å^3)	1401.8 (6)
Z	4
Radiation type	Synchrotron, λ = 0.610 Å
μ (mm^{-1})	0.61
Crystal size (mm)	0.02 × 0.02 × 0.01
Data collection	
Diffractometer	ADSC Q210 CCD area-detector
Absorption correction	Empirical (using intensity measurements) (*HKL3000sm SCALEPACK*; Otwinowski & Minor, 1997)
$T_{\rm min}$, $T_{\rm max}$	0.989, 0.995
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	6984, 1833, 1519
$R_{\rm int}$	0.028
(sin $\theta/\lambda)_{\rm max}$ (Å^{-1})	0.693
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.035, 0.100, 1.05
No. of reflections	1833
No. of parameters	110
No. of restraints	3
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\rm max}$, $\Delta\rho_{\rm min}$ (e Å^{-3})	0.40, −0.69

Computer programs: *PAL BL2D-SMDC* (Shin *et al.*, 2016), *HKL3000sm* (Otwinowski & Minor, 1997), *SHELXT2014* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *DIAMOND* (Putz & Brandenburg, 2014) and *publCIF* (Westrip, 2010).

Acknowledgements

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References

Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* B**72**, 171–179.
Li, H., Xu, D.-J. & Yin, K.-L. (2003). *Acta Cryst.* E**59**, m671–m673.
Moon, D. & Choi, J.-H. (2015). *Acta Cryst.* E**71**, 210–212.
Mootz, D. & Wunderlich, H. (1980). *Acta Cryst.* B**36**, 445–447.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Putz, H. & Brandenburg, K. (2014). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.
Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.
Shin, J. W., Eom, K. & Moon, D. (2016). *J. Synchrotron Rad.* **23**, 369–373.

Shkol'nikova, L. M., Donchenko, N. V., Poznyak, A. L., Dyatlova, N. M. & Zavodnik, E. V. (1986). *Zh. Strukt. Khim.* **27**, 89–94.

Suh, I.-H., Lee, J.-H., Song, J.-H., Oh, M.-R., Suh, J.-S., Park, S.-J. & Lee, K.-W. (1996). *J. Korean Phys. Soc.* **29**, 739–744.

Suh, J.-S., Park, S.-J., Lee, K.-W., Suh, I.-H., Lee, J.-H., Song, J.-H. & Oh, M.-R. (1997). *Acta Cryst.* C**53**, 432–434.

Uehara, A., Kyuno, E. & Tsuchiya, R. (1970). *Bull. Chem. Soc. Jpn*, **43**, 1394–1397.

Wernicke, R., Schmidtke, H.-H. & Hoggard, P. E. (1977). *Inorg. Chim. Acta*, **24**, 145–148.

Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

supporting information

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Crystal structure of ammonium/potassium *trans*-bis(*N*-methyliminodiacetato-κ^3O,N,O')chromate(III) from synchrotron 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, 2015*a*); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015*b*); molecular graphics: *DIAMOND* (Putz & Brandenburg, 2014); software used to prepare material for publication: *publCIF* (Westrip, 2010).

Ammonium/potassium *trans*-bis(*N*-methyliminodiacetato-κ^3O,N,O')chromate(III)

Crystal data

$[(NH_4)_{0.8}K_{0.2}][Cr(C_5H_7NO_4)_2]$
$M_r = 364.48$
Monoclinic, $C2/c$
$a = 16.786$ (3) Å
$b = 6.5240$ (13) Å
$c = 13.925$ (3) Å
$\beta = 113.19$ (3)°
$V = 1401.8$ (6) Å^3
$Z = 4$
$F(000) = 754$
$D_x = 1.727$ Mg m^{-3}
Synchrotron radiation, $\lambda = 0.610$ Å
Cell parameters from 23758 reflections
$\theta = 0.4$–33.7°
$\mu = 0.61$ mm^{-1}
$T = 243$ K
Plate, pale pink
0.02 × 0.02 × 0.01 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)
$T_{min} = 0.989$, $T_{max} = 0.995$
6984 measured reflections
1833 independent reflections
1519 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.7°$
$h = -23 \rightarrow 23$
$k = -9 \rightarrow 9$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.100$
$S = 1.05$
1833 reflections
110 parameters
3 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0702P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.40$ e Å^{-3}
$\Delta\rho_{min} = -0.69$ e Å^{-3}

Extinction correction: SHELXL2014
(Sheldrick, 2015*b*),
$Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0118 (19)

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)
Cr1	0.5000	0.5000	0.5000	0.01108 (14)	
O1	0.53582 (8)	0.3160 (2)	0.61977 (10)	0.0228 (3)	
O2	0.64463 (9)	0.2272 (2)	0.76630 (10)	0.0267 (3)	
O3	0.54917 (8)	0.3115 (2)	0.42746 (10)	0.0228 (3)	
O4	0.65906 (10)	0.2681 (2)	0.37913 (13)	0.0361 (4)	
N1	0.62700 (8)	0.6057 (2)	0.56638 (10)	0.0129 (3)	
C1	0.61393 (11)	0.3333 (3)	0.68756 (13)	0.0175 (3)	
C2	0.67027 (13)	0.4929 (3)	0.66707 (15)	0.0326 (5)	
H2A	0.6886	0.5922	0.7245	0.039*	
H2B	0.7225	0.4260	0.6668	0.039*	
C3	0.62435 (11)	0.3600 (3)	0.42841 (13)	0.0196 (4)	
C4	0.66622 (12)	0.5484 (3)	0.49128 (15)	0.0232 (4)	
H4A	0.7283	0.5226	0.5297	0.028*	
H4B	0.6601	0.6633	0.4435	0.028*	
C5	0.63441 (13)	0.8296 (3)	0.58441 (19)	0.0333 (5)	
H5A	0.6948	0.8699	0.6086	0.050*	
H5B	0.6121	0.8653	0.6368	0.050*	
H5C	0.6012	0.9003	0.5197	0.050*	
K1S	0.5000	−0.0201 (2)	0.7500	0.0275 (3)	0.2
N1S	0.5000	−0.0201 (2)	0.7500	0.0275 (3)	0.8
H1NS	0.5139 (17)	−0.091 (4)	0.8115 (14)	0.033*	0.8
H2NS	0.5476 (13)	0.052 (4)	0.757 (2)	0.033*	0.8

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cr1	0.00847 (19)	0.01210 (19)	0.0120 (2)	−0.00089 (12)	0.00332 (13)	−0.00138 (12)
O1	0.0173 (6)	0.0248 (6)	0.0209 (6)	−0.0064 (5)	0.0018 (5)	0.0078 (5)
O2	0.0231 (7)	0.0300 (7)	0.0217 (7)	0.0003 (5)	0.0030 (5)	0.0107 (5)
O3	0.0201 (6)	0.0223 (6)	0.0290 (7)	−0.0052 (5)	0.0130 (5)	−0.0119 (5)
O4	0.0328 (8)	0.0430 (9)	0.0416 (9)	−0.0005 (7)	0.0243 (7)	−0.0173 (7)
N1	0.0110 (6)	0.0145 (6)	0.0134 (6)	−0.0023 (5)	0.0050 (5)	−0.0013 (5)
C1	0.0174 (8)	0.0198 (8)	0.0152 (8)	−0.0010 (6)	0.0063 (6)	0.0000 (6)
C2	0.0205 (9)	0.0530 (14)	0.0160 (9)	−0.0150 (8)	−0.0018 (7)	0.0125 (8)
C3	0.0201 (8)	0.0219 (8)	0.0185 (9)	0.0005 (6)	0.0094 (6)	−0.0027 (6)

C4	0.0203 (8)	0.0327 (9)	0.0226 (10)	−0.0081 (7)	0.0148 (7)	−0.0083 (7)
C5	0.0221 (9)	0.0186 (9)	0.0574 (14)	−0.0070 (7)	0.0137 (9)	−0.0129 (8)
K1S	0.0277 (7)	0.0278 (8)	0.0270 (8)	0.000	0.0107 (6)	0.000
N1S	0.0277 (7)	0.0278 (8)	0.0270 (8)	0.000	0.0107 (6)	0.000

Geometric parameters (Å, º)

Cr1—O1	1.9479 (13)	C2—H2B	0.9800
Cr1—O1^{i}	1.9479 (13)	C3—C4	1.512 (2)
Cr1—O3	1.9673 (12)	C3—K1S^{ii}	3.371 (2)
Cr1—O3^{i}	1.9673 (12)	C4—H4A	0.9800
Cr1—N1	2.0792 (14)	C4—H4B	0.9800
Cr1—N1^{i}	2.0792 (14)	C5—H5A	0.9700
O1—C1	1.284 (2)	C5—H5B	0.9700
O1—K1S	3.0524 (17)	C5—H5C	0.9700
O2—C1	1.226 (2)	K1S—O2^{iii}	2.8484 (16)
O2—K1S	2.8485 (17)	K1S—O3^{iv}	2.9658 (16)
O3—C3	1.296 (2)	K1S—O3^{ii}	2.9658 (16)
O3—K1S^{ii}	2.9658 (16)	K1S—O4^{iv}	3.033 (2)
O4—C3	1.219 (2)	K1S—O4^{ii}	3.033 (2)
O4—K1S^{ii}	3.033 (2)	K1S—O1^{iii}	3.0524 (17)
N1—C5	1.479 (2)	K1S—C1^{iii}	3.322 (2)
N1—C4	1.486 (2)	K1S—C3^{ii}	3.371 (2)
N1—C2	1.494 (2)	K1S—C3^{iv}	3.371 (2)
C1—C2	1.508 (3)	N1S—H1NS	0.920 (10)
C1—K1S	3.322 (2)	N1S—H2NS	0.899 (10)
C2—H2A	0.9800		
O1—Cr1—O1^{i}	180.00 (5)	O2—K1S—O3^{ii}	112.23 (4)
O1—Cr1—O3	90.23 (6)	O3^{iv}—K1S—O3^{ii}	100.26 (7)
O1^{i}—Cr1—O3	89.77 (6)	O2^{iii}—K1S—O4^{iv}	150.81 (4)
O1—Cr1—O3^{i}	89.77 (6)	O2—K1S—O4^{iv}	74.40 (4)
O1^{i}—Cr1—O3^{i}	90.23 (6)	O3^{iv}—K1S—O4^{iv}	43.35 (4)
O3—Cr1—O3^{i}	180.0	O3^{ii}—K1S—O4^{iv}	92.48 (6)
O1—Cr1—N1	84.66 (6)	O2^{iii}—K1S—O4^{ii}	74.40 (4)
O1^{i}—Cr1—N1	95.34 (6)	O2—K1S—O4^{ii}	150.82 (4)
O3—Cr1—N1	82.62 (5)	O3^{iv}—K1S—O4^{ii}	92.48 (6)
O3^{i}—Cr1—N1	97.38 (5)	O3^{ii}—K1S—O4^{ii}	43.36 (4)
O1—Cr1—N1^{i}	95.34 (6)	O4^{iv}—K1S—O4^{ii}	115.53 (8)
O1^{i}—Cr1—N1^{i}	84.66 (6)	O2^{iii}—K1S—O1^{iii}	43.90 (4)
O3—Cr1—N1^{i}	97.38 (5)	O2—K1S—O1^{iii}	84.65 (6)
O3^{i}—Cr1—N1^{i}	82.62 (5)	O3^{iv}—K1S—O1^{iii}	91.19 (4)
N1—Cr1—N1^{i}	180.00 (7)	O3^{ii}—K1S—O1^{iii}	154.19 (4)
C1—O1—Cr1	117.20 (11)	O4^{iv}—K1S—O1^{iii}	111.36 (5)
C1—O1—K1S	90.53 (10)	O4^{ii}—K1S—O1^{iii}	113.71 (4)
Cr1—O1—K1S	152.23 (6)	O2^{iii}—K1S—O1	84.65 (6)
C1—O2—K1S	101.75 (11)	O2—K1S—O1	43.90 (4)
C3—O3—Cr1	116.48 (11)	O3^{iv}—K1S—O1	154.19 (4)

C3—O3—K1S^{ii}	96.63 (10)	O3^{ii}—K1S—O1	91.19 (4)
Cr1—O3—K1S^{ii}	142.48 (6)	O4^{iv}—K1S—O1	113.71 (4)
C3—O4—K1S^{ii}	95.27 (12)	O4^{ii}—K1S—O1	111.36 (5)
C5—N1—C4	109.69 (15)	O1^{iii}—K1S—O1	88.15 (7)
C5—N1—C2	110.46 (15)	O2^{iii}—K1S—C1^{iii}	21.17 (4)
C4—N1—C2	110.50 (15)	O2—K1S—C1^{iii}	98.43 (6)
C5—N1—Cr1	113.95 (11)	O3^{iv}—K1S—C1^{iii}	103.11 (4)
C4—N1—Cr1	105.34 (10)	O3^{ii}—K1S—C1^{iii}	131.52 (4)
C2—N1—Cr1	106.75 (10)	O4^{iv}—K1S—C1^{iii}	132.69 (5)
O2—C1—O1	123.79 (16)	O4^{ii}—K1S—C1^{iii}	93.56 (4)
O2—C1—C2	119.04 (16)	O1^{iii}—K1S—C1^{iii}	22.73 (4)
O1—C1—C2	117.17 (15)	O1—K1S—C1^{iii}	85.72 (6)
O2—C1—K1S	57.08 (10)	O2^{iii}—K1S—C1	98.43 (6)
O1—C1—K1S	66.74 (9)	O2—K1S—C1	21.17 (4)
C2—C1—K1S	175.95 (12)	O3^{iv}—K1S—C1	131.52 (4)
N1—C2—C1	114.11 (15)	O3^{ii}—K1S—C1	103.11 (4)
N1—C2—H2A	108.7	O4^{iv}—K1S—C1	93.56 (4)
C1—C2—H2A	108.7	O4^{ii}—K1S—C1	132.69 (5)
N1—C2—H2B	108.7	O1^{iii}—K1S—C1	85.72 (6)
C1—C2—H2B	108.7	O1—K1S—C1	22.73 (4)
H2A—C2—H2B	107.6	C1^{iii}—K1S—C1	92.12 (7)
O4—C3—O3	123.62 (17)	O2^{iii}—K1S—C3^{ii}	93.03 (5)
O4—C3—C4	120.65 (16)	O2—K1S—C3^{ii}	133.81 (4)
O3—C3—C4	115.66 (15)	O3^{iv}—K1S—C3^{ii}	94.64 (6)
O4—C3—K1S^{ii}	63.63 (11)	O3^{ii}—K1S—C3^{ii}	22.45 (4)
O3—C3—K1S^{ii}	60.92 (9)	O4^{iv}—K1S—C3^{ii}	103.37 (7)
C4—C3—K1S^{ii}	166.70 (13)	O4^{ii}—K1S—C3^{ii}	21.10 (4)
N1—C4—C3	112.21 (14)	O1^{iii}—K1S—C3^{ii}	134.49 (4)
N1—C4—H4A	109.2	O1—K1S—C3^{ii}	104.15 (4)
C3—C4—H4A	109.2	C1^{iii}—K1S—C3^{ii}	113.34 (5)
N1—C4—H4B	109.2	C1—K1S—C3^{ii}	121.12 (4)
C3—C4—H4B	109.2	O2^{iii}—K1S—C3^{iv}	133.81 (4)
H4A—C4—H4B	107.9	O2—K1S—C3^{iv}	93.03 (5)
N1—C5—H5A	109.5	O3^{iv}—K1S—C3^{iv}	22.45 (4)
N1—C5—H5B	109.5	O3^{ii}—K1S—C3^{iv}	94.64 (6)
H5A—C5—H5B	109.5	O4^{iv}—K1S—C3^{iv}	21.10 (4)
N1—C5—H5C	109.5	O4^{ii}—K1S—C3^{iv}	103.37 (7)
H5A—C5—H5C	109.5	O1^{iii}—K1S—C3^{iv}	104.15 (4)
H5B—C5—H5C	109.5	O1—K1S—C3^{iv}	134.49 (4)
O2^{iii}—K1S—O2	111.01 (8)	C1^{iii}—K1S—C3^{iv}	121.12 (4)
O2^{iii}—K1S—O3^{iv}	112.23 (4)	C1—K1S—C3^{iv}	113.34 (5)
O2—K1S—O3^{iv}	110.35 (4)	C3^{ii}—K1S—C3^{iv}	97.73 (8)
O2^{iii}—K1S—O3^{ii}	110.35 (4)	H1NS—N1S—H2NS	105.9 (19)

K1S—O2—C1—O1	−1.9 (2)	K1S^{ii}—O4—C3—C4	−165.65 (16)
K1S—O2—C1—C2	178.67 (16)	Cr1—O3—C3—O4	−173.25 (15)
Cr1—O1—C1—O2	−179.97 (14)	K1S^{ii}—O3—C3—O4	−11.5 (2)
K1S—O1—C1—O2	1.73 (19)	Cr1—O3—C3—C4	3.7 (2)

Cr1—O1—C1—C2	−0.5 (2)	K1S^{ii}—O3—C3—C4	165.51 (14)
K1S—O1—C1—C2	−178.83 (16)	Cr1—O3—C3—K1S^{ii}	−161.79 (12)
Cr1—O1—C1—K1S	178.30 (12)	C5—N1—C4—C3	−151.02 (16)
C5—N1—C2—C1	127.82 (19)	C2—N1—C4—C3	86.96 (18)
C4—N1—C2—C1	−110.61 (19)	Cr1—N1—C4—C3	−27.99 (18)
Cr1—N1—C2—C1	3.4 (2)	O4—C3—C4—N1	−165.12 (17)
O2—C1—C2—N1	177.29 (16)	O3—C3—C4—N1	17.8 (2)
O1—C1—C2—N1	−2.2 (3)	K1S^{ii}—C3—C4—N1	89.7 (5)
K1S^{ii}—O4—C3—O3	11.2 (2)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1*S*—H1*NS*···O3^{iv}	0.92 (1)	2.07 (1)	2.9658 (16)	166 (3)
N1*S*—H2*NS*···O2	0.90 (1)	1.95 (1)	2.8485 (17)	175 (3)

Symmetry code: (iv) *x*, −*y*, *z*+1/2.