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Crystal structure and Hirshfeld surface analysis of poly[[bis[μ_4 -*N,N'*-(1,3,5-oxadiazinane-3,5-diyl)bis-(carbamoylmethanoato)]nickel(II)tetrapotassium] 4.8-hydrate]

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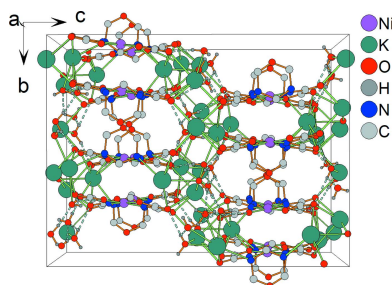
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Murmanska 1, 02094 Kiev, Ukraine, and ^dThe Faculty of Physics, Tajik National University, Rudaki Avenue 17, 734025 Dushanbe, Tajikistan. *Correspondence e-mail: plutenkom@gmail.com, voruch@eml.ru

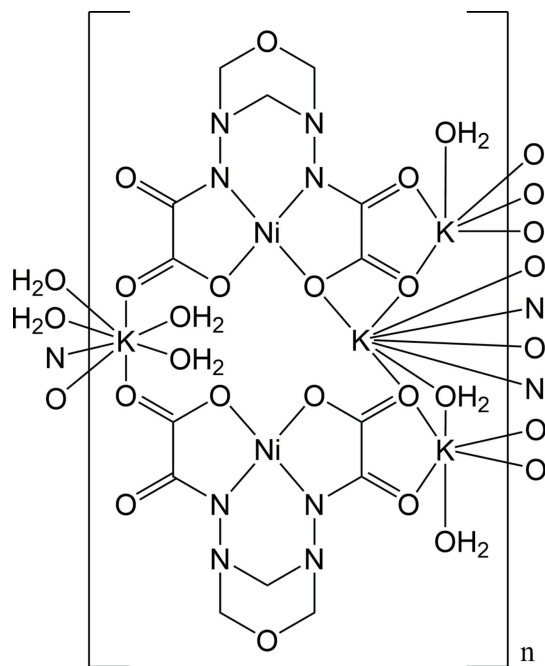
The title compound, $\{[K_4Ni_2(C_7H_6N_4O_7)_2] \cdot 4.8H_2O\}_n$, was obtained as a result of a template reaction between oxalohydrazidehydroxamic acid, formaldehyde and nickel(II) nitrate followed by partial hydrolysis of the formed intermediate. The two independent $[Ni(C_7H_6N_4O_7)]^{2-}$ complex anions exhibit pseudo- C_s symmetry and consist of an almost planar metal-containing fragment and a 1,3,5-oxadiazinane ring with a chair conformation disposed nearly perpendicularly with respect to the former. The central Ni^{II} atom has a square-planar N_2O_2 coordination arrangement formed by two amide N and two carboxylate O atoms. In the crystal, the nickel(II) complex anions form layers parallel to the *ab* plane. Neighboring complex anion layers are connected by layers of potassium cations for which two of the four independent cations are disordered over two sites [ratios of 0.54 (3):0.46 (3) and 0.9643 (15):0.0357 (15)]. The framework is stabilized by an extensive system of hydrogen bonds where the water molecules act as donors and the carboxylic O atoms, the amide O atoms and the oxadiazinane N atoms act as acceptors.

1. Chemical context

Coordination compounds of paramagnetic metal ions based on polydentate ligands comprising amide, hydrazide and hydroxamate functional groups are of great interest as they often form novel oligonuclear structures with interesting supramolecular features (Mezei *et al.*, 2007; Strotmeyer *et al.*, 2003). Frequently, these compounds exhibit unusual magnetic properties (Pavlishchuk *et al.*, 2010; Gumienna-Kontecka *et al.*, 2007; Pavlishchuk *et al.*, 2011; Huang *et al.*, 2014) and have potential biological activity (Raja *et al.*, 2012). The use of hydrazide metal complexes as synthons for template reactions has allowed coordination compounds with more complicated, sometimes unpredictable molecular structures to be obtained (Clark *et al.*, 1976). In particular, for ring-closure reactions, aldehydes (especially formaldehyde) can be used successfully as capping reagents for template condensation, as has been shown in several studies (Fritsky *et al.*, 1998, 2006; Tomyn *et al.*, 2017). Importantly, depending on the nature and coordination preference of the metal ion, the products of the ring-closure reactions can be both macrocyclic or pseudomacrocyclic (Ni^{2+} , Cu^{2+} ; Fritsky *et al.*, 1998, 2006; Tomyn *et al.*, 2017) and macrobicyclic (Fe^{4+} ; Tomyn *et al.*, 2017; Shylin *et al.*, 2019a,b).



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Here, we report the crystal structure of the polymeric title compound $\{K_4[Ni(L-2H)]_2 \cdot 4.8H_2O\}_n$ [$L = N,N'$ -(1,3,5-oxadiazinane-3,5-diyl)bis(carbamoylmethanoic acid)] (**1**) obtained as a result of a template reaction between oxalohydrazidehydroxamic acid, formaldehyde and nickel(II) nitrate followed by partial hydrolysis of the formed intermediate. The plausible mechanism of formation for (**1**) includes the deprotonation of oxalohydrazidehydroxamic acid and coordination to the metal ions in a tetradentate mode, followed by template condensation of two hydrazide moieties with three molecules of formaldehyde and metal-promoted hydrolysis of the hydroxamate group of the formed intermediate, which

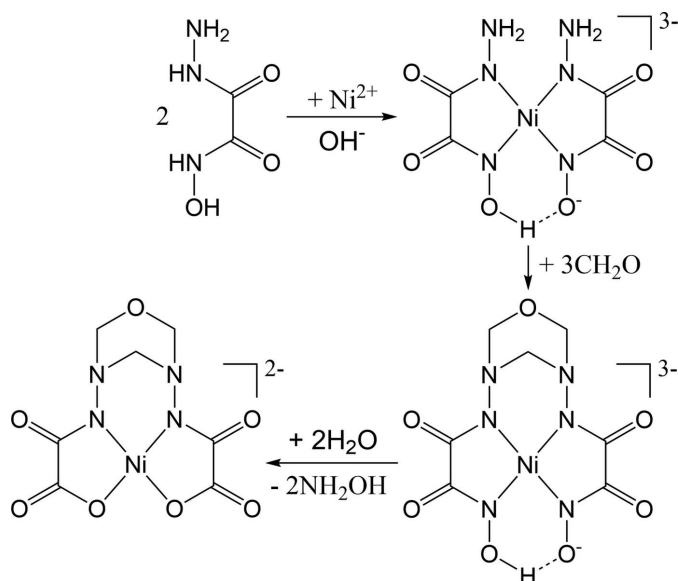


Figure 1
The plausible mechanism for the formation of the $[Ni(L-2H)]^{2-}$ anion.

eventually results in the formation of the nickel(II) complex anion $[Ni(L-2H)]^{2-}$ (Fig. 1). The crystallization process causes the bonding of such anions with the potassium counter-cations and the water solvent molecules, forming the three-dimensional coordination polymer $\{K_4[Ni(L-2H)]_2 \cdot 4.8H_2O\}_n$ (**1**).

2. Structural commentary

The asymmetric unit of (**1**) (Fig. 2) contains two complex anions $[Ni(L-2H)]^{2-}$ (which contain Ni1 and Ni1B, respectively), four potassium cations (two of which, K3 and K4, are disordered over two sites) and five solvent water molecules, one of which is disordered over two sets of sites (O4WA and O4WB in a ratio of 0.8:0.2), and one (O5W) that has an occupancy of 0.8.

Both complex anions $[Ni(C_7H_6N_4O_7)]^{2-}$ have a pseudo- C_s symmetry with similar bond lengths and angles. Each anion consists of an almost planar metal-containing $\{NiN_2O_2\}$ fragment [the maximum deviation of the atoms involved in the anion from the least-squares plane is 0.1232 (12) Å for the anion centred by Ni1 and -0.1510 (13) Å for the anion centred by Ni1B] and an 1,3,5-oxadiazinane ring disposed nearly perpendicularly with respect to the former. The 1,3,5-oxadiazinane ring in each anion adopts a chair conformation. The dihedral angle between the mean planes formed by the non-hydrogen atoms of these two fragments is 87.22 (5)° for the Ni1 anion and 86.89 (5)° for the Ni1B anion. Thus, the complex anions reveal an L-like shape.

The ligand molecule (*L-2H*) coordinates in a tetradentate $\{O_{\text{carboxyl}}, N_{\text{amide}}, N_{\text{amide}}, O_{\text{carboxyl}}\}$ mode, thus forming three fused chelate rings (two five-membered and one six-membered). The central Ni^{II} atom of the complex anion has a square-planar coordination arrangement with an N_2O_2 chromophore. The deviation of the Ni^{II} atom from the mean plane

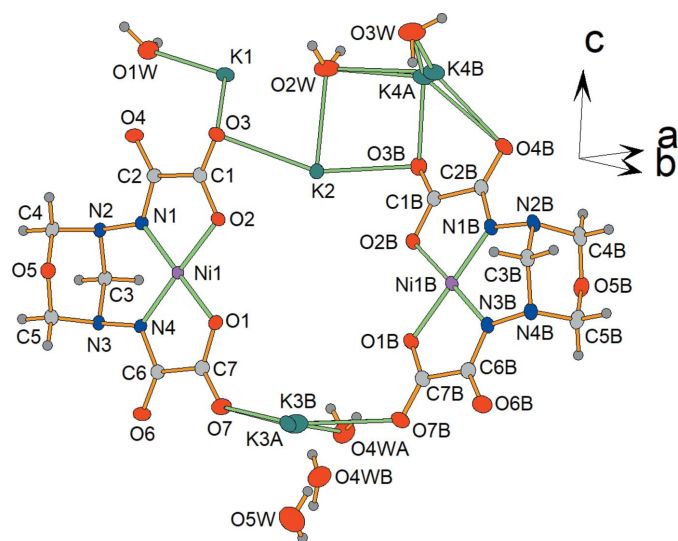


Figure 2
The asymmetric unit of (**1**) with displacement ellipsoids shown at the 50% probability level. The potassium cations K3 and K4 and the solvate water molecule O4W are disordered over two positions, namely K3A and K3B, K4A and K4B, O4WA and O4WB, respectively.

Table 1
Selected geometric parameters (Å, °).

Ni1–N4	1.8429 (15)	Ni1B–N1B	1.8436 (16)
Ni1–N1	1.8479 (15)	Ni1B–N4B	1.8463 (16)
Ni1–O1	1.8830 (13)	Ni1B–O2B	1.8922 (14)
Ni1–O2	1.9012 (13)	Ni1B–O1B	1.8975 (14)
N4–Ni1–N1	96.01 (7)	N1B–Ni1B–N4B	95.98 (7)
N4–Ni1–O1	85.25 (6)	N1B–Ni1B–O2B	85.01 (6)
N1–Ni1–O1	178.74 (6)	N4B–Ni1B–O2B	177.89 (7)
N4–Ni1–O2	178.28 (7)	N1B–Ni1B–O1B	178.86 (7)
N1–Ni1–O2	85.10 (6)	N4B–Ni1B–O1B	85.08 (7)
O1–Ni1–O2	93.65 (6)	O2B–Ni1B–O1B	93.94 (6)

defined by the four donor atoms is 0.0098 (8) and 0.0116 (9) Å for Ni1 and Ni1B, respectively. The Ni–N and Ni–O bond lengths (Table 1) are in the range 1.8429 (15)–1.8479 (15) and 1.8830 (13)–1.9012 (13) Å, respectively, typical for square-planar nickel(II) complexes with similar tetradentate ligands (Fritsky *et al.*, 2004; Sliva *et al.*, 1997*a,b*; Duda *et al.*, 1997). The bite angles O1–Ni1–N4, N1–Ni1–O2 and N1–Ni1–N4 for both anions (Table 1) deviate from the ideal value of 90°, conditioned by the formation of five-membered chelate rings. N–N', N–C and C–O and C=O bond lengths within the (*L*-2H) ligand indicate values typical for the coordinating deprotonated hydrazide and carboxyl groups.

3. Supramolecular features

In the crystal, the nickel(II) complex anions [Ni(*L*-2H)]²⁻ form layers parallel to *ab* plane (Fig. 3). Neighboring complex anion layers are sandwiched by layers of potassium counter-cations (Fig. 4). Thus, complex anion layers and potassium layers are stacked along the *c*-axis direction (Fig. 5).

The potassium cations are bound to the nickel(II) complex anions through the amide and the carboxylic O atoms (K1, K4A) or through the amide O and the oxadiazinane N atoms (K2, K3B). In addition, the potassium cations have contacts with the O atoms of the water molecules, with the amide and the carboxylic O atoms, and with the oxadiazinane N atoms of

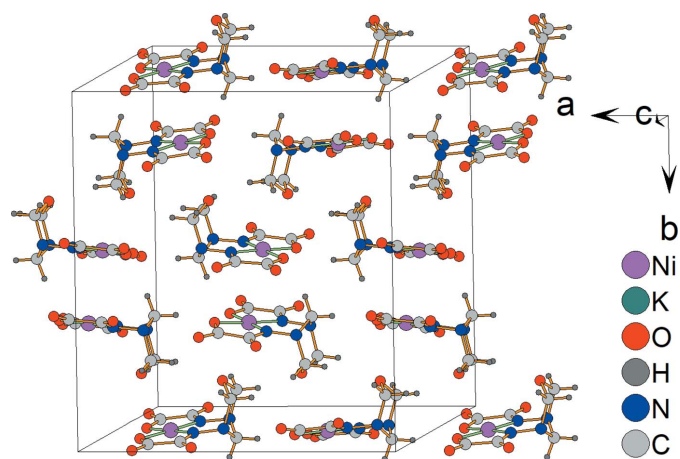


Figure 3
Layers formed by the anionic nickel(II) complexes.

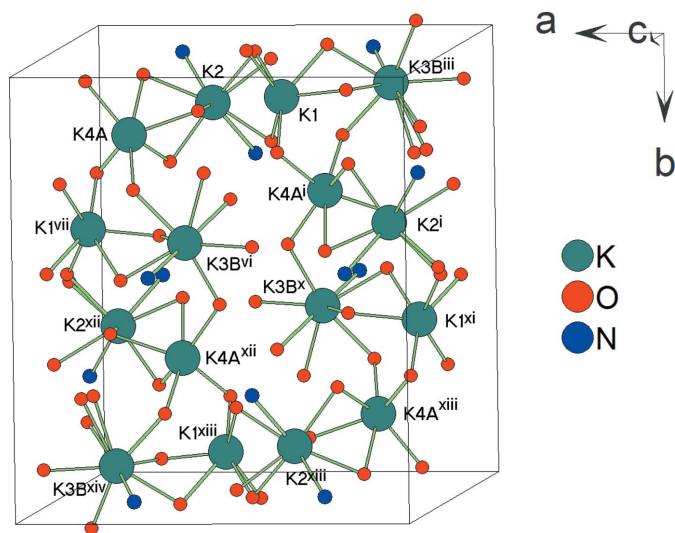


Figure 4
The layer of potassium cations and their coordination arrangement. The minor disordered components of K3 and K4 atoms (K3A and K4B) are omitted for clarity. [Symmetry codes: (i) $x - \frac{1}{2}, -y + \frac{1}{2}, -z + 1$; (iii) $x - \frac{1}{2}, y, -z + \frac{1}{2}$; (vi) $x, -y + \frac{1}{2}, z + \frac{1}{2}$; (vii) $x + \frac{1}{2}, -y + \frac{1}{2}, -z + 1$; (x) $-x + 1, y + \frac{1}{2}, -z + \frac{1}{2}$; (xi) $-x + \frac{1}{2}, y + \frac{1}{2}, -z$; (xii) $-x + \frac{3}{2}, y + \frac{1}{2}, z$; (xiii) $x + 1, -y + 1, -z + 1$; (xiv) $-x + \frac{2}{3}, -y + 1, z + \frac{1}{2}$].

neighboring complex anions. For definition of the coordination spheres around the cations, K–O and K–N contacts that do not exceed the sum of the ionic radii by more than 0.2 Å were defined as bonding contacts [the values of the ionic radii were taken from Shannon (1976)]. The K1, K2 and K4A cations exhibit O₆, O₆N₂ and O₆ coordination sets. As a result of the disorder of the water molecules, the K3B site has an O₆N or O₇N coordination set. In addition, there are K1...O2W, K1...O7B and K2...O2B remote non-bonding contacts, which are significantly greater than the sum of the ionic radii.

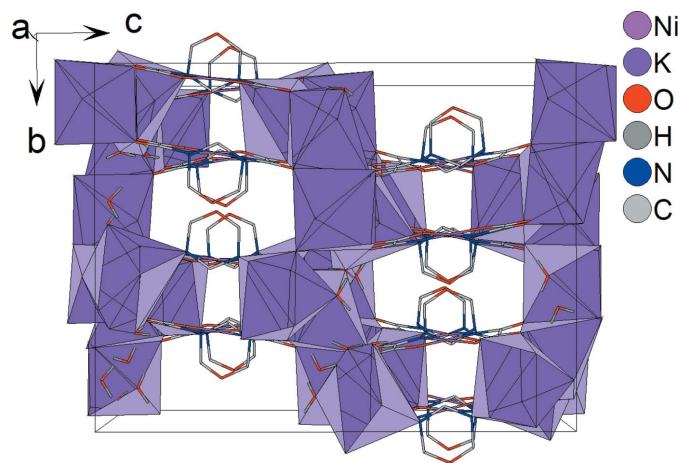


Figure 5
Crystal packing of the title compound in a stick model, showing the coordination polyhedra of the potassium cations in lilac. H atoms of the C–H groups and minor disordered components (K3A and K4B, O4WB water molecule) are omitted for clarity.

Table 2

Values for continuous shapes measures (CShM) of the polyhedra centred by the potassium cations (only major components for the disordered parts are considered).

Shape	CShM	
	K1	K4A
Hexagon (D_{6h})	30.965	33.688
Pentagonal pyramid (C_{5v})	9.924	22.357
Octahedron (Oh)	13.859	4.985
Trigonal prism (D_{3h})	4.697	10.581
Johnson pentagonal pyramid J2 (C_{5v})	13.919	26.100
	K2	K3B
Octagon (D_{8h})	32.591	28.712
Heptagonal pyramid (C_{7v})	18.314	20.510
Hexagonal bipyramid (D_{6h})	14.891	13.393
Cube (Oh)	14.913	14.525
Square antiprism (D_{4d})	6.805	19.105
Triangular dodecahedron (D_{2d})	4.992	17.608
Johnson gyrobifastigium J26 (D_{2d})	10.479	16.378
Johnson elongated triangular bipyramid J14 (D_{3h})	23.441	18.219
Biaugmented trigonal prism J50 (C_{2v})	6.800	16.341
Biaugmented trigonal prism (C_{2v})	5.698	16.739
Snub diphenoid J84 (D_{2d})	6.894	15.895
Triakis tetrahedron (Td)	15.016	14.550
Elongated trigonal bipyramid (D_{3h})	17.893	13.966

For an evaluation of the coordination geometry of each potassium cation, the *SHAPE 2.1* software (Llunell *et al.*, 2013) was used. A *SHAPE* analysis of the potassium coordination sphere (Table 2, Fig. 6) yields the lowest continuous shape measure (CShM) value for a distorted trigonal prism (4.697 for K1), a distorted triangular dodecahedron (4.992 for K2), a distorted hexagonal bipyramid (13.393 for K3B) and a distorted octahedron (4.985 for K4A). For K2 and K3B, comparable CShM values were obtained for a biaugmented trigonal prism (5.698) and an elongated trigonal bipyramid (13.966), respectively.

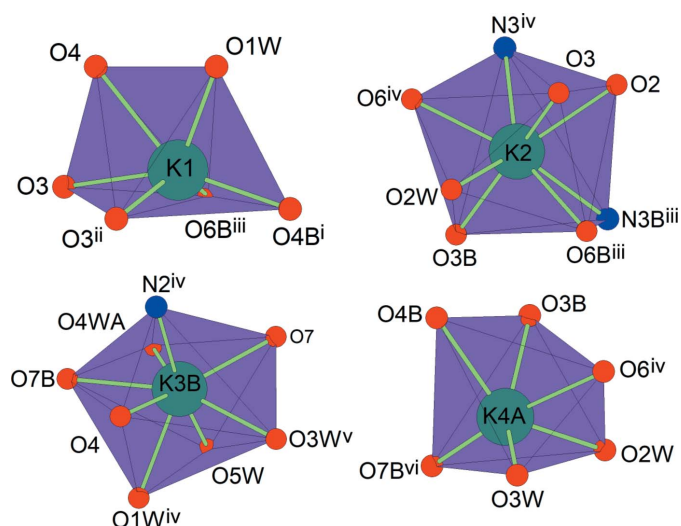


Figure 6

Polyhedral views of the coordination environments for the potassium cations; the minor disordered components of atoms K3 and K4 (K3A and K4B) are omitted for clarity. [Symmetry codes: (i) $x - \frac{1}{2}, -y + \frac{1}{2}, -z + 1$; (ii) $-x + 1, -y, -z + 1$; (iii) $x - \frac{1}{2}, y, -z + \frac{1}{2}$; (iv) $x + \frac{1}{2}, y, -z + \frac{1}{2}$; (v) $-x + \frac{1}{2}, -y, z - \frac{1}{2}$; (vi) $x, -y + \frac{1}{2}, z + \frac{1}{2}$].

Table 3

Hydrogen-bond geometry ($\text{\AA}, ^\circ$).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O1W—H1W1 \cdots O6 ⁱ	0.92	1.94	2.840 (2)	166
O1W—H2W1 \cdots O5W ⁱⁱ	0.83	2.61	3.120 (4)	121
O1W—H2W1 \cdots O3B ⁱⁱⁱ	0.83	2.23	3.001 (2)	154
O2W—H2W2 \cdots O4 ^{iv}	0.93	1.83	2.754 (2)	171
O4WA—H2W4 \cdots O4B ⁱⁱ	0.91	2.44	2.993 (2)	119
O4WA—H2W4 \cdots N2B ⁱⁱ	0.91	2.02	2.895 (3)	161
O5W—H5WC \cdots O6B ^v	0.85	1.95	2.774 (3)	162
O4WB—H3W4 \cdots O4B ⁱⁱ	0.85	2.09	2.848 (9)	149
O4WB—H4W4 \cdots O6B ^v	0.88	2.15	3.024 (9)	174

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

The polyhedra around neighboring potassium cations are connected with each other through common vertices (K1 with K2, K1 with K4A, K3B with K4A), edges (K1 with K1, K1 with K2, K1 with K3B) and faces (K2 with K4A). The K—O and K—N bond lengths are normal for potassium cations and close to those reported in the structures of related carboxylate and amide complexes (Fritsky *et al.*, 1998; Świątek-Kozłowska *et al.*, 2000; Mokhir *et al.*, 2002).

The polymeric framework is stabilized by an extensive system of hydrogen-bonding interactions where the water molecules act as donors and the carboxylic O atoms, the amide O atoms and the oxadizidine N atoms act as acceptors (Table 3, Fig. 7).

4. Hirshfeld analysis

The Hirshfeld surface analysis (Spackman & Jayatilaka, 2009) was performed and the associated two-dimensional fingerprint plots (McKinnon *et al.*, 2007) were obtained with *Crystal-Explorer17* (Turner *et al.*, 2017). The Hirshfeld surfaces of the complex anions are colour-mapped with the normalized contact distance (d_{norm}) from red (distances shorter than the

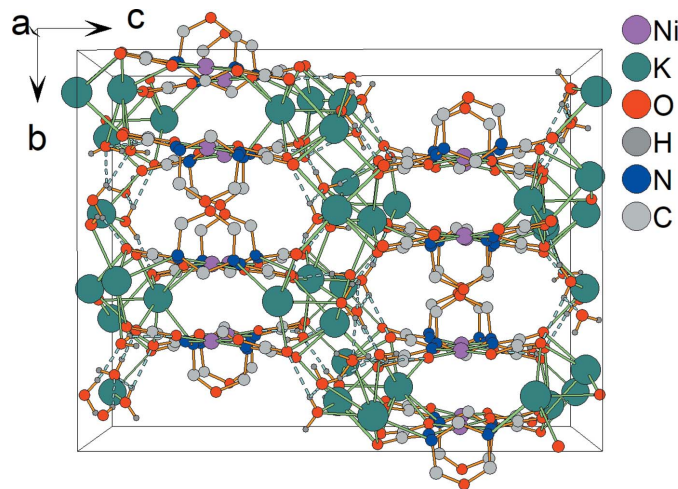


Figure 7

Crystal packing of the title compound. Hydrogen bonds are indicated by dashed lines. H atoms of the C—H groups and minor disordered components (K3A and K4B, O4WB water molecule) are omitted for clarity.

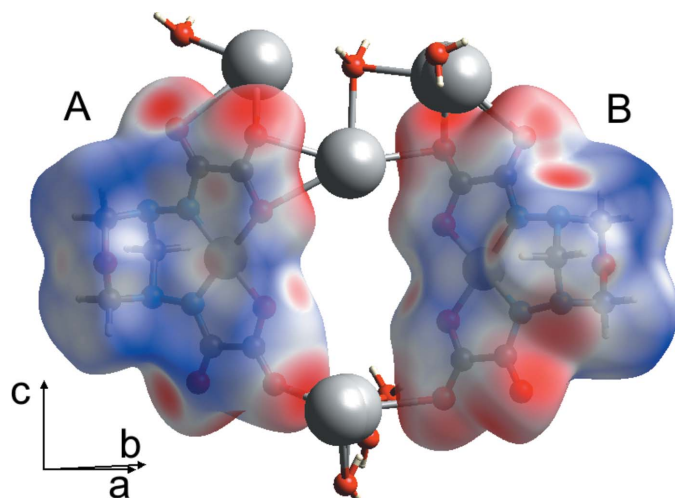


Figure 8
The Hirshfeld surfaces of the two complex anions (A = Ni and B = NiB) mapped over d_{norm} .

sum of the van der Waals radii) through white to blue (distances longer than the sum of the van der Waals radii). The Hirshfeld surface mapped over d_{norm} , in the colour range -0.6411 to 0.9651 a.u. for the anion centred by Ni1 (A) and -0.6382 to 0.9607 a.u. for the anion centred by Ni1B (B) is shown in Fig. 8. Both complex anions are connected to the other moieties of the crystal structure mainly through the amide and the carboxylic O atoms.

A two-dimensional fingerprint plot contains information related to specific intermolecular interactions. The blue colour refers to the frequency of occurrence of the (d_i , d_e) pair with

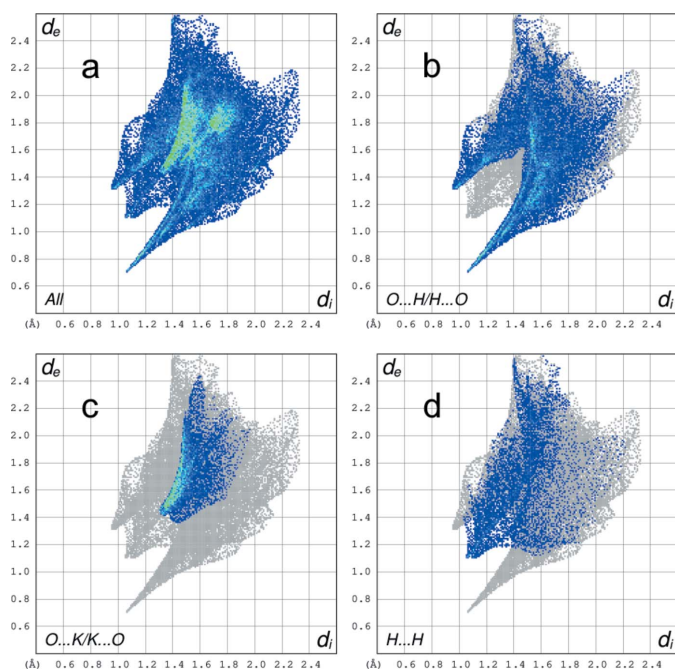


Figure 9
(a) Full two-dimensional fingerprint plot of the A complex anion (Ni1), and delineated into (b) $\text{O}\cdots\text{H}/\text{H}\cdots\text{O}$ (41.3%) (c) $\text{O}\cdots\text{K}/\text{K}\cdots\text{O}$ (15.8%) and (d) $\text{H}\cdots\text{H}$ (13.7%) contacts.

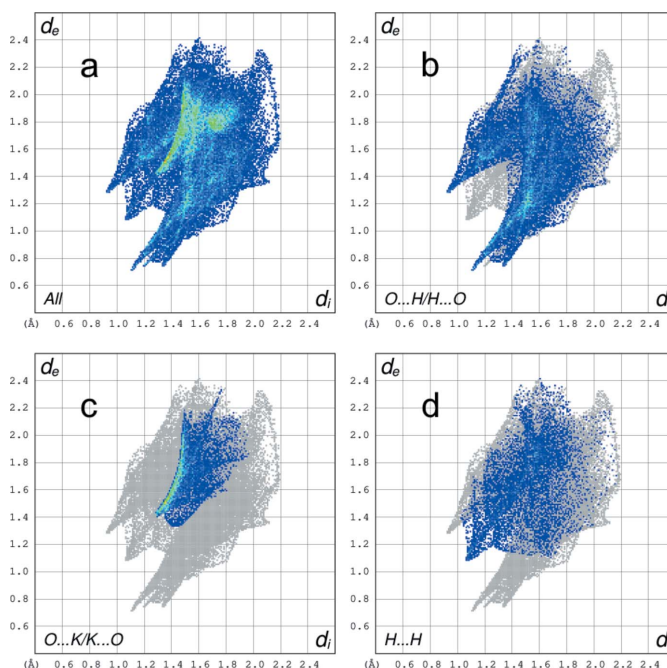


Figure 10
(a) Full two-dimensional fingerprint plot of the B complex anion (Ni1B), and delineated into (b) $\text{O}\cdots\text{H}/\text{H}\cdots\text{O}$ (41.0%) (c) $\text{O}\cdots\text{K}/\text{K}\cdots\text{O}$ (15.8%) and (d) $\text{H}\cdots\text{H}$ (15.1%) contacts.

the full fingerprint plot outlined in gray. Figs. 9a and 10a show the two-dimensional fingerprint plots for the anion centred by Ni1 (A) and by Ni1B (B), represented by the sum of the contacts contributing to the Hirshfeld surface in normal mode. The most significant contribution to the Hirshfeld surface is from $\text{O}\cdots\text{H}/\text{H}\cdots\text{O}$ contacts (41.3% for complex A and 41.0% for complex B, respectively; Fig. 9b and 10b). In addition, $\text{O}\cdots\text{K}/\text{K}\cdots\text{O}$ (15.8% for complex anions A and B; Fig. 9c and 10c) and $\text{H}\cdots\text{H}$ (13.7% for complex anion A and 15.1% for complex anion B; Fig. 9d and 10d) are other significant contributions to the total Hirshfeld surface.

5. Database survey

A search of the Cambridge Structural Database (CSD version 5.41, update of November 2019; Groom *et al.*, 2016) for complexes obtained by hydrazide, aldehyde and 3d-metal salt interactions gave eleven hits for structures with full atomic coordinates. All these compounds include macrocyclic or pseudo-macrocyclic ligands formed by template binding of several hydrazide groups by aldehyde molecules. The 3d-metal ions of these complexes are often in high oxidation states: Cu^{III} (Oliver *et al.*, 1982; Fritsky *et al.*, 1998, 2006) and Fe^{IV} (Tomyn *et al.*, 2017) complexes have been described.

6. Synthesis and crystallization

A solution of $\text{Ni}(\text{NO}_3)_2\cdot 6\text{H}_2\text{O}$ (0.073 g, 0.25 mmol) in 5 ml of water was added to a warm solution of oxalohydrazide-hydroxamic acid (0.06 g, 0.5 mmol) in 5 ml of water. The

Table 4
Experimental details.

Crystal data	
Chemical formula	[K ₄ Ni ₂ (C ₇ H ₆ N ₄ O ₇) ₂].4.8H ₂ O
<i>M_r</i>	876.66
Crystal system, space group	Orthorhombic, <i>Pbca</i>
Temperature (K)	100
<i>a</i> , <i>b</i> , <i>c</i> (Å)	15.0694 (3), 16.9659 (3), 22.1920 (4)
<i>V</i> (Å ³)	5673.74 (18)
<i>Z</i>	8
Radiation type	Mo <i>K</i> α
<i>μ</i> (mm ⁻¹)	2.01
Crystal size (mm)	0.31 × 0.26 × 0.23
Data collection	
Diffractometer	Bruker Kappa APEXII CCD
Absorption correction	Multi-scan (<i>SADABS</i> ; Krause <i>et al.</i> , 2015)
<i>T_{min}</i> , <i>T_{max}</i>	0.572, 0.653
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	51429, 8287, 7371
<i>R_{int}</i>	0.037
(sin θ/λ) _{max} (Å ⁻¹)	0.707
Refinement	
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.033, 0.076, 1.10
No. of reflections	8287
No. of parameters	448
H-atom treatment	H-atom parameters constrained
Δρ _{max} , Δρ _{min} (e Å ⁻³)	0.81, -0.69

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *SHELXS97* (Sheldrick, 2008), *SHELXL2018/1* (Sheldrick, 2015), *DIAMOND* (Brandenburg, 2009) and *publCIF* (Westrip, 2010).

resulting light-green mixture was stirred with heating (320–330 K) for 20 min, and then 1 ml of a 4M KOH solution was added. As a result, the color of the solution changed to pink. After 5 min of stirring, 0.03 g of paraformaldehyde (1 mmol) were added, followed by stirring with heating (320–330 K) for 30 min. The resulting orange solution was left for crystallization by slow diffusion of methanol vapor. After two months, orange crystals suitable for X-ray diffraction studies were obtained. The crystals were filtered off, washed with diethyl ether and dried in air.

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 4. The potassium cations K3 and K4 were found to be disordered over two positions with occupancy factors for the major disorder component of 0.54 (3) (K3B) and 0.9643 (15) (K4A). The solvate water molecule O4W appeared to be disordered over two positions with relative occupancies of 0.805 (4) (O4WA) and 0.195 (4) (O4WB). The solvate water molecule O5W was found to be incompatible with the second positions of the water molecule O4W and thus was refined with the same occupancy factor as the major fraction of O4W as they are linked by a hydrogen bond. The O–H hydrogen atoms were located from a difference-Fourier map and constrained to ride on their parent atoms with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$. The methylene C–H

hydrogen atoms were positioned geometrically and were constrained to ride on their parent atoms, with C–H = 0.99 Å, and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

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Crystal structure and Hirshfeld surface analysis of poly[[bis[μ_4 -*N,N'*-(1,3,5-oxadiazinane-3,5-diyl)bis(carbamoylmethanoato)]nickel(II)tetrapotassium] 4.8-hydrate]

Maksym O. Plutenko, Matti Haukka, Alina O. Husak, Turganbay S. Iskenderov and Nurullo U. Mulloev

Computing details

Data collection: *APEX3* (Bruker, 2016); cell refinement: *SAINTE* (Bruker, 2016); data reduction: *SAINTE* (Bruker, 2016); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2018/1* (Sheldrick, 2015); molecular graphics: *DIAMOND* (Brandenburg, 2009); software used to prepare material for publication: *publCIF* (Westrip, 2010).

Poly[[bis[μ_4 -*N,N'*-(1,3,5-oxadiazinane-3,5-diyl)bis(carbamoylmethanoato)]nickel(II)tetrapotassium] 4.8-hydrate]

Crystal data

$[\text{K}_4\text{Ni}_2(\text{C}_7\text{H}_6\text{N}_4\text{O}_7)_2] \cdot 4.8\text{H}_2\text{O}$

$M_r = 876.66$

Orthorhombic, *Pbca*

$a = 15.0694$ (3) Å

$b = 16.9659$ (3) Å

$c = 22.1920$ (4) Å

$V = 5673.74$ (18) Å³

$Z = 8$

$F(000) = 3552$

$D_x = 2.053$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 18674 reflections

$\theta = 1.0$ – 30.0°

$\mu = 2.01$ mm⁻¹

$T = 100$ K

Block, orange

$0.31 \times 0.26 \times 0.23$ mm

Data collection

Bruker Kappa APEXII CCD
diffractometer

Radiation source: fine-focus sealed tube

Horizontally mounted graphite crystal
monochromator

Detector resolution: 16 pixels mm⁻¹

φ scans and ω scans with κ offset

Absorption correction: multi-scan
(SADABS; Krause *et al.*, 2015)

$T_{\min} = 0.572$, $T_{\max} = 0.653$

51429 measured reflections

8287 independent reflections

7371 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.037$

$\theta_{\max} = 30.2^\circ$, $\theta_{\min} = 2.0^\circ$

$h = -20 \rightarrow 21$

$k = -23 \rightarrow 19$

$l = -31 \rightarrow 31$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.033$

$wR(F^2) = 0.076$

$S = 1.10$

8287 reflections

448 parameters

0 restraints

Hydrogen site location: mixed

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0165P)^2 + 9.9285P]$

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

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

$$\Delta\rho_{\max} = 0.81 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.69 \text{ e } \text{\AA}^{-3}$$

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)
Ni1	0.39976 (2)	0.03560 (2)	0.25909 (2)	0.01144 (6)	
K1	0.42066 (3)	0.10115 (3)	0.50619 (2)	0.01944 (9)	
K2	0.62602 (3)	0.12976 (3)	0.37947 (2)	0.01642 (8)	
K3A	0.6573 (3)	0.0634 (2)	0.0628 (2)	0.0156 (10)	0.46 (3)
K3B	0.6592 (3)	0.0786 (9)	0.0646 (2)	0.0447 (9)	0.54 (3)
K4A	0.81367 (4)	0.18864 (3)	0.48907 (2)	0.02787 (14)	0.9643 (15)
K4B	0.8620 (12)	0.1672 (10)	0.4887 (6)	0.02787 (14)	0.0357 (15)
O1	0.48022 (9)	0.05193 (8)	0.19539 (6)	0.0158 (3)	
O2	0.48968 (8)	0.03838 (8)	0.31915 (6)	0.0147 (3)	
O3	0.50728 (9)	0.01073 (9)	0.41691 (6)	0.0175 (3)	
O4	0.32237 (9)	-0.00551 (9)	0.42549 (6)	0.0187 (3)	
O5	0.23246 (9)	-0.10170 (8)	0.25535 (6)	0.0175 (3)	
O6	0.29549 (9)	0.06429 (10)	0.09851 (6)	0.0209 (3)	
O7	0.48153 (10)	0.07263 (11)	0.09625 (7)	0.0290 (4)	
O1W	0.24931 (12)	0.08231 (11)	0.54478 (7)	0.0294 (4)	
H1W1	0.236269	0.039493	0.568245	0.044*	
H2W1	0.226969	0.122493	0.559745	0.044*	
O2W	0.63486 (14)	0.13337 (10)	0.50382 (8)	0.0361 (4)	
H1W2	0.624292	0.171955	0.530219	0.054*	
H2W2	0.647697	0.086965	0.524420	0.054*	
O3W	0.91669 (13)	0.06558 (12)	0.52097 (8)	0.0383 (4)	
H1W3	0.926510	0.062280	0.482596	0.057*	
H2W3	0.967921	0.084541	0.536516	0.057*	
O4WA	0.60586 (13)	0.23598 (13)	0.07782 (10)	0.0309 (6)	0.805 (4)
H1W4	0.655353	0.218452	0.090399	0.046*	0.805 (4)
H2W4	0.567382	0.239687	0.109145	0.046*	0.805 (4)
O5W	0.5651 (2)	0.16499 (15)	-0.03385 (13)	0.0532 (9)	0.805 (4)
H5WC	0.546573	0.207539	-0.049704	0.080*	0.805 (4)
H5WB	0.588393	0.177329	-0.000284	0.080*	0.805 (4)
O4WB	0.5638 (6)	0.2234 (6)	0.0249 (4)	0.032 (2)	0.195 (4)
H3W4	0.531930	0.234322	0.055384	0.048*	0.195 (4)
H4W4	0.532796	0.240423	-0.006182	0.048*	0.195 (4)
N1	0.32286 (10)	0.01942 (9)	0.32277 (7)	0.0125 (3)	
N2	0.22857 (10)	0.01217 (10)	0.31956 (7)	0.0129 (3)	
N3	0.22075 (10)	0.02637 (10)	0.20888 (7)	0.0137 (3)	
N4	0.31391 (10)	0.03568 (10)	0.19995 (7)	0.0128 (3)	

C1	0.46081 (12)	0.01956 (11)	0.37164 (8)	0.0134 (3)
C2	0.35945 (12)	0.00948 (11)	0.37637 (8)	0.0131 (3)
C3	0.19564 (12)	0.05825 (11)	0.26801 (8)	0.0138 (3)
H3A	0.130103	0.060914	0.270332	0.017*
H3B	0.218626	0.112739	0.271334	0.017*
C4	0.20301 (13)	-0.07011 (12)	0.31134 (9)	0.0167 (4)
H4A	0.228229	-0.101882	0.344615	0.020*
H4B	0.137568	-0.074362	0.313553	0.020*
C5	0.19597 (13)	-0.05675 (12)	0.20682 (9)	0.0177 (4)
H5A	0.130467	-0.061003	0.208006	0.021*
H5B	0.216417	-0.079534	0.168170	0.021*
C6	0.34091 (12)	0.05407 (11)	0.14507 (8)	0.0146 (3)
C7	0.44272 (12)	0.06056 (12)	0.14422 (9)	0.0166 (4)
Ni1B	0.85260 (2)	0.23735 (2)	0.24199 (2)	0.01304 (6)
O1B	0.77709 (9)	0.22102 (9)	0.17472 (6)	0.0180 (3)
O2B	0.75916 (9)	0.23086 (8)	0.29873 (6)	0.0162 (3)
O3B	0.73206 (9)	0.26008 (9)	0.39495 (7)	0.0212 (3)
O4B	0.91522 (10)	0.28278 (10)	0.41008 (7)	0.0220 (3)
O5B	1.01572 (10)	0.37800 (8)	0.24472 (7)	0.0203 (3)
O6B	0.96972 (11)	0.21209 (10)	0.08487 (7)	0.0253 (3)
O7B	0.78613 (11)	0.19730 (10)	0.07598 (7)	0.0251 (3)
N1B	0.92407 (10)	0.25393 (10)	0.30822 (7)	0.0148 (3)
N2B	1.01790 (10)	0.26347 (10)	0.30814 (7)	0.0151 (3)
N3B	1.03515 (10)	0.25123 (10)	0.19792 (7)	0.0160 (3)
N4B	0.94292 (10)	0.23990 (10)	0.18580 (7)	0.0153 (3)
C1B	0.78284 (12)	0.25187 (11)	0.35171 (9)	0.0152 (3)
C2B	0.88312 (12)	0.26501 (11)	0.36010 (9)	0.0153 (3)
C3B	1.05635 (12)	0.21901 (12)	0.25760 (9)	0.0159 (3)
H3B1	1.121650	0.217835	0.262372	0.019*
H3B2	1.034773	0.163958	0.259596	0.019*
C4B	1.04157 (13)	0.34622 (12)	0.30124 (9)	0.0196 (4)
H4B1	1.012978	0.376994	0.333794	0.024*
H4B2	1.106608	0.351872	0.305799	0.024*
C5B	1.05732 (13)	0.33496 (12)	0.19717 (10)	0.0200 (4)
H5B1	1.122471	0.340939	0.200622	0.024*
H5B2	1.038927	0.357734	0.158040	0.024*
C6B	0.92131 (13)	0.22112 (12)	0.13005 (9)	0.0171 (4)
C7B	0.82017 (13)	0.21177 (12)	0.12558 (9)	0.0178 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.00784 (10)	0.01467 (12)	0.01179 (11)	-0.00082 (8)	-0.00019 (7)	0.00124 (8)
K1	0.0243 (2)	0.0196 (2)	0.01449 (18)	0.00216 (16)	-0.00152 (15)	-0.00092 (15)
K2	0.01387 (17)	0.0187 (2)	0.01671 (18)	-0.00075 (14)	0.00052 (14)	0.00027 (14)
K3A	0.0119 (9)	0.018 (2)	0.0172 (9)	0.0053 (6)	-0.0008 (6)	-0.0050 (9)
K3B	0.0199 (9)	0.083 (3)	0.0313 (13)	-0.0053 (16)	-0.0054 (8)	0.0187 (14)
K4A	0.0356 (3)	0.0307 (3)	0.0173 (2)	0.0017 (2)	-0.00088 (19)	-0.00179 (19)

K4B	0.0356 (3)	0.0307 (3)	0.0173 (2)	0.0017 (2)	-0.00088 (19)	-0.00179 (19)
O1	0.0110 (6)	0.0210 (7)	0.0154 (6)	-0.0016 (5)	0.0011 (5)	0.0024 (5)
O2	0.0110 (6)	0.0193 (7)	0.0138 (6)	-0.0006 (5)	-0.0022 (5)	0.0009 (5)
O3	0.0155 (6)	0.0211 (7)	0.0160 (6)	0.0007 (5)	-0.0042 (5)	0.0005 (5)
O4	0.0162 (6)	0.0268 (8)	0.0132 (6)	0.0031 (6)	0.0017 (5)	0.0019 (5)
O5	0.0177 (7)	0.0146 (7)	0.0203 (7)	-0.0005 (5)	-0.0007 (5)	0.0000 (5)
O6	0.0153 (6)	0.0338 (9)	0.0136 (6)	-0.0009 (6)	-0.0015 (5)	0.0034 (6)
O7	0.0168 (7)	0.0527 (11)	0.0176 (7)	-0.0055 (7)	0.0049 (6)	0.0025 (7)
O1W	0.0343 (9)	0.0321 (9)	0.0219 (7)	-0.0018 (7)	0.0047 (7)	-0.0029 (7)
O2W	0.0642 (13)	0.0242 (9)	0.0199 (8)	0.0043 (8)	-0.0021 (8)	0.0021 (6)
O3W	0.0388 (10)	0.0525 (12)	0.0236 (8)	0.0080 (9)	0.0036 (7)	0.0022 (8)
O4WA	0.0185 (9)	0.0401 (13)	0.0341 (12)	0.0043 (8)	0.0101 (8)	0.0022 (9)
O5W	0.073 (2)	0.0320 (14)	0.0542 (17)	-0.0034 (13)	-0.0292 (15)	0.0080 (12)
O4WB	0.022 (4)	0.039 (5)	0.035 (5)	0.010 (4)	0.003 (3)	0.012 (4)
N1	0.0084 (6)	0.0152 (8)	0.0139 (7)	0.0007 (5)	0.0009 (5)	0.0004 (6)
N2	0.0081 (6)	0.0157 (8)	0.0150 (7)	-0.0004 (5)	0.0000 (5)	0.0016 (6)
N3	0.0072 (6)	0.0181 (8)	0.0158 (7)	-0.0021 (5)	0.0002 (5)	0.0014 (6)
N4	0.0083 (6)	0.0167 (8)	0.0133 (7)	-0.0014 (5)	0.0004 (5)	0.0021 (6)
C1	0.0137 (8)	0.0108 (8)	0.0159 (8)	0.0015 (6)	-0.0011 (6)	-0.0014 (6)
C2	0.0123 (8)	0.0126 (8)	0.0143 (8)	0.0026 (6)	0.0006 (6)	0.0001 (6)
C3	0.0103 (7)	0.0153 (9)	0.0159 (8)	0.0008 (6)	0.0004 (6)	0.0022 (6)
C4	0.0146 (8)	0.0171 (9)	0.0184 (9)	-0.0031 (7)	0.0008 (7)	0.0036 (7)
C5	0.0157 (8)	0.0192 (10)	0.0183 (9)	-0.0039 (7)	-0.0008 (7)	-0.0002 (7)
C6	0.0120 (8)	0.0169 (9)	0.0151 (8)	-0.0006 (6)	-0.0002 (6)	0.0008 (7)
C7	0.0139 (8)	0.0190 (10)	0.0169 (8)	-0.0011 (7)	0.0014 (7)	0.0004 (7)
Ni1B	0.00973 (10)	0.01497 (12)	0.01442 (11)	-0.00085 (8)	-0.00058 (8)	-0.00124 (8)
O1B	0.0152 (6)	0.0210 (7)	0.0179 (6)	-0.0014 (5)	-0.0030 (5)	-0.0007 (5)
O2B	0.0114 (6)	0.0196 (7)	0.0175 (6)	-0.0014 (5)	-0.0002 (5)	-0.0011 (5)
O3B	0.0159 (6)	0.0268 (8)	0.0210 (7)	-0.0019 (6)	0.0040 (5)	-0.0030 (6)
O4B	0.0181 (7)	0.0302 (8)	0.0177 (7)	-0.0016 (6)	-0.0019 (5)	-0.0070 (6)
O5B	0.0196 (7)	0.0133 (7)	0.0280 (8)	-0.0002 (5)	0.0034 (6)	-0.0014 (6)
O6B	0.0247 (8)	0.0331 (9)	0.0180 (7)	-0.0001 (6)	0.0047 (6)	-0.0061 (6)
O7B	0.0276 (8)	0.0291 (9)	0.0186 (7)	-0.0036 (6)	-0.0072 (6)	0.0003 (6)
N1B	0.0099 (7)	0.0166 (8)	0.0178 (7)	-0.0002 (6)	-0.0007 (5)	-0.0028 (6)
N2B	0.0091 (6)	0.0160 (8)	0.0202 (8)	-0.0001 (6)	-0.0006 (6)	-0.0044 (6)
N3B	0.0118 (7)	0.0170 (8)	0.0191 (8)	-0.0014 (6)	0.0017 (6)	-0.0022 (6)
N4B	0.0122 (7)	0.0154 (8)	0.0183 (7)	-0.0016 (6)	0.0004 (6)	-0.0021 (6)
C1B	0.0144 (8)	0.0139 (9)	0.0172 (8)	-0.0006 (7)	0.0011 (6)	-0.0004 (7)
C2B	0.0136 (8)	0.0146 (9)	0.0178 (8)	-0.0006 (6)	0.0001 (6)	-0.0016 (7)
C3B	0.0109 (8)	0.0144 (9)	0.0224 (9)	0.0018 (6)	0.0005 (6)	-0.0024 (7)
C4B	0.0153 (8)	0.0188 (10)	0.0248 (10)	-0.0027 (7)	0.0010 (7)	-0.0064 (8)
C5B	0.0158 (9)	0.0186 (10)	0.0255 (10)	-0.0024 (7)	0.0047 (7)	-0.0002 (8)
C6B	0.0187 (9)	0.0143 (9)	0.0183 (9)	-0.0002 (7)	0.0008 (7)	-0.0009 (7)
C7B	0.0210 (9)	0.0131 (9)	0.0193 (9)	-0.0012 (7)	-0.0034 (7)	-0.0003 (7)

Geometric parameters (Å, °)

Ni1—N4	1.8429 (15)	K4B—O3B	3.263 (17)
Ni1—N1	1.8479 (15)	K4B—C2B	3.316 (14)
Ni1—O1	1.8830 (13)	K4B—O6B ^{vi}	3.374 (15)
Ni1—O2	1.9012 (13)	O1—C7	1.277 (2)
K1—O4B ⁱ	2.7086 (15)	O2—C1	1.284 (2)
K1—O1W	2.7392 (18)	O3—C1	1.234 (2)
K1—O3 ⁱⁱ	2.7739 (14)	O4—C2	1.251 (2)
K1—O3	2.8254 (15)	O5—C4	1.424 (2)
K1—O6B ⁱⁱⁱ	2.8588 (16)	O5—C5	1.430 (2)
K1—O4	2.9456 (15)	O6—C6	1.252 (2)
K1—O7B ⁱⁱⁱ	3.1774 (17)	O7—C7	1.232 (2)
K1—O2W	3.274 (2)	O1W—H1W1	0.9152
K1—C1	3.3464 (19)	O1W—H2W1	0.8297
K1—C2	3.4015 (19)	O2W—H1W2	0.8929
K1—K4A ⁱ	3.9153 (7)	O2W—H2W2	0.9306
K1—K4B ⁱ	4.030 (16)	O3W—H1W3	0.8662
K2—O3B	2.7495 (16)	O3W—H2W3	0.9048
K2—O2W	2.7634 (17)	O4WA—H1W4	0.8501
K2—O3	2.8232 (15)	O4WA—H2W4	0.9075
K2—O6 ^{iv}	2.8273 (15)	O5W—H5WC	0.8499
K2—O6B ⁱⁱⁱ	2.8505 (17)	O5W—H5WB	0.8499
K2—O2	2.9013 (14)	O4WB—H3W4	0.8501
K2—N3 ^{iv}	2.9932 (16)	O4WB—H4W4	0.8827
K2—N3B ⁱⁱⁱ	3.0121 (17)	N1—C2	1.322 (2)
K2—C1	3.1184 (19)	N1—N2	1.428 (2)
K2—O2B	3.1903 (14)	N2—C4	1.460 (2)
K2—C1B	3.2025 (19)	N2—C3	1.472 (2)
K2—C6B ⁱⁱⁱ	3.459 (2)	N3—N4	1.427 (2)
K3A—O3W ^v	2.625 (4)	N3—C5	1.460 (3)
K3A—O7	2.755 (4)	N3—C3	1.469 (2)
K3A—O4 ^{iv}	2.761 (4)	N4—C6	1.321 (2)
K3A—O1W ^{iv}	2.779 (5)	C1—C2	1.541 (2)
K3A—N2 ^{iv}	2.954 (5)	C3—H3A	0.9900
K3A—O7B	3.003 (5)	C3—H3B	0.9900
K3A—O4WA	3.047 (4)	C4—H4A	0.9900
K3A—O5W	3.082 (5)	C4—H4B	0.9900
K3A—O4WB	3.171 (9)	C5—H5A	0.9900
K3A—C2 ^{iv}	3.456 (5)	C5—H5B	0.9900
K3A—K4B ^v	4.254 (16)	C6—C7	1.538 (3)
K3B—O7	2.769 (5)	Ni1B—N1B	1.8436 (16)
K3B—O1W ^{iv}	2.783 (5)	Ni1B—N4B	1.8463 (16)
K3B—O7B	2.789 (13)	Ni1B—O2B	1.8922 (14)
K3B—O4WA	2.804 (15)	Ni1B—O1B	1.8975 (14)
K3B—O4 ^{iv}	2.851 (8)	O1B—C7B	1.279 (2)
K3B—O3W ^v	2.868 (15)	O2B—C1B	1.279 (2)
K3B—O4WB	2.979 (15)	O3B—C1B	1.235 (2)

K3B—O5W	2.990 (7)	O4B—C2B	1.247 (2)
K3B—N2 ^{iv}	2.995 (6)	O5B—C4B	1.420 (3)
K3B—C2 ^{iv}	3.492 (6)	O5B—C5B	1.428 (2)
K3B—K4B ^v	4.51 (2)	O6B—C6B	1.249 (2)
K4A—K4B	0.815 (18)	O7B—C7B	1.239 (2)
K4A—O3W	2.696 (2)	N1B—C2B	1.320 (2)
K4A—O3B	2.7100 (16)	N1B—N2B	1.423 (2)
K4A—O7B ^{vi}	2.7633 (17)	N2B—C4B	1.457 (3)
K4A—O4B	2.8223 (17)	N2B—C3B	1.471 (2)
K4A—O2W	2.872 (2)	N3B—N4B	1.429 (2)
K4A—O6 ^{iv}	2.8815 (16)	N3B—C5B	1.459 (3)
K4A—C1B	3.265 (2)	N3B—C3B	1.468 (3)
K4A—C2B	3.311 (2)	N4B—C6B	1.318 (2)
K4A—C7B ^{vi}	3.470 (2)	C1B—C2B	1.539 (3)
K4B—O3W	2.040 (17)	C3B—H3B1	0.9900
K4B—O4B	2.744 (15)	C3B—H3B2	0.9900
K4B—O6 ^{iv}	2.792 (14)	C4B—H4B1	0.9900
K4B—O7 ^{iv}	3.061 (17)	C4B—H4B2	0.9900
K4B—O4WB ^{iv}	3.201 (19)	C5B—H5B1	0.9900
K4B—O7B ^{vi}	3.216 (16)	C5B—H5B2	0.9900
K4B—O5W ^{iv}	3.220 (18)	C6B—C7B	1.536 (3)
N4—Ni1—N1	96.01 (7)	O3W—K4A—C7B ^{vi}	97.56 (5)
N4—Ni1—O1	85.25 (6)	O3B—K4A—C7B ^{vi}	117.90 (5)
N1—Ni1—O1	178.74 (6)	O7B ^{vi} —K4A—C7B ^{vi}	18.91 (4)
N4—Ni1—O2	178.28 (7)	O4B—K4A—C7B ^{vi}	104.56 (5)
N1—Ni1—O2	85.10 (6)	O2W—K4A—C7B ^{vi}	94.93 (5)
O1—Ni1—O2	93.65 (6)	O6 ^{iv} —K4A—C7B ^{vi}	161.44 (5)
O4B ⁱ —K1—O1W	80.90 (5)	C1B—K4A—C7B ^{vi}	131.24 (5)
O4B ⁱ —K1—O3 ⁱⁱ	95.01 (5)	C2B—K4A—C7B ^{vi}	123.72 (5)
O1W—K1—O3 ⁱⁱ	95.56 (5)	O3W—K4A—K2	112.75 (5)
O4B ⁱ —K1—O3	152.85 (5)	O3B—K4A—K2	45.40 (3)
O1W—K1—O3	126.25 (5)	O7B ^{vi} —K4A—K2	120.72 (4)
O3 ⁱⁱ —K1—O3	83.05 (4)	O4B—K4A—K2	98.76 (3)
O4B ⁱ —K1—O6B ⁱⁱⁱ	90.81 (5)	O2W—K4A—K2	45.59 (3)
O1W—K1—O6B ⁱⁱⁱ	122.79 (5)	O6 ^{iv} —K4A—K2	46.85 (3)
O3 ⁱⁱ —K1—O6B ⁱⁱⁱ	141.65 (5)	C1B—K4A—K2	52.60 (3)
O3—K1—O6B ⁱⁱⁱ	75.07 (5)	C2B—K4A—K2	77.78 (3)
O4B ⁱ —K1—O4	148.05 (5)	C7B ^{vi} —K4A—K2	134.15 (4)
O1W—K1—O4	69.17 (4)	O3W—K4A—K1 ^{vii}	117.46 (5)
O3 ⁱⁱ —K1—O4	98.66 (4)	O3B—K4A—K1 ^{vii}	78.47 (4)
O3—K1—O4	58.15 (4)	O7B ^{vi} —K4A—K1 ^{vii}	53.51 (4)
O6B ⁱⁱⁱ —K1—O4	96.01 (4)	O4B—K4A—K1 ^{vii}	43.77 (3)
O4B ⁱ —K1—O7B ⁱⁱⁱ	90.06 (5)	O2W—K4A—K1 ^{vii}	132.91 (4)
O1W—K1—O7B ⁱⁱⁱ	68.77 (5)	O6 ^{iv} —K4A—K1 ^{vii}	136.40 (3)
O3 ⁱⁱ —K1—O7B ⁱⁱⁱ	162.58 (4)	C1B—K4A—K1 ^{vii}	77.55 (4)
O3—K1—O7B ⁱⁱⁱ	99.85 (4)	C2B—K4A—K1 ^{vii}	62.40 (3)
O6B ⁱⁱⁱ —K1—O7B ⁱⁱⁱ	54.64 (4)	C7B ^{vi} —K4A—K1 ^{vii}	61.41 (4)

O4—K1—O7B ⁱⁱⁱ	69.24 (4)	K2—K4A—K1 ^{vii}	123.652 (17)
O4B ⁱ —K1—O2W	85.38 (5)	O3W—K4B—O4B	135.2 (8)
O1W—K1—O2W	162.52 (5)	O3W—K4B—O6 ^{iv}	82.0 (5)
O3 ⁱⁱ —K1—O2W	74.82 (4)	O4B—K4B—O6 ^{iv}	96.4 (4)
O3—K1—O2W	67.91 (4)	O3W—K4B—O7 ^{iv}	62.4 (4)
O6B ⁱⁱⁱ —K1—O2W	67.90 (4)	O4B—K4B—O7 ^{iv}	79.1 (4)
O4—K1—O2W	126.05 (4)	O6 ^{iv} —K4B—O7 ^{iv}	57.1 (3)
O7B ⁱⁱⁱ —K1—O2W	122.28 (4)	O3W—K4B—O4WB ^{iv}	84.3 (6)
O4B ⁱ —K1—C1	156.68 (5)	O4B—K4B—O4WB ^{iv}	56.6 (4)
O1W—K1—C1	113.65 (5)	O6 ^{iv} —K4B—O4WB ^{iv}	117.5 (5)
O3 ⁱⁱ —K1—C1	101.25 (5)	O7 ^{iv} —K4B—O4WB ^{iv}	62.6 (4)
O3—K1—C1	20.96 (4)	O3W—K4B—O7B ^{vi}	122.4 (5)
O6B ⁱⁱⁱ —K1—C1	66.10 (5)	O4B—K4B—O7B ^{vi}	88.6 (4)
O4—K1—C1	45.11 (4)	O6 ^{iv} —K4B—O7B ^{vi}	137.4 (7)
O7B ⁱⁱⁱ —K1—C1	79.38 (4)	O7 ^{iv} —K4B—O7B ^{vi}	162.7 (6)
O2W—K1—C1	82.91 (4)	O3W—K4B—O5W ^{iv}	59.8 (4)
O4B ⁱ —K1—C2	154.85 (5)	O4B—K4B—O5W ^{iv}	85.9 (4)
O1W—K1—C2	87.46 (5)	O6 ^{iv} —K4B—O5W ^{iv}	123.3 (6)
O3 ⁱⁱ —K1—C2	108.32 (4)	O7 ^{iv} —K4B—O5W ^{iv}	68.1 (4)
O3—K1—C2	44.20 (4)	O7B ^{vi} —K4B—O5W ^{iv}	99.1 (4)
O6B ⁱⁱⁱ —K1—C2	76.85 (5)	O3W—K4B—O3B	150.9 (7)
O4—K1—C2	21.21 (4)	O4B—K4B—O3B	54.9 (3)
O7B ⁱⁱⁱ —K1—C2	64.89 (4)	O6 ^{iv} —K4B—O3B	69.2 (3)
O2W—K1—C2	109.27 (4)	O7 ^{iv} —K4B—O3B	102.3 (4)
C1—K1—C2	26.38 (4)	O4WB ^{iv} —K4B—O3B	111.5 (5)
O4B ⁱ —K1—K4A ⁱ	46.13 (3)	O7B ^{vi} —K4B—O3B	79.9 (4)
O1W—K1—K4A ⁱ	73.12 (4)	O5W ^{iv} —K4B—O3B	140.8 (5)
O3 ⁱⁱ —K1—K4A ⁱ	140.17 (3)	O3W—K4B—C2B	133.3 (7)
O3—K1—K4A ⁱ	134.72 (3)	O4B—K4B—C2B	21.17 (12)
O6B ⁱⁱⁱ —K1—K4A ⁱ	61.69 (4)	O6 ^{iv} —K4B—C2B	75.6 (3)
O4—K1—K4A ⁱ	111.64 (3)	O7 ^{iv} —K4B—C2B	71.1 (3)
O7B ⁱⁱⁱ —K1—K4A ⁱ	44.36 (3)	O4WB ^{iv} —K4B—C2B	71.3 (4)
O2W—K1—K4A ⁱ	104.74 (3)	O7B ^{vi} —K4B—C2B	101.2 (5)
C1—K1—K4A ⁱ	118.37 (3)	O5W ^{iv} —K4B—C2B	100.5 (4)
C2—K1—K4A ⁱ	109.12 (3)	O3B—K4B—C2B	42.9 (2)
O4B ⁱ —K1—K4B ⁱ	42.7 (2)	O3W—K4B—O6B ^{vi}	95.5 (5)
O1W—K1—K4B ⁱ	84.2 (3)	O4B—K4B—O6B ^{vi}	80.1 (4)
O3 ⁱⁱ —K1—K4B ⁱ	137.4 (2)	O6 ^{iv} —K4B—O6B ^{vi}	172.1 (7)
O3—K1—K4B ⁱ	130.6 (2)	O7 ^{iv} —K4B—O6B ^{vi}	115.1 (5)
O6B ⁱⁱⁱ —K1—K4B ⁱ	55.6 (2)	O7B ^{vi} —K4B—O6B ^{vi}	49.9 (2)
O4—K1—K4B ⁱ	120.4 (2)	O5W ^{iv} —K4B—O6B ^{vi}	49.7 (2)
O7B ⁱⁱⁱ —K1—K4B ⁱ	51.4 (2)	O3B—K4B—O6B ^{vi}	113.5 (5)
O2W—K1—K4B ⁱ	93.1 (3)	C2B—K4B—O6B ^{vi}	101.2 (4)
C1—K1—K4B ⁱ	117.9 (2)	O3W—K4B—K1 ^{vii}	136.5 (6)
C2—K1—K4B ⁱ	114.2 (2)	O4B—K4B—K1 ^{vii}	42.0 (2)
K4A ⁱ —K1—K4B ⁱ	11.7 (3)	O6 ^{iv} —K4B—K1 ^{vii}	135.1 (5)
O3B—K2—O2W	80.18 (5)	O7 ^{iv} —K4B—K1 ^{vii}	113.5 (4)
O3B—K2—O3	155.07 (4)	O7B ^{vi} —K4B—K1 ^{vii}	50.5 (2)

O2W—K2—O3	75.67 (5)	O5W ^{iv} —K4B—K1 ^{vii}	78.1 (3)
O3B—K2—O6 ^{iv}	76.67 (5)	O3B—K4B—K1 ^{vii}	71.2 (3)
O2W—K2—O6 ^{iv}	78.03 (5)	C2B—K4B—K1 ^{vii}	61.0 (3)
O3—K2—O6 ^{iv}	103.92 (4)	O6B ^{vi} —K4B—K1 ^{vii}	44.33 (19)
O3B—K2—O6B ⁱⁱⁱ	92.95 (5)	C7—O1—Ni1	113.55 (12)
O2W—K2—O6B ⁱⁱⁱ	75.63 (5)	C1—O2—Ni1	112.85 (11)
O3—K2—O6B ⁱⁱⁱ	75.23 (5)	C1—O2—K2	87.38 (10)
O6 ^{iv} —K2—O6B ⁱⁱⁱ	152.98 (4)	Ni1—O2—K2	147.29 (7)
O3B—K2—O2	154.02 (4)	C1—O3—K1 ⁱⁱ	143.74 (13)
O2W—K2—O2	120.44 (5)	C1—O3—K2	91.90 (11)
O3—K2—O2	45.94 (4)	K1 ⁱⁱ —O3—K2	114.99 (5)
O6 ^{iv} —K2—O2	120.63 (4)	C1—O3—K1	104.05 (12)
O6B ⁱⁱⁱ —K2—O2	78.74 (4)	K1 ⁱⁱ —O3—K1	96.95 (4)
O3B—K2—N3 ^{iv}	106.02 (4)	K2—O3—K1	96.36 (5)
O2W—K2—N3 ^{iv}	130.10 (5)	C2—O4—K3A ⁱⁱⁱ	113.48 (14)
O3—K2—N3 ^{iv}	94.35 (4)	C2—O4—K3B ⁱⁱⁱ	110.5 (2)
O6 ^{iv} —K2—N3 ^{iv}	56.79 (4)	C2—O4—K1	100.39 (12)
O6B ⁱⁱⁱ —K2—N3 ^{iv}	149.67 (5)	K3A ⁱⁱⁱ —O4—K1	97.80 (11)
O2—K2—N3 ^{iv}	73.88 (4)	K3B ⁱⁱⁱ —O4—K1	94.6 (2)
O3B—K2—N3B ⁱⁱⁱ	77.60 (5)	C4—O5—C5	109.67 (15)
O2W—K2—N3B ⁱⁱⁱ	125.18 (5)	C6—O6—K4B ⁱⁱⁱ	117.5 (4)
O3—K2—N3B ⁱⁱⁱ	111.66 (4)	C6—O6—K2 ⁱⁱⁱ	113.93 (12)
O6 ^{iv} —K2—N3B ⁱⁱⁱ	141.08 (5)	K4B ⁱⁱⁱ —O6—K2 ⁱⁱⁱ	101.5 (4)
O6B ⁱⁱⁱ —K2—N3B ⁱⁱⁱ	56.45 (4)	C6—O6—K4A ⁱⁱⁱ	127.32 (13)
O2—K2—N3B ⁱⁱⁱ	77.33 (4)	K2 ⁱⁱⁱ —O6—K4A ⁱⁱⁱ	85.11 (4)
N3 ^{iv} —K2—N3B ⁱⁱⁱ	104.14 (5)	C7—O7—K3A	132.83 (17)
O3B—K2—C1	162.38 (5)	C7—O7—K3B	133.15 (17)
O2W—K2—C1	96.17 (6)	C7—O7—K4B ⁱⁱⁱ	109.9 (3)
O3—K2—C1	23.29 (4)	K3A—O7—K4B ⁱⁱⁱ	115.4 (3)
O6 ^{iv} —K2—C1	119.69 (5)	K3B—O7—K4B ⁱⁱⁱ	113.2 (3)
O6B ⁱⁱⁱ —K2—C1	69.48 (5)	K1—O1W—K3A ⁱⁱⁱ	102.44 (11)
O2—K2—C1	24.28 (4)	K1—O1W—K3B ⁱⁱⁱ	100.96 (14)
N3 ^{iv} —K2—C1	89.59 (5)	K1—O1W—H1W1	118.2
N3B ⁱⁱⁱ —K2—C1	90.89 (5)	K3A ⁱⁱⁱ —O1W—H1W1	106.9
O3B—K2—O2B	43.31 (4)	K3B ⁱⁱⁱ —O1W—H1W1	111.9
O2W—K2—O2B	121.24 (5)	K1—O1W—H2W1	114.3
O3—K2—O2B	161.52 (4)	K3A ⁱⁱⁱ —O1W—H2W1	103.7
O6 ^{iv} —K2—O2B	74.95 (4)	K3B ⁱⁱⁱ —O1W—H2W1	99.8
O6B ⁱⁱⁱ —K2—O2B	114.34 (4)	H1W1—O1W—H2W1	109.7
O2—K2—O2B	118.26 (4)	K2—O2W—K4A	86.48 (5)
N3 ^{iv} —K2—O2B	69.34 (4)	K2—O2W—K1	87.99 (5)
N3B ⁱⁱⁱ —K2—O2B	66.28 (4)	K4A—O2W—K1	168.91 (7)
C1—K2—O2B	142.47 (4)	K2—O2W—H1W2	131.5
O3B—K2—C1B	22.33 (4)	K4A—O2W—H1W2	90.2
O2W—K2—C1B	98.18 (6)	K1—O2W—H1W2	86.3
O3—K2—C1B	170.77 (5)	K2—O2W—H2W2	118.8
O6 ^{iv} —K2—C1B	67.72 (5)	K4A—O2W—H2W2	97.9
O6B ⁱⁱⁱ —K2—C1B	110.25 (5)	K1—O2W—H2W2	93.2

O2—K2—C1B	141.22 (5)	H1W2—O2W—H2W2	109.6
N3 ^{iv} —K2—C1B	84.33 (5)	K4B—O3W—K3A ^{viii}	131.1 (5)
N3B ⁱⁱⁱ —K2—C1B	77.48 (5)	K3A ^{viii} —O3W—K4A	119.59 (11)
C1—K2—C1B	165.07 (5)	K4B—O3W—K3B ^{viii}	132.7 (5)
O2B—K2—C1B	23.09 (4)	K4A—O3W—K3B ^{viii}	121.30 (15)
O3B—K2—C6B ⁱⁱⁱ	99.54 (5)	K4B—O3W—H1W3	77.2
O2W—K2—C6B ⁱⁱⁱ	95.40 (5)	K3A ^{viii} —O3W—H1W3	111.5
O3—K2—C6B ⁱⁱⁱ	76.89 (4)	K4A—O3W—H1W3	83.7
O6 ^{iv} —K2—C6B ⁱⁱⁱ	172.82 (5)	K3B ^{viii} —O3W—H1W3	110.2
O6B ⁱⁱⁱ —K2—C6B ⁱⁱⁱ	20.01 (4)	K4B—O3W—H2W3	100.3
O2—K2—C6B ⁱⁱⁱ	65.14 (4)	K3A ^{viii} —O3W—H2W3	121.6
N3 ^{iv} —K2—C6B ⁱⁱⁱ	130.38 (5)	K4A—O3W—H2W3	108.4
N3B ⁱⁱⁱ —K2—C6B ⁱⁱⁱ	41.68 (4)	K3B ^{viii} —O3W—H2W3	121.0
C1—K2—C6B ⁱⁱⁱ	63.46 (5)	H1W3—O3W—H2W3	104.6
O2B—K2—C6B ⁱⁱⁱ	106.60 (4)	K3B—O4WA—H1W4	56.6
C1B—K2—C6B ⁱⁱⁱ	110.89 (5)	K3A—O4WA—H1W4	58.4
O3W ^v —K3A—O7	74.60 (13)	K3B—O4WA—H2W4	109.2
O3W ^v —K3A—O4 ^{iv}	93.60 (12)	K3A—O4WA—H2W4	108.2
O7—K3A—O4 ^{iv}	149.9 (2)	H1W4—O4WA—H2W4	109.5
O3W ^v —K3A—O1W ^{iv}	90.25 (12)	K3B—O5W—K4B ⁱⁱⁱ	103.2 (3)
O7—K3A—O1W ^{iv}	134.80 (18)	K3B—O5W—H5WC	151.0
O4 ^{iv} —K3A—O1W ^{iv}	71.35 (10)	K3A—O5W—H5WC	155.6
O3W ^v —K3A—N2 ^{iv}	102.80 (15)	K4B ⁱⁱⁱ —O5W—H5WC	78.9
O7—K3A—N2 ^{iv}	97.36 (14)	K3A—O5W—H5WB	48.8
O4 ^{iv} —K3A—N2 ^{iv}	57.62 (10)	K4B ⁱⁱⁱ —O5W—H5WB	96.7
O1W ^{iv} —K3A—N2 ^{iv}	127.71 (15)	H5WC—O5W—H5WB	106.8
O3W ^v —K3A—O7B	159.98 (16)	K3B—O4WB—K4B ⁱⁱⁱ	103.9 (5)
O7—K3A—O7B	123.54 (12)	K3A—O4WB—K4B ⁱⁱⁱ	101.1 (4)
O4 ^{iv} —K3A—O7B	74.25 (12)	K3B—O4WB—H3W4	102.6
O1W ^{iv} —K3A—O7B	70.95 (12)	K3A—O4WB—H3W4	103.2
N2 ^{iv} —K3A—O7B	84.34 (12)	K4B ⁱⁱⁱ —O4WB—H3W4	66.6
O3W ^v —K3A—O4WA	137.03 (17)	K3B—O4WB—H4W4	139.2
O7—K3A—O4WA	70.82 (9)	K3A—O4WB—H4W4	136.3
O4 ^{iv} —K3A—O4WA	128.73 (16)	K4B ⁱⁱⁱ —O4WB—H4W4	61.3
O1W ^{iv} —K3A—O4WA	96.31 (15)	H3W4—O4WB—H4W4	104.5
N2 ^{iv} —K3A—O4WA	106.17 (12)	C2—N1—N2	116.68 (15)
O7B—K3A—O4WA	55.03 (8)	C2—N1—Ni1	116.46 (12)
O3W ^v —K3A—O5W	91.65 (14)	N2—N1—Ni1	126.77 (12)
O7—K3A—O5W	73.88 (12)	N1—N2—C4	110.56 (14)
O4 ^{iv} —K3A—O5W	135.11 (18)	N1—N2—C3	109.18 (14)
O1W ^{iv} —K3A—O5W	64.08 (12)	C4—N2—C3	108.78 (15)
N2 ^{iv} —K3A—O5W	160.64 (13)	N1—N2—K3A ⁱⁱⁱ	107.01 (13)
O7B—K3A—O5W	86.35 (13)	C4—N2—K3A ⁱⁱⁱ	107.21 (13)
O4WA—K3A—O5W	54.85 (10)	C3—N2—K3A ⁱⁱⁱ	114.08 (12)
O3W ^v —K3A—O4WB	115.5 (2)	N1—N2—K3B ⁱⁱⁱ	105.80 (15)
O7—K3A—O4WB	66.17 (18)	C4—N2—K3B ⁱⁱⁱ	112.0 (3)
O4 ^{iv} —K3A—O4WB	142.0 (2)	C3—N2—K3B ⁱⁱⁱ	110.5 (3)
O1W ^{iv} —K3A—O4WB	84.0 (2)	N4—N3—C5	110.76 (15)

N2 ^{iv} —K3A—O4WB	130.4 (2)	N4—N3—C3	109.68 (14)
O7B—K3A—O4WB	70.46 (19)	C5—N3—C3	108.53 (15)
O3W ^v —K3A—C2 ^{iv}	106.96 (13)	N4—N3—K2 ⁱⁱⁱ	108.26 (10)
O7—K3A—C2 ^{iv}	139.22 (17)	C5—N3—K2 ⁱⁱⁱ	115.07 (11)
O4 ^{iv} —K3A—C2 ^{iv}	19.39 (5)	C3—N3—K2 ⁱⁱⁱ	104.28 (10)
O1W ^{iv} —K3A—C2 ^{iv}	85.77 (11)	C6—N4—N3	117.21 (15)
N2 ^{iv} —K3A—C2 ^{iv}	41.94 (7)	C6—N4—Ni1	116.14 (12)
O7B—K3A—C2 ^{iv}	65.93 (10)	N3—N4—Ni1	126.28 (12)
O4WA—K3A—C2 ^{iv}	115.84 (13)	O3—C1—O2	125.22 (17)
O5W—K3A—C2 ^{iv}	144.82 (17)	O3—C1—C2	119.59 (17)
O3W ^v —K3A—K4B ^v	21.2 (3)	O2—C1—C2	115.18 (15)
O7—K3A—K4B ^v	95.2 (3)	O3—C1—K2	64.80 (10)
O4 ^{iv} —K3A—K4B ^v	73.1 (3)	O2—C1—K2	68.34 (10)
O1W ^{iv} —K3A—K4B ^v	78.9 (2)	C2—C1—K2	148.69 (12)
N2 ^{iv} —K3A—K4B ^v	95.5 (2)	O3—C1—K1	54.99 (10)
O7B—K3A—K4B ^v	141.0 (3)	O2—C1—K1	140.18 (13)
O4WA—K3A—K4B ^v	155.3 (3)	C2—C1—K1	78.81 (10)
O5W—K3A—K4B ^v	102.4 (2)	K2—C1—K1	81.17 (4)
C2 ^{iv} —K3A—K4B ^v	88.2 (3)	O4—C2—N1	128.58 (17)
O3W ^v —K3A—K1 ^{iv}	112.95 (11)	O4—C2—C1	121.66 (16)
O7—K3A—K1 ^{iv}	167.00 (15)	N1—C2—C1	109.76 (15)
O4 ^{iv} —K3A—K1 ^{iv}	42.72 (7)	O4—C2—K1	58.41 (10)
O1W ^{iv} —K3A—K1 ^{iv}	38.45 (7)	N1—C2—K1	144.78 (13)
N2 ^{iv} —K3A—K1 ^{iv}	91.31 (11)	C1—C2—K1	74.82 (10)
O7B—K3A—K1 ^{iv}	47.58 (7)	O4—C2—K3A ⁱⁱⁱ	47.13 (11)
O4WA—K3A—K1 ^{iv}	97.50 (12)	N1—C2—K3A ⁱⁱⁱ	87.14 (12)
O5W—K3A—K1 ^{iv}	94.88 (13)	C1—C2—K3A ⁱⁱⁱ	150.62 (14)
C2 ^{iv} —K3A—K1 ^{iv}	50.58 (6)	K1—C2—K3A ⁱⁱⁱ	77.71 (9)
K4B ^v —K3A—K1 ^{iv}	93.6 (2)	O4—C2—K3B ⁱⁱⁱ	49.9 (2)
O3W ^v —K3A—H1W4	152.5	N1—C2—K3B ⁱⁱⁱ	86.22 (14)
O7—K3A—H1W4	82.7	C1—C2—K3B ⁱⁱⁱ	147.7 (2)
O4 ^{iv} —K3A—H1W4	113.6	K1—C2—K3B ⁱⁱⁱ	76.31 (12)
O1W ^{iv} —K3A—H1W4	95.0	N3—C3—N2	114.33 (15)
N2 ^{iv} —K3A—H1W4	95.2	N3—C3—H3A	108.7
O7B—K3A—H1W4	41.2	N2—C3—H3A	108.7
O4WA—K3A—H1W4	15.6	N3—C3—H3B	108.7
O5W—K3A—H1W4	66.9	N2—C3—H3B	108.7
C2 ^{iv} —K3A—H1W4	100.3	H3A—C3—H3B	107.6
K4B ^v —K3A—H1W4	169.3	O5—C4—N2	112.75 (15)
K1 ^{iv} —K3A—H1W4	86.9	O5—C4—H4A	109.0
O3W ^v —K3A—H5WB	105.0	N2—C4—H4A	109.0
O7—K3A—H5WB	73.7	O5—C4—H4B	109.0
O4 ^{iv} —K3A—H5WB	136.4	N2—C4—H4B	109.0
O1W ^{iv} —K3A—H5WB	69.6	H4A—C4—H4B	107.8
N2 ^{iv} —K3A—H5WB	147.0	O5—C5—N3	113.17 (15)
O7B—K3A—H5WB	75.4	O5—C5—H5A	108.9
O4WA—K3A—H5WB	40.9	N3—C5—H5A	108.9
O5W—K3A—H5WB	14.2	O5—C5—H5B	108.9

C2 ^{iv} —K3A—H5WB	139.3	N3—C5—H5B	108.9
K4B ^v —K3A—H5WB	116.6	H5A—C5—H5B	107.8
K1 ^{iv} —K3A—H5WB	93.8	O6—C6—N4	128.70 (17)
H1W4—K3A—H5WB	52.7	O6—C6—C7	121.70 (17)
O7—K3B—O1W ^{iv}	133.9 (2)	N4—C6—C7	109.59 (16)
O7—K3B—O7B	131.8 (5)	O6—C6—K2 ⁱⁱⁱ	47.14 (10)
O1W ^{iv} —K3B—O7B	74.2 (2)	N4—C6—K2 ⁱⁱⁱ	86.88 (11)
O7—K3B—O4WA	74.4 (2)	C7—C6—K2 ⁱⁱⁱ	152.63 (13)
O1W ^{iv} —K3B—O4WA	102.1 (4)	O7—C7—O1	125.28 (18)
O7B—K3B—O4WA	60.0 (3)	O7—C7—C6	119.73 (17)
O7—K3B—O4 ^{iv}	142.7 (5)	O1—C7—C6	114.98 (16)
O1W ^{iv} —K3B—O4 ^{iv}	69.98 (15)	N1B—Ni1B—N4B	95.98 (7)
O7B—K3B—O4 ^{iv}	76.28 (15)	N1B—Ni1B—O2B	85.01 (6)
O4WA—K3B—O4 ^{iv}	135.7 (5)	N4B—Ni1B—O2B	177.89 (7)
O7—K3B—O3W ^v	70.7 (3)	N1B—Ni1B—O1B	178.86 (7)
O1W ^{iv} —K3B—O3W ^v	85.4 (3)	N4B—Ni1B—O1B	85.08 (7)
O7B—K3B—O3W ^v	156.9 (3)	O2B—Ni1B—O1B	93.94 (6)
O4WA—K3B—O3W ^v	137.15 (18)	C7B—O1B—Ni1B	112.61 (12)
O4 ^{iv} —K3B—O3W ^v	86.7 (4)	C1B—O2B—Ni1B	112.81 (12)
O7—K3B—O4WB	68.8 (2)	C1B—O2B—K2	78.99 (10)
O1W ^{iv} —K3B—O4WB	87.6 (3)	Ni1B—O2B—K2	150.81 (7)
O7B—K3B—O4WB	76.2 (4)	C1B—O3B—K4A	105.49 (12)
O4 ^{iv} —K3B—O4WB	148.3 (4)	C1B—O3B—K2	99.92 (12)
O3W ^v —K3B—O4WB	114.3 (3)	K4A—O3B—K2	90.02 (5)
O7—K3B—O5W	75.20 (14)	C1B—O3B—K4B	93.9 (3)
O1W ^{iv} —K3B—O5W	65.33 (14)	K2—O3B—K4B	92.3 (3)
O7B—K3B—O5W	92.1 (4)	C2B—O4B—K1 ^{vii}	142.90 (14)
O4WA—K3B—O5W	58.2 (2)	C2B—O4B—K4B	106.2 (3)
O4 ^{iv} —K3B—O5W	135.30 (19)	K1 ^{vii} —O4B—K4B	95.3 (3)
O3W ^v —K3B—O5W	89.0 (2)	C2B—O4B—K4A	101.84 (12)
O7—K3B—N2 ^{iv}	96.10 (18)	K1 ^{vii} —O4B—K4A	90.10 (5)
O1W ^{iv} —K3B—N2 ^{iv}	125.9 (2)	C4B—O5B—C5B	109.77 (15)
O7B—K3B—N2 ^{iv}	87.4 (2)	C6B—O6B—K2 ^{iv}	108.65 (13)
O4WA—K3B—N2 ^{iv}	111.6 (4)	C6B—O6B—K1 ^{iv}	119.80 (13)
O4 ^{iv} —K3B—N2 ^{iv}	56.26 (13)	K2 ^{iv} —O6B—K1 ^{iv}	95.01 (5)
O3W ^v —K3B—N2 ^{iv}	96.2 (4)	C6B—O6B—K4B ^{ix}	98.8 (3)
O5W—K3B—N2 ^{iv}	167.8 (4)	K2 ^{iv} —O6B—K4B ^{ix}	150.5 (3)
O7—K3B—C2 ^{iv}	136.7 (3)	K1 ^{iv} —O6B—K4B ^{ix}	80.1 (3)
O1W ^{iv} —K3B—C2 ^{iv}	85.01 (14)	C7B—O7B—K4A ^{ix}	114.78 (13)
O7B—K3B—C2 ^{iv}	67.41 (13)	C7B—O7B—K3B	120.48 (16)
O4WA—K3B—C2 ^{iv}	121.9 (4)	K4A ^{ix} —O7B—K3B	123.07 (13)
O4 ^{iv} —K3B—C2 ^{iv}	19.60 (5)	C7B—O7B—K3A	120.27 (16)
O3W ^v —K3B—C2 ^{iv}	100.7 (4)	K4A ^{ix} —O7B—K3A	123.98 (10)
O5W—K3B—C2 ^{iv}	148.1 (2)	C7B—O7B—K1 ^{iv}	110.33 (13)
N2 ^{iv} —K3B—C2 ^{iv}	41.43 (8)	K4A ^{ix} —O7B—K1 ^{iv}	82.14 (4)
O7—K3B—K1 ^{iv}	172.3 (2)	K3B—O7B—K1 ^{iv}	90.9 (2)
O1W ^{iv} —K3B—K1 ^{iv}	39.15 (8)	K3A—O7B—K1 ^{iv}	88.18 (8)
O7B—K3B—K1 ^{iv}	48.23 (8)	C7B—O7B—K4B ^{ix}	104.3 (3)

O4WA—K3B—K1 ^{iv}	102.6 (3)	K3B—O7B—K4B ^{ix}	134.9 (3)
O4 ^{iv} —K3B—K1 ^{iv}	43.58 (8)	K3A—O7B—K4B ^{ix}	135.4 (3)
O3W ^v —K3B—K1 ^{iv}	108.7 (3)	K1 ^{iv} —O7B—K4B ^{ix}	78.1 (3)
O5W—K3B—K1 ^{iv}	97.18 (18)	C2B—N1B—N2B	116.70 (16)
N2 ^{iv} —K3B—K1 ^{iv}	91.58 (11)	C2B—N1B—Ni1B	116.37 (13)
C2 ^{iv} —K3B—K1 ^{iv}	50.88 (7)	N2B—N1B—Ni1B	126.63 (12)
O7—K3B—K4B ^v	89.6 (4)	N1B—N2B—C4B	110.68 (15)
O1W ^{iv} —K3B—K4B ^v	74.3 (4)	N1B—N2B—C3B	109.51 (14)
O7B—K3B—K4B ^v	138.6 (3)	C4B—N2B—C3B	108.53 (15)
O4WA—K3B—K4B ^v	154.1 (3)	N4B—N3B—C5B	110.58 (15)
O4 ^{iv} —K3B—K4B ^v	68.1 (4)	N4B—N3B—C3B	109.35 (15)
O3W ^v —K3B—K4B ^v	19.4 (3)	C5B—N3B—C3B	108.84 (16)
O5W—K3B—K4B ^v	98.4 (3)	N4B—N3B—K2 ^{iv}	104.06 (10)
N2 ^{iv} —K3B—K4B ^v	89.9 (4)	C5B—N3B—K2 ^{iv}	123.74 (11)
C2 ^{iv} —K3B—K4B ^v	83.7 (4)	C3B—N3B—K2 ^{iv}	99.25 (11)
K1 ^{iv} —K3B—K4B ^v	90.6 (3)	C6B—N4B—N3B	116.71 (16)
O7—K3B—H1W4	87.3	C6B—N4B—Ni1B	116.48 (13)
O1W ^{iv} —K3B—H1W4	101.2	N3B—N4B—Ni1B	126.38 (12)
O7B—K3B—H1W4	45.0	O3B—C1B—O2B	124.92 (18)
O4WA—K3B—H1W4	16.9	O3B—C1B—C2B	119.87 (17)
O4 ^{iv} —K3B—H1W4	119.2	O2B—C1B—C2B	115.19 (16)
O3W ^v —K3B—H1W4	154.0	O3B—C1B—K2	57.75 (10)
O5W—K3B—H1W4	71.5	O2B—C1B—K2	77.92 (10)
N2 ^{iv} —K3B—H1W4	100.0	C2B—C1B—K2	142.67 (13)
C2 ^{iv} —K3B—H1W4	105.0	O3B—C1B—K4A	53.12 (10)
K1 ^{iv} —K3B—H1W4	91.2	O2B—C1B—K4A	143.73 (13)
K4B ^v —K3B—H1W4	170.0	C2B—C1B—K4A	78.17 (10)
O7—K3B—H5WB	75.7	K2—C1B—K4A	73.31 (4)
O1W ^{iv} —K3B—H5WB	71.6	O4B—C2B—N1B	128.98 (18)
O7B—K3B—H5WB	81.8	O4B—C2B—C1B	121.58 (17)
O4WA—K3B—H5WB	44.5	N1B—C2B—C1B	109.44 (16)
O4 ^{iv} —K3B—H5WB	139.6	O4B—C2B—K4A	56.53 (11)
O3W ^v —K3B—H5WB	102.2	N1B—C2B—K4A	147.78 (14)
O5W—K3B—H5WB	14.0	C1B—C2B—K4A	74.78 (10)
N2 ^{iv} —K3B—H5WB	155.9	O4B—C2B—K4B	52.6 (3)
C2 ^{iv} —K3B—H5WB	145.6	N1B—C2B—K4B	136.4 (3)
K1 ^{iv} —K3B—H5WB	97.1	C1B—C2B—K4B	86.4 (3)
K4B ^v —K3B—H5WB	112.4	N3B—C3B—N2B	114.29 (15)
H1W4—K3B—H5WB	57.6	N3B—C3B—H3B1	108.7
O3W—K4A—O3B	144.09 (5)	N2B—C3B—H3B1	108.7
O3W—K4A—O7B ^{vi}	116.45 (5)	N3B—C3B—H3B2	108.7
O3B—K4A—O7B ^{vi}	99.01 (5)	N2B—C3B—H3B2	108.7
O3W—K4A—O4B	106.80 (6)	H3B1—C3B—H3B2	107.6
O3B—K4A—O4B	60.94 (4)	O5B—C4B—N2B	113.06 (16)
O7B ^{vi} —K4A—O4B	96.81 (5)	O5B—C4B—H4B1	109.0
O3W—K4A—O2W	104.92 (6)	N2B—C4B—H4B1	109.0
O3B—K4A—O2W	78.94 (5)	O5B—C4B—H4B2	109.0
O7B ^{vi} —K4A—O2W	90.47 (5)	N2B—C4B—H4B2	109.0

O4B—K4A—O2W	139.85 (5)	H4B1—C4B—H4B2	107.8
O3W—K4A—O6 ^{iv}	70.42 (5)	O5B—C5B—N3B	112.88 (16)
O3B—K4A—O6 ^{iv}	76.37 (5)	O5B—C5B—H5B1	109.0
O7B ^{vi} —K4A—O6 ^{iv}	165.73 (5)	N3B—C5B—H5B1	109.0
O4B—K4A—O6 ^{iv}	92.69 (4)	O5B—C5B—H5B2	109.0
O2W—K4A—O6 ^{iv}	75.44 (5)	N3B—C5B—H5B2	109.0
O3W—K4A—C1B	125.55 (5)	H5B1—C5B—H5B2	107.8
O3B—K4A—C1B	21.38 (4)	O6B—C6B—N4B	129.67 (19)
O7B ^{vi} —K4A—C1B	113.59 (5)	O6B—C6B—C7B	121.00 (18)
O4B—K4A—C1B	46.47 (4)	N4B—C6B—C7B	109.32 (16)
O2W—K4A—C1B	94.60 (5)	O6B—C6B—K2 ^{iv}	51.34 (11)
O6 ^{iv} —K4A—C1B	66.25 (5)	N4B—C6B—K2 ^{iv}	86.86 (12)
O3W—K4A—C2B	110.36 (5)	C7B—C6B—K2 ^{iv}	146.62 (13)
O3B—K4A—C2B	45.75 (5)	O7B—C7B—O1B	124.88 (19)
O7B ^{vi} —K4A—C2B	112.13 (5)	O7B—C7B—C6B	119.26 (18)
O4B—K4A—C2B	21.63 (4)	O1B—C7B—C6B	115.85 (17)
O2W—K4A—C2B	121.52 (5)	O7B—C7B—K4A ^{ix}	46.30 (11)
O6 ^{iv} —K4A—C2B	74.55 (5)	O1B—C7B—K4A ^{ix}	132.10 (13)
C1B—K4A—C2B	27.05 (5)	C6B—C7B—K4A ^{ix}	91.96 (11)
O3B—K4A—K4B—O3W	-136.2 (12)	K2—C1—C2—N1	92.1 (2)
O7B ^{vi} —K4A—K4B—O3W	97.0 (17)	K1—C1—C2—N1	143.32 (14)
O4B—K4A—K4B—O3W	-172.4 (19)	O3—C1—C2—K1	38.29 (16)
O2W—K4A—K4B—O3W	-23 (2)	O2—C1—C2—K1	-140.55 (16)
O6 ^{iv} —K4A—K4B—O3W	-75.9 (16)	K2—C1—C2—K1	-51.2 (2)
C1B—K4A—K4B—O3W	-135.8 (14)	O3—C1—C2—K3A ⁱⁱⁱ	59.6 (3)
C2B—K4A—K4B—O3W	-151.6 (18)	O2—C1—C2—K3A ⁱⁱⁱ	-119.2 (2)
C7B ^{vi} —K4A—K4B—O3W	85.3 (17)	K2—C1—C2—K3A ⁱⁱⁱ	-29.9 (4)
K2—K4A—K4B—O3W	-80.6 (18)	K1—C1—C2—K3A ⁱⁱⁱ	21.3 (2)
K1 ^{vii} —K4A—K4B—O3W	146.5 (16)	O3—C1—C2—K3B ⁱⁱⁱ	65.7 (5)
O3W—K4A—K4B—O4B	172.4 (19)	O2—C1—C2—K3B ⁱⁱⁱ	-113.1 (4)
O3B—K4A—K4B—O4B	36.2 (10)	K2—C1—C2—K3B ⁱⁱⁱ	-23.8 (5)
O7B ^{vi} —K4A—K4B—O4B	-90.7 (6)	K1—C1—C2—K3B ⁱⁱⁱ	27.4 (4)
O2W—K4A—K4B—O4B	149.0 (7)	N4—N3—C3—N2	-69.27 (19)
O6 ^{iv} —K4A—K4B—O4B	96.5 (4)	C5—N3—C3—N2	51.86 (19)
C1B—K4A—K4B—O4B	36.6 (5)	K2 ⁱⁱⁱ —N3—C3—N2	174.98 (11)
C2B—K4A—K4B—O4B	20.78 (14)	N1—N2—C3—N3	68.50 (19)
C7B ^{vi} —K4A—K4B—O4B	-102.3 (3)	C4—N2—C3—N3	-52.22 (19)
K2—K4A—K4B—O4B	91.7 (7)	K3A ⁱⁱⁱ —N2—C3—N3	-171.85 (14)
K1 ^{vii} —K4A—K4B—O4B	-41.1 (3)	K3B ⁱⁱⁱ —N2—C3—N3	-175.6 (3)
O3W—K4A—K4B—O6 ^{iv}	75.9 (16)	C5—O5—C4—N2	-59.19 (19)
O3B—K4A—K4B—O6 ^{iv}	-60.3 (10)	N1—N2—C4—O5	-64.63 (19)
O7B ^{vi} —K4A—K4B—O6 ^{iv}	172.8 (3)	C3—N2—C4—O5	55.24 (19)
O4B—K4A—K4B—O6 ^{iv}	-96.5 (4)	K3A ⁱⁱⁱ —N2—C4—O5	179.06 (14)
O2W—K4A—K4B—O6 ^{iv}	52.5 (11)	K3B ⁱⁱⁱ —N2—C4—O5	177.64 (17)
C1B—K4A—K4B—O6 ^{iv}	-59.9 (5)	C4—O5—C5—N3	59.2 (2)
C2B—K4A—K4B—O6 ^{iv}	-75.7 (3)	N4—N3—C5—O5	65.7 (2)
C7B ^{vi} —K4A—K4B—O6 ^{iv}	161.16 (10)	C3—N3—C5—O5	-54.8 (2)

K2—K4A—K4B—O6 ^{iv}	-4.8 (9)	K2 ⁱⁱⁱ —N3—C5—O5	-171.15 (11)
K1 ^{vii} —K4A—K4B—O6 ^{iv}	-137.6 (2)	K4B ⁱⁱⁱ —O6—C6—N4	151.4 (4)
O3W—K4A—K4B—O7 ^{iv}	103 (3)	K2 ⁱⁱⁱ —O6—C6—N4	33.0 (3)
O3B—K4A—K4B—O7 ^{iv}	-33 (3)	K4A ⁱⁱⁱ —O6—C6—N4	135.77 (18)
O7B ^{vi} —K4A—K4B—O7 ^{iv}	-159.8 (13)	K4B ⁱⁱⁱ —O6—C6—C7	-29.9 (5)
O4B—K4A—K4B—O7 ^{iv}	-69.2 (17)	K2 ⁱⁱⁱ —O6—C6—C7	-148.30 (14)
O2W—K4A—K4B—O7 ^{iv}	80 (2)	K4A ⁱⁱⁱ —O6—C6—C7	-45.5 (3)
O6 ^{iv} —K4A—K4B—O7 ^{iv}	27.3 (15)	K4B ⁱⁱⁱ —O6—C6—K2 ⁱⁱⁱ	118.4 (4)
C1B—K4A—K4B—O7 ^{iv}	-33 (2)	K4A ⁱⁱⁱ —O6—C6—K2 ⁱⁱⁱ	102.78 (16)
C2B—K4A—K4B—O7 ^{iv}	-48.4 (17)	N3—N4—C6—O6	-0.5 (3)
C7B ^{vi} —K4A—K4B—O7 ^{iv}	-171.5 (16)	Ni1—N4—C6—O6	-173.90 (17)
K2—K4A—K4B—O7 ^{iv}	23 (2)	N3—N4—C6—C7	-179.34 (15)
K1 ^{vii} —K4A—K4B—O7 ^{iv}	-110.2 (18)	Ni1—N4—C6—C7	7.3 (2)
O3W—K4A—K4B—O4WB ^{iv}	-155 (3)	N3—N4—C6—K2 ⁱⁱⁱ	23.05 (15)
O3B—K4A—K4B—O4WB ^{iv}	68.6 (18)	Ni1—N4—C6—K2 ⁱⁱⁱ	-150.34 (10)
O7B ^{vi} —K4A—K4B—O4WB ^{iv}	-58.3 (17)	K3A—O7—C7—O1	12.2 (4)
O4B—K4A—K4B—O4WB ^{iv}	32.4 (12)	K3B—O7—C7—O1	4.8 (6)
O2W—K4A—K4B—O4WB ^{iv}	-178.6 (5)	K4B ⁱⁱⁱ —O7—C7—O1	-151.0 (4)
O6 ^{iv} —K4A—K4B—O4WB ^{iv}	128.9 (15)	K3A—O7—C7—C6	-167.15 (17)
C1B—K4A—K4B—O4WB ^{iv}	69.0 (15)	K3B—O7—C7—C6	-174.6 (5)
C2B—K4A—K4B—O4WB ^{iv}	53.2 (13)	K4B ⁱⁱⁱ —O7—C7—C6	29.6 (4)
C7B ^{vi} —K4A—K4B—O4WB ^{iv}	-69.9 (14)	Ni1—O1—C7—O7	179.31 (18)
K2—K4A—K4B—O4WB ^{iv}	124.2 (12)	Ni1—O1—C7—C6	-1.3 (2)
K1 ^{vii} —K4A—K4B—O4WB ^{iv}	-8.7 (14)	O6—C6—C7—O7	-3.3 (3)
O3W—K4A—K4B—O7B ^{vi}	-97.0 (17)	N4—C6—C7—O7	175.6 (2)
O3B—K4A—K4B—O7B ^{vi}	126.8 (13)	K2 ⁱⁱⁱ —C6—C7—O7	-60.2 (4)
O4B—K4A—K4B—O7B ^{vi}	90.7 (6)	O6—C6—C7—O1	177.25 (18)
O2W—K4A—K4B—O7B ^{vi}	-120.4 (13)	N4—C6—C7—O1	-3.8 (2)
O6 ^{iv} —K4A—K4B—O7B ^{vi}	-172.8 (3)	K2 ⁱⁱⁱ —C6—C7—O1	120.4 (2)
C1B—K4A—K4B—O7B ^{vi}	127.3 (8)	N4B—Ni1B—O1B—C7B	6.27 (14)
C2B—K4A—K4B—O7B ^{vi}	111.5 (5)	O2B—Ni1B—O1B—C7B	-171.86 (14)
C7B ^{vi} —K4A—K4B—O7B ^{vi}	-11.7 (3)	N1B—Ni1B—O2B—C1B	9.98 (14)
K2—K4A—K4B—O7B ^{vi}	-177.6 (12)	O1B—Ni1B—O2B—C1B	-169.56 (14)
K1 ^{vii} —K4A—K4B—O7B ^{vi}	49.6 (5)	N1B—Ni1B—O2B—K2	-99.18 (14)
O3W—K4A—K4B—O5W ^{iv}	-110 (3)	O1B—Ni1B—O2B—K2	81.28 (13)
O3B—K4A—K4B—O5W ^{iv}	113.5 (19)	N4B—Ni1B—N1B—C2B	172.86 (15)
O7B ^{vi} —K4A—K4B—O5W ^{iv}	-13 (3)	O2B—Ni1B—N1B—C2B	-9.00 (15)
O4B—K4A—K4B—O5W ^{iv}	77 (2)	N4B—Ni1B—N1B—N2B	-0.66 (16)
O2W—K4A—K4B—O5W ^{iv}	-133.8 (18)	O2B—Ni1B—N1B—N2B	177.48 (16)
O6 ^{iv} —K4A—K4B—O5W ^{iv}	174 (2)	C2B—N1B—N2B—C4B	-83.8 (2)
C1B—K4A—K4B—O5W ^{iv}	114 (2)	Ni1B—N1B—N2B—C4B	89.67 (18)
C2B—K4A—K4B—O5W ^{iv}	98 (2)	C2B—N1B—N2B—C3B	156.56 (17)
C7B ^{vi} —K4A—K4B—O5W ^{iv}	-25 (2)	Ni1B—N1B—N2B—C3B	-29.9 (2)
K2—K4A—K4B—O5W ^{iv}	169.0 (15)	C5B—N3B—N4B—C6B	98.5 (2)
K1 ^{vii} —K4A—K4B—O5W ^{iv}	36 (2)	C3B—N3B—N4B—C6B	-141.70 (17)
O3W—K4A—K4B—O3B	136.2 (12)	K2 ^{iv} —N3B—N4B—C6B	-36.42 (18)
O7B ^{vi} —K4A—K4B—O3B	-126.8 (13)	C5B—N3B—N4B—Ni1B	-89.30 (18)
O4B—K4A—K4B—O3B	-36.2 (10)	C3B—N3B—N4B—Ni1B	30.5 (2)

O2W—K4A—K4B—O3B	112.8 (14)	K2 ^{iv} —N3B—N4B—Ni1B	135.82 (11)
O6 ^{iv} —K4A—K4B—O3B	60.3 (10)	N1B—Ni1B—N4B—C6B	172.60 (15)
C1B—K4A—K4B—O3B	0.4 (5)	O1B—Ni1B—N4B—C6B	-7.82 (15)
C2B—K4A—K4B—O3B	-15.4 (9)	N1B—Ni1B—N4B—N3B	0.35 (17)
C7B ^{vi} —K4A—K4B—O3B	-138.5 (10)	O1B—Ni1B—N4B—N3B	179.92 (16)
K2—K4A—K4B—O3B	55.6 (6)	K4A—O3B—C1B—O2B	134.87 (18)
K1 ^{vii} —K4A—K4B—O3B	-77.3 (8)	K2—O3B—C1B—O2B	42.1 (2)
O3W—K4A—K4B—C2B	151.6 (18)	K4B—O3B—C1B—O2B	135.1 (3)
O3B—K4A—K4B—C2B	15.4 (9)	K4A—O3B—C1B—C2B	-43.4 (2)
O7B ^{vi} —K4A—K4B—C2B	-111.5 (5)	K2—O3B—C1B—C2B	-136.21 (15)
O4B—K4A—K4B—C2B	-20.78 (14)	K4B—O3B—C1B—C2B	-43.2 (3)
O2W—K4A—K4B—C2B	128.2 (9)	K4A—O3B—C1B—K2	92.80 (8)
O6 ^{iv} —K4A—K4B—C2B	75.7 (3)	K4B—O3B—C1B—K2	93.0 (3)
C1B—K4A—K4B—C2B	15.8 (4)	K2—O3B—C1B—K4A	-92.80 (8)
C7B ^{vi} —K4A—K4B—C2B	-123.12 (18)	K4B—O3B—C1B—K4A	0.2 (3)
K2—K4A—K4B—C2B	71.0 (7)	Ni1B—O2B—C1B—O3B	172.58 (16)
K1 ^{vii} —K4A—K4B—C2B	-61.87 (15)	K2—O2B—C1B—O3B	-35.41 (19)
O3W—K4A—K4B—O6B ^{vi}	-108.1 (18)	Ni1B—O2B—C1B—C2B	-9.1 (2)
O3B—K4A—K4B—O6B ^{vi}	115.7 (7)	K2—O2B—C1B—C2B	142.93 (16)
O7B ^{vi} —K4A—K4B—O6B ^{vi}	-11.2 (10)	Ni1B—O2B—C1B—K2	-152.01 (9)
O4B—K4A—K4B—O6B ^{vi}	79.5 (5)	Ni1B—O2B—C1B—K4A	-114.02 (18)
O2W—K4A—K4B—O6B ^{vi}	-131.5 (8)	K2—O2B—C1B—K4A	37.99 (18)
O6 ^{iv} —K4A—K4B—O6B ^{vi}	176.0 (7)	K1 ^{vii} —O4B—C2B—N1B	113.4 (2)
C1B—K4A—K4B—O6B ^{vi}	116.1 (4)	K4B—O4B—C2B—N1B	-123.6 (4)
C2B—K4A—K4B—O6B ^{vi}	100.3 (5)	K4A—O4B—C2B—N1B	-140.3 (2)
C7B ^{vi} —K4A—K4B—O6B ^{vi}	-22.8 (6)	K1 ^{vii} —O4B—C2B—C1B	-67.2 (3)
K2—K4A—K4B—O6B ^{vi}	171.2 (3)	K4B—O4B—C2B—C1B	55.8 (4)
K1 ^{vii} —K4A—K4B—O6B ^{vi}	38.4 (4)	K4A—O4B—C2B—C1B	39.1 (2)
O3W—K4A—K4B—K1 ^{vii}	-146.5 (16)	K1 ^{vii} —O4B—C2B—K4A	-106.3 (2)
O3B—K4A—K4B—K1 ^{vii}	77.3 (8)	K4B—O4B—C2B—K4A	16.7 (4)
O7B ^{vi} —K4A—K4B—K1 ^{vii}	-49.6 (5)	K1 ^{vii} —O4B—C2B—K4B	-123.0 (5)
O4B—K4A—K4B—K1 ^{vii}	41.1 (3)	K4A—O4B—C2B—K4B	-16.7 (4)
O2W—K4A—K4B—K1 ^{vii}	-170.0 (10)	N2B—N1B—C2B—O4B	-0.2 (3)
O6 ^{iv} —K4A—K4B—K1 ^{vii}	137.6 (2)	Ni1B—N1B—C2B—O4B	-174.38 (18)
C1B—K4A—K4B—K1 ^{vii}	77.7 (3)	N2B—N1B—C2B—C1B	-179.70 (15)
C2B—K4A—K4B—K1 ^{vii}	61.87 (15)	Ni1B—N1B—C2B—C1B	6.1 (2)
C7B ^{vi} —K4A—K4B—K1 ^{vii}	-61.3 (2)	N2B—N1B—C2B—K4A	-87.5 (3)
K2—K4A—K4B—K1 ^{vii}	132.8 (7)	Ni1B—N1B—C2B—K4A	98.3 (2)
N4—Ni1—O1—C7	4.19 (14)	N2B—N1B—C2B—K4B	-73.8 (5)
O2—Ni1—O1—C7	-174.48 (14)	Ni1B—N1B—C2B—K4B	112.1 (4)
N4—Ni1—N1—C2	175.16 (14)	O3B—C1B—C2B—O4B	1.1 (3)
O2—Ni1—N1—C2	-6.16 (14)	O2B—C1B—C2B—O4B	-177.33 (18)
N4—Ni1—N1—N2	-1.06 (16)	K2—C1B—C2B—O4B	-73.7 (3)
O2—Ni1—N1—N2	177.62 (15)	K4A—C1B—C2B—O4B	-33.06 (18)
C2—N1—N2—C4	-86.08 (19)	O3B—C1B—C2B—N1B	-179.34 (18)
Ni1—N1—N2—C4	90.13 (17)	O2B—C1B—C2B—N1B	2.2 (2)
C2—N1—N2—C3	154.28 (16)	K2—C1B—C2B—N1B	105.8 (2)
Ni1—N1—N2—C3	-29.5 (2)	K4A—C1B—C2B—N1B	146.49 (15)

C2—N1—N2—K3A ⁱⁱⁱ	30.35 (19)	O3B—C1B—C2B—K4A	34.17 (17)
Ni1—N1—N2—K3A ⁱⁱⁱ	-153.43 (12)	O2B—C1B—C2B—K4A	-144.27 (17)
C2—N1—N2—K3B ⁱⁱⁱ	35.4 (4)	K2—C1B—C2B—K4A	-40.66 (17)
Ni1—N1—N2—K3B ⁱⁱⁱ	-148.4 (3)	O3B—C1B—C2B—K4B	42.3 (3)
C5—N3—N4—C6	98.02 (19)	O2B—C1B—C2B—K4B	-136.1 (3)
C3—N3—N4—C6	-142.21 (17)	K2—C1B—C2B—K4B	-32.5 (3)
K2 ⁱⁱⁱ —N3—N4—C6	-29.02 (19)	K4A—C1B—C2B—K4B	8.1 (3)
C5—N3—N4—Ni1	-89.34 (18)	N4B—N3B—C3B—N2B	-69.1 (2)
C3—N3—N4—Ni1	30.4 (2)	C5B—N3B—C3B—N2B	51.8 (2)
K2 ⁱⁱⁱ —N3—N4—Ni1	143.62 (10)	K2 ^{iv} —N3B—C3B—N2B	-177.62 (12)
N1—Ni1—N4—C6	173.36 (15)	N1B—N2B—C3B—N3B	68.8 (2)
O1—Ni1—N4—C6	-6.73 (14)	C4B—N2B—C3B—N3B	-52.1 (2)
N1—Ni1—N4—N3	0.65 (16)	C5B—O5B—C4B—N2B	-59.4 (2)
O1—Ni1—N4—N3	-179.44 (15)	N1B—N2B—C4B—O5B	-64.8 (2)
K1 ⁱⁱ —O3—C1—O2	-105.7 (2)	C3B—N2B—C4B—O5B	55.4 (2)
K2—O3—C1—O2	33.8 (2)	C4B—O5B—C5B—N3B	58.9 (2)
K1—O3—C1—O2	130.80 (17)	N4B—N3B—C5B—O5B	65.6 (2)
K1 ⁱⁱ —O3—C1—C2	75.6 (3)	C3B—N3B—C5B—O5B	-54.5 (2)
K2—O3—C1—C2	-144.95 (15)	K2 ^{iv} —N3B—C5B—O5B	-170.11 (11)
K1—O3—C1—C2	-47.91 (18)	K2 ^{iv} —O6B—C6B—N4B	41.0 (3)
K1 ⁱⁱ —O3—C1—K2	-139.4 (2)	K1 ^{iv} —O6B—C6B—N4B	148.43 (18)
K1—O3—C1—K2	97.04 (7)	K4B ^{ix} —O6B—C6B—N4B	-128.0 (4)
K1 ⁱⁱ —O3—C1—K1	123.5 (2)	K2 ^{iv} —O6B—C6B—C7B	-140.07 (15)
K2—O3—C1—K1	-97.04 (7)	K1 ^{iv} —O6B—C6B—C7B	-32.6 (2)
Ni1—O2—C1—O3	173.77 (16)	K4B ^{ix} —O6B—C6B—C7B	50.9 (4)
K2—O2—C1—O3	-32.75 (19)	K1 ^{iv} —O6B—C6B—K2 ^{iv}	107.42 (14)
Ni1—O2—C1—C2	-7.5 (2)	K4B ^{ix} —O6B—C6B—K2 ^{iv}	-169.0 (3)
K2—O2—C1—C2	146.01 (14)	N3B—N4B—C6B—O6B	-0.7 (3)
Ni1—O2—C1—K2	-153.48 (10)	Ni1B—N4B—C6B—O6B	-173.75 (18)
Ni1—O2—C1—K1	-110.71 (16)	N3B—N4B—C6B—C7B	-179.74 (15)
K2—O2—C1—K1	42.77 (17)	Ni1B—N4B—C6B—C7B	7.2 (2)
K3A ⁱⁱⁱ —O4—C2—N1	-34.1 (3)	N3B—N4B—C6B—K2 ^{iv}	30.15 (15)
K3B ⁱⁱⁱ —O4—C2—N1	-38.5 (4)	Ni1B—N4B—C6B—K2 ^{iv}	-142.88 (10)
K1—O4—C2—N1	-137.41 (19)	K4A ^{ix} —O7B—C7B—O1B	117.66 (19)
K3A ⁱⁱⁱ —O4—C2—C1	145.53 (17)	K3B—O7B—C7B—O1B	-48.1 (4)
K3B ⁱⁱⁱ —O4—C2—C1	141.1 (3)	K3A—O7B—C7B—O1B	-51.5 (3)
K1—O4—C2—C1	42.23 (18)	K1 ^{iv} —O7B—C7B—O1B	-151.74 (17)
K3A ⁱⁱⁱ —O4—C2—K1	103.30 (14)	K4B ^{ix} —O7B—C7B—O1B	125.8 (4)
K3B ⁱⁱⁱ —O4—C2—K1	98.9 (3)	K4A ^{ix} —O7B—C7B—C6B	-61.2 (2)
K3B ⁱⁱⁱ —O4—C2—K3A ⁱⁱⁱ	-4.4 (3)	K3B—O7B—C7B—C6B	133.0 (3)
K1—O4—C2—K3A ⁱⁱⁱ	-103.30 (14)	K3A—O7B—C7B—C6B	129.56 (17)
K3A ⁱⁱⁱ —O4—C2—K3B ⁱⁱⁱ	4.4 (3)	K1 ^{iv} —O7B—C7B—C6B	29.4 (2)
K1—O4—C2—K3B ⁱⁱⁱ	-98.9 (3)	K4B ^{ix} —O7B—C7B—C6B	-53.1 (4)
N2—N1—C2—O4	-0.1 (3)	K3B—O7B—C7B—K4A ^{ix}	-165.7 (3)
Ni1—N1—C2—O4	-176.74 (17)	K3A—O7B—C7B—K4A ^{ix}	-169.2 (2)
N2—N1—C2—C1	-179.80 (15)	K1 ^{iv} —O7B—C7B—K4A ^{ix}	90.60 (11)
Ni1—N1—C2—C1	3.6 (2)	K4B ^{ix} —O7B—C7B—K4A ^{ix}	8.2 (3)
N2—N1—C2—K1	-88.3 (2)	Ni1B—O1B—C7B—O7B	177.15 (17)

Ni1—N1—C2—K1	95.1 (2)	Ni1B—O1B—C7B—C6B	-3.9 (2)
N2—N1—C2—K3A ⁱⁱⁱ	-24.43 (16)	Ni1B—O1B—C7B—K4A ^{ix}	-123.19 (13)
Ni1—N1—C2—K3A ⁱⁱⁱ	158.96 (12)	O6B—C6B—C7B—O7B	-2.1 (3)
N2—N1—C2—K3B ⁱⁱⁱ	-28.6 (3)	N4B—C6B—C7B—O7B	177.02 (18)
Ni1—N1—C2—K3B ⁱⁱⁱ	154.8 (3)	K2 ^{iv} —C6B—C7B—O7B	-67.7 (3)
O3—C1—C2—O4	1.9 (3)	O6B—C6B—C7B—O1B	178.91 (19)
O2—C1—C2—O4	-176.93 (17)	N4B—C6B—C7B—O1B	-2.0 (2)
K2—C1—C2—O4	-87.6 (3)	K2 ^{iv} —C6B—C7B—O1B	113.3 (2)
K1—C1—C2—O4	-36.38 (17)	O6B—C6B—C7B—K4A ^{ix}	-41.5 (2)
O3—C1—C2—N1	-178.40 (17)	N4B—C6B—C7B—K4A ^{ix}	137.66 (14)
O2—C1—C2—N1	2.8 (2)	K2 ^{iv} —C6B—C7B—K4A ^{ix}	-107.1 (2)

Symmetry codes: (i) $x-1/2, -y+1/2, -z+1$; (ii) $-x+1, -y, -z+1$; (iii) $x-1/2, y, -z+1/2$; (iv) $x+1/2, y, -z+1/2$; (v) $-x+3/2, -y, z-1/2$; (vi) $x, -y+1/2, z+1/2$; (vii) $x+1/2, -y+1/2, -z+1$; (viii) $-x+3/2, -y, z+1/2$; (ix) $x, -y+1/2, z-1/2$.

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1W—H1W1 \cdots O6 ^x	0.92	1.94	2.840 (2)	166
O1W—H2W1 \cdots O5W ⁱⁱⁱ	0.83	2.61	3.120 (4)	121
O1W—H2W1 \cdots O3B ⁱ	0.83	2.23	3.001 (2)	154
O2W—H2W2 \cdots O4 ⁱⁱ	0.93	1.83	2.754 (2)	171
O4WA—H2W4 \cdots O4B ⁱⁱⁱ	0.91	2.44	2.993 (2)	119
O4WA—H2W4 \cdots N2B ⁱⁱⁱ	0.91	2.02	2.895 (3)	161
O5W—H5WC \cdots O6B ^{xi}	0.85	1.95	2.774 (3)	162
O4WB—H3W4 \cdots O4B ⁱⁱⁱ	0.85	2.09	2.848 (9)	149
O4WB—H4W4 \cdots O6B ^{xi}	0.88	2.15	3.024 (9)	174

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