metal-organic compounds

Dipotassium tetraaquabis(μ-citrato-κ^4O:O',O'',O''')nickelate(II) tetrahydrate

Hua-Gang Yao,* Jia-Na Huang, Run-Kang Deng and Zhi-Bang Yao

School of Chemistry and Chemical Engineering, Guangdong Pharmaceutical University, Guangdong 528458, People's Republic of China
Correspondence e-mail: yaohg518@126.com

The title complex, $K_2[Ni_2(C_6H_5O_7)_2(H_2O)_4]\cdot 4H_2O$, is a dinuclear centrosymmetric anionic octahedral complex, involving citrates as tridentate and bridging ligands, and coordinating water molecules. An extensive network of hydrogen bonds connects the complex anions through the two unique uncoordinating water molecules. The K^+ counter cation is surrounded by seven O atoms in the form of an irregular polyhedron and further stabilizes the crystal packing.

Related literature

For applications of structures with metal-organic frameworks, see: Chui *et al.* (1999); Kahn & Martinez (1998); Kiang *et al.* (1999); Lin *et al.* (1999). For metal coordination polymers with a variety of topologies, see: Kondo *et al.* (2000); Shin *et al.* (2003); Wu *et al.* (2003); Yao *et al.* (2007). For the nickel–citrate complex $K_2[Ni(C_6H_5O_7)(H_2O)_2]_2\cdot 4H_2O$, which crystallized in the triclinic space group $P\bar{1}$, see: Baker *et al.* (1983).
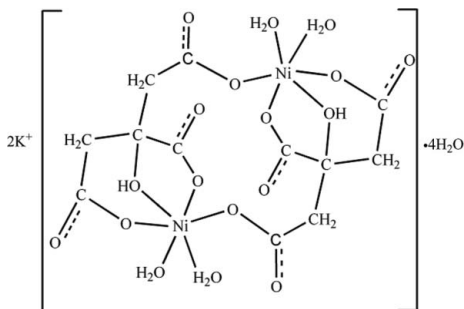
Experimental

Crystal data

$K_2[Ni_2(C_6H_5O_7)_2(H_2O)_4]\cdot 4H_2O$
$M_r = 717.94$
Monoclinic, $P2_1/c$
$a = 10.616$ (2) Å
$b = 13.006$ (3) Å
$c = 9.0513$ (18) Å
$\beta = 93.09$ (3)°
$V = 1247.8$ (4) Å^3

$Z = 2$
Mo $K\alpha$ radiation
$\mu = 1.94$ mm^{-1}
$T = 293$ K
$0.30 \times 0.20 \times 0.15$ mm

Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
$T_{min} = 0.636$, $T_{max} = 0.741$

9515 measured reflections
3128 independent reflections
2916 reflections with $I > 2\sigma(I)$
$R_{int} = 0.017$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.023$
$wR(F^2) = 0.063$
$S = 1.02$
3128 reflections
224 parameters

2 restraints
H-atom parameters constrained
$\Delta\rho_{max} = 0.43$ e Å^{-3}
$\Delta\rho_{min} = -0.55$ e Å^{-3}

Table 1
Selected bond lengths (Å).

Ni1—O4	2.0322 (12)	Ni1—O2W	2.0677 (12)
Ni1—O2	2.0330 (11)	Ni1—O1W	2.0709 (11)
Ni1—O6	2.0345 (10)	Ni1—O3	2.0927 (10)

Table 2
Hydrogen-bond geometry (Å, °).

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O1W—H1WB⋯O2^i	0.79 (3)	1.96 (3)	2.7322 (16)	167 (2)
O2W—H2WB⋯O1^{ii}	0.84 (3)	1.93 (3)	2.7638 (16)	170 (2)
O4W—H4WB⋯O5^{iii}	0.85 (3)	1.91 (3)	2.7459 (17)	171 (2)
O2W—H2WA⋯O4W^{iv}	0.88 (3)	1.83 (3)	2.7064 (18)	174 (2)
O4W—H4WA⋯O5^v	0.72 (3)	2.20 (2)	2.8714 (19)	155 (2)
O3—H1⋯O6^{vi}	0.75 (2)	2.13 (2)	2.7152 (15)	135 (2)
O3W—H3WA⋯O2W^{vii}	0.85 (1)	2.25 (4)	2.912 (2)	135 (5)

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

References

Baker, E. N., Baker, H. N., Anderson, B. F. & Reevs, R. D. (1983). *Inorg. Chim. Acta*, **78**, 281–285.
Bruker (2004). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Chui, S. S. Y., Lo, S. M. F., Charmant, J. P. H., Orpen, A. G. & Williams, I. D. (1999). *Science*, **283**, 1148–1150.
Kahn, O. & Martinez, C. J. (1998). *Science*, **279**, 44–46.
Kiang, Y.-H., Gardner, G. B., Lee, S., Xu, Z. & Lobkovsky, E. B. (1999). *J. Am. Chem. Soc.* **121**, 8204–8206.
Kondo, M., Shimamura, M., Noro, S. I., Minakoshi, S., Asami, A., Seki, K. & Kitagawa, S. (2000). *Chem. Mater.* **12**, 1288–1295.
Lin, W., Wang, Z. & Ma, L.-J. (1999). *J. Am. Chem. Soc.* **121**, 11249–11251.

Sheldrick, G. M. (2001). *SADABS*. University of Gottingen, Germany.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Shin, D.-M., Lee, I.-S., Lee, Y.-A. & Chung, Y.-K. (2003). *Inorg. Chem.* **42**, 2977–2981.

Wu, C.-D., Lu, C.-Z., Lin, X., Wu, D.-M., Lu, S.-F., Zhang, H.-H. & Huang, J.-S. (2003). *Chem. Commun.* pp. 1254–1255.

Yao, H.-G., Ji, M., Ji, S.-H., Jiang, Y.-S., Li, L. & An, Y.-L. (2007). *Inorg. Chem. Commun.* **10**, 440–442.

supporting information

Dipotassium tetraaquabis(*μ*-citrato-*κ*⁴*O*:*O*′,*O*″,*O*‴)nickelate(II) tetrahydrate

Hua-Gang Yao, Jia-Na Huang, Run-Kang Deng and Zhi-Bang Yao

S1. Comment

The construction of metal–organic frameworks (MOFs) is an area of intense research activity due to their intriguing structural diversity and potential applications as zeolitic, optoelectronic, magnetic and conducting materials (Chui *et al.*, 1999; Kiang *et al.*, 1999; Kahn & Martinez, 1998; Lin *et al.*, 1999). Depending on the conformation of carbon chains, the functional group of organic ligands and the type of metal ions, a variety of metal coordination polymers with different topological structures, such as one-dimensional chains (Shin *et al.*, 2003), two-dimensional grids (Kondo *et al.*, 2000), three-dimensional porous motifs (Yao *et al.*, 2007) and helical strands (Wu *et al.*, 2003) were observed. In this paper, we report the synthesis of a dimeric nickel(II) citrate complex by self-assembly under hydrothermal conditions.

In the crystal the centrosymmetric structural unit is a dinuclear Ni^{II} anion (Fig. 1) and the two potassium cations, and crystalline water molecules. The crystallographic unit is a half of the structural unit. The Ni^{II} ion adopts an octahedral coordination mode. One citrate ligand is bound with an hydroxyl and two carboxylate groups to the Ni^{II} ion, whereas one O atom (O6) from a carboxylate group of a symmetry-related citrate ligand occupies another apex, and two water molecules complete the octahedral environment. The Ni—O distances range from 2.0322 (12) Å to 2.0927 (10) Å (Table 1). Neighbouring dimeric complexes are consolidated into a three-dimensional structure by hydrogen bonds (Table 2, Fig. 2). The crystallographically independent potassium cation, K1, is seven-coordinated by O atoms, with an average contact distance of 2.852 Å.

The corresponding nickel–citrate complex with the triclinic space group $P\bar{1}$, $K_2[Ni(C_6H_5O_7)(H_2O)_2]_2 \cdot 4H_2O$, has been reported (Baker *et al.*, 1983). The complex exists as centrosymmetric dimers, which has identical structure with the title complex, but a difference is that the potassium ions and water molecules of crystallization occupy the spaces between the nickel–citrate dimers in the two cases, resulting in the different formation of the geometry of potassium ion and hydrogen bonds.

S2. Experimental

Citric acid monohydrate (0.048 g), $NiCl_2 \cdot 6H_2O$ (0.042 g) and KOH (0.027 g) were dissolved in 6 ml mixed solvent of DMF–H_2O (2:1 *v/v*), which were placed in a small vial. The mixture was heated at 351 K for 3 d and then cooled to room temperature. Green block crystals of the product were collected by filtration and washed with ethanol several times (88% based on Ni). This synthetic route allowed us to obtain a pure phase. Elemental analysis, calculated (%) for title compound: C 20.06, H 3.62; found C 20.35, H 3.44.

S3. Refinement

H atoms were positioned geometrically, with C—H = 0.93 Å, and allowed to ride during subsequent refinement, with $U_{iso}(H) = 1.2U_{eq}(C)$.
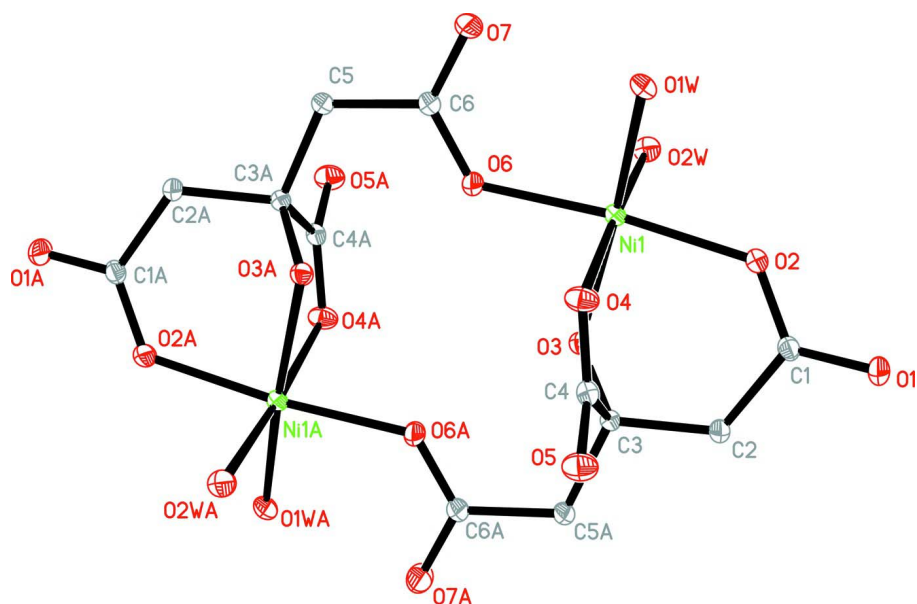
Figure 1

The structure of the dimeric complex anion, with the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity.
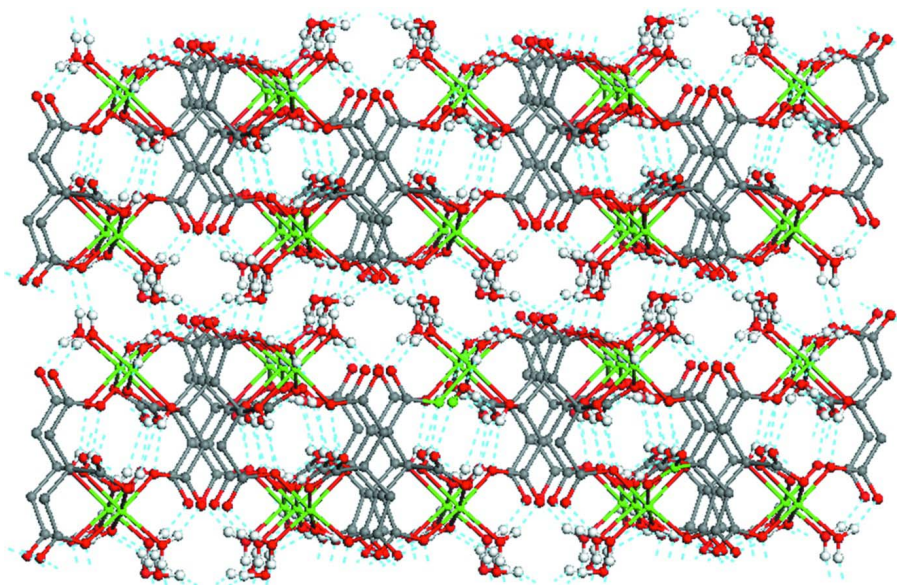
Figure 2

Dimeric complexes are consolidated into three-dimensional structures by hydrogen bonds. Symmetry code: -*x*, 1/2 + *y*, 1/2 - *z*

Dipotassium tetraaquabis(*µ*-citrato-*κ*⁴*O*:*O′*,*O″*,*O‴*)nickelate(II) tetrahydrate

Crystal data

K₂[Ni₂(C₆H₅O₇)₂(H₂O)₄]·4H₂O
$M_r = 717.94$
Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc
$a = 10.616 (2)$ Å
$b = 13.006 (3)$ Å

$c = 9.0513\ (18)$ Å

$\beta = 93.09\ (3)°$

$V = 1247.8\ (4)$ Å^3

$Z = 2$

$F(000) = 736$

$D_x = 1.911$ Mg m^{-3}

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

$\mu = 1.94$ mm^{-1}

$T = 293$ K

Block, green

$0.30 \times 0.20 \times 0.15$ mm

Data collection

Bruker SMART APEXII CCD
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan
(*SADABS*; Sheldrick, 2001)

$T_{min} = 0.636$, $T_{max} = 0.741$

9515 measured reflections

3128 independent reflections

2916 reflections with $I > 2\sigma(I)$

$R_{int} = 0.017$

$\theta_{max} = 28.4°$, $\theta_{min} = 1.9°$

$h = -14\rightarrow12$

$k = -17\rightarrow17$

$l = -12\rightarrow12$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.023$

$wR(F^2) = 0.063$

$S = 1.02$

3128 reflections

224 parameters

2 restraints

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0378P)^2 + 0.4504P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.43$ e Å^{-3}

$\Delta\rho_{min} = -0.55$ e Å^{-3}

Special details

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

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

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

	x	y	z	U_{iso}*/U_{eq}
Ni1	0.243734 (14)	0.519202 (13)	−0.006123 (17)	0.01642 (7)
K1	−0.13777 (3)	0.90086 (3)	0.14726 (4)	0.03020 (9)
O1	0.07698 (9)	0.76820 (8)	0.19256 (13)	0.0290 (2)
O2	0.12630 (9)	0.63014 (8)	0.06472 (12)	0.0256 (2)
O3	0.38530 (9)	0.63022 (8)	−0.02002 (10)	0.01756 (18)
O4	0.32413 (10)	0.51024 (8)	0.20239 (12)	0.0247 (2)
O5	0.43183 (11)	0.60531 (8)	0.37004 (11)	0.0278 (2)
O6	0.37540 (9)	0.41901 (8)	−0.07541 (12)	0.0240 (2)
O7	0.25885 (11)	0.27918 (10)	−0.11481 (19)	0.0503 (4)
O1W	0.11765 (10)	0.40268 (8)	0.03601 (12)	0.0235 (2)
O2W	0.16893 (11)	0.54135 (9)	−0.21942 (12)	0.0238 (2)

O3W	−0.00587 (17)	0.89783 (14)	−0.11374 (17)	0.0549 (4)
O4W	−0.32069 (13)	0.97832 (11)	−0.06745 (14)	0.0336 (3)
C1	0.15547 (12)	0.71310 (10)	0.13242 (14)	0.0187 (2)
C2	0.29093 (12)	0.75171 (10)	0.13945 (16)	0.0202 (3)
C3	0.39866 (11)	0.67514 (10)	0.12633 (13)	0.0160 (2)
C4	0.38568 (12)	0.58926 (10)	0.24245 (14)	0.0181 (2)
C5	0.47751 (12)	0.26558 (11)	−0.14594 (16)	0.0197 (3)
C6	0.36067 (12)	0.32483 (11)	−0.11019 (15)	0.0212 (3)
H1	0.444 (2)	0.6015 (17)	−0.036 (2)	0.040 (6)*
H2A	0.2987 (17)	0.8014 (15)	0.067 (2)	0.030 (5)*
H2B	0.3039 (17)	0.7872 (16)	0.227 (2)	0.031 (5)*
H5A	0.4817 (18)	0.2059 (16)	−0.090 (2)	0.033 (5)*
H5B	0.4680 (17)	0.2407 (15)	−0.249 (2)	0.029 (5)*
H1WB	0.047 (2)	0.4026 (18)	0.003 (2)	0.048 (6)*
H1WA	0.152 (2)	0.354 (2)	−0.006 (3)	0.065 (8)*
H2WA	0.220 (2)	0.525 (2)	−0.290 (3)	0.057 (7)*
H2WB	0.140 (2)	0.601 (2)	−0.235 (3)	0.055 (7)*
H3WC	−0.003 (3)	0.8359 (11)	−0.141 (4)	0.096 (11)*
H3WA	−0.073 (3)	0.906 (4)	−0.168 (5)	0.18 (2)*
H4WA	−0.372 (2)	0.948 (2)	−0.100 (3)	0.041 (6)*
H4WB	−0.351 (2)	1.0134 (19)	0.001 (3)	0.053 (7)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.01296 (10)	0.01699 (11)	0.01930 (10)	−0.00081 (6)	0.00068 (6)	−0.00159 (6)
K1	0.02800 (17)	0.03044 (18)	0.03236 (17)	0.00154 (13)	0.00338 (13)	−0.00315 (13)
O4	0.0277 (5)	0.0229 (5)	0.0230 (5)	−0.0075 (4)	−0.0032 (4)	0.0043 (4)
O3	0.0162 (4)	0.0189 (4)	0.0176 (4)	0.0000 (4)	0.0014 (3)	−0.0016 (3)
O6	0.0158 (4)	0.0174 (5)	0.0390 (6)	0.0003 (4)	0.0041 (4)	−0.0049 (4)
O2	0.0151 (4)	0.0257 (5)	0.0358 (5)	0.0002 (4)	0.0014 (4)	−0.0107 (4)
O5	0.0335 (6)	0.0305 (6)	0.0188 (5)	−0.0038 (4)	−0.0047 (4)	−0.0002 (4)
O1	0.0182 (5)	0.0257 (5)	0.0438 (6)	0.0019 (4)	0.0077 (4)	−0.0090 (5)
C5	0.0148 (6)	0.0170 (6)	0.0273 (7)	−0.0008 (5)	0.0012 (5)	−0.0021 (5)
C6	0.0155 (6)	0.0201 (6)	0.0281 (6)	−0.0004 (5)	0.0018 (5)	−0.0029 (5)
C2	0.0152 (6)	0.0169 (6)	0.0286 (7)	0.0014 (5)	0.0011 (5)	−0.0030 (5)
O7	0.0178 (5)	0.0321 (7)	0.1024 (12)	−0.0080 (5)	0.0151 (6)	−0.0266 (7)
C1	0.0149 (6)	0.0201 (6)	0.0209 (6)	0.0015 (5)	0.0006 (4)	0.0004 (5)
C3	0.0136 (5)	0.0167 (6)	0.0175 (5)	0.0004 (4)	0.0004 (4)	−0.0021 (4)
C4	0.0147 (6)	0.0196 (6)	0.0201 (6)	0.0019 (5)	0.0009 (4)	0.0005 (5)
O1W	0.0150 (5)	0.0245 (5)	0.0308 (5)	−0.0034 (4)	0.0016 (4)	−0.0013 (4)
O2W	0.0265 (5)	0.0223 (5)	0.0224 (5)	−0.0007 (4)	−0.0003 (4)	0.0012 (4)
O3W	0.0621 (10)	0.0611 (10)	0.0421 (8)	0.0235 (8)	0.0082 (7)	−0.0023 (7)
O4W	0.0313 (6)	0.0412 (7)	0.0287 (6)	−0.0052 (5)	0.0048 (5)	−0.0084 (5)

Geometric parameters (Å, º)

Ni1—O4	2.0322 (12)	O1—C1	1.2462 (16)
Ni1—O2	2.0330 (11)	C5—C6	1.5100 (18)
Ni1—O6	2.0345 (10)	C5—C3�v	1.5259 (18)
Ni1—O2W	2.0677 (12)	C5—H5B	0.984 (18)
Ni1—O1W	2.0709 (11)	C5—H5A	0.93 (2)
Ni1—O3	2.0927 (10)	C6—O7	1.2320 (17)
K1—O7ⁱ	2.6796 (13)	C2—C1	1.5213 (18)
K1—O3W	2.8108 (17)	C2—C3	1.5259 (17)
K1—O4ⁱⁱ	2.8425 (12)	C2—H2B	0.925 (19)
K1—O4W	2.8557 (16)	C2—H2A	0.926 (19)
K1—O1Wⁱⁱ	2.8633 (13)	O7—K1ⁱ	2.6796 (13)
K1—O1	2.8708 (12)	C3—C5�v	1.5259 (18)
K1—O3Wⁱⁱⁱ	3.053 (2)	C3—C4	1.5449 (18)
O4—C4	1.2606 (17)	O1W—K1ⁱᵛ	2.8633 (13)
O4—K1ⁱᵛ	2.8425 (12)	O1W—H1WB	0.79 (3)
O3—C3	1.4477 (15)	O1W—H1WA	0.83 (3)
O3—H1	0.75 (2)	O4W—H4WB	0.85 (3)
O6—C6	1.2723 (17)	O4W—H4WA	0.72 (3)
O2W—H2WB	0.84 (3)	O3W—K1ⁱⁱⁱ	3.053 (2)
O2W—H2WA	0.88 (3)	O3W—H3WC	0.842 (10)
O2—C1	1.2707 (17)	O3W—H3WA	0.845 (10)
O5—C4	1.2475 (17)		
O4—Ni1—O2	88.95 (5)	C1—O2—Ni1	128.10 (9)
O4—Ni1—O6	89.34 (5)	C1—O1—K1	145.86 (10)
O2—Ni1—O6	174.20 (4)	C6—C5—C3�v	115.44 (11)
O4—Ni1—O2W	174.86 (4)	C6—C5—H5B	109.1 (11)
O2—Ni1—O2W	89.11 (5)	C3�v—C5—H5B	108.7 (11)
O6—Ni1—O2W	92.12 (5)	C6—C5—H5A	109.1 (12)
O4—Ni1—O1W	91.73 (5)	C3�v—C5—H5A	110.1 (12)
O2—Ni1—O1W	92.74 (5)	H5B—C5—H5A	103.7 (16)
O6—Ni1—O1W	92.85 (5)	O7—C6—O6	124.63 (13)
O2W—Ni1—O1W	93.12 (5)	O7—C6—C5	118.43 (13)
O4—Ni1—O3	80.12 (4)	O6—C6—C5	116.93 (12)
O2—Ni1—O3	89.07 (5)	C1—C2—C3	119.47 (11)
O6—Ni1—O3	85.17 (4)	C1—C2—H2B	107.3 (12)
O2W—Ni1—O3	95.08 (4)	C3—C2—H2B	108.4 (12)
O1W—Ni1—O3	171.62 (4)	C1—C2—H2A	108.7 (11)
O7ⁱ—K1—O3W	98.82 (5)	C3—C2—H2A	107.8 (11)
O7ⁱ—K1—O4ⁱⁱ	98.49 (4)	H2B—C2—H2A	104.2 (16)
O3W—K1—O4ⁱⁱ	143.18 (4)	C6—O7—K1ⁱ	147.10 (10)
O7ⁱ—K1—O4W	85.94 (5)	O1—C1—O2	123.20 (12)
O3W—K1—O4W	77.54 (5)	O1—C1—C2	116.40 (12)
O4ⁱⁱ—K1—O4W	71.58 (4)	O2—C1—C2	120.38 (11)
O7ⁱ—K1—O1Wⁱⁱ	97.26 (5)	O3—C3—C2	107.32 (10)
O3W—K1—O1Wⁱⁱ	145.89 (5)	O3—C3—C5�v	110.58 (10)

O4^{ii}—K1—O1W^{ii}	62.15 (4)	C2—C3—C5^{v}	107.80 (11)
O4W—K1—O1W^{ii}	133.61 (4)	O3—C3—C4	108.84 (10)
O7^{i}—K1—O1	82.13 (4)	C2—C3—C4	108.91 (10)
O3W—K1—O1	71.57 (5)	C5^{v}—C3—C4	113.21 (11)
O4^{ii}—K1—O1	143.15 (3)	O5—C4—O4	125.05 (13)
O4W—K1—O1	144.57 (4)	O5—C4—C3	117.64 (12)
O1W^{ii}—K1—O1	81.15 (4)	O4—C4—C3	117.23 (11)
O7^{i}—K1—O3W^{iii}	167.85 (5)	Ni1—O1W—K1^{iv}	100.16 (5)
O3W—K1—O3W^{iii}	69.78 (6)	Ni1—O1W—H1WB	122.5 (17)
O4^{ii}—K1—O3W^{iii}	89.00 (4)	K1^{iv}—O1W—H1WB	113.6 (16)
O4W—K1—O3W^{iii}	87.41 (5)	Ni1—O1W—H1WA	99.7 (18)
O1W^{ii}—K1—O3W^{iii}	94.69 (4)	K1^{iv}—O1W—H1WA	116.3 (18)
O1—K1—O3W^{iii}	97.62 (4)	H1WB—O1W—H1WA	104 (2)
C4—O4—Ni1	113.92 (9)	K1—O4W—H4WB	87.9 (17)
C4—O4—K1^{iv}	129.32 (9)	K1—O4W—H4WA	123.4 (19)
Ni1—O4—K1^{iv}	101.83 (4)	H4WB—O4W—H4WA	107 (2)
C3—O3—Ni1	104.99 (7)	K1—O3W—K1^{iii}	110.22 (6)
C3—O3—H1	109.4 (17)	K1—O3W—H3WC	107 (2)
Ni1—O3—H1	106.2 (17)	K1^{iii}—O3W—H3WC	140 (2)
C6—O6—Ni1	128.00 (9)	K1—O3W—H3WA	92 (3)
Ni1—O2W—H2WB	114.0 (16)	K1^{iii}—O3W—H3WA	104 (4)
Ni1—O2W—H2WA	115.0 (16)	H3WC—O3W—H3WA	90 (4)
H2WB—O2W—H2WA	109 (2)		

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

Hydrogen-bond geometry (Å, º)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O1W—H1WB⋯O2^{i}	0.79 (3)	1.96 (3)	2.7322 (16)	167 (2)
O2W—H2WB⋯O1^{vi}	0.84 (3)	1.93 (3)	2.7638 (16)	170 (2)
O4W—H4WB⋯O5^{ii}	0.85 (3)	1.91 (3)	2.7459 (17)	171 (2)
O2W—H2WA⋯O4W^{vii}	0.88 (3)	1.83 (3)	2.7064 (18)	174 (2)
O4W—H4WA⋯O5^{viii}	0.72 (3)	2.20 (2)	2.8714 (19)	155 (2)
O3—H1⋯O6^{v}	0.75 (2)	2.13 (2)	2.7152 (15)	135 (2)
O3W—H3WA⋯O2W^{ix}	0.85 (1)	2.25 (4)	2.912 (2)	135 (5)

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