IUCrData

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# Poly[[bis($\mu_4$-acetato-$\kappa^4O$:$O$:$O'$:$O'$)tetrakis($\mu_3$-acetato-$\kappa^3O$:$O$:$O'$)bis($\mu_2$-acetato-$\kappa^2O$:$O'$)bis($\mu_3$-hydroxido)pentanickel(II)] 2.60-hydrate]

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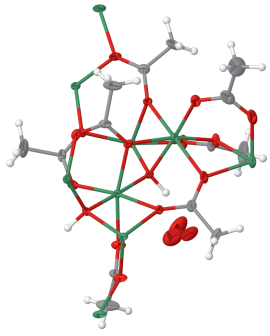
**Keywords:** crystal structure; nickel; basic acetate; disorder; hydrogen-bonding.

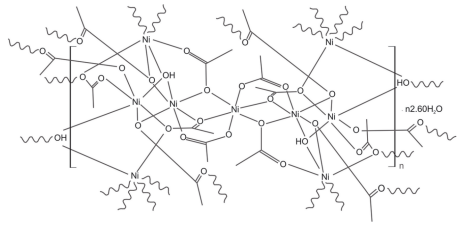
**Structural data:** full structural data are available from iucrdata.iucr.org

The title compound, $\{[Ni_5(C_2H_3O_2)_8(OH)_2]\cdot 2.60H_2O\}_n$ or $[Ni_5(OAc)_8(OH)_2]\cdot 2.60H_2O$ (OAc is the acetate anion, $C_2H_3O_2$), represents a hydrated basic acetate. Its asymmetric unit comprises half of the formula unit, with one of the three unique $Ni^{II}$ cations situated at an inversion centre. The $Ni^{II}$ atoms are in octahedral coordination environments by O atoms of the acetato ligands and by the basic OH group. The different kinds of bridging modes ($\mu_2$, $2\times\mu_3$, and $\mu_4$ for the acetato ligands; $\mu_3$ for the OH group) lead to the formation of a framework structure with hydrophobic channels extending parallel to the main crystallographic axes. Disordered water molecules are situated in pockets close to the OH groups and are held in place by hydrogen-bonding interactions.

3D view



Chemical scheme



## Structure description

Nickel acetate, $Ni(OAc)_2$, is a common precursor for the synthesis of oxygen-containing nickel compounds and is usually employed in form of its tetrahydrate. As it decomposes easily when the temperature is increased, it is used for typical solid-state reactions. As a result of its good solubility in water, nickel acetate can also be used for syntheses in aqueous media or under hydrothermal conditions. Precisely for this purpose, $Ni(OAc)_2$ was employed as a precursor intended for phase-formation studies of nickel arsenates under hydrothermal conditions. However, a basic nickel acetate of composition $[Ni_5(OAc)_8(OH)_2]\cdot 2.60H_2O$ had formed serendipitously instead, and its crystal structure is reported here.

In general, basic acetates comprise metal cations bound to a collection of acetate anions and to an $O^{2-}$ ion or an $OH^-$ group. The latter bridge several metal atoms ($M$) and thus form oxido-centred coordination polyhedra, usually with $\{OM_3\}$-/$\{(HO)M_2\}$-trigonal–planar or $\{OM_4\}$-/$\{(HO)M_3\}$-tetrahedral shapes. These kinds of structural

**Table 1**
Selected bond lengths (Å).

| | | | |
|---|---|---|---|
| Ni1—O2 | 1.993 (2) | Ni2—O6$^{ii}$ | 2.1140 (18) |
| Ni1—O3 | 2.1037 (18) | Ni3—O9 | 1.9747 (18) |
| Ni1—O5 | 2.1257 (19) | Ni3—O9$^{i}$ | 1.9902 (17) |
| Ni2—O1 | 1.993 (2) | Ni3—O7 | 2.0474 (19) |
| Ni2—O9 | 1.9962 (18) | Ni3—O4$^{i}$ | 2.0750 (19) |
| Ni2—O3 | 2.0698 (18) | Ni3—O8$^{i}$ | 2.1023 (19) |
| Ni2—O5 | 2.0937 (19) | Ni3—O6 | 2.1259 (18) |
| Ni2—O8$^{i}$ | 2.1016 (18) | | |

Symmetry codes: (i) $y+\frac{1}{4}, -x+\frac{5}{4}, z+\frac{1}{4}$; (ii) $-y+\frac{5}{4}, x-\frac{1}{4}, z-\frac{1}{4}$.

features are observed, for example, in the acetate compounds $Be_4O(OAc)_6$ (Pauling & Sherman, 1934), $Mg_3O(OAc)_4$ (Scheurell *et al.*, 2015), $[Cr_8(OH)_8(OAc)_{16}]\cdot 30H_2O$ (Eshel & Bino, 2001), $Fe_3O(OAc)_7(HOAc)$ (Abrahams *et al.*, 2024), $Cu_2(OH)_3(OAc)\cdot H_2O$ (Švarcová *et al.*, 2011), $Pb_3O_2(OAc)_2\cdot 0.5H_2O$ (Mauck *et al.*, 2010), $Pb_4O(OAc)_6$ or $Pb_2O(OAc)_2$ (Martínez Casado *et al.*, 2016). In the title compound, an {(HO)Ni$_3$} unit with tetrahedral shape is present, as discussed in more detail below.

$[Ni_5(OAc)_8(OH)_2]\cdot 2.60H_2O$ is isostructural with the magnesium analogue, $[Mg_5(OAc)_8(OH)_2]\cdot 1.19H_2O$ (Scheurell *et al.*, 2015). The asymmetric unit of $[Ni_5(OAc)_8(OH)_2]\cdot 2.60H_2O$ comprises of half of the formula unit, with Ni1 situated at a special position (multiplicity 8, Wyckoff letter *d*, site symmetry $\overline{1}$) of space group $I4_1/a$. The three Ni$^{II}$ cations are octahedrally surrounded by O atoms, with Ni1 only by carboxylate O atoms, Ni2 by five carboxylate O atoms and one O atom (O9) of the OH group, and Ni3 by four carboxylate O atoms and two OH groups (Fig. 1Table 1). The Ni—O distances range from 1.993 (2) to 2.1259 (18) Å, with a mean of 2.063 (55) Å, which is close to the literature value of 2.070 (54) calculated for 242 [NiO$_6$] polyhedra (Gagné & Hawthorne, 2020).

From the seven different possible coordination modes of acetato ligands to central $M^{II}$ cations shown in Fig. 2, the acetate groups in the structure of the title compound feature only three. Coordination mode (*a*) is bridging two Ni$^{II}$ cations in a bis-monodentate manner, $\mu_2$-(-$\kappa^1O,\kappa^1O'$), and realized for carboxylate group C2(O1)O2; mode (*b*) is bridging three Ni$^{II}$

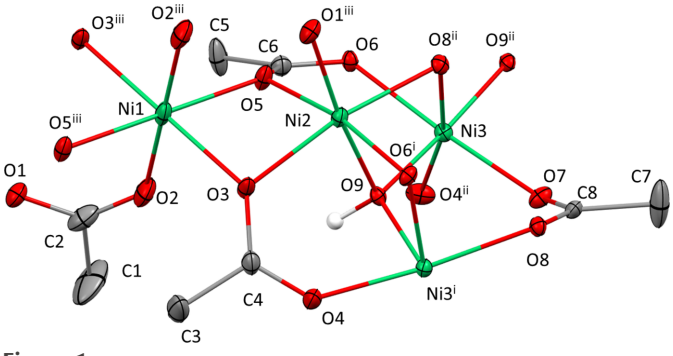



**Figure 1**
The coordination of the Ni$^{II}$ atoms in the crystal structure of $[Ni_5(OAc)_8(OH)_2]\cdot 2.6H_2O$. Displacement ellipsoids are drawn at the 40% probability level; for clarity, methyl H atoms and the O atoms of disordered water molecules are not shown. [Symmetry codes: (i) $-y+\frac{5}{4}, x-\frac{1}{4}, z-\frac{1}{4}$; (ii) $y+\frac{1}{4}, -x+\frac{5}{4}, z+\frac{1}{4}$; (iii) $-x+2, -y+1, -z+1$.]

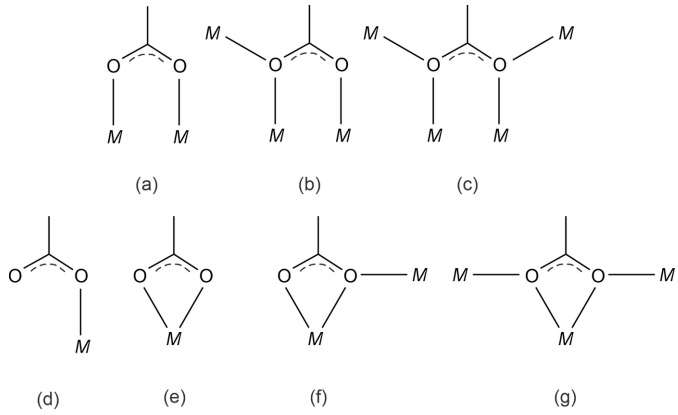



**Figure 2**
Possible coordination modes of the acetato ligand to metal cations *M*.

cations in a monodentate-bis-monodentate manner, $\mu_3$-(-$\kappa^1O\kappa^2O'$), and realized for carboxylate groups C4(O3)O4 and C7(O8)O7; mode (*c*) is bridging four Ni$^{II}$ cations in a bis(bis-monodentate) manner, $\mu_4$-(-$\kappa^2O\kappa^2O'$, and realized for carboxylate group C5(O5)O6. Monodentate coordination mode (*d*), or any of the chelating coordination modes (*e–g*) detailed in Fig. 2 are not realized, but are known for other divalent first-row transition metals *M*, *e.g.* for anhydrous iron(II) acetate (Weber *et al.*, 2011). The oxygen atom of the hydroxy group, O9H9, bridges three Ni$^{II}$ cations (Ni2, Ni3, Ni3′). Together with the attached H9 atom, the environment of O9 is distorted tetrahedral, with Ni—O—Ni angles ranging from 97.13 (8) to 121.18 (9)°.

The $\mu_2$- $\mu_3$-, $\mu_4$- and $\mu_3$-bridging modes of the acetato ligands and the $\mu_3$-mode of the OH group, respectively, lead to the formation of a framework structure, whereby the arrangement of the acetato ligands with the methyl groups pointing away from the Ni$^{II}$ cations creates hydrophobic channels extending parallel to the main crystallographic axes

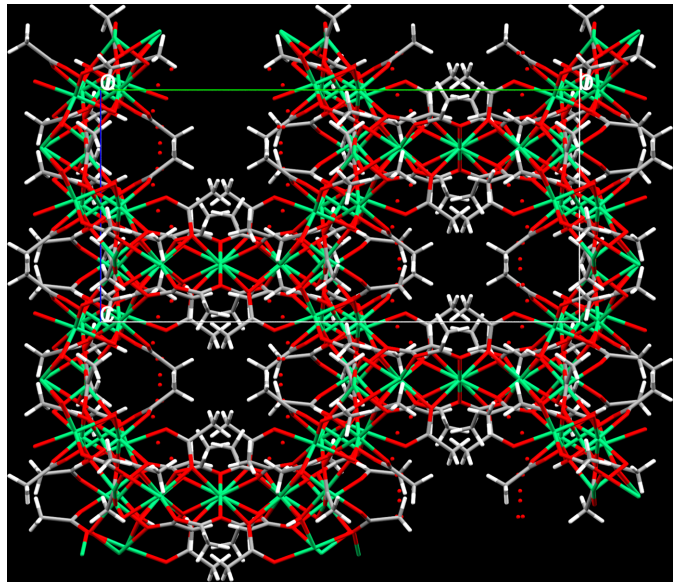



**Figure 3**
Packing plot of $[Ni_5(OAc)_8(OH)_2]\cdot 2.60H_2O$ along [100].

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O9—H9···O*W*1 | 1.00 | 2.06 | 2.957 (4) | 147 |
| O9—H9···O*W*3 | 1.00 | 1.85 | 2.827 (8) | 166 |
| O7···O*W*2$^{ii}$ | | | 2.87 | |
| O*W*2···O*W*3 | | | 2.85 | |

Symmetry code: (ii) $-y+\frac{5}{4}, x-\frac{1}{4}, z-\frac{1}{4}$.

(Figs. 3, 4). The disordered water molecules of crystallization are situated in pockets near to the hydroxy group to which they are hydrogen-bonded (Table 2, Fig. 4). In addition, typical donor···acceptor distances suitable for hydrogen bonds of moderate strength are present between O7···O*W*2 and O2*W*···O3*W* (Table 2). These interactions might further consolidate the crystal structure.

## Synthesis and crystallization

Single crystals of $[Ni_5(OAc)_8(OH)_2]\cdot 2.60H_2O$ were inadvertently obtained by reacting $Ni(OAc)_2\cdot 4H_2O$, KOH (>85%$_{wt}$) and ∼80%$_{wt}$ $H_3AsO_4$ under hydrothermal conditions in an approximate 3:2:3 molar ratio. The reactants were introduced in a Teflon lined steal autoclave and heated at 493 K for 3 d. After cooling to room temperature, large faint greenish plates of $[Ni_5(OAc)_8(OH)_2]\cdot 2.60H_2O$ were directly isolated from the mother liquor under a polarizing microscope.

## Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. The H atom of the hydroxide group was located in a difference-Fourier map and was refined as riding on the parent O atom with $U_{iso}(H) = 1.5U_{eq}(O)$. Free refinement of the occupation factors of the three crystal water O-atom positions indicated underoccupation for all of them. For the final structure model, the two least occupied positions (O*W*2, O*W*3) were paired and coupled with the occupation factor of the most occupied site (O*W*1) so that the sum of site occupation factors equals 1. The water H atoms could not be located and were excluded from the structural model, but are included for calculation of crystal data.

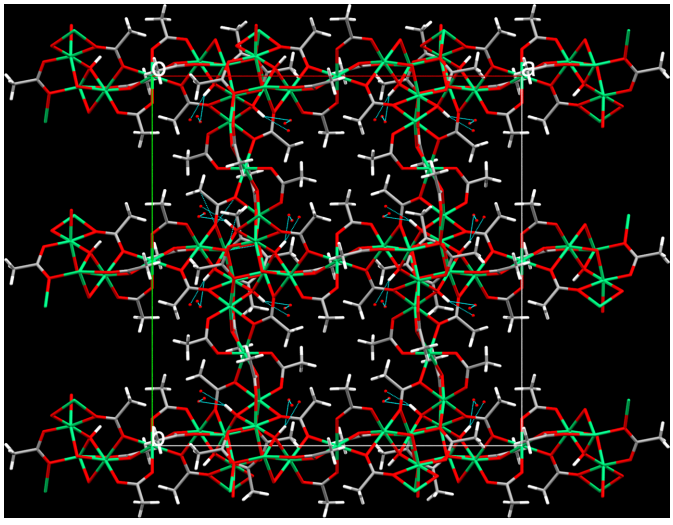


**Figure 4**
Packing plot of $[Ni_5(OAc)_8(OH)_2]\cdot 2.60H_2O$ along [001] with hydrogen-bonding interactions between the hydroxy group and the O atoms of disordered water molecules (shown as blue dotted lines).

**Table 3**
Experimental details.

| | |
|---|---|
| Crystal data | |
| Chemical formula | $[Ni_5(C_2H_3O_2)_8(OH)_2]\cdot 2.60H2O$ |
| $M_r$ | 846.80 |
| Crystal system, space group | Tetragonal, $I4_1/a$ |
| Temperature (K) | 100 |
| $a$, $c$ (Å) | 23.3025 (11), 11.2648 (5) |
| $V$ (Å$^3$) | 6116.9 (6) |
| $Z$ | 8 |
| Radiation type | Mo $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 3.10 |
| Crystal size (mm) | 0.20 × 0.12 × 0.05 × 0.04 (radius) |
| Data collection | |
| Diffractometer | Stoe Stadivari |
| Absorption correction | Multi-scan (*LANA*; Koziskova *et al.*, 2016) |
| $T_{min}$, $T_{max}$ | 0.581, 0.710 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 20999, 5145, 2744 |
| $R_{int}$ | 0.069 |
| (sin $\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.756 |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.038, 0.069, 0.83 |
| No. of reflections | 5145 |
| No. of parameters | 210 |
| H-atom treatment | H-atom parameters constrained |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.53, −0.91 |

Computer programs: *X-AREA* (Stoe & Cie, 2024), *SHELXT* (Sheldrick, 2015*a*), *SHELXL* (Sheldrick, 2015*b*), *Mercury* (Macrae *et al.*, 2020) and *publCIF* (Westrip, 2010).

## Acknowledgements

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# full crystallographic data

*IUCrData* (2025). **10**, x250082 [https://doi.org/10.1107/S2414314625000823]

## Poly[[bis($\mu_4$-acetato-$\kappa^4O$:$O$:$O'$:$O'$)tetrakis($\mu_3$-acetato-$\kappa^3O$:$O$:$O'$)bis($\mu_2$-acetato-$\kappa^2O$:$O'$)bis($\mu_3$-hydroxido)pentanickel(II)] 2.60-hydrate]

**Maximilian Pfeiffer, Berthold Stöger and Matthias Weil**

**Poly[[bis($\mu_4$-acetato-$\kappa^4O$:$O$:$O'$:$O'$)tetrakis($\mu_3$-acetato-$\kappa^3O$:$O$:$O'$)bis($\mu_2$-acetato-$\kappa^2O$:$O'$)bis($\mu_3$-hydroxido)pentanickel(II)] 2.60-hydrate]**

*Crystal data*

$[Ni_5(C_2H_3O_2)_8(OH)_2]\cdot 2.60H2O$
$M_r$ = 846.80
Tetragonal, $I4_1/a$
$a$ = 23.3025 (11) Å
$c$ = 11.2648 (5) Å
$V$ = 6116.9 (6) Å$^3$
$Z$ = 8
$F(000)$ = 3456
$D_x$ = 1.839 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 9821 reflections
$\theta$ = 1.8–30.9°
$\mu$ = 3.10 mm$^{-1}$
$T$ = 100 K
Plate, light green
0.20 × 0.12 × 0.05 × 0.04 (radius) mm

*Data collection*

Stoe Stadivari diffractometer
Radiation source: Axo_Mo
Graded multilayer mirror monochromator
Detector resolution: 13.33 pixels mm$^{-1}$
rotation method, $\omega$ scans
Absorption correction: multi-scan (*LANA*; Koziskova *et al*., 2016)
$T_{min}$ = 0.581, $T_{max}$ = 0.710
20999 measured reflections
5145 independent reflections
2744 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.069
$\theta_{max}$ = 32.5°, $\theta_{min}$ = 2.0°
$h$ = −31→35
$k$ = −34→33
$l$ = −16→7

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.069
$S$ = 0.83
5145 reflections
210 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.026P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.53 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.91 e Å$^{-3}$

*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 (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| Ni1 | 1.000000 | 0.500000 | 0.500000 | 0.01941 (13) | |
| Ni2 | 0.87305 (2) | 0.53890 (2) | 0.49712 (3) | 0.01354 (8) | |
| Ni3 | 0.77923 (2) | 0.44682 (2) | 0.52555 (3) | 0.01217 (8) | |
| O1 | 0.90748 (9) | 0.59881 (9) | 0.60134 (16) | 0.0211 (5) | |
| O2 | 1.00011 (9) | 0.57249 (10) | 0.59387 (17) | 0.0284 (5) | |
| O3 | 0.94065 (8) | 0.53418 (9) | 0.37833 (14) | 0.0183 (5) | |
| O4 | 0.88695 (8) | 0.53130 (9) | 0.21325 (15) | 0.0214 (5) | |
| O5 | 0.92122 (8) | 0.47408 (9) | 0.57859 (15) | 0.0191 (5) | |
| O6 | 0.84795 (8) | 0.42100 (8) | 0.63759 (14) | 0.0134 (4) | |
| O7 | 0.70649 (8) | 0.46180 (8) | 0.42844 (15) | 0.0178 (4) | |
| O8 | 0.72147 (8) | 0.54712 (8) | 0.34548 (14) | 0.0136 (4) | |
| O9 | 0.83183 (8) | 0.47852 (8) | 0.40476 (14) | 0.0122 (4) | |
| H9 | 0.858792 | 0.448659 | 0.373873 | 0.018* | |
| OW1 | 0.8698 (2) | 0.37614 (17) | 0.2711 (4) | 0.0573 (14) | 0.699 (5) |
| OW2 | 0.8781 (4) | 0.3908 (4) | 0.0907 (8) | 0.070 (4) | 0.301 (5) |
| OW3 | 0.8977 (4) | 0.3816 (4) | 0.3396 (8) | 0.041 (3) | 0.301 (5) |
| C1 | 0.97488 (17) | 0.65457 (18) | 0.7052 (4) | 0.0710 (15) | |
| H1A | 0.965806 | 0.690456 | 0.663914 | 0.106* | |
| H1B | 1.015970 | 0.653458 | 0.723552 | 0.106* | |
| H1C | 0.952762 | 0.652406 | 0.779061 | 0.106* | |
| C2 | 0.95967 (16) | 0.60452 (16) | 0.6271 (3) | 0.0316 (8) | |
| C3 | 0.98514 (14) | 0.51384 (18) | 0.1903 (3) | 0.0435 (11) | |
| H3A | 0.994597 | 0.547377 | 0.141613 | 0.065* | |
| H3B | 0.976865 | 0.481045 | 0.138644 | 0.065* | |
| H3C | 1.017715 | 0.504596 | 0.241930 | 0.065* | |
| C4 | 0.93369 (12) | 0.52682 (13) | 0.2648 (2) | 0.0205 (6) | |
| C5 | 0.94067 (13) | 0.37866 (14) | 0.6475 (3) | 0.0338 (9) | |
| H5A | 0.938084 | 0.369374 | 0.732144 | 0.051* | |
| H5B | 0.980102 | 0.389891 | 0.628096 | 0.051* | |
| H5C | 0.929924 | 0.344947 | 0.600447 | 0.051* | |
| C6 | 0.90074 (13) | 0.42727 (13) | 0.6195 (2) | 0.0183 (7) | |
| C7 | 0.62615 (14) | 0.52147 (16) | 0.4064 (3) | 0.0487 (11) | |
| H7A | 0.618561 | 0.561801 | 0.387364 | 0.073* | |
| H7B | 0.614544 | 0.513707 | 0.488435 | 0.073* | |
| H7C | 0.604258 | 0.496792 | 0.352400 | 0.073* | |
| C8 | 0.68878 (12) | 0.50943 (13) | 0.3925 (2) | 0.0167 (6) | |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Ni1 | 0.0095 (3) | 0.0283 (3) | 0.0204 (3) | −0.0013 (2) | −0.0010 (2) | 0.0099 (2) |
| Ni2 | 0.01025 (19) | 0.0152 (2) | 0.01523 (16) | −0.00078 (14) | −0.00080 (15) | 0.00298 (15) |
| Ni3 | 0.0127 (2) | 0.01059 (19) | 0.01326 (15) | −0.00031 (14) | 0.00098 (14) | 0.00057 (14) |
| O1 | 0.0166 (12) | 0.0224 (13) | 0.0244 (10) | −0.0019 (9) | −0.0067 (9) | −0.0002 (9) |
| O2 | 0.0157 (13) | 0.0348 (15) | 0.0348 (12) | −0.0056 (10) | −0.0080 (10) | 0.0042 (11) |

| | | | | | | |
|---|---|---|---|---|---|---|
| O3 | 0.0107 (11) | 0.0305 (13) | 0.0138 (9) | −0.0011 (9) | −0.0005 (8) | 0.0093 (9) |
| O4 | 0.0134 (11) | 0.0343 (14) | 0.0165 (9) | 0.0008 (9) | −0.0017 (8) | 0.0020 (9) |
| O5 | 0.0110 (11) | 0.0244 (13) | 0.0219 (10) | −0.0022 (9) | −0.0023 (8) | 0.0117 (9) |
| O6 | 0.0107 (11) | 0.0138 (11) | 0.0157 (9) | −0.0002 (8) | 0.0016 (8) | 0.0037 (8) |
| O7 | 0.0201 (12) | 0.0142 (11) | 0.0192 (9) | −0.0033 (9) | −0.0056 (9) | 0.0021 (9) |
| O8 | 0.0114 (11) | 0.0141 (11) | 0.0154 (8) | −0.0020 (8) | 0.0005 (8) | 0.0001 (8) |
| O9 | 0.0125 (11) | 0.0103 (10) | 0.0138 (8) | 0.0021 (8) | 0.0014 (8) | 0.0005 (8) |
| OW1 | 0.062 (3) | 0.050 (3) | 0.060 (3) | 0.013 (2) | 0.009 (3) | 0.001 (2) |
| OW2 | 0.057 (8) | 0.087 (9) | 0.065 (6) | 0.037 (6) | −0.022 (5) | −0.016 (6) |
| OW3 | 0.044 (6) | 0.033 (5) | 0.047 (5) | 0.016 (4) | −0.029 (4) | −0.020 (4) |
| C1 | 0.049 (3) | 0.060 (3) | 0.104 (3) | 0.001 (2) | −0.044 (3) | −0.038 (3) |
| C2 | 0.034 (2) | 0.031 (2) | 0.0298 (18) | −0.0104 (17) | −0.0141 (16) | 0.0046 (15) |
| C3 | 0.019 (2) | 0.087 (3) | 0.0240 (16) | 0.0062 (19) | 0.0037 (15) | 0.0094 (19) |
| C4 | 0.0133 (15) | 0.0274 (18) | 0.0208 (14) | 0.0023 (12) | 0.0019 (13) | 0.0057 (13) |
| C5 | 0.0184 (19) | 0.036 (2) | 0.0472 (19) | 0.0124 (15) | 0.0106 (16) | 0.0220 (17) |
| C6 | 0.0160 (17) | 0.0234 (18) | 0.0154 (13) | 0.0011 (13) | 0.0001 (12) | 0.0036 (12) |
| C7 | 0.015 (2) | 0.056 (3) | 0.075 (3) | 0.0044 (18) | 0.0072 (19) | 0.044 (2) |
| C8 | 0.0114 (15) | 0.0257 (18) | 0.0129 (12) | 0.0006 (13) | −0.0030 (11) | 0.0013 (12) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Ni1—O2$^{i}$ | 1.993 (2) | O5—C6 | 1.277 (3) |
| Ni1—O2 | 1.993 (2) | O6—C6 | 1.255 (3) |
| Ni1—O3$^{i}$ | 2.1037 (18) | O7—C8 | 1.251 (3) |
| Ni1—O3 | 2.1037 (18) | O8—C8 | 1.278 (3) |
| Ni1—O5 | 2.1257 (19) | O9—H9 | 1.0000 |
| Ni1—O5$^{i}$ | 2.1257 (19) | C1—H1A | 0.9800 |
| Ni2—O1 | 1.993 (2) | C1—H1B | 0.9800 |
| Ni2—O9 | 1.9962 (18) | C1—H1C | 0.9800 |
| Ni2—O3 | 2.0698 (18) | C1—C2 | 1.503 (5) |
| Ni2—O5 | 2.0937 (19) | C3—H3A | 0.9800 |
| Ni2—O8$^{ii}$ | 2.1016 (18) | C3—H3B | 0.9800 |
| Ni2—O6$^{iii}$ | 2.1140 (18) | C3—H3C | 0.9800 |
| Ni3—O9 | 1.9747 (18) | C3—C4 | 1.494 (4) |
| Ni3—O9$^{ii}$ | 1.9902 (17) | C5—H5A | 0.9800 |
| Ni3—O7 | 2.0474 (19) | C5—H5B | 0.9800 |
| Ni3—O4$^{ii}$ | 2.0750 (19) | C5—H5C | 0.9800 |
| Ni3—O8$^{ii}$ | 2.1023 (19) | C5—C6 | 1.499 (4) |
| Ni3—O6 | 2.1259 (18) | C7—H7A | 0.9800 |
| O1—C2 | 1.257 (4) | C7—H7B | 0.9800 |
| O2—C2 | 1.259 (4) | C7—H7C | 0.9800 |
| O3—C4 | 1.301 (3) | C7—C8 | 1.495 (4) |
| O4—C4 | 1.238 (3) | | |
| | | | |
| O2$^{i}$—Ni1—O2 | 180.0 | C6—O5—Ni1 | 135.72 (19) |
| O2—Ni1—O3 | 91.47 (8) | C6—O5—Ni2 | 125.03 (18) |
| O2—Ni1—O3$^{i}$ | 88.53 (8) | Ni2$^{ii}$—O6—Ni3 | 89.64 (7) |
| O2$^{i}$—Ni1—O3 | 88.53 (8) | C6—O6—Ni2$^{ii}$ | 142.11 (18) |

| | | | |
|---|---|---|---|
| O2$^{i}$—Ni1—O3$^{i}$ | 91.47 (8) | C6—O6—Ni3 | 127.48 (17) |
| O2—Ni1—O5 | 91.20 (8) | C8—O7—Ni3 | 126.65 (19) |
| O2—Ni1—O5$^{i}$ | 88.80 (8) | Ni2$^{iii}$—O8—Ni3$^{iii}$ | 94.22 (7) |
| O2$^{i}$—Ni1—O5$^{i}$ | 91.20 (8) | C8—O8—Ni2$^{iii}$ | 136.79 (19) |
| O2$^{i}$—Ni1—O5 | 88.80 (8) | C8—O8—Ni3$^{iii}$ | 123.99 (18) |
| O3—Ni1—O3$^{i}$ | 180.0 | Ni2—O9—H9 | 111.7 |
| O3$^{i}$—Ni1—O5 | 100.88 (7) | Ni3—O9—Ni2 | 101.72 (7) |
| O3—Ni1—O5 | 79.12 (7) | Ni3$^{iii}$—O9—Ni2 | 97.13 (8) |
| O3$^{i}$—Ni1—O5$^{i}$ | 79.12 (7) | Ni3—O9—Ni3$^{iii}$ | 121.18 (9) |
| O3—Ni1—O5$^{i}$ | 100.89 (7) | Ni3—O9—H9 | 111.7 |
| O5—Ni1—O5$^{i}$ | 180.0 | Ni3$^{iii}$—O9—H9 | 111.7 |
| O1—Ni2—O3 | 96.42 (8) | H1A—C1—H1B | 109.5 |
| O1—Ni2—O5 | 91.80 (8) | H1A—C1—H1C | 109.5 |
| O1—Ni2—O6$^{iii}$ | 94.78 (7) | H1B—C1—H1C | 109.5 |
| O1—Ni2—O8$^{ii}$ | 96.25 (8) | C2—C1—H1A | 109.5 |
| O1—Ni2—O9 | 174.04 (8) | C2—C1—H1B | 109.5 |
| O3—Ni2—O5 | 80.62 (7) | C2—C1—H1C | 109.5 |
| O3—Ni2—O6$^{iii}$ | 91.54 (7) | O1—C2—O2 | 126.4 (3) |
| O3—Ni2—O8$^{ii}$ | 167.29 (7) | O1—C2—C1 | 116.4 (3) |
| O5—Ni2—O6$^{iii}$ | 170.29 (7) | O2—C2—C1 | 117.2 (3) |
| O5—Ni2—O8$^{ii}$ | 97.89 (7) | H3A—C3—H3B | 109.5 |
| O8$^{ii}$—Ni2—O6$^{iii}$ | 88.49 (7) | H3A—C3—H3C | 109.5 |
| O9—Ni2—O3 | 89.53 (7) | H3B—C3—H3C | 109.5 |
| O9—Ni2—O5 | 88.74 (7) | C4—C3—H3A | 109.5 |
| O9—Ni2—O6$^{iii}$ | 85.46 (7) | C4—C3—H3B | 109.5 |
| O9—Ni2—O8$^{ii}$ | 77.80 (7) | C4—C3—H3C | 109.5 |
| O4$^{ii}$—Ni3—O6 | 85.24 (7) | O3—C4—C3 | 118.6 (3) |
| O4$^{ii}$—Ni3—O8$^{ii}$ | 166.95 (8) | O4—C4—O3 | 124.0 (3) |
| O7—Ni3—O4$^{ii}$ | 89.94 (8) | O4—C4—C3 | 117.4 (2) |
| O7—Ni3—O6 | 171.54 (7) | H5A—C5—H5B | 109.5 |
| O7—Ni3—O8$^{ii}$ | 102.12 (7) | H5A—C5—H5C | 109.5 |
| O8$^{ii}$—Ni3—O6 | 83.37 (7) | H5B—C5—H5C | 109.5 |
| O9$^{ii}$—Ni3—O4$^{ii}$ | 86.88 (7) | C6—C5—H5A | 109.5 |
| O9—Ni3—O4$^{ii}$ | 95.99 (7) | C6—C5—H5B | 109.5 |
| O9—Ni3—O6 | 92.72 (7) | C6—C5—H5C | 109.5 |
| O9$^{ii}$—Ni3—O6 | 85.30 (7) | O5—C6—C5 | 119.3 (3) |
| O9—Ni3—O7 | 94.70 (7) | O6—C6—O5 | 121.6 (3) |
| O9$^{ii}$—Ni3—O7 | 87.50 (7) | O6—C6—C5 | 119.1 (3) |
| O9—Ni3—O8$^{ii}$ | 78.25 (7) | H7A—C7—H7B | 109.5 |
| O9$^{ii}$—Ni3—O8$^{ii}$ | 98.49 (7) | H7A—C7—H7C | 109.5 |
| O9—Ni3—O9$^{ii}$ | 176.38 (9) | H7B—C7—H7C | 109.5 |
| C2—O1—Ni2 | 126.8 (2) | C8—C7—H7A | 109.5 |
| C2—O2—Ni1 | 131.2 (2) | C8—C7—H7B | 109.5 |
| Ni2—O3—Ni1 | 95.69 (7) | C8—C7—H7C | 109.5 |
| C4—O3—Ni1 | 132.29 (19) | O7—C8—O8 | 123.2 (3) |
| C4—O3—Ni2 | 123.22 (18) | O7—C8—C7 | 117.0 (3) |
| C4—O4—Ni3$^{iii}$ | 131.83 (17) | O8—C8—C7 | 119.8 (3) |
| Ni2—O5—Ni1 | 94.33 (7) | | |

| | | | |
|---|---|---|---|
| Ni1—O2—C2—O1 | −4.1 (5) | Ni2$^{ii}$—O6—C6—O5 | −150.4 (2) |
| Ni1—O2—C2—C1 | 175.8 (2) | Ni2$^{ii}$—O6—C6—C5 | 29.1 (5) |
| Ni1—O3—C4—O4 | 151.5 (2) | Ni2$^{iii}$—O8—C8—O7 | 166.11 (17) |
| Ni1—O3—C4—C3 | −30.4 (4) | Ni2$^{iii}$—O8—C8—C7 | −13.8 (4) |
| Ni1—O5—C6—O6 | −164.02 (18) | Ni3$^{iii}$—O4—C4—O3 | −18.6 (5) |
| Ni1—O5—C6—C5 | 16.4 (4) | Ni3$^{iii}$—O4—C4—C3 | 163.2 (2) |
| Ni2—O1—C2—O2 | −1.8 (5) | Ni3—O6—C6—O5 | 43.1 (4) |
| Ni2—O1—C2—C1 | 178.3 (2) | Ni3—O6—C6—C5 | −137.4 (2) |
| Ni2—O3—C4—O4 | 12.4 (4) | Ni3—O7—C8—O8 | 44.2 (3) |
| Ni2—O3—C4—C3 | −169.5 (2) | Ni3—O7—C8—C7 | −135.9 (2) |
| Ni2—O5—C6—O6 | −15.7 (4) | Ni3$^{iii}$—O8—C8—O7 | 18.2 (4) |
| Ni2—O5—C6—C5 | 164.8 (2) | Ni3$^{iii}$—O8—C8—C7 | −161.7 (2) |

Symmetry codes: (i) −*x*+2, −*y*+1, −*z*+1; (ii) *y*+1/4, −*x*+5/4, *z*+1/4; (iii) −*y*+5/4, *x*−1/4, *z*−1/4.

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O9—H9···O*W*1 | 1.00 | 2.06 | 2.957 (4) | 147 |
| O9—H9···O*W*3 | 1.00 | 1.85 | 2.827 (8) | 166 |
| O7···O*W*2$^{iii}$ | | | 2.87 | |
| O*W*2···O*W*3 | | | 2.85 | |

Symmetry code: (iii) −*y*+5/4, *x*−1/4, *z*−1/4.