# metal-organic compounds

# Diaquabis[*N*-(2-fluorobenzyl)-*N*-nitroso-hydroxylaminato-$\kappa^2 O,O'$]nickel(II)

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In the centrosymmetric title compound, $[Ni(C_7H_6FN_2O_2)_2(H_2O)_2]$, the Ni$^{II}$ cation is in a slightly distorted octahedral environment and is surrounded by four O atoms from the N—O groups of the organic ligands [Ni—O = 2.0179 (13) and 2.0283 (12) Å], and two water molecules [Ni—O = 2.0967 (14) Å]. The *N*-(2-fluorobenzyl)-*N*-nitrosohydroxy-laminate monoanions act as bidentate chelating ligands. In the crystal, the Ni cations in the columns are shifted in such a way that the coordinated water molecules are involved in the formation of hydrogen bonds with the O atoms of the organic species of neighbouring molecules. Thus, a two-dimensional network parallel to (100) is built up by hydrogen-bonded molecules.

## Related literature

For the synthesis of the potassium *N*-(2-fluorobenzyl)-*N*-nitrosohydroxylaminate salt, see: Zyuzin *et al.* (1997) and of the Ni complex of *N*-(2-fluorobenzyl)-*N*-nitrosohydroxy-laminate, see: Kovalchukova *et al.* (2013). For the structures of some 3d-metal complexes with *N*-nitrosohydroxylamine derivatives, see: Deák *et al.* (1998); Okabe & Tamaki (1995); Tamaki & Okabe (1996, 1998). For the synthesis, properties and applications of other metal nitrosohydroxylaminates, see: Okabe *et al.* (1995); Abraham *et al.* (1987); Venter *et al.* (2009); Popov & Wendlandt (1954); Lundell & Knowles (1920); Buscarons & Canela (1974); Oztekin & Erim (2000); Yi *et al.* (1995); McGill *et al.* (2000); Shiino *et al.* (2001).
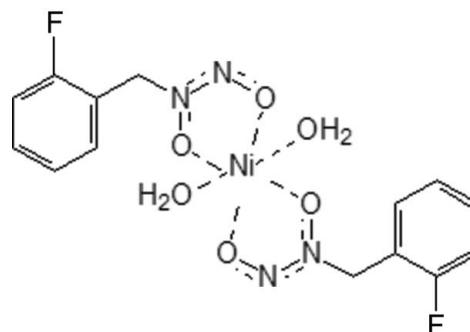


## Experimental

### Crystal data

| | |
|---|---|
| $[Ni(C_7H_6FN_2O_2)_2(H_2O)_2]$ | $V$ = 847.5 (3) Å$^3$ |
| $M_r$ = 433.02 | $Z$ = 2 |
| Monoclinic, $P2_1/c$ | Mo $K\alpha$ radiation |
| $a$ = 15.411 (3) Å | $\mu$ = 1.21 mm$^{-1}$ |
| $b$ = 7.235 (1) Å | $T$ = 293 K |
| $c$ = 7.604 (1) Å | $0.75 \times 0.20 \times 0.05$ mm |
| $\beta$ = 91.65 (3)° | |

### Data collection

| | |
|---|---|
| Enraf–Nonius CAD-4 diffractometer | 1703 measured reflections |
| Absorption correction: part of the refinement model ($\Delta F$) (Walker & Stuart, 1983) | 1571 independent reflections |
| | 1181 reflections with $I > 2\sigma(I)$ |
| | $R_{int}$ = 0.022 |
| $T_{min}$ = 0.427, $T_{max}$ = 0.809 | 3 standard reflections every 60 min intensity decay: 0.0% |

### Refinement

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)]$ = 0.022 | H atoms treated by a mixture of independent and constrained refinement |
| $wR(F^2)$ = 0.066 | |
| $S$ = 1.01 | |
| 1571 reflections | $\Delta\rho_{max}$ = 0.31 e Å$^{-3}$ |
| 132 parameters | $\Delta\rho_{min}$ = −0.32 e Å$^{-3}$ |
| 2 restraints | |

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O3—H31···O1$^i$ | 0.84 (1) | 1.97 (1) | 2.7987 (18) | 169 (3) |
| O3—H32···O2$^{ii}$ | 0.84 (1) | 1.98 (1) | 2.8078 (18) | 170 (2) |

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

Data collection: *CAD-4-PC* (Enraf–Nonius, 1993); cell refinement: *CAD-4-PC*; data reduction: *CAD-4-PC*; 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: *CIFTAB97* (Sheldrick, 2008) and *SHELX97*.

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# Diaquabis[*N*-(2-fluorobenzyl)-*N*-nitrosohydroxylaminato-*κ²O,O′*]nickel(II)

## Olga Kovalchukova, Ali Sheikh Bostanabad, Adam Stash, Svetlana Strashnova and Igor Zyuzin

## 1. Comment

*N*-nitrosohydroxylamine derivatives are good chelating agents which form stable complexes with a wide range of metal ions (Okabe *et al.*, 1995; Abraham *et al.*, 1987; Venter *et al.*, 2009; Popov & Wendlandt, 1954). The phenyl and naphthyl derivatives, known as cupferron and neocupferron, are reported as good analytical reagents for the determination of zirconium in its ores and metallurgical products, as well as for the separation of iron and titanium from manganese and aluminum in limestone analysis, separation and direct UV detection of lanthanides and other analyses (Lundell & Knowles, 1920; Buscarons & Canela, 1974; Oztekin & Erim, 2000). $R_2N[N_2O_2]$ anions are smooth nonenzymatic releasers of nitric oxide in physiological media (Yi *et al.*, 1995; McGill *et al.*, 2000) and possess the property of inhibition of mushroom tyrosinase (Shiino *et al.*, 2001).

In the title compound, $C_{14}H_{16}F_2N_4NiO_6$, the Ni cation of the centrosymmetrical structure is in a slightly distorted octahedral coordination and is surrounded by four oxo O atoms of the N—O groups of the organic ligands [Ni—O = 2.0179 (13) and 2.0283 (12) Å], and two water molecules in the axial positions [Ni—O = 2.0967 (14) Å]. The described coordination type of the central atom correlates with those described previously for the dimethanolo-bis(N– nitroso-*N*-phenyl-hydroxylaminato-o,o) cobalt(II) (Deak *et al.*, 1998) and the dimethanolo-bis (*N*-nitroso-*N*-phenylhydroxy-laminato-O,O′)nickel(II) (Okabe & Tamaki, 1995). On the other hand, in the reported structure of the diaquabis[*N*-(1-naphthyl)-*N*-nitrosohydroxylaminato- O, O′] cobalt(II) (Tamaki & Okabe, 1998), the two coordinated water molecules are in the *cis* arrangement. In addition in the bis(*N*-nitroso-*N*-phenylhydroxylaminato)manganese, or manganese cupferronate (Tamaki & Okabe, 1996), the saturation of the coordination sphere of the Mn(II) cation occurs with the two O atoms of the nitroso groups of two adjacent cupferron ligands. The *N*-(2-fluorobenzyl)-*N*-nitrosohydroxylaminate monoanions act as bidentate chelating ligands. The Ni cations in the columns are shifted in such a way that the coordinated $H_2O$ molecules are involved in the formation of hydrogen bonds with the O atoms of the organic species of the neighbouring molecules in the columns. Thus, the crystal lattice of the reported structure is a supramolecular architecture built up by infinite one dimensional chains of hydrogen bonded molecules.

## 2. Experimental

The potassium *N*-(2-fluorobenzyl)-*N*-nitrosohydroxylaminate salt and its Ni complex was prepared (see Fig. 3) according to procedures described previously (Zyuzin *et al.*, 1997; Kovalchukova *et al.*, 2013). Single crystals of $C_{14}H_{16}F_2N_4O_6$ were grown by the slow evaporation of the ethanol solution of the diaquabis[N-(2-fluorobenzyl)-*N*-nitrosohydroxy-laminato-*O,O′*]nickel(II) powdered sample.

## 3. Refinement

The structure of of $C_{14}H_{16}F_2N_4O_6$ was solved by direct method and all non-hydrogen atoms were located and refined in anisotropically. All the hydrogen atoms were located in difference electron density syntheses and included in refinement

with fixed parameters.

## Computing details

Data collection: *CAD-4-PC* (Enraf–Nonius, 1993); cell refinement: *CAD-4-PC* (Enraf–Nonius, 1993); data reduction: *CAD-4-PC* (Enraf–Nonius, 1993); 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: *CIFTAB97* and *SHELX97* (Sheldrick, 2008).
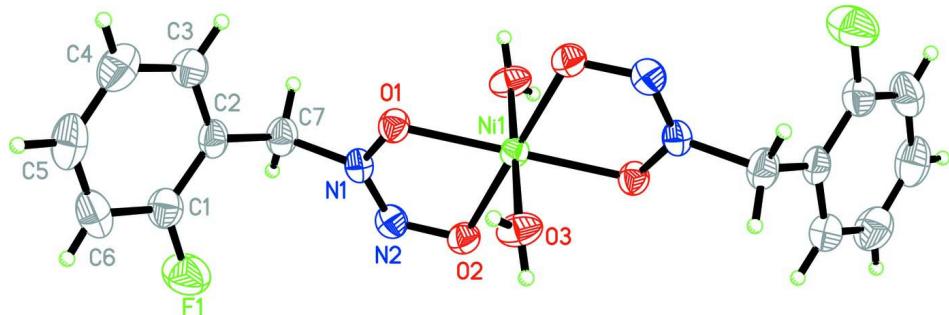


**Figure 1**

*ORTEP* view of $C_{14}H_{16}F_2N_4O_6$ with atom labeling scheme (displacement ellipsoids are drawn at the 50% probability level for non-hydrogen atoms).
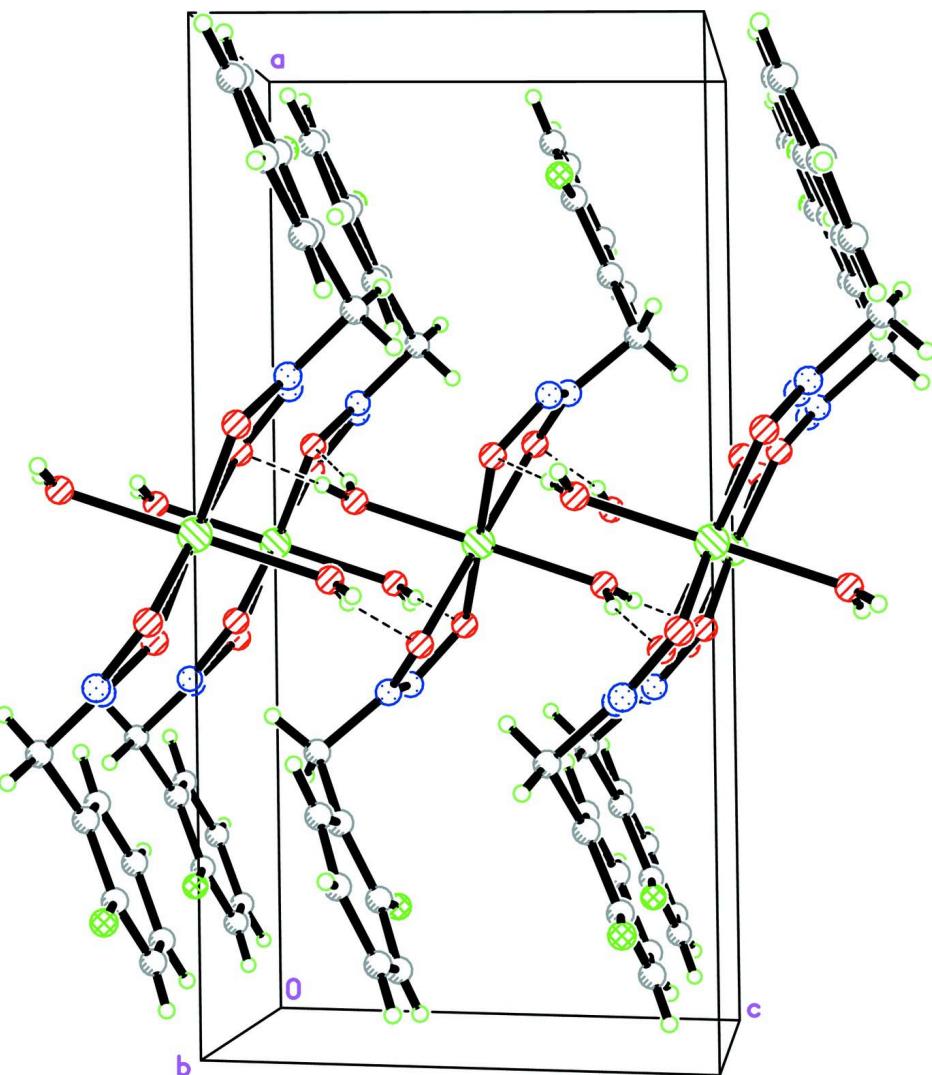
**Figure 2**

Molecular packing in the crystal of the complex along the crystallographic axis b.
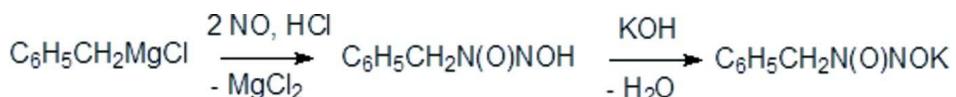


**Figure 3**

The synthesis of the potassium *N*-(2-fluorobenzyl)-Nnitrosohydroxylaminate salt

## Diaquabis[*N*-(2-fluorobenzyl)-*N*-nitrosohydroxylaminato-*κ²O,O′*]nickel(II)

*Crystal data*

| | |
|---|---|
| [Ni(C$_7$H$_6$FN$_2$O$_2$)$_2$(H$_2$O)$_2$] | $V = 847.5$ (3) Å$^3$ |
| $M_r = 433.02$ | $Z = 2$ |
| Monoclinic, $P2_1/c$ | $F(000) = 444$ |
| $a = 15.411$ (3) Å | $D_x = 1.697$ Mg m$^{-3}$ |
| $b = 7.235$ (1) Å | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| $c = 7.604$ (1) Å | Cell parameters from 24 reflections |
| $\beta = 91.65$ (3)° | $\theta = 10.9$–12.5° |

$\mu = 1.21$ mm$^{-1}$

$T = 293$ K

Plate, green

$0.75 \times 0.20 \times 0.05$ mm

*Data collection*

| | |
|---|---|
| Enraf–Nonius CAD-4 diffractometer | 1571 independent reflections |
| Radiation source: fine-focus tube | 1181 reflections with $I > 2\sigma(I)$ |
| $\beta$-filter monochromator | $R_{int} = 0.022$ |
| $\omega/2\theta$ scans | $\theta_{max} = 25.5°$, $\theta_{min} = 2.6°$ |
| Absorption correction: part of the refinement model ($\Delta F$) | $h = -18 \rightarrow 18$ |
| | $k = 0 \rightarrow 8$ |
| (Walker & Stuart, 1983) | $l = 0 \rightarrow 9$ |
| $T_{min} = 0.427$, $T_{max} = 0.809$ | 3 standard reflections every 60 min |
| 1703 measured reflections | intensity decay: 0.0% |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | |
| $R[F^2 > 2\sigma(F^2)] = 0.022$ | Hydrogen site location: difference Fourier map |
| $wR(F^2) = 0.066$ | H atoms treated by a mixture of independent and constrained refinement |
| $S = 1.01$ | |
| 1571 reflections | $w = 1/[\sigma^2(F_o^2) + (0.0471P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| 132 parameters | |
| 2 restraints | $(\Delta/\sigma)_{max} < 0.001$ |
| Primary atom site location: structure-invariant direct methods | $\Delta\rho_{max} = 0.31$ e Å$^{-3}$ |
| | $\Delta\rho_{min} = -0.32$ 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 | 0.5000 | 0.5000 | 0.5000 | 0.02414 (12) |
| F1 | 0.12983 (10) | 0.46369 (18) | 0.3199 (2) | 0.0610 (4) |
| O1 | 0.39701 (8) | 0.63578 (16) | 0.39297 (15) | 0.0292 (3) |
| O2 | 0.41642 (8) | 0.28946 (16) | 0.44659 (16) | 0.0312 (3) |
| N1 | 0.34766 (9) | 0.51068 (19) | 0.31185 (19) | 0.0278 (3) |
| N2 | 0.35419 (10) | 0.3370 (2) | 0.3362 (2) | 0.0317 (3) |
| C1 | 0.14337 (13) | 0.6479 (3) | 0.3049 (3) | 0.0386 (4) |
| C2 | 0.22021 (12) | 0.7072 (3) | 0.2360 (2) | 0.0329 (4) |
| C3 | 0.23158 (14) | 0.8967 (3) | 0.2205 (3) | 0.0406 (5) |
| H3 | 0.2824 | 0.9423 | 0.1738 | 0.049* |
| C4 | 0.16858 (17) | 1.0184 (3) | 0.2733 (3) | 0.0519 (6) |
| H4 | 0.1773 | 1.1451 | 0.2629 | 0.062* |
| C5 | 0.09283 (16) | 0.9527 (3) | 0.3412 (3) | 0.0531 (6) |

| | | | | |
|---|---|---|---|---|
| H5 | 0.0504 | 1.0354 | 0.3760 | 0.064* |
| C6 | 0.07923 (13) | 0.7649 (3) | 0.3581 (3) | 0.0477 (5) |
| H6 | 0.0282 | 0.7193 | 0.4041 | 0.057* |
| C7 | 0.28684 (13) | 0.5742 (3) | 0.1721 (2) | 0.0369 (4) |
| H71 | 0.2575 | 0.4679 | 0.1206 | 0.044* |
| H72 | 0.3194 | 0.6333 | 0.0805 | 0.044* |
| O3 | 0.45896 (10) | 0.53062 (17) | 0.75889 (18) | 0.0371 (3) |
| H31 | 0.4344 (17) | 0.628 (3) | 0.790 (4) | 0.075 (9)* |
| H32 | 0.4418 (15) | 0.443 (2) | 0.821 (3) | 0.052 (7)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Ni1 | 0.02426 (18) | 0.02405 (17) | 0.02409 (17) | 0.00166 (12) | 0.00050 (11) | 0.00039 (11) |
| F1 | 0.0577 (8) | 0.0447 (7) | 0.0812 (10) | −0.0079 (6) | 0.0135 (7) | −0.0001 (6) |
| O1 | 0.0292 (6) | 0.0261 (5) | 0.0321 (6) | 0.0014 (5) | −0.0027 (5) | −0.0010 (5) |
| O2 | 0.0330 (7) | 0.0271 (6) | 0.0335 (6) | −0.0003 (5) | −0.0004 (5) | 0.0022 (5) |
| N1 | 0.0243 (7) | 0.0317 (7) | 0.0274 (7) | 0.0030 (6) | −0.0003 (6) | −0.0027 (6) |
| N2 | 0.0304 (8) | 0.0317 (8) | 0.0331 (7) | 0.0006 (6) | 0.0021 (6) | −0.0030 (6) |
| C1 | 0.0366 (11) | 0.0405 (10) | 0.0382 (10) | 0.0008 (8) | −0.0049 (8) | −0.0023 (8) |
| C2 | 0.0286 (9) | 0.0419 (10) | 0.0279 (9) | 0.0051 (7) | −0.0059 (7) | −0.0005 (7) |
| C3 | 0.0381 (11) | 0.0441 (11) | 0.0394 (10) | 0.0001 (8) | −0.0046 (9) | 0.0047 (8) |
| C4 | 0.0595 (14) | 0.0403 (11) | 0.0553 (13) | 0.0083 (10) | −0.0067 (11) | 0.0006 (9) |
| C5 | 0.0494 (13) | 0.0590 (14) | 0.0504 (13) | 0.0228 (11) | −0.0050 (10) | −0.0083 (10) |
| C6 | 0.0317 (11) | 0.0679 (15) | 0.0434 (11) | 0.0052 (10) | 0.0013 (9) | −0.0062 (10) |
| C7 | 0.0351 (10) | 0.0484 (10) | 0.0269 (9) | 0.0077 (9) | −0.0029 (8) | −0.0001 (8) |
| O3 | 0.0518 (8) | 0.0300 (7) | 0.0303 (6) | 0.0022 (6) | 0.0127 (6) | 0.0008 (5) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Ni1—O1i | 2.0179 (13) | C2—C3 | 1.388 (3) |
| Ni1—O1 | 2.0179 (13) | C2—C7 | 1.498 (3) |
| Ni1—O2 | 2.0283 (12) | C3—C4 | 1.379 (3) |
| Ni1—O2i | 2.0283 (12) | C3—H3 | 0.9300 |
| Ni1—O3 | 2.0967 (14) | C4—C5 | 1.375 (4) |
| Ni1—O3i | 2.0967 (14) | C4—H4 | 0.9300 |
| F1—C1 | 1.354 (2) | C5—C6 | 1.381 (3) |
| O1—N1 | 1.3233 (19) | C5—H5 | 0.9300 |
| O2—N2 | 1.302 (2) | C6—H6 | 0.9300 |
| N1—N2 | 1.274 (2) | C7—H71 | 0.9700 |
| N1—C7 | 1.470 (2) | C7—H72 | 0.9700 |
| C1—C6 | 1.371 (3) | O3—H31 | 0.838 (10) |
| C1—C2 | 1.377 (3) | O3—H32 | 0.837 (10) |
| | | | |
| O1i—Ni1—O1 | 180.0 | C1—C2—C3 | 116.90 (18) |
| O1i—Ni1—O2 | 101.70 (5) | C1—C2—C7 | 121.86 (17) |
| O1—Ni1—O2 | 78.30 (5) | C3—C2—C7 | 121.17 (18) |
| O1i—Ni1—O2i | 78.30 (5) | C4—C3—C2 | 121.0 (2) |
| O1—Ni1—O2i | 101.70 (5) | C4—C3—H3 | 119.5 |
| O2—Ni1—O2i | 180.0 | C2—C3—H3 | 119.5 |

| | | | |
|---|---|---|---|
| O1ⁱ—Ni1—O3 | 85.86 (6) | C5—C4—C3 | 120.1 (2) |
| O1—Ni1—O3 | 94.14 (6) | C5—C4—H4 | 120.0 |
| O2—Ni1—O3 | 93.46 (6) | C3—C4—H4 | 120.0 |
| O2ⁱ—Ni1—O3 | 86.54 (6) | C4—C5—C6 | 120.5 (2) |
| O1ⁱ—Ni1—O3ⁱ | 94.14 (6) | C4—C5—H5 | 119.7 |
| O1—Ni1—O3ⁱ | 85.86 (6) | C6—C5—H5 | 119.7 |
| O2—Ni1—O3ⁱ | 86.54 (6) | C1—C6—C5 | 117.8 (2) |
| O2ⁱ—Ni1—O3ⁱ | 93.46 (6) | C1—C6—H6 | 121.1 |
| O3—Ni1—O3ⁱ | 180.0 | C5—C6—H6 | 121.1 |
| N1—O1—Ni1 | 106.85 (9) | N1—C7—C2 | 113.27 (14) |
| N2—O2—Ni1 | 112.47 (10) | N1—C7—H71 | 108.9 |
| N2—N1—O1 | 124.40 (14) | C2—C7—H71 | 108.9 |
| N2—N1—C7 | 117.37 (14) | N1—C7—H72 | 108.9 |
| O1—N1—C7 | 117.96 (14) | C2—C7—H72 | 108.9 |
| N1—N2—O2 | 114.08 (14) | H71—C7—H72 | 107.7 |
| F1—C1—C6 | 117.94 (19) | Ni1—O3—H31 | 120 (2) |
| F1—C1—C2 | 118.37 (17) | Ni1—O3—H32 | 124.0 (18) |
| C6—C1—C2 | 123.69 (19) | H31—O3—H32 | 109 (3) |

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+1.

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O3—H31···O1ⁱⁱ | 0.84 (1) | 1.97 (1) | 2.7987 (18) | 169 (3) |
| O3—H32···O2ⁱⁱⁱ | 0.84 (1) | 1.98 (1) | 2.8078 (18) | 170 (2) |

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