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[μ-10,21-Dimethyl-3,6,14,17-tetraazatricyclo[17.3.1.1^{8,12}]tetracosa-1(23),8(24),9,11,19,21-hexaene-23,24-diolato-$\kappa^8N^3,N^6,O^{23},O^{24}$:$N^{14},N^{17},O^{23},O^{24}$]-bis[(nitrato-$\kappa^2O,O'$)nickel(II)]

Quan-Jun Li, Jian-Fang Ma,* Jie Liu and Ting-Ting Han

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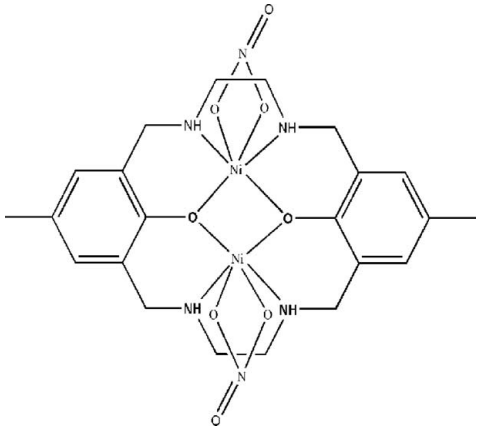
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.043; wR factor = 0.117; data-to-parameter ratio = 12.4.

In the title centrosymmetric dinuclear nickel complex, $[Ni_2(C_{22}H_{30}N_4O_2)(NO_3)_2]$, each of the two Ni^{II} atoms has a distorted octahedral geometry, defined by two N atoms and two O atoms from the macrocyclic ligand and two O atoms from a chelating nitrate anion. The two Ni atoms are bridged by two phenolate O atoms, forming a four-membered Ni_2O_2 ring.

Related literature

For general background, see: Caldwell & Crumbliss (1998); Rosa *et al.* (1998). For related structures, see: Aromi *et al.* (2002). For the ligand synthesis, see: Mandal & Nag (1986).



Experimental

Crystal data

$[Ni_2(C_{22}H_{30}N_4O_2)(NO_3)_2]$
M_r = 623.90
Trigonal, $R\overline{3}$
a = 25.020 (5) Å
c = 10.616 (5) Å
V = 5755 (3) Å^3
Z = 9
Mo $K\alpha$ radiation
μ = 1.53 mm^{-1}
T = 293 K
0.40 × 0.30 × 0.25 mm

Data collection

Bruker APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.495, T_{max} = 0.609
(expected range = 0.554–0.682)
9432 measured reflections
2213 independent reflections
1745 reflections with $I > 2\sigma(I)$
R_{int} = 0.107

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.117
S = 1.03
2213 reflections
178 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 1.06 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

Table 1
Selected bond lengths (Å).

Ni1—O1	2.000 (2)	Ni1—N1	2.054 (3)
Ni1—O1^{i}	2.006 (2)	Ni1—O3	2.134 (3)
Ni1—N2	2.038 (3)	Ni1—O2	2.183 (3)

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

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); 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: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HY2188).

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supporting information

Acta Cryst. (2009). E**65**, m522 [doi:10.1107/S1600536809010174]

[μ-10,21-Dimethyl-3,6,14,17-tetraazatricyclo-[17.3.1.1^{8,12}]tetracosa-1(23),8(24),9,11,19,21-hexaene-23,24-diolato-$\kappa^8N^3,N^6,O^{23},O^{24}$:$N^{14},N^{17},O^{23},O^{24}$]bis[(nitrato-$\kappa^2O,O'$)nickel(II)]

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S1. Comment

Crown ether compounds have attracted much interest as a result of their significance in biological transport systems (Caldwell & Crumbliss, 1998). In addition, crown ether compounds are found to have functions in selective molecular recognition (Rosa *et al.*, 1998). To further widen the scope of applications of crown ether, there is a need to prepare new series of crown ether compounds. In this work, a new dinuclear nickel(II) compound has been synthesized and its struture is reported here.

As shown in Fig. 1, the title compound is a centrosymmetric dinuclear nickel complex. The coordination environment around each Ni^{II} atom is distorted octahedral, with one N atom and one O atom from the macrocyclic ligand and two O atoms from the nitrate anion occupying the equatorial plane, and the other N atom and O atom from the ligand occupying the axial positions. In the complex molecule, two Ni atoms are bridged by two phenolate O atoms, generating a four-membered Ni_2O_2 ring, with a Ni···Ni distance of 2.9737 (10) Å. The Ni—O and Ni—N distances are normal (Aromi *et al.*, 2002).

S2. Experimental

The macrocyclic ligand, $C_{22}H_{32}N_4O_2$ (H_2L), was prepared by the reported procedure (Mandal & Nag, 1986). A mixture of H_2L (0.10 g, 0.26 mmol) and $Ni(NO_3)_2.6H_2O$ (0.15 g, 0.52 mmol) in methanol (20 ml) was stirred for 10 min. The resulting solution was filtered. Green single crystals were obtained by slow evaporation of the filtrate at room temperature (yield 56%).

S3. Refinement

H atoms bound to C atoms were positioned geometrically and refined using a riding model, with C—H = 0.93 (aromatic), 0.97 (CH_2) and 0.96 (CH_3) Å and with U_{iso} = 1.2(1.5 for methyl)U_{eq}(C). The imino H atoms were located in a difference Fourier map and refined with U_{iso}(H) = 0.128 Å^2. The highest residual electron density was found 1.03Å from Ni1 and the deepest hole 0.76 Å from Ni1.

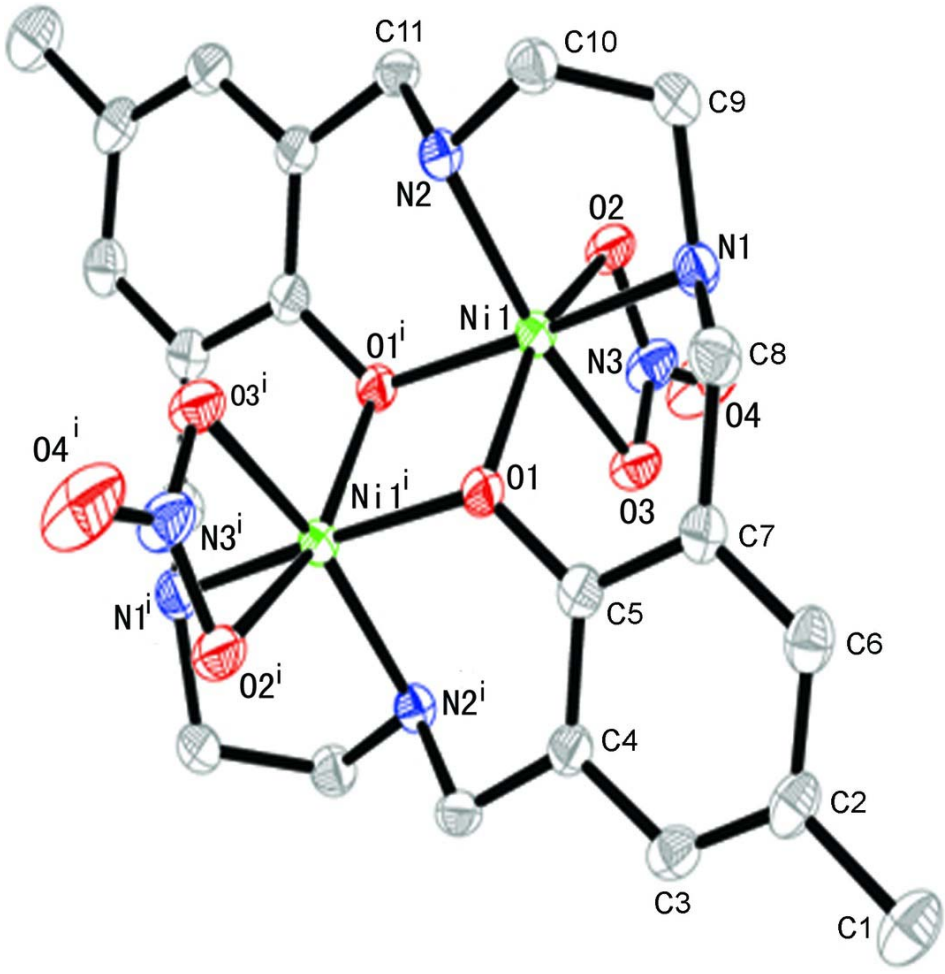

Figure 1

Molecular structure of the title compound. Displacement ellipsoids are draw at the 30% probability level. H atoms have been omitted for clarity. [Symmetry code: (i) -*x* + 2/3, -*y* + 1/3, -*z* + 1/3.]

[*μ*-10,21-Dimethyl-3,6,14,17-tetraazatricyclo[17.3.1.1^{8,12}]tetracosa- 1(23),8(24),9,11,19,21-hexaene-23,24-diolato- $\kappa^8N^3,N^6,O^{23},O^{24}$:$N^{14}$, N^{17},O^{23},O^{24}]bis[(nitrato- κ^2O,O')nickel(II)]

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a = 25.020 (5) Å
c = 10.616 (5) Å
V = 5755 (3) Å^3
Z = 9
F(000) = 2916
D_x = 1.620 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71069 Å
Cell parameters from 3000 reflections
θ = 2.4–28.4°
μ = 1.53 mm^{-1}
T = 293 K
Block, green
0.40 × 0.30 × 0.25 mm

Data collection

Bruker APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.495, T_{max} = 0.609
9432 measured reflections
2213 independent reflections
1745 reflections with $I > 2\sigma(I)$
R_{int} = 0.107
θ_{max} = 24.9°, θ_{min} = 1.6°
h = −15→29
k = −29→23
l = −12→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.117
S = 1.03
2213 reflections
178 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.066P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.007
$\Delta\rho_{max}$ = 1.06 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Ni1	0.355561 (19)	0.13534 (2)	0.25357 (4)	0.03150 (19)
C1	0.04882 (19)	−0.0620 (2)	0.3314 (4)	0.0658 (13)
H1A	0.0488	−0.0770	0.4147	0.099*
H1B	0.0372	−0.0948	0.2717	0.099*
H1C	0.0199	−0.0475	0.3276	0.099*
C2	0.11262 (17)	−0.00973 (17)	0.3005 (3)	0.0431 (9)
C3	0.12561 (16)	0.01995 (17)	0.1862 (3)	0.0410 (9)
H3	0.0947	0.0056	0.1255	0.049*
C4	0.18315 (15)	0.07079 (16)	0.1570 (3)	0.0353 (8)
C5	0.22847 (15)	0.09460 (15)	0.2510 (3)	0.0347 (8)
C6	0.16018 (17)	0.01062 (17)	0.3868 (3)	0.0446 (9)
H6	0.1536	−0.0111	0.4617	0.054*
C7	0.21738 (16)	0.06230 (16)	0.3656 (3)	0.0374 (8)
C8	0.26663 (17)	0.08766 (18)	0.4652 (3)	0.0443 (9)
H8A	0.2746	0.1284	0.4892	0.053*
H8B	0.2515	0.0614	0.5391	0.053*
C9	0.37778 (18)	0.13034 (17)	0.5104 (3)	0.0430 (9)
H9A	0.4112	0.1221	0.4949	0.052*
H9B	0.3646	0.1196	0.5972	0.052*
C10	0.40046 (18)	0.19842 (17)	0.4892 (3)	0.0421 (9)
H10A	0.3704	0.2085	0.5224	0.050*
H10B	0.4390	0.2230	0.5342	0.050*
C11	0.47350 (15)	0.23666 (16)	0.3077 (3)	0.0383 (8)
H11A	0.4824	0.2031	0.3091	0.046*

H11B	0.5022	0.2685	0.3644	0.046*
N1	0.32528 (14)	0.09201 (14)	0.4244 (3)	0.0377 (7)
N2	0.41009 (14)	0.21377 (13)	0.3532 (2)	0.0355 (7)
N3	0.37310 (14)	0.05216 (15)	0.1596 (3)	0.0479 (8)
O1	0.28230 (10)	0.14687 (10)	0.23716 (19)	0.0342 (5)
O2	0.41509 (12)	0.09599 (12)	0.2220 (2)	0.0455 (6)
O3	0.32302 (12)	0.05220 (12)	0.1480 (2)	0.0459 (6)
O4	0.38022 (15)	0.01126 (15)	0.1141 (4)	0.0853 (11)
HN1	0.320 (3)	0.055 (3)	0.423 (6)	0.128*
HN2	0.401 (3)	0.242 (2)	0.336 (6)	0.128*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0299 (3)	0.0299 (3)	0.0353 (3)	0.0154 (2)	−0.00202 (16)	0.00065 (16)
C1	0.041 (3)	0.060 (3)	0.081 (3)	0.014 (2)	0.014 (2)	0.004 (2)
C2	0.037 (2)	0.035 (2)	0.052 (2)	0.0137 (17)	0.0119 (16)	−0.0027 (16)
C3	0.033 (2)	0.038 (2)	0.055 (2)	0.0200 (18)	−0.0023 (16)	−0.0069 (16)
C4	0.0299 (19)	0.0322 (19)	0.0451 (18)	0.0165 (16)	0.0011 (14)	−0.0002 (14)
C5	0.0322 (19)	0.033 (2)	0.0424 (18)	0.0193 (17)	0.0038 (14)	−0.0001 (14)
C6	0.044 (2)	0.042 (2)	0.049 (2)	0.022 (2)	0.0114 (17)	0.0067 (16)
C7	0.037 (2)	0.037 (2)	0.0402 (18)	0.0193 (17)	0.0053 (15)	0.0017 (14)
C8	0.043 (2)	0.051 (2)	0.0380 (18)	0.024 (2)	0.0040 (15)	0.0054 (16)
C9	0.047 (2)	0.045 (2)	0.0366 (18)	0.0226 (19)	−0.0053 (15)	0.0029 (15)
C10	0.048 (2)	0.043 (2)	0.0359 (18)	0.0227 (19)	−0.0046 (15)	−0.0049 (15)
C11	0.033 (2)	0.034 (2)	0.049 (2)	0.0181 (17)	−0.0105 (15)	−0.0059 (15)
N1	0.0404 (18)	0.0370 (18)	0.0381 (14)	0.0211 (16)	−0.0010 (12)	0.0024 (13)
N2	0.0366 (17)	0.0327 (17)	0.0383 (15)	0.0182 (15)	−0.0035 (12)	−0.0001 (12)
N3	0.0349 (19)	0.040 (2)	0.067 (2)	0.0180 (16)	−0.0012 (15)	−0.0111 (16)
O1	0.0289 (13)	0.0311 (13)	0.0408 (12)	0.0137 (11)	−0.0007 (10)	0.0025 (10)
O2	0.0341 (14)	0.0377 (15)	0.0634 (16)	0.0171 (13)	−0.0070 (12)	−0.0076 (12)
O3	0.0329 (15)	0.0434 (16)	0.0570 (15)	0.0159 (13)	−0.0059 (11)	−0.0075 (11)
O4	0.058 (2)	0.062 (2)	0.139 (3)	0.0324 (18)	−0.0003 (19)	−0.048 (2)

Geometric parameters (Å, º)

Ni1—O1	2.000 (2)	C7—C8	1.502 (5)
Ni1—O1^{i}	2.006 (2)	C8—N1	1.481 (5)
Ni1—N2	2.038 (3)	C8—H8A	0.9700
Ni1—N1	2.054 (3)	C8—H8B	0.9700
Ni1—O3	2.134 (3)	C9—N1	1.489 (4)
Ni1—O2	2.183 (3)	C9—C10	1.519 (5)
Ni1—Ni1^{i}	2.9737 (10)	C9—H9A	0.9700
C1—C2	1.510 (5)	C9—H9B	0.9700
C1—H1A	0.9600	C10—N2	1.483 (4)
C1—H1B	0.9600	C10—H10A	0.9700
C1—H1C	0.9600	C10—H10B	0.9700
C2—C3	1.374 (5)	C11—N2	1.473 (4)

C2—C6	1.382 (5)	C11—C4^{i}	1.505 (5)
C3—C4	1.398 (5)	C11—H11A	0.9700
C3—H3	0.9300	C11—H11B	0.9700
C4—C5	1.400 (5)	N1—HN1	0.87 (6)
C4—C11^{i}	1.505 (5)	N2—HN2	0.86 (6)
C5—O1	1.336 (4)	N3—O4	1.223 (4)
C5—C7	1.408 (5)	N3—O3	1.260 (4)
C6—C7	1.386 (5)	N3—O2	1.262 (4)
C6—H6	0.9300		

O1—Ni1—O1^{i}	84.17 (9)	N1—C8—C7	113.5 (3)
O1—Ni1—N2	97.29 (10)	N1—C8—H8A	108.9
O1^{i}—Ni1—N2	87.95 (10)	C7—C8—H8A	108.9
O1—Ni1—N1	91.74 (10)	N1—C8—H8B	108.9
O1^{i}—Ni1—N1	172.81 (10)	C7—C8—H8B	108.9
N2—Ni1—N1	86.72 (12)	H8A—C8—H8B	107.7
O1—Ni1—O3	99.46 (9)	N1—C9—C10	110.3 (3)
O1^{i}—Ni1—O3	91.39 (10)	N1—C9—H9A	109.6
N2—Ni1—O3	163.09 (11)	C10—C9—H9A	109.6
N1—Ni1—O3	95.11 (11)	N1—C9—H9B	109.6
O1—Ni1—O2	158.83 (9)	C10—C9—H9B	109.6
O1^{i}—Ni1—O2	92.93 (9)	H9A—C9—H9B	108.1
N2—Ni1—O2	103.57 (11)	N2—C10—C9	110.8 (3)
N1—Ni1—O2	92.99 (11)	N2—C10—H10A	109.5
O3—Ni1—O2	59.57 (10)	C9—C10—H10A	109.5
O1—Ni1—Ni1^{i}	42.16 (6)	N2—C10—H10B	109.5
O1^{i}—Ni1—Ni1^{i}	42.01 (6)	C9—C10—H10B	109.5
N2—Ni1—Ni1^{i}	93.51 (8)	H10A—C10—H10B	108.1
N1—Ni1—Ni1^{i}	133.62 (9)	N2—C11—C4^{i}	112.7 (3)
O3—Ni1—Ni1^{i}	97.29 (7)	N2—C11—H11A	109.0
O2—Ni1—Ni1^{i}	131.44 (7)	C4^{i}—C11—H11A	109.0
C2—C1—H1A	109.5	N2—C11—H11B	109.0
C2—C1—H1B	109.5	C4^{i}—C11—H11B	109.0
H1A—C1—H1B	109.5	H11A—C11—H11B	107.8
C2—C1—H1C	109.5	C8—N1—C9	113.0 (3)
H1A—C1—H1C	109.5	C8—N1—Ni1	112.8 (2)
H1B—C1—H1C	109.5	C9—N1—Ni1	103.2 (2)
C3—C2—C6	117.3 (3)	C8—N1—HN1	108 (4)
C3—C2—C1	121.5 (4)	C9—N1—HN1	108 (4)
C6—C2—C1	121.2 (4)	Ni1—N1—HN1	112 (4)
C2—C3—C4	123.0 (3)	C11—N2—C10	115.1 (3)
C2—C3—H3	118.5	C11—N2—Ni1	106.0 (2)
C4—C3—H3	118.5	C10—N2—Ni1	108.2 (2)
C3—C4—C5	118.4 (3)	C11—N2—HN2	107 (4)
C3—C4—C11^{i}	118.1 (3)	C10—N2—HN2	110 (4)
C5—C4—C11^{i}	123.5 (3)	Ni1—N2—HN2	110 (4)
O1—C5—C4	122.9 (3)	O4—N3—O3	121.5 (3)
O1—C5—C7	118.0 (3)	O4—N3—O2	121.9 (3)

C4—C5—C7	119.1 (3)	O3—N3—O2	116.6 (3)
C2—C6—C7	122.2 (3)	C5—O1—Ni1	113.40 (19)
C2—C6—H6	118.9	C5—O1—Ni1^{i}	125.52 (19)
C7—C6—H6	118.9	Ni1—O1—Ni1^{i}	95.83 (9)
C6—C7—C5	119.4 (3)	N3—O2—Ni1	90.8 (2)
C6—C7—C8	121.7 (3)	N3—O3—Ni1	93.11 (19)
C5—C7—C8	118.7 (3)		

C6—C2—C3—C4	−3.1 (5)	O2—Ni1—N2—C11	32.8 (2)
C1—C2—C3—C4	176.2 (4)	Ni1^{i}—Ni1—N2—C11	−101.34 (19)
C2—C3—C4—C5	−3.7 (5)	O1—Ni1—N2—C10	92.5 (2)
C2—C3—C4—C11^{i}	175.9 (3)	O1^{i}—Ni1—N2—C10	176.3 (2)
C3—C4—C5—O1	−172.4 (3)	N1—Ni1—N2—C10	1.1 (2)
C11^{i}—C4—C5—O1	8.1 (5)	O3—Ni1—N2—C10	−95.6 (4)
C3—C4—C5—C7	7.8 (5)	O2—Ni1—N2—C10	−91.1 (2)
C11^{i}—C4—C5—C7	−171.7 (3)	Ni1^{i}—Ni1—N2—C10	134.7 (2)
C3—C2—C6—C7	5.8 (5)	C4—C5—O1—Ni1	−120.4 (3)
C1—C2—C6—C7	−173.5 (4)	C7—C5—O1—Ni1	59.4 (3)
C2—C6—C7—C5	−1.7 (5)	C4—C5—O1—Ni1^{i}	−4.0 (4)
C2—C6—C7—C8	173.8 (3)	C7—C5—O1—Ni1^{i}	175.8 (2)
O1—C5—C7—C6	174.9 (3)	O1^{i}—Ni1—O1—C5	132.9 (2)
C4—C5—C7—C6	−5.2 (5)	N2—Ni1—O1—C5	−139.9 (2)
O1—C5—C7—C8	−0.7 (5)	N1—Ni1—O1—C5	−53.0 (2)
C4—C5—C7—C8	179.1 (3)	O3—Ni1—O1—C5	42.4 (2)
C6—C7—C8—N1	123.4 (4)	O2—Ni1—O1—C5	49.8 (3)
C5—C7—C8—N1	−61.0 (4)	Ni1^{i}—Ni1—O1—C5	132.9 (2)
N1—C9—C10—N2	−48.9 (4)	O1^{i}—Ni1—O1—Ni1^{i}	0.0
C7—C8—N1—C9	166.4 (3)	N2—Ni1—O1—Ni1^{i}	87.17 (11)
C7—C8—N1—Ni1	49.9 (4)	N1—Ni1—O1—Ni1^{i}	174.08 (11)
C10—C9—N1—C8	−76.0 (4)	O3—Ni1—O1—Ni1^{i}	−90.44 (10)
C10—C9—N1—Ni1	46.1 (3)	O2—Ni1—O1—Ni1^{i}	−83.0 (3)
N2—Ni1—N1—C8	96.4 (2)	O4—N3—O2—Ni1	−179.5 (4)
O3—Ni1—N1—C8	−100.4 (2)	O3—N3—O2—Ni1	−0.4 (3)
O2—Ni1—N1—C8	−160.1 (2)	O1—Ni1—O2—N3	−8.2 (4)
Ni1^{i}—Ni1—N1—C8	4.7 (3)	O1^{i}—Ni1—O2—N3	−89.6 (2)
N2—Ni1—N1—C9	−25.8 (2)	N2—Ni1—O2—N3	−178.2 (2)
O3—Ni1—N1—C9	137.3 (2)	N1—Ni1—O2—N3	94.4 (2)
O2—Ni1—N1—C9	77.6 (2)	O3—Ni1—O2—N3	0.24 (19)
Ni1^{i}—Ni1—N1—C9	−117.5 (2)	Ni1^{i}—Ni1—O2—N3	−71.0 (2)
C4^{i}—C11—N2—C10	−169.3 (3)	O4—N3—O3—Ni1	179.5 (4)
C4^{i}—C11—N2—Ni1	71.2 (3)	O2—N3—O3—Ni1	0.4 (3)
C9—C10—N2—C11	−94.0 (4)	O1—Ni1—O3—N3	176.7 (2)
C9—C10—N2—Ni1	24.3 (4)	O1^{i}—Ni1—O3—N3	92.3 (2)
O1—Ni1—N2—C11	−143.54 (19)	N2—Ni1—O3—N3	4.8 (5)
O1^{i}—Ni1—N2—C11	−59.7 (2)	N1—Ni1—O3—N3	−90.7 (2)

N1—Ni1—N2—C11	125.1 (2)	O2—Ni1—O3—N3	−0.24 (19)
O3—Ni1—N2—C11	28.3 (5)	Ni1^{i}—Ni1—O3—N3	134.08 (19)

Symmetry code: (i) −*x*+2/3, −*y*+1/3, −*z*+1/3.