Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Bis{μ-2,2′-[ethane-1,2-diylbis(nitrilomethylidyne)]diphenolato}dinickel(II)

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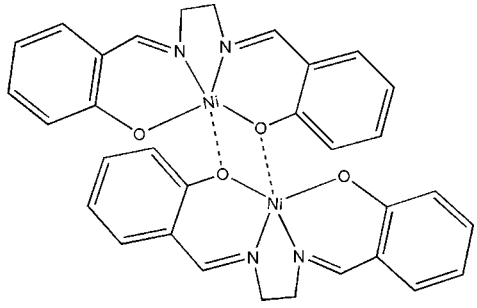
Received 15 November 2007; accepted 4 December 2007

Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.003 Å; R factor = 0.024; wR factor = 0.056; data-to-parameter ratio = 16.2.

The asymmetric unit of the title compound, $[Ni_2(C_{16}H_{14}N_2O_2)_2]$, contains an Ni^{II} cation which is coordinated by two imine N atoms and by two phenolate O atoms of the salen ligand {salen = *N*,*N*′-bis(salicylidene)ethane-1,2-diamine or 2,2′-[ethane-1,2-diylbis(nitrilomethylidyne)]diphenol}, leading to a distorted square-planar conformation. When a secondary Ni—O interaction > 2.41 Å to the neighbouring phenolate O atom is considered, two molecules are linked into a centrosymmetric dimer with an overall square-pyramidal coordination for the Ni^{II} cation. Weak π–π interactions with a shortest interplanar distance of 3.704 Å help to stabilize the crystal structure.

Related literature

For a review on metal–salen complexes used in catalysis, see: Cozzi (2004).



Experimental

Crystal data

$[Ni_2(C_{16}H_{14}N_2O_2)_2]$
M_r = 650.00
Monoclinic, $C2/c$
a = 26.639 (2) Å
b = 6.9775 (6) Å
c = 14.7094 (12) Å
β = 97.501 (1)°
V = 2710.7 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.44 mm^{-1}
T = 273 (2) K
0.34 × 0.21 × 0.07 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.641, T_{max} = 0.906
8437 measured reflections
3090 independent reflections
2607 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.024
$wR(F^2)$ = 0.056
S = 1.03
3090 reflections
191 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni1—O2	1.9115 (11)	Ni1—N2	1.9484 (13)
Ni1—O1	1.9412 (10)	Ni1—N1	1.9560 (13)
O2—Ni1—O1	91.35 (5)	O2—Ni1—N1	92.53 (5)
O2—Ni1—N2	171.05 (5)	O1—Ni1—N1	170.36 (5)
O1—Ni1—N2	91.21 (5)	N2—Ni1—N1	83.67 (6)

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* ((Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

This work was supported by the Natural Science Foundation of Xiaogan University (Z2008012).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2168).

References

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Cozzi, P. G. (2004). *Chem. Soc. Rev.* **33**, 410–421.
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supporting information

Acta Cryst. (2008). E**64**, m173 [https://doi.org/10.1107/S1600536807065506]

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S1. Comment

A review on metal-salen complexes in catalysis was given recently by Cozzi (2004). Herein, we report on synthesis and crystal structure of $[Ni(salen)]_2$, (I).

As shown in Fig. 1, the molecular structure of (I) is made up of a centrosymmetric dimer. The Ni^{II} cation is surrounded by two N atoms and two O atoms from the salen ligands leading to a distorted square coordination with mean Ni—O distances of 1.927 Å, and somewhat longer mean Ni—N distances of 1.952 Å. Secondary Ni1—O1(-*x*, *y*, 1/2 - *z*) interactions of 2.4106 (11) Å of one salen ligand to the neighbouring Ni^{II} center link two molecules to a centrosymmetric dimer with an Ni1···Ni1(-*x*, *y*, 1/2 - *z*) separation of 3.1946 (4) Å. The resulting overall coordination sphere of the Ni^{II} cation can thus be described as a distorted square pyramid.

As shown in Fig. 2, there are weak π-π interactions, with plane-to-plane distances and displacement angles for the planes *Cg*4···*Cg*5 and *Cg*5···*Cg*6 of 3.704 Å and 11.85 °, and 4.022 Å and 6.72 °, respectively. The planes *Cg*4, *Cg*5 and *Cg*6 consist of atoms C3—O2/N1, C1—C6 and C7—C12.

S2. Experimental

Compound (I) was prepared by adding $Ni(Ac)_2{\cdot}2H_2O$ (0.110 g, 0.5 mmol) to a solution of H_2(salen) 0.122 mg (0.5 mmol) in methanol (20 mL) and DMF (20 ml). After stirring the mixture for 2 h, the solution was filtered and kept for several days at ambient temperature to evaporate. Brown block-like crystals were obtained.

S3. Refinement

H atoms were placed in geometrically idealized positions and were refined in the riding mode with C–H = 0.97Å and U_{iso}(H)=1.5U_{eq}(C) for C14 and C15. All other H atoms were refined with C–H = 0.93Å and U_{iso}(H) = 1.2U_{eq}(C).

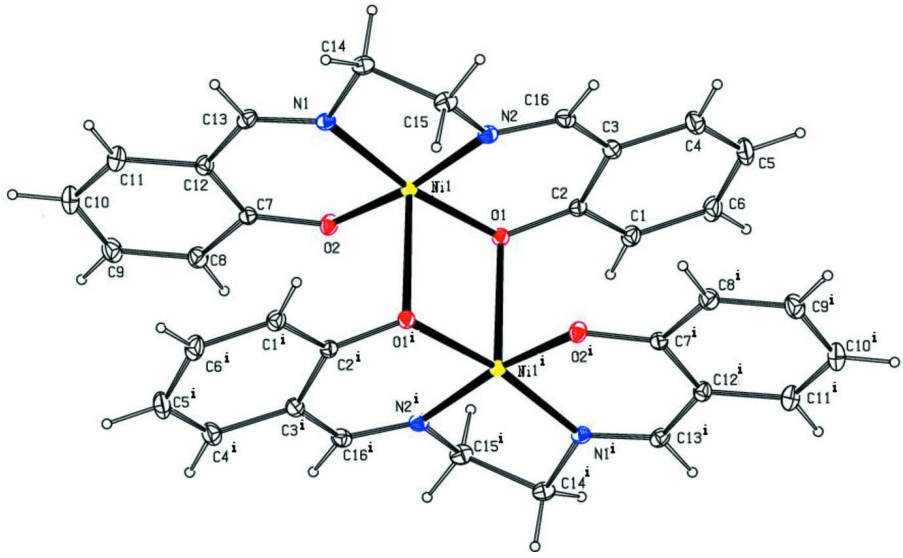

Figure 1

The molecular structure of (I), showing displacement ellipsoids at the 30% probability level [symmetry code: (i) -*x*, *y*, 1/2 - *z*].

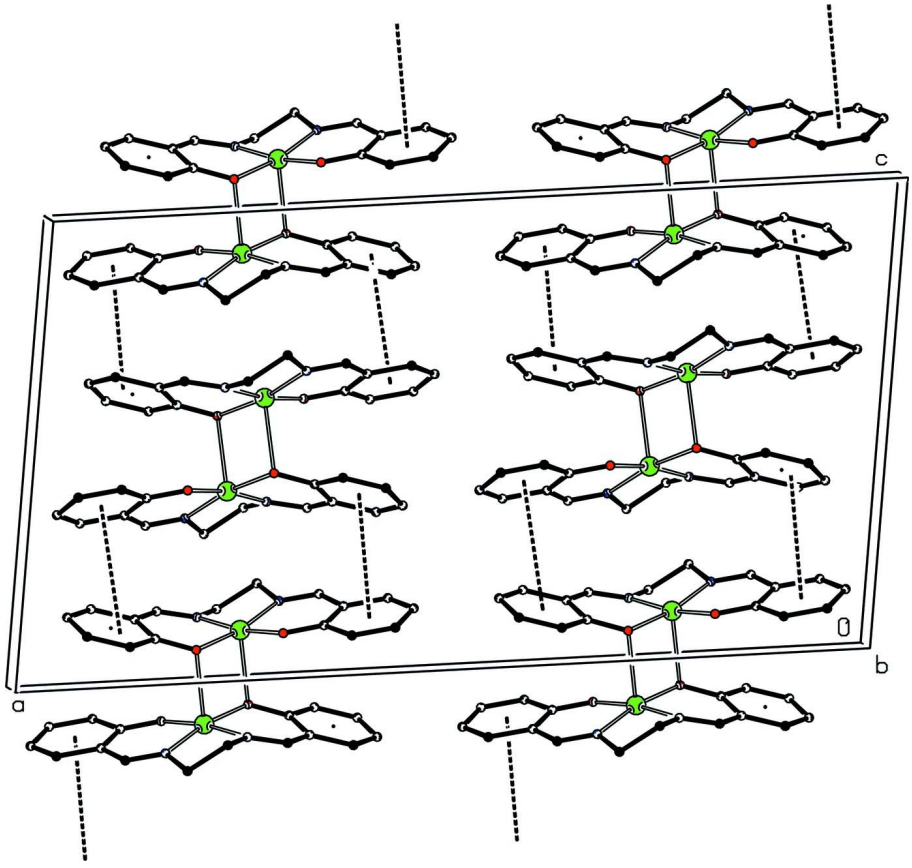

Figure 2

The packing diagram of (I).

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Crystal data

$[Ni_2(C_{16}H_{14}N_2O_2)_2]$
M_r = 650.00
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
a = 26.639 (2) Å
b = 6.9775 (6) Å
c = 14.7094 (12) Å
β = 97.501 (1)°
V = 2710.7 (4) Å^3
Z = 4
F(000) = 1344
D_x = 1.593 Mg m^{-3}
Mo *K*α radiation, λ = 0.71073 Å
Cell parameters from 3127 reflections
θ = 2.8–26.6°
μ = 1.44 mm^{-1}
T = 273 K
Block, brown
0.34 × 0.21 × 0.07 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ– and ω– scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.641, T_{max} = 0.906
8437 measured reflections
3090 independent reflections
2607 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 27.5°, θ_{min} = 2.8°
h = −34→32
k = −7→9
l = −16→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.024
$wR(F^2)$ = 0.056
S = 1.03
3090 reflections
191 parameters
0 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
$w = 1/[\sigma^2(F_o^2) + (0.0243P)^2 + 0.8084P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Extinction correction: *SHELXL*, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.00018 (7)

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	0.267171 (7)	0.20529 (3)	0.402119 (12)	0.02626 (7)
C1	0.13571 (6)	0.5214 (3)	0.42203 (12)	0.0431 (4)
H1	0.1505	0.6316	0.4492	0.052*

O1	0.21557 (4)	0.37606 (15)	0.43757 (7)	0.0331 (2)
C3	0.14245 (6)	0.1972 (2)	0.36791 (11)	0.0381 (4)
C2	0.16650 (6)	0.3629 (2)	0.40947 (10)	0.0333 (3)
C6	0.08435 (7)	0.5180 (3)	0.39534 (14)	0.0560 (5)
H6	0.0650	0.6253	0.4050	0.067*
C4	0.08991 (7)	0.1999 (3)	0.34122 (14)	0.0520 (5)
H4	0.0743	0.0914	0.3138	0.062*
C5	0.06105 (7)	0.3565 (3)	0.35422 (15)	0.0611 (6)
H5	0.0263	0.3551	0.3358	0.073*
C7	0.36315 (6)	0.3962 (2)	0.39857 (10)	0.0341 (4)
C12	0.38590 (6)	0.2332 (2)	0.36206 (12)	0.0383 (4)
C9	0.44526 (7)	0.5552 (3)	0.40933 (14)	0.0560 (5)
H9	0.4651	0.6624	0.4261	0.067*
C8	0.39502 (7)	0.5555 (3)	0.42134 (13)	0.0459 (4)
H8	0.3815	0.6644	0.4453	0.055*
C11	0.43741 (7)	0.2396 (3)	0.34965 (15)	0.0538 (5)
H11	0.4518	0.1331	0.3251	0.065*
N1	0.31197 (5)	0.03112 (18)	0.34648 (9)	0.0342 (3)
O2	0.31572 (4)	0.40839 (15)	0.41080 (8)	0.0386 (3)
N2	0.21745 (5)	0.00240 (18)	0.37265 (9)	0.0360 (3)
C13	0.35815 (6)	0.0617 (2)	0.33554 (11)	0.0392 (4)
H13	0.3750	−0.0349	0.3082	0.047*
C14	0.28630 (7)	−0.1438 (2)	0.30878 (12)	0.0430 (4)
H14A	0.3096	−0.2513	0.3158	0.052*
H14B	0.2747	−0.1270	0.2440	0.052*
C15	0.24182 (7)	−0.1823 (2)	0.35987 (13)	0.0443 (4)
H15A	0.2181	−0.2688	0.3250	0.053*
H15B	0.2532	−0.2407	0.4188	0.053*
C10	0.46695 (7)	0.3965 (3)	0.37237 (16)	0.0618 (6)
H10	0.5009	0.3974	0.3634	0.074*
C16	0.16935 (7)	0.0228 (2)	0.35544 (11)	0.0402 (4)
H16	0.1504	−0.0832	0.3332	0.048*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.02833 (11)	0.02401 (11)	0.02671 (11)	0.00003 (8)	0.00461 (7)	−0.00381 (8)
C1	0.0403 (9)	0.0467 (10)	0.0431 (10)	0.0081 (8)	0.0082 (8)	0.0044 (8)
O1	0.0303 (6)	0.0334 (6)	0.0356 (6)	0.0004 (5)	0.0048 (4)	−0.0036 (5)
C3	0.0359 (9)	0.0435 (10)	0.0343 (9)	−0.0052 (7)	0.0030 (7)	0.0054 (7)
C2	0.0326 (8)	0.0405 (9)	0.0271 (8)	0.0013 (7)	0.0056 (6)	0.0068 (7)
C6	0.0404 (10)	0.0672 (14)	0.0615 (13)	0.0172 (10)	0.0112 (9)	0.0118 (11)
C4	0.0390 (10)	0.0646 (13)	0.0510 (12)	−0.0112 (9)	0.0007 (8)	0.0094 (10)
C5	0.0288 (9)	0.0842 (16)	0.0691 (14)	0.0010 (10)	0.0019 (9)	0.0164 (12)
C7	0.0328 (8)	0.0386 (9)	0.0309 (8)	−0.0011 (7)	0.0037 (7)	0.0026 (7)
C12	0.0357 (9)	0.0395 (9)	0.0403 (10)	0.0041 (7)	0.0066 (7)	0.0034 (7)
C9	0.0434 (11)	0.0600 (13)	0.0637 (13)	−0.0162 (9)	0.0043 (9)	0.0022 (10)
C8	0.0433 (10)	0.0445 (10)	0.0505 (11)	−0.0054 (8)	0.0082 (8)	−0.0047 (8)

C11	0.0390 (10)	0.0576 (12)	0.0667 (13)	0.0083 (9)	0.0143 (9)	0.0018 (10)
N1	0.0396 (7)	0.0309 (7)	0.0323 (7)	0.0024 (6)	0.0052 (6)	−0.0032 (6)
O2	0.0349 (6)	0.0327 (6)	0.0499 (7)	−0.0017 (5)	0.0119 (5)	−0.0051 (5)
N2	0.0425 (8)	0.0318 (7)	0.0342 (7)	−0.0048 (6)	0.0071 (6)	−0.0026 (6)
C13	0.0438 (10)	0.0365 (9)	0.0387 (9)	0.0109 (7)	0.0110 (7)	−0.0005 (7)
C14	0.0542 (11)	0.0333 (9)	0.0419 (10)	0.0018 (8)	0.0083 (8)	−0.0087 (7)
C15	0.0583 (12)	0.0301 (9)	0.0455 (10)	−0.0035 (8)	0.0102 (9)	−0.0035 (7)
C10	0.0330 (10)	0.0720 (14)	0.0817 (16)	−0.0024 (10)	0.0124 (10)	0.0065 (12)
C16	0.0447 (10)	0.0394 (9)	0.0361 (9)	−0.0142 (8)	0.0037 (7)	−0.0004 (7)

Geometric parameters (Å, º)

Ni1—O2	1.9115 (11)	C12—C13	1.433 (2)
Ni1—O1	1.9412 (10)	C9—C8	1.373 (3)
Ni1—N2	1.9484 (13)	C9—C10	1.392 (3)
Ni1—N1	1.9560 (13)	C9—H9	0.9300
C1—C6	1.373 (2)	C8—H8	0.9300
C1—C2	1.403 (2)	C11—C10	1.364 (3)
C1—H1	0.9300	C11—H11	0.9300
O1—C2	1.3217 (18)	N1—C13	1.279 (2)
C3—C4	1.403 (2)	N1—C14	1.472 (2)
C3—C2	1.421 (2)	N2—C16	1.281 (2)
C3—C16	1.436 (2)	N2—C15	1.466 (2)
C6—C5	1.387 (3)	C13—H13	0.9300
C6—H6	0.9300	C14—C15	1.508 (3)
C4—C5	1.364 (3)	C14—H14A	0.9700
C4—H4	0.9300	C14—H14B	0.9700
C5—H5	0.9300	C15—H15A	0.9700
C7—O2	1.3021 (18)	C15—H15B	0.9700
C7—C8	1.412 (2)	C10—H10	0.9300
C7—C12	1.426 (2)	C16—H16	0.9300
C12—C11	1.409 (2)		
O2—Ni1—O1	91.35 (5)	C9—C8—C7	122.30 (18)
O2—Ni1—N2	171.05 (5)	C9—C8—H8	118.9
O1—Ni1—N2	91.21 (5)	C7—C8—H8	118.9
O2—Ni1—N1	92.53 (5)	C10—C11—C12	122.30 (19)
O1—Ni1—N1	170.36 (5)	C10—C11—H11	118.8
N2—Ni1—N1	83.67 (6)	C12—C11—H11	118.8
C6—C1—C2	121.78 (18)	C13—N1—C14	119.87 (14)
C6—C1—H1	119.1	C13—N1—Ni1	126.75 (11)
C2—C1—H1	119.1	C14—N1—Ni1	113.21 (10)
C2—O1—Ni1	125.41 (10)	C7—O2—Ni1	127.05 (10)
C4—C3—C2	119.15 (16)	C16—N2—C15	121.38 (14)
C4—C3—C16	118.13 (16)	C16—N2—Ni1	126.61 (12)
C2—C3—C16	122.67 (15)	C15—N2—Ni1	111.57 (11)
O1—C2—C1	118.39 (15)	N1—C13—C12	125.11 (15)
O1—C2—C3	124.28 (15)	N1—C13—H13	117.4

C1—C2—C3	117.33 (15)	C12—C13—H13	117.4
C1—C6—C5	120.74 (18)	N1—C14—C15	108.46 (14)
C1—C6—H6	119.6	N1—C14—H14A	110.0
C5—C6—H6	119.6	C15—C14—H14A	110.0
C5—C4—C3	122.08 (19)	N1—C14—H14B	110.0
C5—C4—H4	119.0	C15—C14—H14B	110.0
C3—C4—H4	119.0	H14A—C14—H14B	108.4
C4—C5—C6	118.93 (17)	N2—C15—C14	107.30 (13)
C4—C5—H5	120.5	N2—C15—H15A	110.3
C6—C5—H5	120.5	C14—C15—H15A	110.3
O2—C7—C8	118.69 (15)	N2—C15—H15B	110.3
O2—C7—C12	124.88 (15)	C14—C15—H15B	110.3
C8—C7—C12	116.43 (15)	H15A—C15—H15B	108.5
C11—C12—C7	119.51 (16)	C11—C10—C9	118.58 (18)
C11—C12—C13	117.88 (16)	C11—C10—H10	120.7
C7—C12—C13	122.60 (15)	C9—C10—H10	120.7
C8—C9—C10	120.87 (19)	N2—C16—C3	124.89 (15)
C8—C9—H9	119.6	N2—C16—H16	117.6
C10—C9—H9	119.6	C3—C16—H16	117.6

O2—Ni1—O1—C2	148.07 (12)	O1—Ni1—N1—C14	−54.8 (3)
N2—Ni1—O1—C2	−23.36 (12)	N2—Ni1—N1—C14	3.41 (11)
N1—Ni1—O1—C2	34.3 (4)	C8—C7—O2—Ni1	−170.58 (11)
Ni1—O1—C2—C1	−164.26 (11)	C12—C7—O2—Ni1	10.4 (2)
Ni1—O1—C2—C3	16.4 (2)	O1—Ni1—O2—C7	177.25 (13)
C6—C1—C2—O1	−179.44 (16)	N2—Ni1—O2—C7	−76.1 (4)
C6—C1—C2—C3	−0.1 (3)	N1—Ni1—O2—C7	−11.58 (13)
C4—C3—C2—O1	179.69 (15)	O2—Ni1—N2—C16	−86.3 (4)
C16—C3—C2—O1	2.5 (3)	O1—Ni1—N2—C16	20.29 (14)
C4—C3—C2—C1	0.4 (2)	N1—Ni1—N2—C16	−151.53 (15)
C16—C3—C2—C1	−176.84 (15)	O2—Ni1—N2—C15	86.0 (3)
C2—C1—C6—C5	−0.4 (3)	O1—Ni1—N2—C15	−167.35 (11)
C2—C3—C4—C5	−0.3 (3)	N1—Ni1—N2—C15	20.84 (11)
C16—C3—C4—C5	177.11 (18)	C14—N1—C13—C12	174.47 (15)
C3—C4—C5—C6	−0.2 (3)	Ni1—N1—C13—C12	−0.4 (2)
C1—C6—C5—C4	0.5 (3)	C11—C12—C13—N1	176.60 (17)
O2—C7—C12—C11	178.16 (16)	C7—C12—C13—N1	−4.5 (3)
C8—C7—C12—C11	−0.8 (2)	C13—N1—C14—C15	158.62 (15)
O2—C7—C12—C13	−0.7 (3)	Ni1—N1—C14—C15	−25.82 (17)
C8—C7—C12—C13	−179.72 (15)	C16—N2—C15—C14	133.16 (16)
C10—C9—C8—C7	1.0 (3)	Ni1—N2—C15—C14	−39.67 (16)
O2—C7—C8—C9	−179.08 (17)	N1—C14—C15—N2	41.36 (18)
C12—C7—C8—C9	0.0 (3)	C12—C11—C10—C9	0.2 (3)
C7—C12—C11—C10	0.8 (3)	C8—C9—C10—C11	−1.1 (3)
C13—C12—C11—C10	179.71 (19)	C15—N2—C16—C3	178.62 (15)
O2—Ni1—N1—C13	6.72 (14)	Ni1—N2—C16—C3	−9.7 (2)
O1—Ni1—N1—C13	120.4 (3)	C4—C3—C16—N2	176.60 (17)
N2—Ni1—N1—C13	178.60 (14)	C2—C3—C16—N2	−6.1 (3)

O2—Ni1—N1—C14	−168.47 (11)