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{2,2'-[1,1'-(Ethylenedioxydinitrilo)-diethylidyne]di-1-naphtholato}nickel(II)

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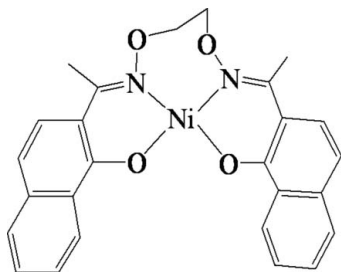
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 Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.007$ Å; R factor = 0.048; wR factor = 0.126; data-to-parameter ratio = 12.7.

In the title complex, $[\text{Ni}(\text{C}_{26}\text{H}_{22}\text{N}_2\text{O}_4)]$, the Ni^{II} atom has a slight distortion toward tetrahedral geometry from a square-planar structure, coordinated by two O and two N atoms of the tetradentate salen-type bisoxime 2,2'-[1,1'-(ethylenedioxydinitrilo)diethylidyne]di-1-naphtholate (L^{2-}) unit, with a mean deviation of 0.022 Å from the N_2O_2 plane. The N - and O -donor atoms are mutually *cis*. The dihedral angle between two naphthalene systems of the L^{2-} ligand is 67.59 (4)°. The crystal structure is stabilized by intermolecular $\text{C}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\pi$ interactions, which link neighbouring molecules into extended chains along the b axis.

Related literature

For multidentate salen-type compounds in coordination chemistry, see: Akine *et al.* (2005); Dong *et al.* (2009*a,b*); Katsuki (1995); Ray *et al.* (2003); Sun *et al.* (2008). For the isostructural Cu complex, see: Dong *et al.* (2009*c*).



Experimental

Crystal data

 $[\text{Ni}(\text{C}_{26}\text{H}_{22}\text{N}_2\text{O}_4)]$
 $M_r = 485.17$

 Monoclinic, $P2_1/n$
 $a = 13.6975$ (13) Å
 $b = 8.2711$ (10) Å
 $c = 19.049$ (2) Å
 $\beta = 95.346$ (1)°
 $V = 2148.7$ (4) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.94$ mm⁻¹
 $T = 298$ K
 $0.43 \times 0.16 \times 0.06$ mm

Data collection

 Siemens SMART 1000 CCD diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\text{min}} = 0.688$, $T_{\text{max}} = 0.946$

 10414 measured reflections
 3782 independent reflections
 2179 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.073$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.048$
 $wR(F^2) = 0.126$
 $S = 1.02$
 3782 reflections

 298 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.45$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.38$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---|-------|-------------|-------------|---------------|
| $\text{C15}-\text{H15A}\cdots\text{O3}^{\text{i}}$ | 0.96 | 2.45 | 3.152 (6) | 130 |
| $\text{C3}-\text{H3C}\cdots\text{Cg1}^{\text{ii}}$ | 0.97 | 3.17 | 4.127 (3) | 172 |
| $\text{C15}-\text{H15A}\cdots\text{Cg2}^{\text{iii}}$ | 0.96 | 3.53 | 4.398 (3) | 152 |

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

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); 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.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2525).

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supplementary materials

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{2,2'-[1,1'-(Ethylenedioxydinitrilo)diethylidyne]di-1-naphtholato}nickel(II)

W.-K. Dong, J.-C. Wu, J. Yao, S.-S. Gong and J.-F. Tong

Comment

Multidentate salen-type compounds play an important role in the development of modern coordination chemistry as they readily form stable complexes with most of the transition metals, in which some could exhibit interesting properties (Katsuki, *et al.*, 1995; Akine, *et al.*, 2005; Ray, *et al.*, 2003). Here, we report a new Ni^{II} complex based on the tetradentate salen-type bisoxime ligand 2,2'-[1,1'-ethylenedioxybis(nitriloethylidyne)]dinaphthol (Dong, *et al.*, 2009a; Dong, *et al.*, 2009b).

In this paper, a new mononuclear nickel(II) complex with salen-type bisoxime chelating ligand, 2,2'-[1,1'-ethylenedioxybis(nitriloethylidyne)]dinaphthol, has been synthesized (Sun, *et al.*, 2008). The dihedral angle between the coordination plane of O3—Ni1—N1 and that of O4—Ni1—N2 is 67.59 (4)°, indicating slight distortion toward tetrahedral geometry from the square planar structure, with a mean deviation of 0.022 Å from the N₂O₂ plane. The crystal structure is further stabilized by intermolecular C15—H15A···O3 hydrogen bond and C3—H3C···π(benzene), C15—H15A···π(naphthalene) interactions (Table 1), which link neighbouring molecules into extended chains along the *b* axis.

Experimental

A solution of nickel(II) chloride tetrahydrate (2.8 mg, 0.0138 mmol) in methanol (3 ml) was added dropwise to a solution of 2,2'-[1,1'-ethylenedioxybis(nitriloethylidyne)]dinaphthol (4.5 mg, 0.0105 mmol) and 99% triethylamine (0.025 ml) in dichloromethane (3 ml). The color of the mixing solution turns to dark-yellow, immediately, and was allowed to stand at room temperature for about three weeks, the solvent was partially evaporated and obtained brown needle-like single crystals suitable for X-ray crystallographic analysis.

Refinement

Non-H atoms were refined anisotropically. H atoms were treated as riding atoms with distances C—H = 0.96 (CH₃), C—H = 0.97 (CH₂), or 0.93 Å (CH), and $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$ and $1.5 U_{\text{eq}}(\text{O})$.

Figures

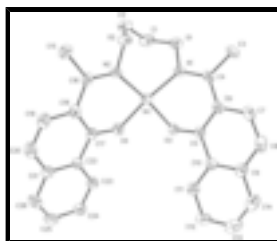


Fig. 1. The molecule structure of the title complex. Displacement ellipsoids for non-hydrogen atoms are drawn at the 30% probability level.

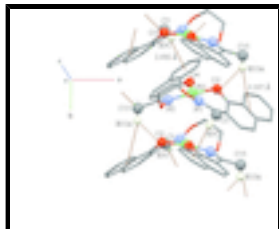


Fig. 2. Part of the supramolecular structure of the title complex Showing the formation of C15—H15A···O3 hydrogen bond and C3—H3C··· π (benzene), C15—H15A··· π (naphthalene) interactions.

{2,2'-[1,1'-(Ethylenedioxydinitrilo)diethylidyne]di-1-naphtholato}nickel(II)

Crystal data

[Ni(C₂₆H₂₂N₂O₄)]

$M_r = 485.17$

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

$a = 13.6975$ (13) Å

$b = 8.2711$ (10) Å

$c = 19.049$ (2) Å

$\beta = 95.3460$ (10)°

$V = 2148.7$ (4) Å³

$Z = 4$

$F_{000} = 1008$

$D_x = 1.500$ Mg m⁻³

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

Cell parameters from 1885 reflections

$\theta = 2.9$ – 22.6 °

$\mu = 0.94$ mm⁻¹

$T = 298$ K

Prismatic, brown

$0.43 \times 0.16 \times 0.06$ mm

Data collection

Siemens SMART 1000 CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 298$ K

φ and ω scans

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

$T_{\min} = 0.688$, $T_{\max} = 0.946$

10414 measured reflections

3782 independent reflections

2179 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.073$

$\theta_{\text{max}} = 25.0$ °

$\theta_{\text{min}} = 1.8$ °

$h = -16$ → 15

$k = -9$ → 9

$l = -22$ → 21

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.048$

$wR(F^2) = 0.126$

$S = 1.02$

3782 reflections

298 parameters

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0457P)^2 + 0.4257P]$$

where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\text{max}} = 0.45$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.38$ e Å⁻³

Primary atom site location: structure-invariant direct methods Extinction correction: none

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 (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|-------------|--------------|----------------------------------|
| Ni1 | 0.75831 (4) | 0.20469 (8) | 0.22591 (3) | 0.0404 (2) |
| N1 | 0.7696 (2) | 0.2159 (5) | 0.12766 (18) | 0.0427 (9) |
| N2 | 0.6275 (2) | 0.2675 (4) | 0.22573 (18) | 0.0414 (9) |
| O1 | 0.7023 (2) | 0.1391 (4) | 0.07716 (16) | 0.0612 (10) |
| O2 | 0.5751 (2) | 0.2984 (4) | 0.15860 (15) | 0.0476 (8) |
| O3 | 0.89260 (19) | 0.1753 (4) | 0.23858 (14) | 0.0468 (8) |
| O4 | 0.7640 (2) | 0.1617 (4) | 0.31931 (15) | 0.0513 (9) |
| C1 | 0.6314 (4) | 0.0466 (6) | 0.1102 (3) | 0.0589 (14) |
| H1A | 0.6614 | 0.0022 | 0.1542 | 0.071* |
| H1B | 0.6093 | -0.0428 | 0.0798 | 0.071* |
| C2 | 0.5455 (3) | 0.1494 (6) | 0.1245 (3) | 0.0548 (14) |
| H2A | 0.5070 | 0.1728 | 0.0804 | 0.066* |
| H2B | 0.5042 | 0.0898 | 0.1542 | 0.066* |
| C3 | 0.8419 (3) | 0.2790 (6) | 0.0185 (2) | 0.0577 (14) |
| H3A | 0.8183 | 0.1769 | -0.0002 | 0.087* |
| H3B | 0.9075 | 0.2965 | 0.0061 | 0.087* |
| H3C | 0.8001 | 0.3640 | -0.0010 | 0.087* |
| C4 | 0.8418 (3) | 0.2780 (6) | 0.0975 (2) | 0.0422 (12) |
| C5 | 0.9487 (3) | 0.2779 (5) | 0.2089 (2) | 0.0390 (11) |
| C6 | 0.9264 (3) | 0.3411 (5) | 0.1412 (2) | 0.0408 (12) |
| C7 | 0.9930 (4) | 0.4501 (6) | 0.1145 (2) | 0.0511 (13) |
| H7 | 0.9782 | 0.4920 | 0.0695 | 0.061* |
| C8 | 1.0781 (4) | 0.4966 (6) | 0.1518 (3) | 0.0561 (14) |
| H8 | 1.1192 | 0.5704 | 0.1325 | 0.067* |
| C9 | 1.1038 (3) | 0.4326 (6) | 0.2199 (3) | 0.0484 (12) |
| C10 | 1.0395 (3) | 0.3241 (5) | 0.2491 (2) | 0.0407 (11) |
| C11 | 1.0631 (3) | 0.2647 (6) | 0.3181 (2) | 0.0497 (13) |
| H11 | 1.0203 | 0.1940 | 0.3376 | 0.060* |
| C12 | 1.1481 (4) | 0.3094 (7) | 0.3565 (3) | 0.0621 (15) |
| H12 | 1.1630 | 0.2698 | 0.4019 | 0.075* |
| C13 | 1.2124 (4) | 0.4155 (7) | 0.3268 (3) | 0.0709 (17) |

supplementary materials

| | | | | |
|------|------------|------------|------------|-------------|
| H13 | 1.2707 | 0.4449 | 0.3526 | 0.085* |
| C14 | 1.1914 (4) | 0.4762 (6) | 0.2610 (3) | 0.0653 (15) |
| H14 | 1.2350 | 0.5474 | 0.2426 | 0.078* |
| C15 | 0.4730 (3) | 0.3664 (6) | 0.2637 (2) | 0.0547 (14) |
| H15A | 0.4715 | 0.4819 | 0.2589 | 0.082* |
| H15B | 0.4364 | 0.3351 | 0.3021 | 0.082* |
| H15C | 0.4443 | 0.3177 | 0.2209 | 0.082* |
| C16 | 0.5777 (3) | 0.3104 (5) | 0.2781 (2) | 0.0396 (11) |
| C17 | 0.7106 (3) | 0.2156 (6) | 0.3666 (2) | 0.0415 (11) |
| C18 | 0.6206 (3) | 0.2971 (5) | 0.3500 (2) | 0.0401 (11) |
| C19 | 0.5721 (3) | 0.3588 (6) | 0.4074 (3) | 0.0544 (13) |
| H19 | 0.5140 | 0.4160 | 0.3975 | 0.065* |
| C20 | 0.6067 (4) | 0.3380 (6) | 0.4755 (3) | 0.0588 (14) |
| H20 | 0.5727 | 0.3823 | 0.5109 | 0.071* |
| C21 | 0.6941 (3) | 0.2496 (6) | 0.4937 (2) | 0.0473 (13) |
| C22 | 0.7463 (3) | 0.1878 (6) | 0.4392 (2) | 0.0408 (11) |
| C23 | 0.8325 (3) | 0.0997 (6) | 0.4558 (2) | 0.0508 (13) |
| H23 | 0.8670 | 0.0590 | 0.4199 | 0.061* |
| C24 | 0.8665 (4) | 0.0730 (7) | 0.5248 (3) | 0.0624 (15) |
| H24 | 0.9239 | 0.0145 | 0.5354 | 0.075* |
| C25 | 0.8152 (4) | 0.1332 (7) | 0.5791 (3) | 0.0692 (17) |
| H25 | 0.8382 | 0.1138 | 0.6258 | 0.083* |
| C26 | 0.7319 (4) | 0.2199 (7) | 0.5639 (3) | 0.0620 (15) |
| H26 | 0.6990 | 0.2607 | 0.6006 | 0.074* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|------------|-------------|-------------|--------------|--------------|
| Ni1 | 0.0349 (3) | 0.0501 (4) | 0.0356 (4) | 0.0030 (3) | 0.0009 (2) | 0.0056 (3) |
| N1 | 0.033 (2) | 0.056 (3) | 0.038 (2) | 0.010 (2) | -0.0003 (17) | -0.002 (2) |
| N2 | 0.041 (2) | 0.046 (3) | 0.035 (2) | 0.0016 (18) | -0.0066 (17) | 0.0077 (18) |
| O1 | 0.048 (2) | 0.091 (3) | 0.043 (2) | -0.004 (2) | -0.0062 (17) | -0.0145 (18) |
| O2 | 0.0465 (18) | 0.047 (2) | 0.0471 (19) | 0.0065 (17) | -0.0069 (15) | 0.0045 (17) |
| O3 | 0.0344 (17) | 0.063 (2) | 0.0422 (19) | 0.0012 (16) | 0.0013 (14) | 0.0161 (16) |
| O4 | 0.0446 (18) | 0.072 (3) | 0.0380 (19) | 0.0152 (17) | 0.0097 (15) | 0.0141 (16) |
| C1 | 0.056 (3) | 0.048 (3) | 0.069 (4) | 0.001 (3) | -0.012 (3) | -0.011 (3) |
| C2 | 0.045 (3) | 0.057 (4) | 0.060 (3) | -0.003 (3) | -0.010 (2) | -0.003 (3) |
| C3 | 0.058 (3) | 0.083 (4) | 0.033 (3) | 0.022 (3) | 0.005 (2) | 0.009 (3) |
| C4 | 0.044 (3) | 0.052 (3) | 0.031 (2) | 0.019 (2) | 0.004 (2) | 0.004 (2) |
| C5 | 0.035 (3) | 0.042 (3) | 0.041 (3) | 0.009 (2) | 0.008 (2) | 0.003 (2) |
| C6 | 0.041 (3) | 0.043 (3) | 0.039 (3) | 0.010 (2) | 0.007 (2) | 0.010 (2) |
| C7 | 0.058 (3) | 0.054 (4) | 0.044 (3) | 0.011 (3) | 0.015 (3) | 0.009 (2) |
| C8 | 0.063 (4) | 0.043 (3) | 0.065 (4) | 0.001 (3) | 0.023 (3) | 0.005 (3) |
| C9 | 0.042 (3) | 0.046 (3) | 0.058 (3) | 0.003 (2) | 0.009 (3) | -0.004 (3) |
| C10 | 0.037 (3) | 0.043 (3) | 0.042 (3) | 0.008 (2) | 0.004 (2) | 0.000 (2) |
| C11 | 0.048 (3) | 0.053 (3) | 0.047 (3) | 0.004 (2) | 0.000 (2) | 0.002 (2) |
| C12 | 0.056 (3) | 0.073 (4) | 0.053 (3) | 0.002 (3) | -0.016 (3) | -0.005 (3) |
| C13 | 0.053 (4) | 0.065 (4) | 0.090 (5) | 0.001 (3) | -0.013 (3) | -0.018 (4) |

| | | | | | | |
|-----|-----------|-----------|-----------|------------|------------|------------|
| C14 | 0.054 (3) | 0.055 (4) | 0.088 (4) | -0.009 (3) | 0.012 (3) | -0.003 (3) |
| C15 | 0.040 (3) | 0.057 (4) | 0.067 (3) | 0.011 (3) | 0.005 (2) | 0.006 (3) |
| C16 | 0.033 (2) | 0.037 (3) | 0.049 (3) | -0.002 (2) | 0.006 (2) | 0.008 (2) |
| C17 | 0.038 (3) | 0.045 (3) | 0.042 (3) | -0.009 (2) | 0.006 (2) | 0.006 (2) |
| C18 | 0.043 (3) | 0.032 (3) | 0.046 (3) | -0.005 (2) | 0.007 (2) | 0.005 (2) |
| C19 | 0.050 (3) | 0.050 (3) | 0.065 (4) | 0.000 (3) | 0.012 (3) | -0.007 (3) |
| C20 | 0.066 (4) | 0.056 (4) | 0.056 (4) | -0.012 (3) | 0.019 (3) | -0.018 (3) |
| C21 | 0.048 (3) | 0.050 (4) | 0.044 (3) | -0.018 (2) | 0.005 (2) | -0.001 (2) |
| C22 | 0.041 (3) | 0.046 (3) | 0.034 (3) | -0.014 (2) | 0.001 (2) | 0.010 (2) |
| C23 | 0.045 (3) | 0.063 (4) | 0.044 (3) | -0.011 (3) | -0.002 (2) | 0.011 (3) |
| C24 | 0.052 (3) | 0.080 (4) | 0.054 (3) | -0.017 (3) | -0.003 (3) | 0.010 (3) |
| C25 | 0.071 (4) | 0.094 (5) | 0.040 (3) | -0.031 (4) | -0.011 (3) | 0.007 (3) |
| C26 | 0.080 (4) | 0.064 (4) | 0.043 (3) | -0.030 (3) | 0.007 (3) | -0.010 (3) |

Geometric parameters (Å, °)

| | | | |
|-----------|------------|------------|-----------|
| Ni1—O4 | 1.809 (3) | C10—C11 | 1.413 (6) |
| Ni1—O3 | 1.849 (3) | C11—C12 | 1.367 (6) |
| Ni1—N2 | 1.865 (3) | C11—H11 | 0.9300 |
| Ni1—N1 | 1.894 (3) | C12—C13 | 1.400 (7) |
| N1—C4 | 1.295 (5) | C12—H12 | 0.9300 |
| N1—O1 | 1.419 (4) | C13—C14 | 1.355 (7) |
| N2—C16 | 1.309 (5) | C13—H13 | 0.9300 |
| N2—O2 | 1.430 (4) | C14—H14 | 0.9300 |
| O1—C1 | 1.427 (5) | C15—C16 | 1.508 (5) |
| O2—C2 | 1.434 (5) | C15—H15A | 0.9600 |
| O3—C5 | 1.308 (5) | C15—H15B | 0.9600 |
| O4—C17 | 1.292 (5) | C15—H15C | 0.9600 |
| C1—C2 | 1.498 (6) | C16—C18 | 1.444 (6) |
| C1—H1A | 0.9700 | C17—C18 | 1.415 (6) |
| C1—H1B | 0.9700 | C17—C22 | 1.442 (6) |
| C2—H2A | 0.9700 | C18—C19 | 1.426 (6) |
| C2—H2B | 0.9700 | C19—C20 | 1.349 (6) |
| C3—C4 | 1.506 (5) | C19—H19 | 0.9300 |
| C3—H3A | 0.9600 | C20—C21 | 1.419 (7) |
| C3—H3B | 0.9600 | C20—H20 | 0.9300 |
| C3—H3C | 0.9600 | C21—C26 | 1.409 (6) |
| C4—C6 | 1.459 (6) | C21—C22 | 1.410 (6) |
| C5—C6 | 1.398 (5) | C22—C23 | 1.398 (6) |
| C5—C10 | 1.449 (6) | C23—C24 | 1.370 (6) |
| C6—C7 | 1.411 (6) | C23—H23 | 0.9300 |
| C7—C8 | 1.362 (6) | C24—C25 | 1.395 (7) |
| C7—H7 | 0.9300 | C24—H24 | 0.9300 |
| C8—C9 | 1.415 (6) | C25—C26 | 1.356 (7) |
| C8—H8 | 0.9300 | C25—H25 | 0.9300 |
| C9—C10 | 1.407 (6) | C26—H26 | 0.9300 |
| C9—C14 | 1.417 (6) | | |
| O4—Ni1—O3 | 83.93 (12) | C9—C10—C5 | 119.8 (4) |
| O4—Ni1—N2 | 90.58 (14) | C11—C10—C5 | 120.7 (4) |

supplementary materials

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| O3—Ni1—N2 | 168.72 (15) | C12—C11—C10 | 121.0 (5) |
| O4—Ni1—N1 | 168.87 (14) | C12—C11—H11 | 119.5 |
| O3—Ni1—N1 | 87.90 (13) | C10—C11—H11 | 119.5 |
| N2—Ni1—N1 | 98.73 (14) | C11—C12—C13 | 119.3 (5) |
| C4—N1—O1 | 110.6 (3) | C11—C12—H12 | 120.3 |
| C4—N1—Ni1 | 126.4 (3) | C13—C12—H12 | 120.3 |
| O1—N1—Ni1 | 122.7 (3) | C14—C13—C12 | 121.2 (5) |
| C16—N2—O2 | 112.3 (3) | C14—C13—H13 | 119.4 |
| C16—N2—Ni1 | 130.0 (3) | C12—C13—H13 | 119.4 |
| O2—N2—Ni1 | 117.0 (2) | C13—C14—C9 | 120.8 (5) |
| N1—O1—C1 | 111.5 (3) | C13—C14—H14 | 119.6 |
| N2—O2—C2 | 110.4 (3) | C9—C14—H14 | 119.6 |
| C5—O3—Ni1 | 118.4 (3) | C16—C15—H15A | 109.5 |
| C17—O4—Ni1 | 130.0 (3) | C16—C15—H15B | 109.5 |
| O1—C1—C2 | 110.7 (4) | H15A—C15—H15B | 109.5 |
| O1—C1—H1A | 109.5 | C16—C15—H15C | 109.5 |
| C2—C1—H1A | 109.5 | H15A—C15—H15C | 109.5 |
| O1—C1—H1B | 109.5 | H15B—C15—H15C | 109.5 |
| C2—C1—H1B | 109.5 | N2—C16—C18 | 120.5 (4) |
| H1A—C1—H1B | 108.1 | N2—C16—C15 | 119.9 (4) |
| O2—C2—C1 | 112.1 (4) | C18—C16—C15 | 119.5 (4) |
| O2—C2—H2A | 109.2 | O4—C17—C18 | 123.1 (4) |
| C1—C2—H2A | 109.2 | O4—C17—C22 | 116.8 (4) |
| O2—C2—H2B | 109.2 | C18—C17—C22 | 120.1 (4) |
| C1—C2—H2B | 109.2 | C17—C18—C19 | 117.2 (4) |
| H2A—C2—H2B | 107.9 | C17—C18—C16 | 121.2 (4) |
| C4—C3—H3A | 109.5 | C19—C18—C16 | 121.5 (4) |
| C4—C3—H3B | 109.5 | C20—C19—C18 | 123.0 (5) |
| H3A—C3—H3B | 109.5 | C20—C19—H19 | 118.5 |
| C4—C3—H3C | 109.5 | C18—C19—H19 | 118.5 |
| H3A—C3—H3C | 109.5 | C19—C20—C21 | 120.9 (5) |
| H3B—C3—H3C | 109.5 | C19—C20—H20 | 119.5 |
| N1—C4—C6 | 119.1 (4) | C21—C20—H20 | 119.5 |
| N1—C4—C3 | 121.2 (4) | C26—C21—C22 | 118.0 (5) |
| C6—C4—C3 | 119.6 (4) | C26—C21—C20 | 123.3 (5) |
| O3—C5—C6 | 123.6 (4) | C22—C21—C20 | 118.7 (4) |
| O3—C5—C10 | 116.9 (4) | C23—C22—C21 | 119.8 (4) |
| C6—C5—C10 | 119.5 (4) | C23—C22—C17 | 120.3 (4) |
| C5—C6—C7 | 118.5 (4) | C21—C22—C17 | 119.9 (4) |
| C5—C6—C4 | 119.1 (4) | C24—C23—C22 | 120.3 (5) |
| C7—C6—C4 | 121.9 (4) | C24—C23—H23 | 119.8 |
| C8—C7—C6 | 122.9 (4) | C22—C23—H23 | 119.8 |
| C8—C7—H7 | 118.5 | C23—C24—C25 | 120.3 (5) |
| C6—C7—H7 | 118.5 | C23—C24—H24 | 119.9 |
| C7—C8—C9 | 120.0 (5) | C25—C24—H24 | 119.9 |
| C7—C8—H8 | 120.0 | C26—C25—C24 | 120.2 (5) |
| C9—C8—H8 | 120.0 | C26—C25—H25 | 119.9 |
| C10—C9—C8 | 119.2 (4) | C24—C25—H25 | 119.9 |
| C10—C9—C14 | 118.3 (5) | C25—C26—C21 | 121.4 (5) |

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|----------------|------------|-----------------|------------|
| C8—C9—C14 | 122.5 (5) | C25—C26—H26 | 119.3 |
| C9—C10—C11 | 119.4 (4) | C21—C26—H26 | 119.3 |
| O4—Ni1—N1—C4 | -77.6 (9) | C14—C9—C10—C5 | 179.4 (4) |
| O3—Ni1—N1—C4 | -34.9 (4) | O3—C5—C10—C9 | 179.2 (4) |
| N2—Ni1—N1—C4 | 135.9 (4) | C6—C5—C10—C9 | 0.4 (6) |
| O4—Ni1—N1—O1 | 95.3 (8) | O3—C5—C10—C11 | -2.4 (6) |
| O3—Ni1—N1—O1 | 138.1 (3) | C6—C5—C10—C11 | 178.8 (4) |
| N2—Ni1—N1—O1 | -51.1 (3) | C9—C10—C11—C12 | -0.8 (7) |
| O4—Ni1—N2—C16 | 18.8 (4) | C5—C10—C11—C12 | -179.2 (4) |
| O3—Ni1—N2—C16 | -41.9 (9) | C10—C11—C12—C13 | -0.1 (7) |
| N1—Ni1—N2—C16 | -167.3 (4) | C11—C12—C13—C14 | 0.9 (8) |
| O4—Ni1—N2—O2 | -171.9 (3) | C12—C13—C14—C9 | -0.7 (8) |
| O3—Ni1—N2—O2 | 127.5 (6) | C10—C9—C14—C13 | -0.2 (7) |
| N1—Ni1—N2—O2 | 2.0 (3) | C8—C9—C14—C13 | 178.3 (5) |
| C4—N1—O1—C1 | 170.0 (4) | O2—N2—C16—C18 | -176.5 (4) |
| Ni1—N1—O1—C1 | -3.9 (5) | Ni1—N2—C16—C18 | -6.7 (6) |
| C16—N2—O2—C2 | -110.3 (4) | O2—N2—C16—C15 | 6.5 (6) |
| Ni1—N2—O2—C2 | 78.6 (3) | Ni1—N2—C16—C15 | 176.3 (3) |
| O4—Ni1—O3—C5 | -136.0 (3) | Ni1—O4—C17—C18 | 14.1 (6) |
| N2—Ni1—O3—C5 | -74.8 (8) | Ni1—O4—C17—C22 | -166.5 (3) |
| N1—Ni1—O3—C5 | 51.5 (3) | O4—C17—C18—C19 | -176.3 (4) |
| O3—Ni1—O4—C17 | 147.7 (4) | C22—C17—C18—C19 | 4.3 (6) |
| N2—Ni1—O4—C17 | -22.5 (4) | O4—C17—C18—C16 | 6.6 (7) |
| N1—Ni1—O4—C17 | -169.4 (7) | C22—C17—C18—C16 | -172.8 (4) |
| N1—O1—C1—C2 | 87.7 (4) | N2—C16—C18—C17 | -9.8 (7) |
| N2—O2—C2—C1 | -62.0 (5) | C15—C16—C18—C17 | 167.3 (4) |
| O1—C1—C2—O2 | -48.8 (5) | N2—C16—C18—C19 | 173.2 (4) |
| O1—N1—C4—C6 | -170.9 (4) | C15—C16—C18—C19 | -9.8 (7) |
| Ni1—N1—C4—C6 | 2.8 (6) | C17—C18—C19—C20 | -2.1 (7) |
| O1—N1—C4—C3 | 6.2 (6) | C16—C18—C19—C20 | 175.0 (4) |
| Ni1—N1—C4—C3 | 179.9 (3) | C18—C19—C20—C21 | -1.0 (7) |
| Ni1—O3—C5—C6 | -39.8 (5) | C19—C20—C21—C26 | -178.0 (4) |
| Ni1—O3—C5—C10 | 141.4 (3) | C19—C20—C21—C22 | 2.0 (7) |
| O3—C5—C6—C7 | -179.5 (4) | C26—C21—C22—C23 | 0.2 (6) |
| C10—C5—C6—C7 | -0.7 (6) | C20—C21—C22—C23 | -179.8 (4) |
| O3—C5—C6—C4 | -6.9 (6) | C26—C21—C22—C17 | -179.8 (4) |
| C10—C5—C6—C4 | 171.9 (4) | C20—C21—C22—C17 | 0.2 (6) |
| N1—C4—C6—C5 | 26.4 (6) | O4—C17—C22—C23 | -2.9 (6) |
| C3—C4—C6—C5 | -150.8 (4) | C18—C17—C22—C23 | 176.6 (4) |
| N1—C4—C6—C7 | -161.3 (4) | O4—C17—C22—C21 | 177.1 (4) |
| C3—C4—C6—C7 | 21.6 (6) | C18—C17—C22—C21 | -3.4 (6) |
| C5—C6—C7—C8 | -0.1 (7) | C21—C22—C23—C24 | 0.1 (7) |
| C4—C6—C7—C8 | -172.5 (4) | C17—C22—C23—C24 | -179.9 (4) |
| C6—C7—C8—C9 | 1.3 (7) | C22—C23—C24—C25 | 0.2 (7) |
| C7—C8—C9—C10 | -1.6 (7) | C23—C24—C25—C26 | -0.7 (8) |
| C7—C8—C9—C14 | 179.9 (5) | C24—C25—C26—C21 | 1.0 (8) |
| C8—C9—C10—C11 | -177.6 (4) | C22—C21—C26—C25 | -0.7 (7) |
| C14—C9—C10—C11 | 1.0 (6) | C20—C21—C26—C25 | 179.2 (5) |
| C8—C9—C10—C5 | 0.8 (6) | | |

supplementary materials

Hydrogen-bond geometry (Å, °)

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|--------------------------------------|-------|-------------|-------------|---------------|
| C15—H15A \cdots O3 ⁱ | 0.96 | 2.45 | 3.152 (6) | 130 |
| C3—H3C \cdots Cg1 ⁱⁱ | 0.97 | 3.17 | 4.127 (3) | 172 |
| C15—H15A \cdots Cg2 ⁱⁱⁱ | 0.96 | 3.53 | 4.398 (3) | 152 |

Symmetry codes: (i) $-x+3/2, y+1/2, -z+1/2$; (ii) $-x+1, -y, -z$; (iii) $x+1, y, z$.

Fig. 2

