# *trans*-Bis(2-benzoylbenzoato-$\kappa O^1$)bis-(ethanol-$\kappa O$)bis(1*H*-imidazole-$\kappa N^3$)-nickel(II)

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In the title centrosymmetric mononuclear nickel(II) complex, [Ni(C$_{14}$H$_9$O$_3$)$_2$(C$_3$H$_4$N$_2$)$_2$(CH$_3$CH$_2$OH)$_2$], the central Ni$^{II}$ ion lies on an inversion centre and is octahedrally coordinated. The equatorial plane is formed by two O atoms from two symmetry-related 2-benzoylbenzoate ligands and two N atoms from two symmetry-related imidazole ligands, whereas the axial positions are occupied by two O atoms from two ethanol ligands. Intramolecular O---H...O hydrogen bonds stabilize this arrangement. The molecules are linked into chains running along the *b* axis by N—H···O hydrogen bonds.

## Related literature

For crystal structures with 2-benzoylbenzoate ligands, see: Diop *et al.* (2006, 2007); Foreman *et al.* (2001); Jones *et al.* (1996); Martin & Valente (1998); Prout *et al.* (1996); Song *et al.* (2005); Yıldırım *et al.* (2009). For the crystal structure of 2-benzoylbenzoic acid, see: Lalancette *et al.* (1990). For graph-set notation, see: Bernstein *et al.* (1995).
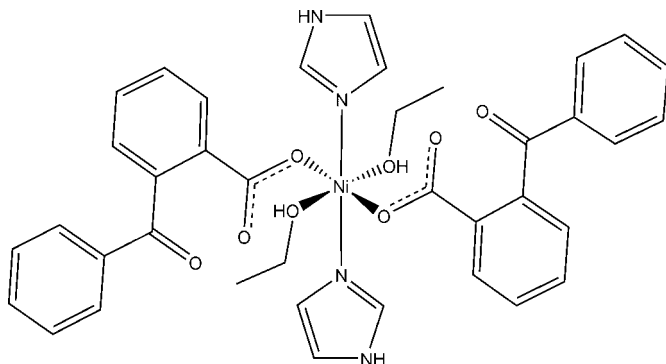


## Experimental

### Crystal data

[Ni(C$_{14}$H$_9$O$_3$)$_2$(C$_3$H$_4$N$_2$)$_2$(C$_2$H$_6$O)$_2$]
$M_r$ = 737.43
Monoclinic, $P2_1/c$
$a$ = 12.8914 (8) Å
$b$ = 8.3908 (5) Å
$c$ = 16.4590 (11) Å
$\beta$ = 90.832 (5)°

$V$ = 1780.17 (19) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.60 mm$^{-1}$
$T$ = 296 K
0.43 × 0.25 × 0.14 mm

### Data collection

Stoe IPDSII diffractometer
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
$T_{min}$ = 0.809, $T_{max}$ = 0.921

15078 measured reflections
3889 independent reflections
2704 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.046

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.150
$S$ = 1.11
3889 reflections
235 parameters

H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.31 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.26 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O1—H1···O3 | 0.78 (5) | 1.86 (5) | 2.627 (4) | 170 (6) |
| N2—H2···O3$^i$ | 0.86 | 2.02 | 2.837 (5) | 159 |

Symmetry code: (i) $x, y - 1, z$.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

## References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* 34, 1555–1573.
Diop, C. A. K., Touré, A., Diop, L., Tinant, B. & Mahieu, B. (2007). *Acta Cryst.* E63, m91–m93.
Diop, C. A. K., Touré, A., Diop, L. & Welter, R. (2006). *Acta Cryst.* E62, m3338–m3340.
Farrugia, L. J. (1997). *J. Appl. Cryst.* 30, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* 32, 837–838.
Foreman, M. R. J., Plater, M. J. & Skakle, M. S. (2001). *J. Chem. Soc. Dalton Trans.* pp. 1897–1903.
Jones, P. B., Pollastri, M. P. & Porter, N. A. (1996). *J. Org. Chem.* 61, 9455–9461.
Lalancette, R. A., Vanderhoff, P. A. & Thompson, H. W. (1990). *Acta Cryst.* C46, 1682–1686.
Martin, S. B. & Valente, J. (1998). *J. Chem. Crystallogr.* 28, 203–207.

# metal-organic compounds

Prout, K., Vaughan-Lee, D., Moloney, M. G. & Prottey, S. C. (1996). *Acta Cryst.* C**52**, 351–354.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Song, Y., Yan, B. & Chen, Z. (2005). *J. Coord. Chem.* **58**, 1417–1421.

Stoe & Cie (2002). *X-RED32* and *X-AREA*. Stoe & Cie, Darmstadt, Germany.

Yıldırım, M. H., Heren, Z., Paşaoğlu, H., Hıra, D. & Büyükgüngör, O. (2009). *Acta Cryst.* E**65**, m638–m639.

**supplementary materials**

# *trans*-Bis(2-benzoylbenzoato-$\kappa O^1$)bis(ethanol-$\kappa O$)bis(1*H*-imidazole-$\kappa N^3$)nickel(II)

## Z. Heren, H. Pasaoglu, M. H. Yildirim and D. Hira

## Comment

We have previously reported the crystal structure of [Cu(2-byba)$_2$(bim)$_2$] (bim = benzimidazole, 2-byba = 2-benzoylbenzoate) (Yıldırım *et al.*, 2009). As an extension of this study, we now report the structure of a new nickel(II) complex with the 2-byba ligand.

In the title complex, the Ni$^{II}$ ion lies on a centre of symmetry and has a octahedral coordination geometry formed by 2-byba, imidazole (im), ethanol ligands and their symmetry-related equivalents. All ligands are monodentate with the 2-byba coordinates through a carboxylate O atom, im coordinates through the aromatic N atom and ethanol coordinates through the O atom. Intramolecular O—H···O hydrogen bonds are observed.

The molecular packing is mainly stabilized by strong intermolecular N—H···O hydrogen bonds (Table 2 and Fig. 2). Atom N2 in the molecule at (*x*, *y*, *z*) acts as a hydrogen-bond donor, *via* H2, to atom O3 in the molecule at (*x*, *y* - 1, *z*) forming C(8) chains with $R_2^2$(16) rings (Bernstein *et al.*, 1995). These chains run parallel to the [010] (Fig. 2).

## Experimental

A solution of 2-benzoylbenzoate (0.45 g, 2 mmol) in ethanol (10 ml) was added to a solution of nickel acetate tetrahydrate (0.25 g, 1 mmol) in ethanol (10 ml) and the solution was stirred for 15 min at 333 K. To this solution, a solution of imidazole (0.23 g, 2 mmol) in ethanol (10 ml) was added and the resultant solution was left to evaporate slowly at room temperature. After one week, single crystals of the title complex were isolated.

## Refinement

Alcohol H atom was located in a difference Fourier map and its positional parameters were refined. The remaining H atoms were placed in calculated positions and constrained to ride on their parents atoms, with C-H = 0.93-0.97 Å, N-H = 0.86 Å and $U_{iso}$(H) = 1.2$U_{eq}$(C,N).
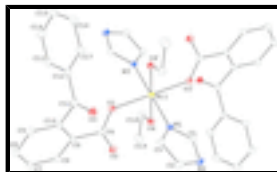
## Figures



Fig. 1. The molecular structure of the title complex, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 20% probability. Symmetry code: (i) 1 -*x*, 1 -*y*, 1 -*z*.
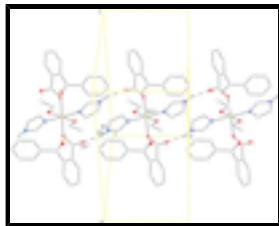
# supplementary materials



Fig. 2. Part of the crystal structure of the title compound showing the chain of $R_2^2(16)$ rings along [010] generated by N—H⋯O hydrogen bonds (dashed lines). H atoms not involved in hydrogen bonds have been omitted for clarity. Symmetry code: (ii) $x, y - 1, z$

## *trans*-Bis(2-benzoylbenzoato-$\kappa O^1$)bis(ethanol- $\kappa O$)bis(1*H*-imidazole-$\kappa N^3$)nickel(II)

*Crystal data*

| | |
|---|---|
| $[Ni(C_{14}H_9O_3)_2(C_3H_4N_2)_2(C_2H_6O)_2]$ | $F_{000} = 772$ |
| $M_r = 737.43$ | $D_x = 1.376$ Mg m$^{-3}$ |
| Monoclinic, $P2_1/c$ | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| Hall symbol: -P 2ybc | Cell parameters from 15078 reflections |
| $a = 12.8914$ (8) Å | $\theta = 1.6–27.6°$ |
| $b = 8.3908$ (5) Å | $\mu = 0.60$ mm$^{-1}$ |
| $c = 16.4590$ (11) Å | $T = 296$ K |
| $\beta = 90.832$ (5)° | Prism, green |
| $V = 1780.17$ (19) Å$^3$ | $0.43 \times 0.25 \times 0.14$ mm |
| $Z = 2$ | |

*Data collection*

| | |
|---|---|
| Stoe IPDSII diffractometer | 3889 independent reflections |
| Radiation source: fine-focus sealed tube | 2704 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{int} = 0.046$ |
| Detector resolution: 6.67 pixels mm$^{-1}$ | $\theta_{max} = 27.0°$ |
| $T = 296$ K | $\theta_{min} = 1.6°$ |
| $\omega$ scans | $h = -16 \rightarrow 16$ |
| Absorption correction: integration (X-RED32; Stoe & Cie, 2002) | $k = -10 \rightarrow 10$ |
| $T_{min} = 0.809$, $T_{max} = 0.921$ | $l = -20 \rightarrow 20$ |
| 15078 measured reflections | |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.055$ | H atoms treated by a mixture of independent and constrained refinement |
| $wR(F^2) = 0.150$ | $w = 1/[\sigma^2(F_o^2) + (0.0435P)^2 + 2.752P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.11$ | $(\Delta/\sigma)_{max} = 0.004$ |

| | |
|---|---|
| 3889 reflections | $\Delta\rho_{max} = 0.31$ e Å$^{-3}$ |
| 235 parameters | $\Delta\rho_{min} = -0.26$ 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 (Å²)*

| | $x$ | $y$ | $z$ | $U_{iso}*/U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.3658 (4) | 0.5474 (6) | 0.3388 (3) | 0.0630 (13) |
| H1A | 0.3057 | 0.4882 | 0.3568 | 0.076* |
| H1B | 0.4138 | 0.4723 | 0.3151 | 0.076* |
| C2 | 0.3320 (5) | 0.6663 (8) | 0.2749 (3) | 0.0848 (18) |
| H2A | 0.2993 | 0.6112 | 0.2303 | 0.127* |
| H2B | 0.3915 | 0.7231 | 0.2558 | 0.127* |
| H2C | 0.2838 | 0.7402 | 0.2979 | 0.127* |
| C3 | 0.5546 (4) | 0.1822 (5) | 0.4290 (3) | 0.0525 (11) |
| H3 | 0.6248 | 0.2074 | 0.4330 | 0.063* |
| C4 | 0.4127 (4) | 0.0507 (6) | 0.4036 (3) | 0.0623 (13) |
| H4 | 0.3661 | −0.0284 | 0.3879 | 0.075* |
| C5 | 0.3897 (4) | 0.1970 (6) | 0.4334 (3) | 0.0563 (11) |
| H5 | 0.3230 | 0.2356 | 0.4416 | 0.068* |
| C6 | 0.6557 (3) | 0.6330 (5) | 0.3827 (2) | 0.0448 (9) |
| C7 | 0.7620 (3) | 0.6345 (5) | 0.3466 (2) | 0.0452 (9) |
| C8 | 0.7755 (4) | 0.7083 (5) | 0.2710 (3) | 0.0548 (11) |
| H8 | 0.7192 | 0.7560 | 0.2448 | 0.066* |
| C9 | 0.8715 (4) | 0.7106 (6) | 0.2352 (3) | 0.0660 (13) |
| H9 | 0.8800 | 0.7617 | 0.1856 | 0.079* |
| C10 | 0.9537 (4) | 0.6380 (7) | 0.2724 (3) | 0.0724 (15) |
| H10 | 1.0181 | 0.6391 | 0.2477 | 0.087* |
| C11 | 0.9426 (4) | 0.5622 (6) | 0.3471 (3) | 0.0658 (13) |
| H11 | 0.9991 | 0.5124 | 0.3720 | 0.079* |
| C12 | 0.8466 (3) | 0.5616 (5) | 0.3841 (2) | 0.0454 (9) |
| C13 | 0.8422 (3) | 0.4876 (6) | 0.4681 (2) | 0.0481 (9) |
| C14 | 0.8461 (3) | 0.3113 (5) | 0.4742 (3) | 0.0463 (10) |
| C15 | 0.8481 (4) | 0.2143 (6) | 0.4068 (3) | 0.0616 (12) |
| H15 | 0.8446 | 0.2587 | 0.3551 | 0.074* |

| | | | | |
|---|---|---|---|---|
| C16 | 0.8554 (5) | 0.0506 (6) | 0.4159 (4) | 0.0829 (17) |
| H16 | 0.8574 | −0.0150 | 0.3704 | 0.100* |
| C17 | 0.8597 (4) | −0.0149 (7) | 0.4934 (4) | 0.0815 (16) |
| H17 | 0.8650 | −0.1249 | 0.4993 | 0.098* |
| C18 | 0.8564 (5) | 0.0769 (7) | 0.5591 (4) | 0.0770 (16) |
| H18 | 0.8585 | 0.0306 | 0.6104 | 0.092* |
| C19 | 0.8498 (4) | 0.2423 (6) | 0.5516 (3) | 0.0613 (12) |
| H19 | 0.8479 | 0.3062 | 0.5978 | 0.074* |
| N1 | 0.4796 (3) | 0.2796 (4) | 0.44961 (19) | 0.0431 (8) |
| N2 | 0.5184 (3) | 0.0434 (4) | 0.4016 (2) | 0.0601 (10) |
| H2 | 0.5548 | −0.0362 | 0.3857 | 0.072* |
| Ni1 | 0.5000 | 0.5000 | 0.5000 | 0.0385 (2) |
| O1 | 0.4142 (2) | 0.6220 (4) | 0.40705 (17) | 0.0489 (7) |
| O2 | 0.6371 (2) | 0.5278 (3) | 0.43480 (16) | 0.0462 (7) |
| O3 | 0.5923 (2) | 0.7370 (4) | 0.35828 (19) | 0.0566 (8) |
| O4 | 0.8429 (3) | 0.5724 (4) | 0.52801 (18) | 0.0585 (8) |
| H1 | 0.465 (4) | 0.665 (7) | 0.395 (3) | 0.070* |

*Atomic displacement parameters ($Å^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.080 (3) | 0.056 (3) | 0.052 (3) | −0.004 (2) | −0.011 (2) | −0.007 (2) |
| C2 | 0.106 (5) | 0.099 (5) | 0.049 (3) | 0.009 (4) | −0.017 (3) | 0.005 (3) |
| C3 | 0.062 (3) | 0.041 (2) | 0.054 (2) | −0.002 (2) | 0.007 (2) | −0.003 (2) |
| C4 | 0.071 (3) | 0.046 (3) | 0.070 (3) | −0.008 (2) | −0.009 (2) | −0.009 (2) |
| C5 | 0.058 (3) | 0.050 (3) | 0.061 (3) | −0.001 (2) | −0.003 (2) | −0.007 (2) |
| C6 | 0.053 (2) | 0.036 (2) | 0.045 (2) | 0.0040 (19) | 0.0011 (18) | −0.0035 (18) |
| C7 | 0.055 (2) | 0.040 (2) | 0.041 (2) | −0.0023 (19) | 0.0071 (18) | −0.0004 (17) |
| C8 | 0.067 (3) | 0.051 (3) | 0.047 (2) | −0.001 (2) | 0.006 (2) | 0.004 (2) |
| C9 | 0.075 (3) | 0.070 (3) | 0.054 (3) | −0.010 (3) | 0.017 (2) | 0.008 (2) |
| C10 | 0.067 (3) | 0.083 (4) | 0.068 (3) | −0.010 (3) | 0.025 (3) | 0.001 (3) |
| C11 | 0.056 (3) | 0.069 (3) | 0.073 (3) | 0.005 (2) | 0.014 (2) | 0.004 (3) |
| C12 | 0.047 (2) | 0.042 (2) | 0.047 (2) | −0.0058 (18) | 0.0040 (18) | −0.0009 (18) |
| C13 | 0.044 (2) | 0.049 (2) | 0.051 (2) | −0.001 (2) | 0.0021 (16) | −0.002 (2) |
| C14 | 0.042 (2) | 0.041 (2) | 0.056 (2) | −0.0002 (18) | 0.0046 (18) | 0.0022 (19) |
| C15 | 0.071 (3) | 0.052 (3) | 0.062 (3) | −0.003 (2) | 0.007 (2) | −0.006 (2) |
| C16 | 0.096 (4) | 0.045 (3) | 0.108 (5) | 0.001 (3) | 0.017 (4) | −0.020 (3) |
| C17 | 0.081 (4) | 0.044 (3) | 0.120 (5) | 0.000 (3) | 0.016 (3) | 0.015 (3) |
| C18 | 0.090 (4) | 0.059 (3) | 0.082 (4) | −0.001 (3) | 0.005 (3) | 0.022 (3) |
| C19 | 0.071 (3) | 0.051 (3) | 0.062 (3) | −0.004 (2) | 0.002 (2) | 0.006 (2) |
| N1 | 0.0491 (19) | 0.0369 (18) | 0.0434 (17) | −0.0001 (16) | 0.0029 (15) | −0.0017 (14) |
| N2 | 0.078 (3) | 0.042 (2) | 0.061 (2) | 0.0093 (19) | 0.0084 (19) | −0.0090 (17) |
| Ni1 | 0.0424 (4) | 0.0347 (4) | 0.0384 (3) | 0.0010 (4) | 0.0027 (3) | −0.0022 (3) |
| O1 | 0.0534 (18) | 0.0486 (19) | 0.0446 (15) | 0.0003 (14) | −0.0038 (13) | −0.0035 (13) |
| O2 | 0.0521 (16) | 0.0393 (17) | 0.0474 (14) | 0.0011 (13) | 0.0080 (12) | 0.0042 (12) |
| O3 | 0.0580 (19) | 0.0476 (18) | 0.0644 (18) | 0.0100 (15) | 0.0056 (15) | 0.0074 (15) |
| O4 | 0.078 (2) | 0.0487 (18) | 0.0485 (17) | 0.0015 (16) | −0.0019 (15) | −0.0077 (14) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| C1—O1 | 1.423 (5) | C10—H10 | 0.93 |
| C1—C2 | 1.509 (7) | C11—C12 | 1.387 (6) |
| C1—H1A | 0.97 | C11—H11 | 0.93 |
| C1—H1B | 0.97 | C12—C13 | 1.517 (6) |
| C2—H2A | 0.96 | C13—O4 | 1.216 (5) |
| C2—H2B | 0.96 | C13—C14 | 1.483 (6) |
| C2—H2C | 0.96 | C14—C15 | 1.377 (6) |
| C3—N1 | 1.315 (5) | C14—C19 | 1.400 (6) |
| C3—N2 | 1.331 (5) | C15—C16 | 1.384 (7) |
| C3—H3 | 0.93 | C15—H15 | 0.93 |
| C4—C5 | 1.356 (6) | C16—C17 | 1.389 (8) |
| C4—N2 | 1.365 (6) | C16—H16 | 0.93 |
| C4—H4 | 0.93 | C17—C18 | 1.329 (8) |
| C5—N1 | 1.373 (5) | C17—H17 | 0.93 |
| C5—H5 | 0.93 | C18—C19 | 1.396 (7) |
| C6—O2 | 1.257 (5) | C18—H18 | 0.93 |
| C6—O3 | 1.257 (5) | C19—H19 | 0.93 |
| C6—C7 | 1.502 (6) | N1—Ni1 | 2.042 (3) |
| C7—C12 | 1.388 (6) | N2—H2 | 0.8600 |
| C7—C8 | 1.403 (6) | Ni1—N1$^i$ | 2.042 (3) |
| C8—C9 | 1.378 (6) | Ni1—O2$^i$ | 2.094 (3) |
| C8—H8 | 0.93 | Ni1—O2 | 2.094 (3) |
| C9—C10 | 1.361 (7) | Ni1—O1$^i$ | 2.136 (3) |
| C9—H9 | 0.93 | Ni1—O1 | 2.136 (3) |
| C10—C11 | 1.394 (7) | O1—H1 | 0.78 (5) |
| O1—C1—C2 | 112.2 (4) | C14—C13—C12 | 117.9 (4) |
| O1—C1—H1A | 109.2 | C15—C14—C19 | 119.2 (4) |
| C2—C1—H1A | 109.2 | C15—C14—C13 | 122.4 (4) |
| O1—C1—H1B | 109.2 | C19—C14—C13 | 118.4 (4) |
| C2—C1—H1B | 109.2 | C14—C15—C16 | 120.1 (5) |
| H1A—C1—H1B | 107.9 | C14—C15—H15 | 119.9 |
| C1—C2—H2A | 109.5 | C16—C15—H15 | 119.9 |
| C1—C2—H2B | 109.5 | C15—C16—C17 | 119.6 (6) |
| H2A—C2—H2B | 109.5 | C15—C16—H16 | 120.2 |
| C1—C2—H2C | 109.5 | C17—C16—H16 | 120.2 |
| H2A—C2—H2C | 109.5 | C18—C17—C16 | 121.1 (6) |
| H2B—C2—H2C | 109.5 | C18—C17—H17 | 119.5 |
| N1—C3—N2 | 112.0 (4) | C16—C17—H17 | 119.5 |
| N1—C3—H3 | 124.0 | C17—C18—C19 | 120.5 (6) |
| N2—C3—H3 | 124.0 | C17—C18—H18 | 119.8 |
| C5—C4—N2 | 105.8 (4) | C19—C18—H18 | 119.8 |
| C5—C4—H4 | 127.1 | C18—C19—C14 | 119.5 (5) |
| N2—C4—H4 | 127.1 | C18—C19—H19 | 120.2 |
| C4—C5—N1 | 109.8 (4) | C14—C19—H19 | 120.2 |
| C4—C5—H5 | 125.1 | C3—N1—C5 | 105.0 (4) |

| | | | |
|---|---|---|---|
| N1—C5—H5 | 125.1 | C3—N1—Ni1 | 125.2 (3) |
| O2—C6—O3 | 125.1 (4) | C5—N1—Ni1 | 129.8 (3) |
| O2—C6—C7 | 117.4 (4) | C3—N2—C4 | 107.4 (4) |
| O3—C6—C7 | 117.4 (4) | C3—N2—H2 | 126.3 |
| C12—C7—C8 | 118.9 (4) | C4—N2—H2 | 126.3 |
| C12—C7—C6 | 122.4 (4) | N1$^i$—Ni1—N1 | 180.0 |
| C8—C7—C6 | 118.7 (4) | N1$^i$—Ni1—O2$^i$ | 89.87 (12) |
| C9—C8—C7 | 120.6 (5) | N1—Ni1—O2$^i$ | 90.13 (12) |
| C9—C8—H8 | 119.7 | N1$^i$—Ni1—O2 | 90.13 (12) |
| C7—C8—H8 | 119.7 | N1—Ni1—O2 | 89.87 (12) |
| C10—C9—C8 | 120.0 (5) | O2$^i$—Ni1—O2 | 180.0 |
| C10—C9—H9 | 120.0 | N1$^i$—Ni1—O1$^i$ | 94.64 (12) |
| C8—C9—H9 | 120.0 | N1—Ni1—O1$^i$ | 85.36 (12) |
| C9—C10—C11 | 120.7 (5) | O2$^i$—Ni1—O1$^i$ | 90.65 (11) |
| C9—C10—H10 | 119.6 | O2—Ni1—O1$^i$ | 89.35 (11) |
| C11—C10—H10 | 119.6 | N1$^i$—Ni1—O1 | 85.36 (12) |
| C12—C11—C10 | 119.6 (5) | N1—Ni1—O1 | 94.64 (12) |
| C12—C11—H11 | 120.2 | O2$^i$—Ni1—O1 | 89.35 (11) |
| C10—C11—H11 | 120.2 | O2—Ni1—O1 | 90.65 (11) |
| C11—C12—C7 | 120.2 (4) | O1$^i$—Ni1—O1 | 180.0 |
| C11—C12—C13 | 116.6 (4) | C1—O1—Ni1 | 124.9 (3) |
| C7—C12—C13 | 123.1 (4) | C1—O1—H1 | 112 (4) |
| O4—C13—C14 | 121.9 (4) | Ni1—O1—H1 | 88 (4) |
| O4—C13—C12 | 120.0 (4) | C6—O2—Ni1 | 126.8 (3) |
| N2—C4—C5—N1 | 0.0 (5) | C17—C18—C19—C14 | 0.4 (8) |
| O2—C6—C7—C12 | −20.1 (6) | C15—C14—C19—C18 | 0.6 (7) |
| O3—C6—C7—C12 | 160.0 (4) | C13—C14—C19—C18 | −178.3 (4) |
| O2—C6—C7—C8 | 158.0 (4) | N2—C3—N1—C5 | −0.6 (5) |
| O3—C6—C7—C8 | −21.9 (6) | N2—C3—N1—Ni1 | 176.6 (3) |
| C12—C7—C8—C9 | −1.1 (7) | C4—C5—N1—C3 | 0.3 (5) |
| C6—C7—C8—C9 | −179.2 (4) | C4—C5—N1—Ni1 | −176.7 (3) |
| C7—C8—C9—C10 | 1.4 (8) | N1—C3—N2—C4 | 0.7 (5) |
| C8—C9—C10—C11 | −0.7 (8) | C5—C4—N2—C3 | −0.4 (5) |
| C9—C10—C11—C12 | −0.4 (8) | C3—N1—Ni1—O2$^i$ | −147.8 (3) |
| C10—C11—C12—C7 | 0.7 (7) | C5—N1—Ni1—O2$^i$ | 28.7 (4) |
| C10—C11—C12—C13 | −175.6 (5) | C3—N1—Ni1—O2 | 32.2 (3) |
| C8—C7—C12—C11 | 0.0 (6) | C5—N1—Ni1—O2 | −151.3 (4) |
| C6—C7—C12—C11 | 178.1 (4) | C3—N1—Ni1—O1$^i$ | −57.2 (3) |
| C8—C7—C12—C13 | 176.1 (4) | C5—N1—Ni1—O1$^i$ | 119.3 (4) |
| C6—C7—C12—C13 | −5.9 (6) | C3—N1—Ni1—O1 | 122.8 (3) |
| C11—C12—C13—O4 | 100.0 (5) | C5—N1—Ni1—O1 | −60.7 (4) |
| C7—C12—C13—O4 | −76.2 (5) | C2—C1—O1—Ni1 | −167.2 (3) |
| C11—C12—C13—C14 | −74.9 (5) | N1$^i$—Ni1—O1—C1 | −179.8 (4) |
| C7—C12—C13—C14 | 108.9 (5) | N1—Ni1—O1—C1 | 0.2 (4) |
| O4—C13—C14—C15 | −178.2 (4) | O2$^i$—Ni1—O1—C1 | −89.8 (3) |

| | | | |
|---|---|---|---|
| C12—C13—C14—C15 | −3.4 (6) | O2—Ni1—O1—C1 | 90.2 (3) |
| O4—C13—C14—C19 | 0.6 (6) | O3—C6—O2—Ni1 | −3.1 (6) |
| C12—C13—C14—C19 | 175.4 (4) | C7—C6—O2—Ni1 | 177.0 (2) |
| C19—C14—C15—C16 | −1.0 (7) | N1$^{i}$—Ni1—O2—C6 | −61.3 (3) |
| C13—C14—C15—C16 | 177.8 (5) | N1—Ni1—O2—C6 | 118.7 (3) |
| C14—C15—C16—C17 | 0.6 (9) | O1$^{i}$—Ni1—O2—C6 | −155.9 (3) |
| C15—C16—C17—C18 | 0.4 (9) | O1—Ni1—O2—C6 | 24.1 (3) |
| C16—C17—C18—C19 | −0.8 (9) | | |

Symmetry codes: (i) −$x$+1, −$y$+1, −$z$+1.

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1—H1···O3 | 0.78 (5) | 1.86 (5) | 2.627 (4) | 170 (6) |
| N2—H2···O3$^{ii}$ | 0.86 | 2.02 | 2.837 (5) | 159 |

Symmetry codes: (ii) $x$, $y$−1, $z$.

**Fig. 1**

**Fig. 2**