

# Title

Shobhana Krishnaswamy

## Abstract

**Table 1**

Experimental details

|                                                                                                                |                                                                                                         |
|----------------------------------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------------|
| Crystal data                                                                                                   |                                                                                                         |
| Chemical formula                                                                                               | C <sub>24</sub> H <sub>32</sub> N <sub>2</sub> NiO <sub>4</sub>                                         |
| <i>M</i> <sub>r</sub>                                                                                          | 471.22                                                                                                  |
| Crystal system, space group                                                                                    | Monoclinic, <i>P</i> 2 <sub>1</sub> / <i>n</i>                                                          |
| Temperature (K)                                                                                                | 303                                                                                                     |
| <i>a</i> , <i>b</i> , <i>c</i> (Å)                                                                             | 6.2835 (5), 17.4797 (14), 21.2542 (17)                                                                  |
| β (°)                                                                                                          | 93.779 (3)                                                                                              |
| <i>V</i> (Å <sup>3</sup> )                                                                                     | 2329.4 (3)                                                                                              |
| <i>Z</i>                                                                                                       | 4                                                                                                       |
| Radiation type                                                                                                 | Mo <i>K</i> α                                                                                           |
| μ (mm <sup>−1</sup> )                                                                                          | 0.87                                                                                                    |
| Crystal size (mm)                                                                                              | 0.29 × 0.12 × 0.05                                                                                      |
| Data collection                                                                                                |                                                                                                         |
| Diffractometer                                                                                                 | Bruker D8 Venture Diffractometer                                                                        |
| Absorption correction                                                                                          | Multi-scan<br>Krause, L., Herbst-Irmer, R., Sheldrick G.M. & Stalke D., J. Appl. Cryst. 48 (2015) 3–10. |
| <i>T</i> <sub>min</sub> , <i>T</i> <sub>max</sub>                                                              | 0.636, 0.745                                                                                            |
| No. of measured, independent<br>and<br>observed [ <i>I</i> > 2σ( <i>I</i> )]<br>reflections                    | 43485, 4423, 3014                                                                                       |
| <i>R</i> <sub>int</sub>                                                                                        | 0.079                                                                                                   |
| (sin θ/λ) <sub>max</sub> (Å <sup>−1</sup> )                                                                    | 0.610                                                                                                   |
| Refinement                                                                                                     |                                                                                                         |
| <i>R</i> [ <i>F</i> <sup>2</sup> > 2σ( <i>F</i> <sup>2</sup> )], <i>wR</i> ( <i>F</i> <sup>2</sup> ), <i>S</i> | 0.047, 0.096, 1.07                                                                                      |
| No. of reflections                                                                                             | 4423                                                                                                    |
| No. of parameters                                                                                              | 286                                                                                                     |
| H-atom treatment                                                                                               | H-atom parameters constrained                                                                           |
| Δρ <sub>max</sub> , Δρ <sub>min</sub> (e Å <sup>−3</sup> )                                                     | 0.39, −0.31                                                                                             |

Computer programs: *APEX4* (Bruker, 2021), *APEX4/SAINT* (Bruker, 2021), *SAINT/XPREF* (Bruker, 2021), *SHELXT* 2018/2 (Sheldrick, 2018), *SHELXL2019/2* (Sheldrick, 2019), *WinGX*, *ORTEP-3* (Farrugia, 2012).

**Table 2**

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H⋯ <i>A</i> | <i>D</i> —H | H⋯ <i>A</i> | <i>D</i> ⋯ <i>A</i> | <i>D</i> —H⋯ <i>A</i> |
|-----------------------|-------------|-------------|---------------------|-----------------------|
| C10—H10⋯O3            | 0.98        | 2.61        | 3.126 (4)           | 113                   |

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|              |      |      |           |     |
|--------------|------|------|-----------|-----|
| C22—H22···O1 | 0.98 | 2.63 | 3.138 (4) | 113 |
|--------------|------|------|-----------|-----|

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**Acknowledgements**

**Funding information**

**References**

**Figure 1**

## supporting information

## Title

## Computing details

Data collection: *APEX4* (Bruker, 2021); cell refinement: *APEX4/SAINT* (Bruker, 2021); data reduction: *SAINT/XPREP* (Bruker, 2021); program(s) used to solve structure: *SHELXT* 2018/2 (Sheldrick, 2018); program(s) used to refine structure: *SHELXL2019/2* (Sheldrick, 2019), *WinGX*; molecular graphics: *ORTEP-3* (Farrugia, 2012).

## (shelx)

## Crystal data

$\text{C}_{24}\text{H}_{32}\text{N}_2\text{NiO}_4$

$M_r = 471.22$

Monoclinic,  $P2_1/n$

$a = 6.2835$  (5) Å

$b = 17.4797$  (14) Å

$c = 21.2542$  (17) Å

$\beta = 93.779$  (3)°

$V = 2329.4$  (3) Å<sup>3</sup>

$Z = 4$

$F(000) = 1000$

$D_x = 1.344$  Mg m<sup>-3</sup>

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

Cell parameters from 8021 reflections

$\theta = 3.0\text{--}25.3^\circ$

$\mu = 0.87$  mm<sup>-1</sup>

$T = 303$  K

Block, green

$0.29 \times 0.12 \times 0.05$  mm

## Data collection

Bruker D8 Venture Diffractometer

Radiation source: fine focus sealed tube

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan

Krause, L., Herbst-Irmer, R., Sheldrick G.M. &

Stalke D., J. Appl. Cryst. 48 (2015) 3-10.

$T_{\min} = 0.636$ ,  $T_{\max} = 0.745$

43485 measured reflections

4423 independent reflections

3014 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.079$

$\theta_{\max} = 25.7^\circ$ ,  $\theta_{\min} = 3.3^\circ$

$h = -6 \rightarrow 7$

$k = -21 \rightarrow 21$

$l = -25 \rightarrow 25$

## Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.047$

$wR(F^2) = 0.096$

$S = 1.07$

4423 reflections

286 parameters

0 restraints

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0188P)^2 + 2.7467P]$

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

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

$\Delta\rho_{\max} = 0.39$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.31$  e Å<sup>-3</sup>

## 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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å<sup>2</sup>)

|     | <i>x</i>    | <i>y</i>    | <i>z</i>    | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|-------------|-------------|-------------|----------------------------------|
| Ni1 | 0.49697 (8) | 0.66443 (2) | 0.25183 (2) | 0.03225 (12)                     |

|      |             |              |              |             |
|------|-------------|--------------|--------------|-------------|
| C1   | 0.7815 (5)  | 0.76319 (17) | 0.19047 (15) | 0.0341 (7)  |
| C2   | 0.9742 (5)  | 0.80743 (17) | 0.19487 (16) | 0.0370 (8)  |
| C3   | 1.0423 (6)  | 0.84376 (18) | 0.14306 (17) | 0.0453 (9)  |
| H3   | 1.167669    | 0.872155     | 0.146611     | 0.054*      |
| C4   | 0.9269 (7)  | 0.8390 (2)   | 0.08484 (18) | 0.0540 (10) |
| H4   | 0.976028    | 0.863902     | 0.049952     | 0.065*      |
| C5   | 0.7426 (6)  | 0.7980 (2)   | 0.07897 (17) | 0.0507 (9)  |
| H5   | 0.666090    | 0.795063     | 0.040064     | 0.061*      |
| C6   | 0.6666 (5)  | 0.75984 (18) | 0.13180 (15) | 0.0381 (8)  |
| C7   | 0.4718 (5)  | 0.71785 (18) | 0.12445 (15) | 0.0392 (8)  |
| H7   | 0.398854    | 0.721039     | 0.085027     | 0.047*      |
| C8   | 1.2618 (6)  | 0.8546 (2)   | 0.26158 (19) | 0.0575 (10) |
| H8A  | 1.368306    | 0.834790     | 0.235600     | 0.086*      |
| H8B  | 1.314874    | 0.853418     | 0.304966     | 0.086*      |
| H8C  | 1.228750    | 0.906415     | 0.249482     | 0.086*      |
| C9   | 0.1854 (5)  | 0.6372 (2)   | 0.14182 (16) | 0.0419 (8)  |
| H9A  | 0.121392    | 0.666478     | 0.106710     | 0.050*      |
| H9B  | 0.085733    | 0.636840     | 0.174773     | 0.050*      |
| C10  | 0.2198 (6)  | 0.5554 (2)   | 0.12018 (17) | 0.0518 (10) |
| H10  | 0.272902    | 0.525097     | 0.156714     | 0.062*      |
| C11  | 0.3781 (7)  | 0.5489 (2)   | 0.0697 (2)   | 0.0734 (13) |
| H11A | 0.326705    | 0.577306     | 0.033105     | 0.110*      |
| H11B | 0.394555    | 0.496104     | 0.058509     | 0.110*      |
| H11C | 0.513314    | 0.569164     | 0.085373     | 0.110*      |
| C12  | 0.0044 (8)  | 0.5230 (3)   | 0.0959 (2)   | 0.0973 (19) |
| H12A | −0.095601   | 0.528032     | 0.127907     | 0.146*      |
| H12B | 0.020184    | 0.469872     | 0.085600     | 0.146*      |
| H12C | −0.047020   | 0.550558     | 0.058913     | 0.146*      |
| C13  | 0.2146 (5)  | 0.56416 (17) | 0.31303 (15) | 0.0346 (7)  |
| C14  | 0.0220 (5)  | 0.52024 (18) | 0.30821 (16) | 0.0401 (8)  |
| C15  | −0.0434 (6) | 0.48094 (19) | 0.35952 (17) | 0.0455 (9)  |
| H15  | −0.167604   | 0.451916     | 0.355556     | 0.055*      |
| C16  | 0.0739 (6)  | 0.4840 (2)   | 0.41737 (18) | 0.0537 (10) |
| H16  | 0.026985    | 0.457741     | 0.451882     | 0.064*      |
| C17  | 0.2586 (6)  | 0.5258 (2)   | 0.42341 (16) | 0.0495 (9)  |
| H17  | 0.336761    | 0.527714     | 0.462079     | 0.059*      |
| C18  | 0.3310 (5)  | 0.56605 (18) | 0.37130 (15) | 0.0379 (8)  |
| C19  | 0.5243 (6)  | 0.60961 (18) | 0.37889 (15) | 0.0404 (8)  |
| H19  | 0.597991    | 0.606417     | 0.418216     | 0.048*      |
| C20  | −0.2703 (6) | 0.4747 (2)   | 0.2415 (2)   | 0.0585 (11) |
| H20A | −0.233913   | 0.422276     | 0.250152     | 0.088*      |
| H20B | −0.328288   | 0.479357     | 0.198700     | 0.088*      |
| H20C | −0.374222   | 0.491424     | 0.269625     | 0.088*      |
| C21  | 0.8017 (5)  | 0.6945 (2)   | 0.36294 (16) | 0.0444 (8)  |
| H21A | 0.902902    | 0.696972     | 0.330468     | 0.053*      |
| H21B | 0.868113    | 0.666394     | 0.398379     | 0.053*      |
| C22  | 0.7512 (6)  | 0.7754 (2)   | 0.38415 (17) | 0.0495 (9)  |
| H22  | 0.689503    | 0.803689     | 0.347514     | 0.059*      |
| C23  | 0.5938 (8)  | 0.7779 (3)   | 0.4352 (2)   | 0.0758 (13) |
| H23A | 0.460968    | 0.755672     | 0.419472     | 0.114*      |
| H23B | 0.570708    | 0.830039     | 0.447139     | 0.114*      |
| H23C | 0.649900    | 0.749497     | 0.471197     | 0.114*      |

|      |             |              |              |             |
|------|-------------|--------------|--------------|-------------|
| C24  | 0.9588 (8)  | 0.8151 (3)   | 0.4073 (2)   | 0.0831 (16) |
| H24A | 0.933990    | 0.868958     | 0.411426     | 0.125*      |
| H24B | 1.064788    | 0.806738     | 0.377462     | 0.125*      |
| H24C | 1.008077    | 0.794341     | 0.447466     | 0.125*      |
| N1   | 0.3835 (4)  | 0.67572 (14) | 0.16584 (12) | 0.0345 (6)  |
| N2   | 0.6089 (4)  | 0.65264 (14) | 0.33807 (12) | 0.0355 (6)  |
| O1   | 0.7208 (4)  | 0.72961 (13) | 0.24107 (10) | 0.0416 (6)  |
| O2   | 1.0740 (4)  | 0.80901 (13) | 0.25368 (11) | 0.0486 (6)  |
| O3   | 0.2716 (4)  | 0.59953 (13) | 0.26288 (10) | 0.0428 (6)  |
| O4   | -0.0841 (4) | 0.52091 (14) | 0.25020 (12) | 0.0506 (6)  |

*Atomic displacement parameters ( $\text{\AA}^2$ )*

|     | $U^{11}$    | $U^{22}$    | $U^{33}$    | $U^{12}$      | $U^{13}$     | $U^{23}$     |
|-----|-------------|-------------|-------------|---------------|--------------|--------------|
| Ni1 | 0.0287 (2)  | 0.0323 (2)  | 0.0358 (2)  | -0.00384 (18) | 0.00290 (14) | 0.00030 (18) |
| C1  | 0.0342 (18) | 0.0274 (16) | 0.0415 (19) | 0.0010 (13)   | 0.0079 (15)  | -0.0022 (14) |
| C2  | 0.0332 (19) | 0.0288 (16) | 0.050 (2)   | 0.0010 (14)   | 0.0117 (16)  | -0.0022 (15) |
| C3  | 0.043 (2)   | 0.037 (2)   | 0.058 (2)   | -0.0049 (15)  | 0.0189 (18)  | -0.0013 (17) |
| C4  | 0.071 (3)   | 0.043 (2)   | 0.051 (2)   | -0.008 (2)    | 0.027 (2)    | 0.0017 (18)  |
| C5  | 0.065 (3)   | 0.046 (2)   | 0.041 (2)   | -0.0052 (19)  | 0.0067 (18)  | 0.0004 (16)  |
| C6  | 0.044 (2)   | 0.0319 (17) | 0.0397 (19) | -0.0020 (15)  | 0.0093 (15)  | -0.0017 (14) |
| C7  | 0.046 (2)   | 0.0353 (18) | 0.0363 (18) | 0.0048 (15)   | -0.0014 (15) | -0.0015 (15) |
| C8  | 0.042 (2)   | 0.058 (2)   | 0.072 (3)   | -0.0152 (18)  | 0.0010 (19)  | 0.002 (2)    |
| C9  | 0.036 (2)   | 0.047 (2)   | 0.0420 (19) | -0.0001 (16)  | -0.0013 (15) | 0.0035 (16)  |
| C10 | 0.061 (3)   | 0.048 (2)   | 0.045 (2)   | -0.0114 (18)  | -0.0057 (18) | -0.0006 (17) |
| C11 | 0.081 (3)   | 0.059 (3)   | 0.082 (3)   | 0.004 (2)     | 0.013 (3)    | -0.018 (2)   |
| C12 | 0.095 (4)   | 0.118 (4)   | 0.078 (3)   | -0.060 (3)    | 0.002 (3)    | -0.030 (3)   |
| C13 | 0.0345 (19) | 0.0323 (17) | 0.0381 (18) | 0.0014 (14)   | 0.0101 (14)  | -0.0026 (14) |
| C14 | 0.037 (2)   | 0.0358 (18) | 0.049 (2)   | -0.0010 (15)  | 0.0089 (16)  | -0.0046 (16) |
| C15 | 0.045 (2)   | 0.0369 (19) | 0.057 (2)   | -0.0088 (16)  | 0.0192 (18)  | -0.0043 (17) |
| C16 | 0.069 (3)   | 0.046 (2)   | 0.049 (2)   | -0.010 (2)    | 0.023 (2)    | 0.0027 (18)  |
| C17 | 0.066 (3)   | 0.047 (2)   | 0.0353 (19) | -0.0074 (19)  | 0.0073 (17)  | 0.0005 (16)  |
| C18 | 0.045 (2)   | 0.0309 (17) | 0.0386 (19) | -0.0042 (15)  | 0.0074 (15)  | -0.0032 (14) |
| C19 | 0.049 (2)   | 0.0366 (19) | 0.0346 (18) | 0.0002 (16)   | -0.0016 (16) | -0.0015 (15) |
| C20 | 0.035 (2)   | 0.062 (3)   | 0.078 (3)   | -0.0151 (18)  | -0.0023 (19) | 0.003 (2)    |
| C21 | 0.037 (2)   | 0.053 (2)   | 0.043 (2)   | -0.0036 (16)  | -0.0073 (15) | 0.0076 (17)  |
| C22 | 0.059 (3)   | 0.044 (2)   | 0.044 (2)   | -0.0093 (18)  | -0.0064 (18) | -0.0011 (17) |
| C23 | 0.078 (3)   | 0.067 (3)   | 0.084 (3)   | -0.009 (2)    | 0.018 (3)    | -0.027 (2)   |
| C24 | 0.092 (4)   | 0.090 (3)   | 0.067 (3)   | -0.053 (3)    | 0.003 (3)    | -0.011 (3)   |
| N1  | 0.0354 (16) | 0.0292 (14) | 0.0390 (15) | -0.0008 (12)  | 0.0029 (12)  | -0.0017 (12) |
| N2  | 0.0315 (16) | 0.0349 (15) | 0.0400 (16) | -0.0032 (12)  | 0.0012 (12)  | -0.0009 (12) |
| O1  | 0.0363 (14) | 0.0497 (14) | 0.0386 (13) | -0.0125 (11)  | 0.0023 (10)  | 0.0051 (11)  |
| O2  | 0.0411 (15) | 0.0502 (14) | 0.0542 (15) | -0.0171 (11)  | -0.0001 (12) | 0.0085 (12)  |
| O3  | 0.0405 (14) | 0.0516 (14) | 0.0360 (13) | -0.0143 (11)  | 0.0000 (10)  | 0.0042 (11)  |
| O4  | 0.0320 (13) | 0.0581 (16) | 0.0612 (16) | -0.0107 (11)  | -0.0001 (12) | 0.0085 (13)  |

*Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )*

|        |           |          |           |
|--------|-----------|----------|-----------|
| Ni1—O1 | 1.836 (2) | C12—H12B | 0.9600    |
| Ni1—O3 | 1.842 (2) | C12—H12C | 0.9600    |
| Ni1—N1 | 1.928 (3) | C13—O3   | 1.303 (4) |
| Ni1—N2 | 1.930 (3) | C13—C18  | 1.397 (5) |

|           |             |               |           |
|-----------|-------------|---------------|-----------|
| C1—O1     | 1.304 (4)   | C13—C14       | 1.431 (4) |
| C1—C6     | 1.400 (5)   | C14—O4        | 1.363 (4) |
| C1—C2     | 1.434 (4)   | C14—C15       | 1.374 (4) |
| C2—O2     | 1.361 (4)   | C15—C16       | 1.392 (5) |
| C2—C3     | 1.364 (4)   | C15—H15       | 0.9300    |
| C3—C4     | 1.395 (5)   | C16—C17       | 1.370 (5) |
| C3—H3     | 0.9300      | C16—H16       | 0.9300    |
| C4—C5     | 1.360 (5)   | C17—C18       | 1.412 (4) |
| C4—H4     | 0.9300      | C17—H17       | 0.9300    |
| C5—C6     | 1.416 (4)   | C18—C19       | 1.434 (5) |
| C5—H5     | 0.9300      | C19—N2        | 1.289 (4) |
| C6—C7     | 1.427 (5)   | C19—H19       | 0.9300    |
| C7—N1     | 1.300 (4)   | C20—O4        | 1.423 (4) |
| C7—H7     | 0.9300      | C20—H20A      | 0.9600    |
| C8—O2     | 1.425 (4)   | C20—H20B      | 0.9600    |
| C8—H8A    | 0.9600      | C20—H20C      | 0.9600    |
| C8—H8B    | 0.9600      | C21—N2        | 1.482 (4) |
| C8—H8C    | 0.9600      | C21—C22       | 1.525 (5) |
| C9—N1     | 1.476 (4)   | C21—H21A      | 0.9700    |
| C9—C10    | 1.522 (5)   | C21—H21B      | 0.9700    |
| C9—H9A    | 0.9700      | C22—C23       | 1.516 (5) |
| C9—H9B    | 0.9700      | C22—C24       | 1.529 (5) |
| C10—C11   | 1.515 (5)   | C22—H22       | 0.9800    |
| C10—C12   | 1.526 (5)   | C23—H23A      | 0.9600    |
| C10—H10   | 0.9800      | C23—H23B      | 0.9600    |
| C11—H11A  | 0.9600      | C23—H23C      | 0.9600    |
| C11—H11B  | 0.9600      | C24—H24A      | 0.9600    |
| C11—H11C  | 0.9600      | C24—H24B      | 0.9600    |
| C12—H12A  | 0.9600      | C24—H24C      | 0.9600    |
| O1—Ni1—O3 | 179.65 (12) | C18—C13—C14   | 117.8 (3) |
| O1—Ni1—N1 | 93.26 (10)  | O4—C14—C15    | 124.6 (3) |
| O3—Ni1—N1 | 86.81 (10)  | O4—C14—C13    | 114.9 (3) |
| O1—Ni1—N2 | 87.16 (10)  | C15—C14—C13   | 120.5 (3) |
| O3—Ni1—N2 | 92.77 (10)  | C14—C15—C16   | 120.8 (3) |
| N1—Ni1—N2 | 179.57 (13) | C14—C15—H15   | 119.6     |
| O1—C1—C6  | 123.6 (3)   | C16—C15—H15   | 119.6     |
| O1—C1—C2  | 118.7 (3)   | C17—C16—C15   | 120.0 (3) |
| C6—C1—C2  | 117.6 (3)   | C17—C16—H16   | 120.0     |
| O2—C2—C3  | 125.6 (3)   | C15—C16—H16   | 120.0     |
| O2—C2—C1  | 114.0 (3)   | C16—C17—C18   | 120.4 (3) |
| C3—C2—C1  | 120.4 (3)   | C16—C17—H17   | 119.8     |
| C2—C3—C4  | 121.1 (3)   | C18—C17—H17   | 119.8     |
| C2—C3—H3  | 119.5       | C13—C18—C17   | 120.4 (3) |
| C4—C3—H3  | 119.5       | C13—C18—C19   | 120.2 (3) |
| C5—C4—C3  | 120.2 (3)   | C17—C18—C19   | 119.4 (3) |
| C5—C4—H4  | 119.9       | N2—C19—C18    | 128.2 (3) |
| C3—C4—H4  | 119.9       | N2—C19—H19    | 115.9     |
| C4—C5—C6  | 120.3 (4)   | C18—C19—H19   | 115.9     |
| C4—C5—H5  | 119.8       | O4—C20—H20A   | 109.5     |
| C6—C5—H5  | 119.8       | O4—C20—H20B   | 109.5     |
| C1—C6—C5  | 120.4 (3)   | H20A—C20—H20B | 109.5     |

|               |            |                 |            |
|---------------|------------|-----------------|------------|
| C1—C6—C7      | 120.5 (3)  | O4—C20—H20C     | 109.5      |
| C5—C6—C7      | 119.1 (3)  | H20A—C20—H20C   | 109.5      |
| N1—C7—C6      | 128.2 (3)  | H20B—C20—H20C   | 109.5      |
| N1—C7—H7      | 115.9      | N2—C21—C22      | 112.5 (3)  |
| C6—C7—H7      | 115.9      | N2—C21—H21A     | 109.1      |
| O2—C8—H8A     | 109.5      | C22—C21—H21A    | 109.1      |
| O2—C8—H8B     | 109.5      | N2—C21—H21B     | 109.1      |
| H8A—C8—H8B    | 109.5      | C22—C21—H21B    | 109.1      |
| O2—C8—H8C     | 109.5      | H21A—C21—H21B   | 107.8      |
| H8A—C8—H8C    | 109.5      | C23—C22—C21     | 113.3 (3)  |
| H8B—C8—H8C    | 109.5      | C23—C22—C24     | 109.9 (3)  |
| N1—C9—C10     | 113.6 (3)  | C21—C22—C24     | 109.0 (3)  |
| N1—C9—H9A     | 108.9      | C23—C22—H22     | 108.2      |
| C10—C9—H9A    | 108.9      | C21—C22—H22     | 108.2      |
| N1—C9—H9B     | 108.9      | C24—C22—H22     | 108.2      |
| C10—C9—H9B    | 108.9      | C22—C23—H23A    | 109.5      |
| H9A—C9—H9B    | 107.7      | C22—C23—H23B    | 109.5      |
| C11—C10—C9    | 113.4 (3)  | H23A—C23—H23B   | 109.5      |
| C11—C10—C12   | 110.0 (3)  | C22—C23—H23C    | 109.5      |
| C9—C10—C12    | 108.1 (4)  | H23A—C23—H23C   | 109.5      |
| C11—C10—H10   | 108.4      | H23B—C23—H23C   | 109.5      |
| C9—C10—H10    | 108.4      | C22—C24—H24A    | 109.5      |
| C12—C10—H10   | 108.4      | C22—C24—H24B    | 109.5      |
| C10—C11—H11A  | 109.5      | H24A—C24—H24B   | 109.5      |
| C10—C11—H11B  | 109.5      | C22—C24—H24C    | 109.5      |
| H11A—C11—H11B | 109.5      | H24A—C24—H24C   | 109.5      |
| C10—C11—H11C  | 109.5      | H24B—C24—H24C   | 109.5      |
| H11A—C11—H11C | 109.5      | C7—N1—C9        | 114.4 (3)  |
| H11B—C11—H11C | 109.5      | C7—N1—Ni1       | 123.5 (2)  |
| C10—C12—H12A  | 109.5      | C9—N1—Ni1       | 122.1 (2)  |
| C10—C12—H12B  | 109.5      | C19—N2—C21      | 114.3 (3)  |
| H12A—C12—H12B | 109.5      | C19—N2—Ni1      | 124.1 (2)  |
| C10—C12—H12C  | 109.5      | C21—N2—Ni1      | 121.6 (2)  |
| H12A—C12—H12C | 109.5      | C1—O1—Ni1       | 130.6 (2)  |
| H12B—C12—H12C | 109.5      | C2—O2—C8        | 116.6 (3)  |
| O3—C13—C18    | 123.9 (3)  | C13—O3—Ni1      | 130.7 (2)  |
| O3—C13—C14    | 118.2 (3)  | C14—O4—C20      | 117.5 (3)  |
| O1—C1—C2—O2   | 0.0 (4)    | O3—C13—C18—C19  | 0.9 (5)    |
| C6—C1—C2—O2   | 178.8 (3)  | C14—C13—C18—C19 | −179.5 (3) |
| O1—C1—C2—C3   | −179.6 (3) | C16—C17—C18—C13 | 0.4 (5)    |
| C6—C1—C2—C3   | −0.9 (4)   | C16—C17—C18—C19 | 179.8 (3)  |
| O2—C2—C3—C4   | −179.5 (3) | C13—C18—C19—N2  | 3.1 (5)    |
| C1—C2—C3—C4   | 0.1 (5)    | C17—C18—C19—N2  | −176.2 (3) |
| C2—C3—C4—C5   | 0.3 (5)    | N2—C21—C22—C23  | 58.9 (4)   |
| C3—C4—C5—C6   | −0.1 (6)   | N2—C21—C22—C24  | −178.4 (3) |
| O1—C1—C6—C5   | 179.8 (3)  | C6—C7—N1—C9     | −177.4 (3) |
| C2—C1—C6—C5   | 1.1 (5)    | C6—C7—N1—Ni1    | 3.2 (5)    |
| O1—C1—C6—C7   | −0.3 (5)   | C10—C9—N1—C7    | 96.0 (3)   |
| C2—C1—C6—C7   | −179.0 (3) | C10—C9—N1—Ni1   | −84.6 (3)  |
| C4—C5—C6—C1   | −0.7 (5)   | C18—C19—N2—C21  | 175.5 (3)  |
| C4—C5—C6—C7   | 179.4 (3)  | C18—C19—N2—Ni1  | −3.6 (5)   |

|                 |            |                |            |
|-----------------|------------|----------------|------------|
| C1—C6—C7—N1     | −4.4 (5)   | C22—C21—N2—C19 | −95.6 (3)  |
| C5—C6—C7—N1     | 175.5 (3)  | C22—C21—N2—Ni1 | 83.5 (3)   |
| N1—C9—C10—C11   | −57.0 (4)  | C6—C1—O1—Ni1   | 5.9 (5)    |
| N1—C9—C10—C12   | −179.3 (3) | C2—C1—O1—Ni1   | −175.5 (2) |
| O3—C13—C14—O4   | −0.6 (4)   | N1—Ni1—O1—C1   | −5.6 (3)   |
| C18—C13—C14—O4  | 179.9 (3)  | N2—Ni1—O1—C1   | 174.4 (3)  |
| O3—C13—C14—C15  | 179.0 (3)  | C3—C2—O2—C8    | 2.1 (5)    |
| C18—C13—C14—C15 | −0.5 (5)   | C1—C2—O2—C8    | −177.5 (3) |
| O4—C14—C15—C16  | −179.4 (3) | C18—C13—O3—Ni1 | −4.1 (5)   |
| C13—C14—C15—C16 | 1.1 (5)    | C14—C13—O3—Ni1 | 176.4 (2)  |
| C14—C15—C16—C17 | −0.9 (5)   | N1—Ni1—O3—C13  | −177.0 (3) |
| C15—C16—C17—C18 | 0.1 (6)    | N2—Ni1—O3—C13  | 3.0 (3)    |
| O3—C13—C18—C17  | −179.7 (3) | C15—C14—O4—C20 | −3.1 (5)   |
| C14—C13—C18—C17 | −0.2 (5)   | C13—C14—O4—C20 | 176.5 (3)  |

*Hydrogen-bond geometry (Å, °)*

| <i>D</i> —H $\cdots$ <i>A</i> | <i>D</i> —H | H $\cdots$ <i>A</i> | <i>D</i> $\cdots$ <i>A</i> | <i>D</i> —H $\cdots$ <i>A</i> |
|-------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| C10—H10 $\cdots$ O3           | 0.98        | 2.61                | 3.126 (4)                  | 113                           |
| C22—H22 $\cdots$ O1           | 0.98        | 2.63                | 3.138 (4)                  | 113                           |