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# *catena*-Poly[[tripyridinenickel(II)]-$\mu$-5-bromoisophthalato]

**Lijun Liu**

Department of Chemistry, Liaocheng University, Liaocheng, Shandong 252059, People's Republic of China
Correspondence e-mail: liulijun@lcu.edu.cn

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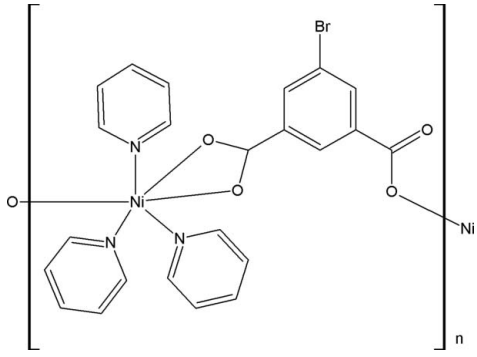
Key indicators: single-crystal X-ray study; $T$ = 296 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.036; $wR$ factor = 0.097; data-to-parameter ratio = 18.7.

The title compound, $[Ni(C_8H_3BrO_4)(C_5H_5N)_3]$, is the first structurally characterized complex with a transition metal coordinated by a 5-bromoisophthalate anion. The $Ni^{II}$ ion is coordinated by three O atoms from the carboxylate groups and three N atoms from three pyridine ligands in a distorted octahedral coordination geometry. The 5-bromoisophthalate anion is planar within 0.057 (2) Å. The two carboxylate groups of the ligand coordinate the $Ni^{II}$ ions in a chelating and monodentate mode, linking the metal atoms into infinite chains along the [010] direction. These chains are stacked together *via* strong $\pi$–$\pi$ interactions between the pyridine rings [centroid–centroid distance 3.601 (4) Å], forming a three-dimensional motif.

## Related literature

For related literature, see: Therrien *et al.* (2005).




## Experimental

### Crystal data

$[Ni(C_8H_3BrO_4)(C_5H_5N)_3]$
$M_r$ = 539.02
Monoclinic, $C2/c$
$a$ = 19.6621 (6) Å
$b$ = 16.0190 (6) Å
$c$ = 14.8755 (5) Å
$\beta$ = 111.504 (2)°
$V$ = 4359.2 (3) Å$^3$
$Z$ = 8
Mo $K\alpha$ radiation
$\mu$ = 2.76 mm$^{-1}$
$T$ = 296 (2) K
0.22 × 0.20 × 0.08 mm

### Data collection

Bruker SMART 1K CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.582, $T_{max}$ = 0.809
22727 measured reflections
5402 independent reflections
4195 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.034

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.096
$S$ = 1.05
5402 reflections
289 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.65 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.34 e Å$^{-3}$

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| Br1—C5 | 1.899 (2) | Ni1—O2 | 2.1655 (17) |
| Ni1—N2 | 2.1439 (19) | Ni1—O1 | 2.2024 (17) |
| | | | |
| N2—Ni1—O2 | 152.98 (7) | O1—Ni1—N1 | 90.02 (7) |
| N2—Ni1—O1 | 92.78 (7) | N2—Ni1—N3 | 91.78 (7) |
| O2—Ni1—O1 | 60.20 (6) | O2—Ni1—N3 | 88.13 (7) |
| N2—Ni1—N1 | 90.11 (7) | O1—Ni1—N3 | 90.54 (7) |
| O2—Ni1—N1 | 90.48 (7) | N1—Ni1—N3 | 178.00 (7) |

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINT-Plus* (Bruker, 2007); data reduction: *SAINT-Plus*; 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*.

The author is grateful to Professor D. Wang for his help.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FJ2127).

## References

Bruker (2007). *SMART* and *SAINT-Plus*. Bruker AXS Inc., Madison, Wisconsin,USA.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Therrien, B., Vieille-Petit, L. & Suss-Fink, G. (2005). *J. Mol. Struct.* **749**, 183–186.

# supporting information

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## *catena*-Poly[[tripyridinenickel(II)]-$\mu$-5-bromoisophthalato]

**Lijun Liu**

### S1. Comment

The 5-bromoisophthalic acid ligand ($H_2$BIPA) was hitherto reported in its molecular form in the cystal structure of its cocrystal complex (Therrien *et al.*, 2005). This paper provides the fist example of its structurally characterized complex with a transition metal; the ligand in this complex is twice deprotonated.

The asymmetric unit of Ni(BIPA)($C_5H_5N)_3$ occupies a general position in the unit cell; the Ni atom is coordinated by four O atoms from the carboxylicate groups and three N atoms from the pyridine ligands (Ni1—N2 2.143 (2), Ni1—O2 2.165 (2), Ni1—O1 2.202 (2), Ni1—N1 2.204 (2), Ni1—N3 2.204 (2) and Ni1—O3 1.996 (2) Å) (Fig. 1). The BIPA ligand has essentially planar conformation, the maximum deviation of the O3 atom from its mean plane being 0.057 (2) Å. The geometry of the ligand is similar to the one observed in Therrien *et al.*, (2005).

No guest molecule or Hydrogen bond was detected in the structure; The two carboxylate groups of the ligand coordinated with the Ni(II) in monodentate and chelated mode, respectively, linking the Ni(II) ion into an infinte chain along the (010) direction. The chains are stacked together *via* the strong $\pi$-$\pi$ interactions between the pyridine rings to form a three-dimensional motif.(Fig. 2).

### S2. Experimental

$NiCl_2.4H_2O$ (0.5 mmol, 101 mg) and 5-bromoisophthalic acid (0.5 mmol, 123 mg) were added to 30 ml of distilled water. After stirring for 15 min at room temperature, the pH value was adjusted to 6 by few drops of pyridine, and clear solution was allowed to evaporate in the ventilating cabinet. Light green plate crystals of the title compound were obtained after 4 days, in yields of 35%. The crystals were filtered, washed by cold EtOH and dried in the air.

### S3. Refinement

All of the H atoms were positioned geometrically and refined using a riding model with C—H = 0.930 Å, with $U_{iso}$(H) = 1.2 times $U_{eq}$(C).

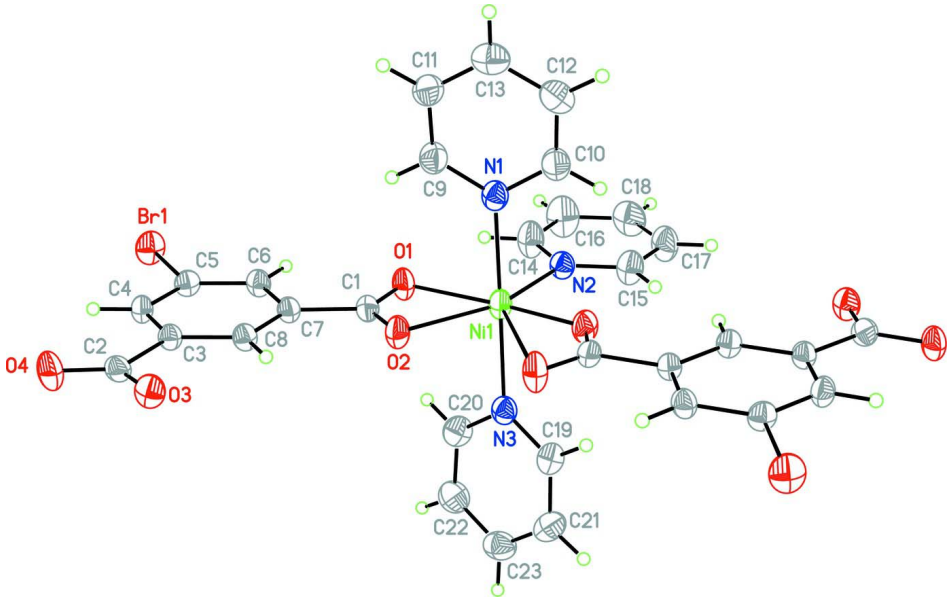


**Figure 1**

Molecular structure showing 50% probability displacement ellipsoids. The unlabeled atoms are derived from the reference atoms by means of the (1.5 - *x*, -1/2 + *y*, 1.5 - *z*) symmetry transformation..

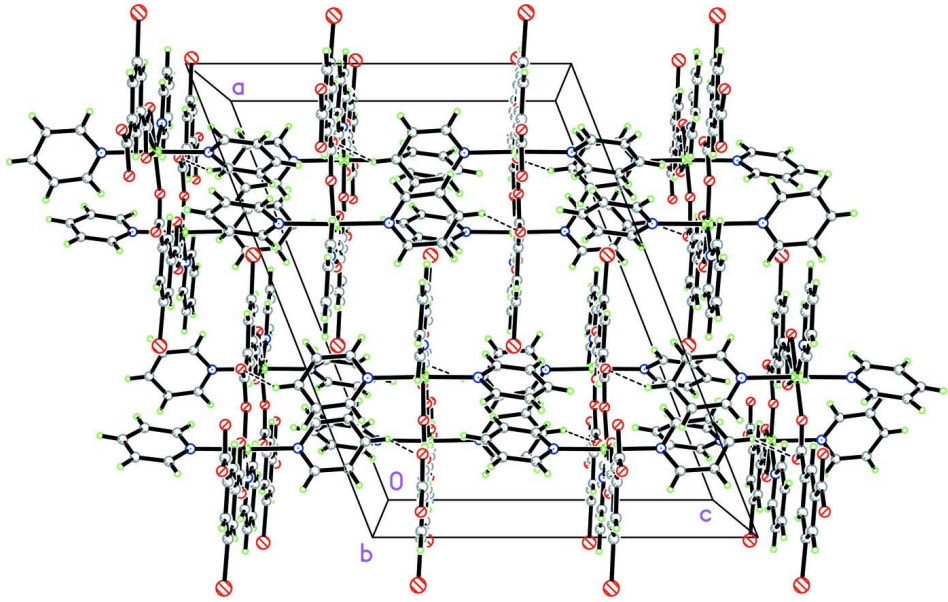


**Figure 2**

Packing diagram viewed down the *b* axis,

**catena-Poly[[tripyridinenickel(II)]-*μ*-5-bromoisophthalato]**

*Crystal data*

| | |
|---|---|
| $[Ni(C_8H_3BrO_4)(C_5H_5N)_3]$ | $a$ = 19.6621 (6) Å |
| $M_r$ = 539.02 | $b$ = 16.0190 (6) Å |
| Monoclinic, *C*2/*c* | $c$ = 14.8755 (5) Å |
| Hall symbol: -C 2yc | $\beta$ = 111.504 (2)° |

$V$ = 4359.2 (3) Å$^3$
$Z$ = 8
$F$(000) = 2176
$D_x$ = 1.643 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 9035 reflections

$\theta$ = 2.7–28.7°
$\mu$ = 2.76 mm$^{-1}$
$T$ = 296 K
Plate, green
0.22 × 0.20 × 0.08 mm

*Data collection*

Bruker SMART 1K CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.582, $T_{max}$ = 0.809

22727 measured reflections
5402 independent reflections
4195 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.034
$\theta_{max}$ = 28.3°, $\theta_{min}$ = 2.0°
$h$ = −26→26
$k$ = −21→21
$l$ = −19→19

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.096
$S$ = 1.05
5402 reflections
289 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
$w = 1/[\sigma^2(F_o^2) + (0.0529P)^2 + 2.0236P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.65 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.34 e Å$^{-3}$

*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$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Br1 | 1.104271 (13) | 1.027190 (17) | 0.93289 (2) | 0.04600 (10) |
| Ni1 | 0.833627 (16) | 0.667412 (18) | 0.78863 (2) | 0.03305 (10) |
| O1 | 0.92550 (9) | 0.75627 (10) | 0.83248 (13) | 0.0385 (4) |
| N1 | 0.83251 (11) | 0.66980 (12) | 0.93619 (15) | 0.0352 (4) |
| C1 | 0.87970 (13) | 0.81533 (14) | 0.80832 (16) | 0.0313 (5) |
| O2 | 0.81214 (9) | 0.80030 (10) | 0.77426 (13) | 0.0386 (4) |
| N2 | 0.90493 (11) | 0.56123 (12) | 0.82571 (14) | 0.0333 (4) |
| C2 | 0.82483 (13) | 1.12204 (14) | 0.78305 (16) | 0.0314 (5) |
| O3 | 0.75769 (9) | 1.09924 (11) | 0.75262 (12) | 0.0396 (4) |
| N3 | 0.83124 (11) | 0.66766 (12) | 0.63935 (15) | 0.0351 (4) |
| C3 | 0.87912 (12) | 1.05099 (14) | 0.81091 (16) | 0.0282 (4) |

| | | | | |
|---|---|---|---|---|
| O4 | 0.84705 (11) | 1.19426 (11) | 0.79268 (15) | 0.0487 (5) |
| C4 | 0.95349 (12) | 1.06846 (14) | 0.85107 (16) | 0.0309 (5) |
| H4 | 0.9698 | 1.1234 | 0.8609 | 0.037* |
| C5 | 1.00279 (12) | 1.00317 (15) | 0.87614 (16) | 0.0311 (5) |
| C6 | 0.98030 (12) | 0.92097 (14) | 0.86223 (17) | 0.0318 (5) |
| H6 | 1.0143 | 0.8778 | 0.8796 | 0.038* |
| C7 | 0.90594 (12) | 0.90378 (13) | 0.82181 (16) | 0.0291 (5) |
| C8 | 0.85606 (13) | 0.96895 (14) | 0.79651 (16) | 0.0298 (5) |
| H8 | 0.8063 | 0.9572 | 0.7694 | 0.036* |
| C9 | 0.85644 (15) | 0.73386 (16) | 0.99719 (19) | 0.0425 (6) |
| H9 | 0.8809 | 0.7770 | 0.9799 | 0.051* |
| C10 | 0.79950 (15) | 0.60768 (16) | 0.9655 (2) | 0.0426 (6) |
| H10 | 0.7834 | 0.5614 | 0.9256 | 0.051* |
| C11 | 0.84689 (16) | 0.73964 (17) | 1.0846 (2) | 0.0467 (6) |
| H11 | 0.8646 | 0.7855 | 1.1247 | 0.056* |
| C12 | 0.78831 (15) | 0.60906 (19) | 1.0510 (2) | 0.0481 (7) |
| H12 | 0.7655 | 0.5644 | 1.0684 | 0.058* |
| C13 | 0.81117 (16) | 0.67711 (18) | 1.1112 (2) | 0.0479 (7) |
| H13 | 0.8025 | 0.6803 | 1.1685 | 0.058* |
| C14 | 0.97593 (15) | 0.57400 (17) | 0.8668 (2) | 0.0509 (7) |
| H14 | 0.9924 | 0.6288 | 0.8794 | 0.061* |
| C15 | 0.88262 (15) | 0.48241 (16) | 0.8088 (2) | 0.0452 (6) |
| H15 | 0.8328 | 0.4721 | 0.7791 | 0.054* |
| C16 | 1.02719 (16) | 0.5108 (2) | 0.8921 (3) | 0.0647 (9) |
| H16 | 1.0768 | 0.5228 | 0.9200 | 0.078* |
| C17 | 0.92961 (17) | 0.41524 (17) | 0.8331 (2) | 0.0520 (7) |
| H17 | 0.9118 | 0.3609 | 0.8213 | 0.062* |
| C18 | 1.00334 (17) | 0.43030 (19) | 0.8752 (2) | 0.0569 (8) |
| H18 | 1.0365 | 0.3863 | 0.8918 | 0.068* |
| C19 | 0.77634 (15) | 0.63156 (16) | 0.56745 (19) | 0.0419 (6) |
| H19 | 0.7427 | 0.6000 | 0.5835 | 0.050* |
| C20 | 0.87919 (15) | 0.71115 (17) | 0.6137 (2) | 0.0434 (6) |
| H20 | 0.9185 | 0.7359 | 0.6621 | 0.052* |
| C21 | 0.76665 (16) | 0.63834 (18) | 0.4714 (2) | 0.0478 (7) |
| H21 | 0.7276 | 0.6118 | 0.4244 | 0.057* |
| C22 | 0.87340 (17) | 0.72126 (19) | 0.5186 (2) | 0.0504 (7) |
| H22 | 0.9080 | 0.7522 | 0.5040 | 0.061* |
| C23 | 0.81539 (16) | 0.68473 (18) | 0.4458 (2) | 0.0488 (7) |
| H23 | 0.8096 | 0.6914 | 0.3813 | 0.059* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Br1 | 0.02529 (13) | 0.04761 (17) | 0.05865 (18) | −0.00510 (11) | 0.00777 (11) | −0.00334 (12) |
| Ni1 | 0.02778 (16) | 0.02420 (15) | 0.04311 (19) | −0.00138 (11) | 0.00821 (13) | −0.00097 (12) |
| O1 | 0.0380 (9) | 0.0225 (8) | 0.0521 (10) | 0.0005 (7) | 0.0130 (8) | −0.0004 (7) |
| N1 | 0.0341 (11) | 0.0288 (10) | 0.0404 (11) | 0.0037 (8) | 0.0107 (9) | −0.0003 (8) |
| C1 | 0.0334 (12) | 0.0258 (11) | 0.0337 (12) | −0.0037 (9) | 0.0113 (10) | 0.0000 (9) |

| | | | | | | |
|---|---|---|---|---|---|---|
| O2 | 0.0331 (9) | 0.0283 (8) | 0.0503 (10) | −0.0051 (7) | 0.0103 (8) | −0.0015 (7) |
| N2 | 0.0304 (10) | 0.0277 (9) | 0.0387 (11) | 0.0032 (8) | 0.0091 (8) | 0.0007 (8) |
| C2 | 0.0350 (12) | 0.0280 (11) | 0.0306 (11) | 0.0075 (9) | 0.0114 (10) | 0.0030 (9) |
| O3 | 0.0276 (9) | 0.0359 (9) | 0.0488 (10) | 0.0089 (7) | 0.0063 (7) | 0.0004 (8) |
| N3 | 0.0340 (11) | 0.0285 (10) | 0.0404 (11) | 0.0035 (8) | 0.0109 (9) | 0.0002 (8) |
| C3 | 0.0281 (11) | 0.0254 (10) | 0.0312 (11) | 0.0016 (8) | 0.0108 (9) | 0.0001 (9) |
| O4 | 0.0485 (11) | 0.0266 (9) | 0.0697 (13) | 0.0064 (8) | 0.0203 (10) | 0.0038 (8) |
| C4 | 0.0316 (12) | 0.0230 (10) | 0.0371 (12) | −0.0023 (9) | 0.0116 (10) | −0.0016 (9) |
| C5 | 0.0215 (10) | 0.0338 (11) | 0.0343 (12) | −0.0012 (9) | 0.0061 (9) | −0.0003 (9) |
| C6 | 0.0267 (11) | 0.0272 (11) | 0.0384 (12) | 0.0050 (9) | 0.0084 (9) | 0.0016 (9) |
| C7 | 0.0300 (11) | 0.0242 (10) | 0.0324 (11) | 0.0006 (9) | 0.0107 (9) | 0.0006 (9) |
| C8 | 0.0257 (11) | 0.0272 (11) | 0.0350 (12) | −0.0004 (9) | 0.0094 (9) | 0.0011 (9) |
| C9 | 0.0464 (15) | 0.0333 (13) | 0.0454 (15) | 0.0002 (11) | 0.0140 (12) | −0.0009 (11) |
| C10 | 0.0405 (14) | 0.0361 (13) | 0.0466 (15) | −0.0023 (11) | 0.0105 (11) | 0.0017 (11) |
| C11 | 0.0585 (17) | 0.0363 (14) | 0.0412 (14) | 0.0086 (13) | 0.0136 (13) | −0.0008 (11) |
| C12 | 0.0406 (15) | 0.0534 (16) | 0.0490 (16) | 0.0000 (13) | 0.0149 (12) | 0.0109 (13) |
| C13 | 0.0452 (15) | 0.0554 (17) | 0.0435 (15) | 0.0153 (13) | 0.0166 (12) | 0.0060 (13) |
| C14 | 0.0346 (14) | 0.0347 (14) | 0.074 (2) | 0.0021 (11) | 0.0089 (13) | 0.0009 (13) |
| C15 | 0.0343 (13) | 0.0351 (13) | 0.0604 (17) | 0.0018 (11) | 0.0107 (12) | −0.0027 (12) |
| C16 | 0.0293 (14) | 0.0547 (19) | 0.097 (3) | 0.0095 (13) | 0.0075 (16) | 0.0062 (18) |
| C17 | 0.0551 (18) | 0.0295 (13) | 0.0676 (19) | 0.0070 (12) | 0.0177 (15) | −0.0009 (13) |
| C18 | 0.0523 (18) | 0.0429 (16) | 0.071 (2) | 0.0222 (14) | 0.0174 (15) | 0.0094 (14) |
| C19 | 0.0419 (14) | 0.0368 (13) | 0.0467 (15) | −0.0030 (11) | 0.0157 (12) | −0.0055 (11) |
| C20 | 0.0361 (13) | 0.0458 (15) | 0.0473 (15) | −0.0007 (11) | 0.0140 (12) | −0.0021 (12) |
| C21 | 0.0442 (16) | 0.0507 (16) | 0.0422 (15) | 0.0023 (13) | 0.0082 (12) | −0.0080 (12) |
| C22 | 0.0504 (17) | 0.0507 (16) | 0.0545 (17) | −0.0018 (13) | 0.0244 (14) | 0.0044 (14) |
| C23 | 0.0555 (17) | 0.0479 (16) | 0.0425 (15) | 0.0132 (13) | 0.0172 (13) | 0.0035 (12) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Br1—C5 | 1.899 (2) | C8—H8 | 0.9300 |
| Ni1—O3$^{i}$ | 1.9963 (16) | C9—C11 | 1.384 (4) |
| Ni1—N2 | 2.1439 (19) | C9—H9 | 0.9300 |
| Ni1—O2 | 2.1655 (17) | C10—C12 | 1.368 (4) |
| Ni1—O1 | 2.2024 (17) | C10—H10 | 0.9300 |
| Ni1—N1 | 2.204 (2) | C11—C13 | 1.362 (4) |
| Ni1—N3 | 2.204 (2) | C11—H11 | 0.9300 |
| O1—C1 | 1.264 (3) | C12—C13 | 1.377 (4) |
| N1—C9 | 1.336 (3) | C12—H12 | 0.9300 |
| N1—C10 | 1.345 (3) | C13—H13 | 0.9300 |
| C1—O2 | 1.259 (3) | C14—C16 | 1.380 (4) |
| C1—C7 | 1.496 (3) | C14—H14 | 0.9300 |
| N2—C14 | 1.318 (3) | C15—C17 | 1.377 (4) |
| N2—C15 | 1.330 (3) | C15—H15 | 0.9300 |
| C2—O4 | 1.226 (3) | C16—C18 | 1.363 (5) |
| C2—O3 | 1.282 (3) | C16—H16 | 0.9300 |
| C2—C3 | 1.511 (3) | C17—C18 | 1.373 (4) |
| O3—Ni1$^{ii}$ | 1.9962 (16) | C17—H17 | 0.9300 |

| | | | |
|---|---|---|---|
| N3—C20 | 1.335 (3) | C18—H18 | 0.9300 |
| N3—C19 | 1.341 (3) | C19—C21 | 1.375 (4) |
| C3—C8 | 1.381 (3) | C19—H19 | 0.9300 |
| C3—C4 | 1.390 (3) | C20—C22 | 1.386 (4) |
| C4—C5 | 1.381 (3) | C20—H20 | 0.9300 |
| C4—H4 | 0.9300 | C21—C23 | 1.372 (4) |
| C5—C6 | 1.380 (3) | C21—H21 | 0.9300 |
| C6—C7 | 1.390 (3) | C22—C23 | 1.383 (4) |
| C6—H6 | 0.9300 | C22—H22 | 0.9300 |
| C7—C8 | 1.387 (3) | C23—H23 | 0.9300 |

| | | | |
|---|---|---|---|
| O3$^{i}$—Ni1—N2 | 94.29 (7) | C3—C8—C7 | 121.0 (2) |
| O3$^{i}$—Ni1—O2 | 112.73 (7) | C3—C8—H8 | 119.5 |
| N2—Ni1—O2 | 152.98 (7) | C7—C8—H8 | 119.5 |
| O3$^{i}$—Ni1—O1 | 172.84 (7) | N1—C9—C11 | 123.7 (3) |
| N2—Ni1—O1 | 92.78 (7) | N1—C9—H9 | 118.2 |
| O2—Ni1—O1 | 60.20 (6) | C11—C9—H9 | 118.2 |
| O3$^{i}$—Ni1—N1 | 88.82 (7) | N1—C10—C12 | 123.4 (3) |
| N2—Ni1—N1 | 90.11 (7) | N1—C10—H10 | 118.3 |
| O2—Ni1—N1 | 90.48 (7) | C12—C10—H10 | 118.3 |
| O1—Ni1—N1 | 90.02 (7) | C13—C11—C9 | 119.1 (3) |
| O3$^{i}$—Ni1—N3 | 90.39 (7) | C13—C11—H11 | 120.5 |
| N2—Ni1—N3 | 91.78 (7) | C9—C11—H11 | 120.5 |
| O2—Ni1—N3 | 88.13 (7) | C10—C12—C13 | 119.4 (3) |
| O1—Ni1—N3 | 90.54 (7) | C10—C12—H12 | 120.3 |
| N1—Ni1—N3 | 178.00 (7) | C13—C12—H12 | 120.3 |
| C1—O1—Ni1 | 88.74 (14) | C11—C13—C12 | 118.3 (3) |
| C9—N1—C10 | 116.1 (2) | C11—C13—H13 | 120.9 |
| C9—N1—Ni1 | 124.07 (17) | C12—C13—H13 | 120.9 |
| C10—N1—Ni1 | 119.51 (17) | N2—C14—C16 | 123.8 (3) |
| O2—C1—O1 | 120.5 (2) | N2—C14—H14 | 118.1 |
| O2—C1—C7 | 119.7 (2) | C16—C14—H14 | 118.1 |
| O1—C1—C7 | 119.7 (2) | N2—C15—C17 | 123.3 (3) |
| C1—O2—Ni1 | 90.54 (14) | N2—C15—H15 | 118.4 |
| C14—N2—C15 | 117.1 (2) | C17—C15—H15 | 118.4 |
| C14—N2—Ni1 | 118.52 (17) | C18—C16—C14 | 118.4 (3) |
| C15—N2—Ni1 | 124.42 (17) | C18—C16—H16 | 120.8 |
| O4—C2—O3 | 125.9 (2) | C14—C16—H16 | 120.8 |
| O4—C2—C3 | 119.5 (2) | C18—C17—C15 | 118.5 (3) |
| O3—C2—C3 | 114.5 (2) | C18—C17—H17 | 120.8 |
| C2—O3—Ni1$^{ii}$ | 130.29 (15) | C15—C17—H17 | 120.8 |
| C20—N3—C19 | 116.3 (2) | C16—C18—C17 | 119.0 (3) |
| C20—N3—Ni1 | 122.15 (17) | C16—C18—H18 | 120.5 |
| C19—N3—Ni1 | 121.10 (17) | C17—C18—H18 | 120.5 |
| C8—C3—C4 | 119.5 (2) | N3—C19—C21 | 124.0 (3) |
| C8—C3—C2 | 121.0 (2) | N3—C19—H19 | 118.0 |
| C4—C3—C2 | 119.5 (2) | C21—C19—H19 | 118.0 |
| C5—C4—C3 | 119.2 (2) | N3—C20—C22 | 123.4 (3) |

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| C5—C4—H4 | 120.4 | N3—C20—H20 | 118.3 |
| C3—C4—H4 | 120.4 | C22—C20—H20 | 118.3 |
| C6—C5—C4 | 121.8 (2) | C23—C21—C19 | 119.1 (3) |
| C6—C5—Br1 | 119.11 (17) | C23—C21—H21 | 120.4 |
| C4—C5—Br1 | 119.06 (17) | C19—C21—H21 | 120.4 |
| C5—C6—C7 | 118.8 (2) | C23—C22—C20 | 119.1 (3) |
| C5—C6—H6 | 120.6 | C23—C22—H22 | 120.5 |
| C7—C6—H6 | 120.6 | C20—C22—H22 | 120.5 |
| C8—C7—C6 | 119.7 (2) | C21—C23—C22 | 118.1 (3) |
| C8—C7—C1 | 120.1 (2) | C21—C23—H23 | 120.9 |
| C6—C7—C1 | 120.1 (2) | C22—C23—H23 | 121.0 |

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| N2—Ni1—O1—C1 | 179.66 (14) | O4—C2—C3—C4 | 2.8 (3) |
| O2—Ni1—O1—C1 | 0.32 (13) | O3—C2—C3—C4 | −175.6 (2) |
| N1—Ni1—O1—C1 | −90.23 (14) | C8—C3—C4—C5 | −0.3 (3) |
| N3—Ni1—O1—C1 | 87.85 (14) | C2—C3—C4—C5 | −179.8 (2) |
| O3$^{i}$—Ni1—N1—C9 | −146.9 (2) | C3—C4—C5—C6 | 0.1 (4) |
| N2—Ni1—N1—C9 | 118.8 (2) | C3—C4—C5—Br1 | −179.01 (17) |
| O2—Ni1—N1—C9 | −34.2 (2) | C4—C5—C6—C7 | 0.0 (4) |
| O1—Ni1—N1—C9 | 26.0 (2) | Br1—C5—C6—C7 | 179.19 (17) |
| O3$^{i}$—Ni1—N1—C10 | 26.11 (19) | C5—C6—C7—C8 | −0.1 (3) |
| N2—Ni1—N1—C10 | −68.18 (19) | C5—C6—C7—C1 | −178.3 (2) |
| O2—Ni1—N1—C10 | 138.84 (19) | O2—C1—C7—C8 | −0.3 (3) |
| O1—Ni1—N1—C10 | −160.96 (18) | O1—C1—C7—C8 | −179.3 (2) |
| Ni1—O1—C1—O2 | −0.5 (2) | O2—C1—C7—C6 | 177.9 (2) |
| Ni1—O1—C1—C7 | 178.45 (19) | O1—C1—C7—C6 | −1.1 (3) |
| O1—C1—O2—Ni1 | 0.6 (2) | C4—C3—C8—C7 | 0.2 (3) |
| C7—C1—O2—Ni1 | −178.44 (19) | C2—C3—C8—C7 | 179.8 (2) |
| O3$^{i}$—Ni1—O2—C1 | 178.35 (13) | C6—C7—C8—C3 | 0.0 (3) |
| N2—Ni1—O2—C1 | −1.8 (2) | C1—C7—C8—C3 | 178.2 (2) |
| O1—Ni1—O2—C1 | −0.32 (13) | C10—N1—C9—C11 | −2.0 (4) |
| N1—Ni1—O2—C1 | 89.43 (14) | Ni1—N1—C9—C11 | 171.2 (2) |
| N3—Ni1—O2—C1 | −92.01 (14) | C9—N1—C10—C12 | 1.8 (4) |
| O3$^{i}$—Ni1—N2—C14 | −174.3 (2) | Ni1—N1—C10—C12 | −171.7 (2) |
| O2—Ni1—N2—C14 | 5.8 (3) | N1—C9—C11—C13 | 0.0 (4) |
| O1—Ni1—N2—C14 | 4.6 (2) | N1—C10—C12—C13 | 0.4 (4) |
| N1—Ni1—N2—C14 | −85.5 (2) | C9—C11—C13—C12 | 2.3 (4) |
| N3—Ni1—N2—C14 | 95.2 (2) | C10—C12—C13—C11 | −2.5 (4) |
| O3$^{i}$—Ni1—N2—C15 | 6.8 (2) | C15—N2—C14—C16 | 0.3 (5) |
| O2—Ni1—N2—C15 | −173.11 (19) | Ni1—N2—C14—C16 | −178.7 (3) |
| O1—Ni1—N2—C15 | −174.4 (2) | C14—N2—C15—C17 | 0.9 (4) |
| N1—Ni1—N2—C15 | 95.6 (2) | Ni1—N2—C15—C17 | 179.8 (2) |
| N3—Ni1—N2—C15 | −83.7 (2) | N2—C14—C16—C18 | −1.0 (6) |
| O4—C2—O3—Ni1$^{ii}$ | −0.5 (4) | N2—C15—C17—C18 | −1.3 (5) |
| C3—C2—O3—Ni1$^{ii}$ | 177.90 (14) | C14—C16—C18—C17 | 0.5 (5) |
| O3$^{i}$—Ni1—N3—C20 | 175.21 (19) | C15—C17—C18—C16 | 0.6 (5) |
| N2—Ni1—N3—C20 | −90.49 (19) | C20—N3—C19—C21 | −1.2 (4) |
| O2—Ni1—N3—C20 | 62.47 (19) | Ni1—N3—C19—C21 | 171.5 (2) |

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| O1—Ni1—N3—C20 | 2.31 (19) | C19—N3—C20—C22 | 1.4 (4) |
| O3$^{i}$—Ni1—N3—C19 | 2.84 (19) | Ni1—N3—C20—C22 | −171.3 (2) |
| N2—Ni1—N3—C19 | 97.14 (19) | N3—C19—C21—C23 | −0.1 (4) |
| O2—Ni1—N3—C19 | −109.90 (19) | N3—C20—C22—C23 | −0.1 (4) |
| O1—Ni1—N3—C19 | −170.06 (18) | C19—C21—C23—C22 | 1.4 (4) |
| O4—C2—C3—C8 | −176.7 (2) | C20—C22—C23—C21 | −1.3 (4) |
| O3—C2—C3—C8 | 4.8 (3) | | |

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