

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

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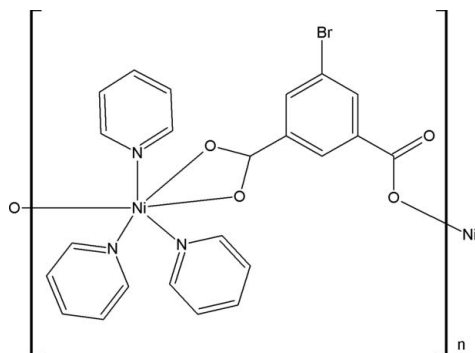
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.036; wR factor = 0.097; data-to-parameter ratio = 18.7.

The title compound, $[\text{Ni}(\text{C}_8\text{H}_3\text{BrO}_4)(\text{C}_5\text{H}_5\text{N})_3]$, 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 π - π 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

$[\text{Ni}(\text{C}_8\text{H}_3\text{BrO}_4)(\text{C}_5\text{H}_5\text{N})_3]$
 $M_r = 539.02$
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) Å³
 $Z = 8$
Mo $K\alpha$ radiation
 $\mu = 2.76$ mm⁻¹
 $T = 296$ (2) K
 $0.22 \times 0.20 \times 0.08$ mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\text{min}} = 0.582$, $T_{\text{max}} = 0.809$

22727 measured reflections
5402 independent reflections
4195 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.034$

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
 $\Delta\rho_{\text{max}} = 0.65$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.34$ 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. A64, 112–122.
Therrien, B., Vieille-Petit, L. & Suss-Fink, G. (2005). J. Mol. Struct. 749, 183–186.

supplementary materials

Acta Cryst. (2008). E64, m974 [doi:10.1107/S1600536808018916]

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Comment

The 5-bromoisophthalic acid ligand (H₂BIPA) was hitherto reported in its molecular form in the crystal structure of its cocrystal complex (Therrien *et al.*, 2005). This paper provides the first example of its structurally characterized complex with a transition metal; the ligand in this complex is twice deprotonated.

The asymmetric unit of Ni(BIPA)(C₅H₅N)₃ occupies a general position in the unit cell; the Ni atom is coordinated by four O atoms from the carboxylate 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 infinite chain along the (010) direction. The chains are stacked together *via* the strong π - π interactions between the pyridine rings to form a three-dimensional motif.(Fig. 2).

Experimental

NiCl₂·4H₂O (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.

Refinement

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

Figures

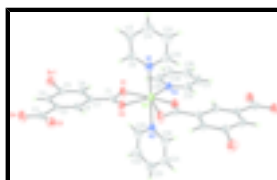


Fig. 1. Molecular structure showing 50% probability displacement ellipsoids. The unlabeled atoms are derived from the reference atoms by means of the (1.5 - x, -1/2 + y, 1.5 - z) symmetry transformation..

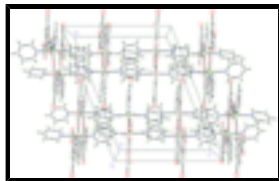


Fig. 2. Packing diagram viewed down the *b* axis,

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Crystal data

[Ni(C₈H₃BrO₄)(C₅H₅N)₃]

$M_r = 539.02$

Monoclinic, *C2/c*

Hall symbol: -C 2yc

$a = 19.6621 (6) \text{ \AA}$

$b = 16.0190 (6) \text{ \AA}$

$c = 14.8755 (5) \text{ \AA}$

$\beta = 111.504 (2)^\circ$

$V = 4359.2 (3) \text{ \AA}^3$

$Z = 8$

$F_{000} = 2176$

$D_x = 1.643 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 9035 reflections

$\theta = 2.7\text{--}28.7^\circ$

$\mu = 2.76 \text{ mm}^{-1}$

$T = 296 (2) \text{ K}$

Plate, green

$0.22 \times 0.20 \times 0.08 \text{ mm}$

Data collection

Bruker SMART 1K CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 296(2) \text{ K}$

φ and ω 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 $I > 2\sigma(I)$

$R_{\text{int}} = 0.034$

$\theta_{\max} = 28.3^\circ$

$\theta_{\min} = 2.0^\circ$

$h = -26 \rightarrow 26$

$k = -21 \rightarrow 21$

$l = -19 \rightarrow 19$

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

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

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

$\Delta\rho_{\max} = 0.65 \text{ e \AA}^{-3}$

$\Delta\rho_{\min} = -0.34 \text{ e \AA}^{-3}$

Primary atom site location: structure-invariant direct methods

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>	$U_{\text{iso}}^*/U_{\text{eq}}$
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)

supplementary materials

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 (\AA^2)

	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, °)

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)

supplementary materials

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)
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
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)
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) $-x+3/2, y+1/2, -z+3/2$.

Fig. 1

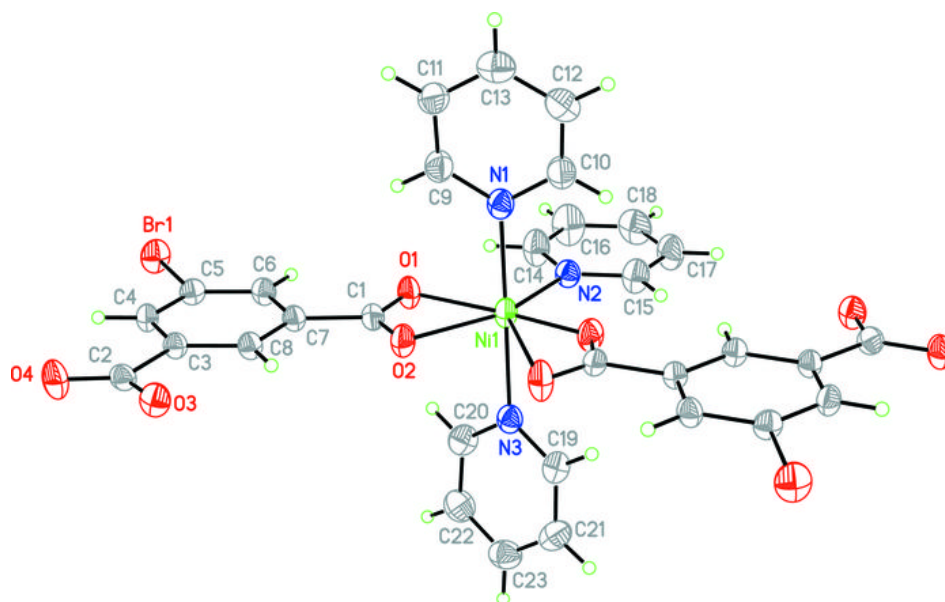


Fig. 2

