Poly[bis[μ₂-4,4′-bis(imidazol-1-ylmethyl)biphenyl-κ²N:N′]dichloridonickel(II)]

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In the title compound, [NiCl₂(C₂₀H₁₈N₄)₂]ₙ, the Ni²⁺ cation is situated on an inversion center and is coordinated by two chloride ions and four imidazole N atoms of four different 4,4′-bis[(1H-imidazol-1-yl)methyl]-1,1′-biphenyl (BIMB), forming a slightly distorted octahedral geometry. Each BIMB ligand adopts a linear linker to connect Ni²⁺ ions, forming a two-dimensional layer with an sql network. In the crystal, neighboring layers repeat in an ABAB stacking mode, and weak intermolecular C—H···Cl hydrogen bonds between alternate layers lead to a three-dimensional, twofold interpenetrated, supramolecular framework with a pcu topology net.

Structure description

Over the last two decades, imidazole and its derivatives have attracted a lot of attention as N-heterocyclic aromatic ligands, since they can easily form metal–imidazole frameworks with special luminescent, magnetic and favorable gas-adsorption abilities (Banerjee et al. 2008; Zhang et al. 2012; Zhu et al. 2012; Chen et al. 2014). As an extended imidazole-type linker, the flexible ligand 4,4′-bis[(1H-imidazol-1-yl)methyl]-1,1′-biphenyl (BIMB) exhibits a geometrical diversity with cis or trans conformations, leading to diverse structures of coordination compounds. Until now, most reported metal–organic compounds based on BIMB ligands have employed organic multicarboxylates as co-ligands because BIMB is a neutral ligand and another anion is needed to balance the charge requirement to form a neutral framework. Common inorganic anions such as Cl⁻, Br⁻, I⁻, NO₃⁻, SO₄²⁻, N₃⁻, etc. can also be used as co-ligands to balance the charge requirement. However, only ten examples of neutral, BIMB-based metal–organic compounds have been reported [according to the Cambridge Structural Database (CSD, Version 5.43 with update of March, 2022; Groom et al., 2016) with inorganic anions as co-ligands.]
The asymmetric unit of the title compound, \([\text{NiCl}_2(\text{C}_{20}\text{H}_{18}\text{N}_4)_2]\), contains one half nickel(II) ion, two half BIMB ligands, and one chloride ion (Fig. 1). The nickel(II) ion sits on an inversion center and is coordinated by four imidazole nitrogen atoms from four different BIMB ligands [\(\text{Ni—N} = 2.100\) (3)–2.108 (3) Å] and two chloride ions [\(\text{Ni—Cl} = 2.4793\) (11) Å], forming a slightly distorted octahedral geometry. In the crystal, the BIMB ligands have twofold rotational symmetry, being bisected by rotation axes, and the biphenyl groups are not coplanar, with dihedral angles of 33.21 (10) and 35.4 (10)° between the ring planes. The dihedral angles between the imidazole ring plane and the average plane of the biphenyl group are 87.71 (14) and 81.93 (14)°.

Each BIMB ligand exhibits a cis-conformation relative to the average plane of the biphenyl group, and acts as a linear linker between \(\text{Ni}^{2+}\) ions, which gives a corrugated two-dimensional layer structure with an sql (square lattice) network as illustrated in Fig. 2. The layers stack in an –ABAB– mode, and the \(\text{Ni}^{2+}\) ion in one layer is located at the center of the grid of adjacent layers. Thus, there are no residual solvent-accessible voids in this compound. Alternate layers between \(A—A\) or \(B—B\) layers are further linked by \(\text{C—H}—\text{Cl}\) hydrogen bonds (Table 1, Figs. 3 and 4) to form a three-dimensional, twofold interpenetrated, supramolecular framework with a pcu (primitive cubic) topology network (Fig. 5).

The structure of the title compound is isomorphous to that of the cadmium(II) compound, whose structure has been studied at 200 K (Zhao et al. 2003). This structural similarity of the \(\text{Cd}^{II}\) and \(\text{Ni}^{II}\) compounds is somewhat unexpected in view of the different effective radii of these ions (Shannon & Prewitt, 1969, 1970), which causes the differences between \(\text{M—N}\) distances [\(\text{Cd—N} = 2.339\) (2)–2.364 (2) Å in the cadmium(II) compound]. It should also be noted that the title compound was easily obtained within one day using solvothermal conditions, whereas the cadmium(II) compound was obtained after several weeks using a slow-diffusion method.

| \(D—H—A\) | \(D—H\) | \(H—A\) | \(D—A\) | \(D—H—A\) |
|-----------------|-------|-------|-------|----------|
| Cl1—H11···Cl1′  | 0.93  | 2.79  | 3.605 (4) | 147 |
| Cl1—H14B···Cl1′ | 0.97  | 2.80  | 3.686 (5) | 153 |

Symmetry code: (i) \(-x+\frac{1}{2}-y+\frac{1}{2}-z+1\).

**Table 1**

Hydrogen-bond geometry (Å, °).

**Figure 3**

The packing of the title compound viewed along the \(b\) axis. H atoms are omitted for clarity.

**Figure 4**

View of the \(\text{C—H}—\text{Cl}\) hydrogen bonds (dashed lines) between alternate layers along the \(c\) axis. H atoms not involved in hydrogen bonding are omitted.
Synthesis and crystallization
A mixture of NiCl₂·2H₂O (24 mg, 0.1 mmol), BIMB (62 mg, 0.2 mmol) and DMF (6 ml) was added to a 20 ml glass vial and then ultrasonicated for 1 minute. The vial was capped tightly and placed in an oven at 120°C. After 12 h, the vial was removed from the oven and allowed to cool to room temperature. The light-green transparent needle-like crystals were collected by filtration, washed with DMF and dried under ambient conditions. About 34 mg of product was obtained (44% yield based on BIMB ligand). The phase purity of the bulk sample was verified by powder X-ray diffraction (PXRD). The experimental and simulated powder XRD patterns of the title compound are displayed in Fig. S1 of the supporting information. Their peak positions are in good agreement with each other, indicating the phase purity of the title compound (slight intensity mismatches due to preferred orientation are observed).

Refinement
Crystal data, data collection and structure refinement details are summarized in Table 2.

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Table 2
Experimental details.

| Parameter | Value |
|-----------|-------|
| M (g/mol) | 758.38 |
| Crystal system, space group | Monoclinic, C2/c |
| Temperature (K) | 296 |
| a, b, c (Å) | 26.453 (3), 7.3571 (7), 18.099 (2) |
| V (Å³) | 3516.8 (7) |
| Z | 4 |
| Radiation type | Mo Kα |
| μ (mm⁻¹) | 0.75 |
| Crystal size (mm) | 0.30 × 0.22 × 0.16 |

Data collection
Diffractometer: Oxford Diffraction, Xcalibur, Eos, Gemini
Absorption correction: Multi-scan (CrysAlis PRO; Rigaku OD, 2015)

| Parameter | Value |
|-----------|-------|
| Tmin, Tmax | 0.856, 1.000 |
| No. of measured, independent and observed [I > 2σ(I)] reflections | 16025, 4343, 2543 |
| Rint | 0.078 |
| wR2 | 0.692 |

Refinement
R[F² > 2σ(F²)], wR(F²), S | 0.072, 0.161, 1.06 |
No. of reflections | 4343 |
No. of parameters | 232 |
H-atom treatment | H-atom parameters constrained |
Δρmax, Δρmin (e Å⁻³) | 0.61, −0.27 |

Computers programs: CrysAlis PRO (Rigaku OD, 2015), SHELXT2014/5 (Sheldrick, 2015a), SHELXL2018/3 (Sheldrick, 2015b) and DIAMOND (Brandenburg, 1999).

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full crystallographic data

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Poly[bis[μ$_2$-4,4'-bis(imidazol-1-ylmethyl)biphenyl-κ²N:N']dichloridonickel(II)]

Crystal data

[NiCl$_2$(C$_{20}$H$_{18}$N$_4$)$_2$]  $F$(000) = 1576
$M_r = 758.38$  $D_\lambda = 1.432$ Mg m$^{-3}$
Monoclinic, C2/c  Mo Kα radiation, $\lambda = 0.71073$ Å
$\alpha = 26.453$ (3) Å  Cell parameters from 3041 reflections
$\beta = 7.3571$ (7) Å  $\theta = 2.7$–22.6°
$\gamma = 18.099$ (2) Å  $\mu = 0.75$ mm$^{-1}$
$\beta = 93.223$ (11)°  $T = 296$ K
V = 3516.8 (7) Å$^3$  Needle, green
Z = 4  0.30 × 0.22 × 0.16 mm

Data collection

Oxford Diffraction, Xcalibur, Eos, Gemini 4343 independent reflections
Xcalibur, Eos, Gemini diffractometer 2543 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed X-ray tube $\theta_{max} = 29.5^\circ$, $\theta_{min} = 2.3^\circ$
\(\omega\) scans  $h = −35→35$
Absorption correction: multi-scan $k = −9→10$
(CrysAlisPro; Rigaku OD, 2015) $l = −24→19$
$T_{min} = 0.856, T_{max} = 1.000$
16025 measured reflections

Refinement

Refinement on $F^2$ Secondary atom site location: difference Fourier
Least-squares matrix: full map
$R[F^2 > 2\sigma(F^2)] = 0.072$ Hydrogen site location: inferred from
$wR(F^2) = 0.161$ neighbouring sites
$S = 1.06$ H-atom parameters constrained
4343 reflections $w = 1/[\sigma^2(F^2) + (0.0438P)^2 + 5.351P]$
232 parameters where $P = (F^2 + 2F_C^2)/3$
0 restraints $(\Delta/\sigma)_{max} < 0.001$
Primary atom site location: structure-invariant
$\Delta\rho_{max} = 0.61$ e Å$^{-3}$
direct methods $\Delta\rho_{min} = −0.27$ e Å$^{-3}$

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. All H atoms were placed in idealized positions (C—H = 0.93 Å for aromatic H; C—H = 0.97 Å for methylene H) and refined as riding atoms with $U_{eq}(H) = 1.2U_{eq}(C)$. 
### Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

|   | x    | y    | z    | U(eq) |
|---|------|------|------|-------|
| Ni1 | 0.250000 | 0.250000 | 0.500000 | 0.0419 (2) |
| Cl1 | 0.21866 (4) | 0.51154 (14) | 0.57071 (6) | 0.0488 (3) |
| N1  | 0.22244 (12) | 0.3739 (5) | 0.40075 (19) | 0.0423 (8) |
| N2  | 0.20188 (12) | 0.5693 (5) | 0.31205 (19) | 0.0454 (9) |
| N3  | 0.32078 (12) | 0.3777 (5) | 0.49579 (19) | 0.0451 (9) |
| N4  | 0.38019 (12) | 0.5864 (5) | 0.4895 (2) | 0.0454 (9) |
| C1  | 0.21920 (15) | 0.5462 (6) | 0.3818 (2) | 0.0480 (11) |
| H1  | 0.228055 | 0.641502 | 0.413725 | 0.058* |
| C2  | 0.19229 (17) | 0.4035 (7) | 0.2839 (3) | 0.0605 (13) |
| H2  | 0.179357 | 0.375818 | 0.236414 | 0.073* |
| C3  | 0.20524 (17) | 0.2848 (7) | 0.3387 (3) | 0.0594 (13) |
| H3  | 0.202711 | 0.159098 | 0.334459 | 0.071* |
| C4  | 0.19061 (16) | 0.7409 (7) | 0.2746 (3) | 0.0598 (13) |
| H4A | 0.205060 | 0.740434 | 0.226555 | 0.072* |
| C5  | 0.13415 (16) | 0.7708 (6) | 0.2650 (3) | 0.0485 (11) |
| C6  | 0.10774 (17) | 0.7322 (6) | 0.1991 (3) | 0.0555 (12) |
| H6  | 0.125205 | 0.697958 | 0.158149 | 0.067* |
| C7  | 0.05550 (17) | 0.7438 (6) | 0.1933 (3) | 0.0511 (11) |
| H7  | 0.038471 | 0.716996 | 0.148230 | 0.061* |
| C8  | 0.02803 (15) | 0.7940 (5) | 0.2523 (2) | 0.0443 (11) |
| C9  | 0.05500 (17) | 0.8394 (7) | 0.3182 (3) | 0.0568 (12) |
| H9  | 0.037767 | 0.877617 | 0.358795 | 0.068* |
| C10 | 0.10719 (17) | 0.8279 (7) | 0.3234 (3) | 0.0601 (13) |
| H10 | 0.124556 | 0.859617 | 0.367591 | 0.072* |
| C11 | 0.33046 (15) | 0.5513 (6) | 0.4876 (2) | 0.0460 (11) |
| H11 | 0.305554 | 0.639986 | 0.481224 | 0.055* |
| C12 | 0.36761 (17) | 0.2989 (6) | 0.5055 (3) | 0.0573 (13) |
| H12 | 0.373160 | 0.175661 | 0.513987 | 0.069* |
| C13 | 0.40491 (18) | 0.4252 (6) | 0.5009 (3) | 0.0580 (13) |
| H13 | 0.439701 | 0.405819 | 0.504830 | 0.070* |
| C14 | 0.40289 (17) | 0.7651 (6) | 0.4815 (3) | 0.0523 (12) |
| H14A| 0.425099 | 0.790809 | 0.524742 | 0.063* |
| H14B| 0.376426 | 0.856560 | 0.478823 | 0.063* |
| C15 | 0.43247 (16) | 0.7761 (6) | 0.4137 (3) | 0.0474 (11) |
| C16 | 0.48277 (16) | 0.8245 (6) | 0.4154 (3) | 0.0558 (12) |
| H16 | 0.499348 | 0.852339 | 0.460692 | 0.067* |
| C17 | 0.50950 (17) | 0.8329 (7) | 0.3518 (3) | 0.0588 (13) |
| H17 | 0.543335 | 0.867828 | 0.354691 | 0.071* |
| C18 | 0.48594 (16) | 0.7894 (5) | 0.2841 (2) | 0.0468 (11) |
| C19 | 0.43553 (17) | 0.7414 (6) | 0.2819 (3) | 0.0544 (12) |
| H19 | 0.418879 | 0.713521 | 0.236701 | 0.065* |
| C20 | 0.40927 (17) | 0.7340 (6) | 0.3457 (3) | 0.0556 (12) |
| H20 | 0.375352 | 0.700011 | 0.342800 | 0.067* |
**Atomic displacement parameters (Å²)**

|     | $U^{11}$  | $U^{22}$  | $U^{33}$  | $U^{12}$  | $U^{13}$  | $U^{23}$  |
|-----|----------|----------|----------|----------|----------|----------|
| Ni1 | 0.0420 (4) | 0.0360 (4) | 0.0478 (5) | 0.0039 (3) | 0.0020 (3) | 0.0062 (4) |
| Cl1 | 0.0494 (6) | 0.0406 (6) | 0.0563 (7) | 0.0077 (5) | 0.0024 (5) | 0.0018 (5) |
| N1  | 0.0364 (19) | 0.042 (2) | 0.048 (2) | −0.0009 (16) | −0.0007 (16) | 0.0031 (17) |
| N2  | 0.0367 (19) | 0.050 (2) | 0.049 (2) | 0.0005 (17) | −0.0055 (16) | 0.0120 (18) |
| N3  | 0.0380 (19) | 0.039 (2) | 0.059 (2) | 0.0035 (16) | 0.0041 (17) | 0.0061 (18) |
| N4  | 0.040 (2) | 0.036 (2) | 0.060 (2) | −0.0003 (16) | 0.0051 (17) | 0.0076 (17) |
| C1  | 0.046 (3) | 0.047 (3) | 0.050 (3) | −0.002 (2) | −0.007 (2) | 0.000 (2) |
| C2  | 0.060 (3) | 0.060 (3) | 0.059 (3) | 0.014 (3) | −0.016 (2) | −0.005 (3) |
| C3  | 0.055 (3) | 0.046 (3) | 0.075 (4) | 0.009 (2) | −0.013 (3) | −0.005 (3) |
| C4  | 0.044 (3) | 0.065 (3) | 0.069 (3) | −0.003 (2) | −0.004 (2) | 0.027 (3) |
| C5  | 0.045 (2) | 0.045 (3) | 0.055 (3) | 0.000 (2) | −0.005 (2) | 0.015 (2) |
| C6  | 0.052 (3) | 0.056 (3) | 0.058 (3) | 0.005 (2) | −0.004 (2) | 0.002 (2) |
| C7  | 0.053 (3) | 0.051 (3) | 0.048 (3) | −0.001 (2) | −0.013 (2) | −0.002 (2) |
| C8  | 0.048 (2) | 0.037 (2) | 0.046 (3) | −0.0001 (18) | −0.009 (2) | 0.004 (2) |
| C9  | 0.053 (3) | 0.068 (3) | 0.048 (3) | 0.002 (2) | −0.006 (2) | −0.001 (3) |
| C10 | 0.052 (3) | 0.072 (3) | 0.055 (3) | −0.002 (3) | −0.018 (2) | 0.001 (3) |
| C11 | 0.037 (2) | 0.045 (3) | 0.056 (3) | 0.0067 (19) | 0.001 (2) | 0.006 (2) |
| C12 | 0.055 (3) | 0.040 (3) | 0.077 (4) | 0.008 (2) | 0.007 (3) | 0.012 (2) |
| C13 | 0.049 (3) | 0.046 (3) | 0.079 (4) | 0.008 (2) | 0.002 (2) | 0.004 (3) |
| C14 | 0.051 (3) | 0.046 (3) | 0.060 (3) | −0.003 (2) | 0.005 (2) | −0.001 (2) |
| C15 | 0.045 (2) | 0.040 (3) | 0.057 (3) | 0.001 (2) | 0.003 (2) | 0.001 (2) |
| C16 | 0.044 (3) | 0.062 (3) | 0.061 (3) | −0.004 (2) | 0.000 (2) | 0.000 (3) |
| C17 | 0.039 (2) | 0.060 (3) | 0.078 (4) | −0.006 (2) | 0.005 (2) | −0.002 (3) |
| C18 | 0.046 (3) | 0.032 (2) | 0.062 (3) | 0.0030 (18) | 0.005 (2) | 0.000 (2) |
| C19 | 0.048 (3) | 0.058 (3) | 0.057 (3) | −0.002 (2) | −0.001 (2) | −0.002 (2) |
| C20 | 0.043 (3) | 0.058 (3) | 0.066 (3) | −0.004 (2) | 0.003 (2) | −0.001 (3) |

**Geometric parameters (Å, °)**

| Bond | Length (Å) | Angle (°) |
|------|-----------|-----------|
| Ni1—N3 | 2.100 (3) | C6—H6 0.9300 |
| Ni1—N3 | 2.100 (3) | C7—C8 1.377 (6) |
| Ni1—N1 | 2.108 (3) | C7—H7 0.9300 |
| Ni1—Ni | 2.108 (3) | C8—C9 1.395 (6) |
| Ni1—Cl1 | 2.4793 (11) | C8—C8 1.480 (8) |
| Ni1—Cl1 | 2.4793 (11) | C9—C10 1.381 (6) |
| Ni1—C1 | 1.315 (5) | C9—H9 0.9300 |
| Ni1—C3 | 1.357 (5) | C10—H10 0.9300 |
| Ni1—C1 | 1.330 (5) | C11—H11 0.9300 |
| Ni1—C2 | 1.341 (6) | C12—C13 1.361 (6) |
| Ni1—C4 | 1.456 (5) | C12—H12 0.9300 |
| Ni1—C11 | 1.312 (5) | C13—H13 0.9300 |
| Ni1—C12 | 1.370 (5) | C14—C15 1.495 (6) |
| Ni1—C1 | 1.339 (5) | C14—H14A 0.9700 |
| Ni1—C13 | 1.365 (5) | C14—H14B 0.9700 |
| Ni1—C14 | 1.456 (5) | C15—C16 1.376 (6) |
C1—H1  0.9300  C15—C20  1.378 (6)  
C2—C3  1.351 (6)  C16—C17  1.386 (6)  
C2—H2  0.9300  C16—H16  0.9300  
C3—H3  0.9300  C17—C18  1.380 (6)  
C4—C5  1.510 (6)  C17—H17  0.9300  
C4—H4A  0.9700  C18—C19  1.378 (6)  
C4—H4B  0.9700  C18—C18iii  1.476 (9)  
C5—C10  1.374 (6)  C19—C20  1.382 (6)  
C5—C6  1.377 (6)  C19—H19  0.9300  
C6—C7  1.383 (6)  C20—H20  0.9300  

N3i—Ni1—N3  180.0  C7—C6—H6  119.7  
N3i—Ni1—N1  87.59 (13)  C8—C7—C6  121.8 (4)  
N3—Ni1—N1  92.41 (13)  C8—C7—H7  119.1  
N3i—Ni1—N1i  92.41 (13)  C6—C7—H7  119.1  
N3—Ni1—N1i  87.59 (13)  C7—C8—C9  117.4 (4)  
N1—Ni1—N1i  180.00 (17)  C7—C8—C8i  121.8 (4)  
N3i—Ni1—Cl1  90.22 (10)  C9—C8—C8i  120.8 (5)  
N3—Ni1—Cl1  89.78 (10)  C10—C9—C8  120.3 (5)  
N1—Ni1—Cl1  89.69 (10)  C10—C9—H9  119.8  
N1i—Ni1—Cl1i  90.31 (10)  C9—C10—C8  119.8  
N3i—Ni1—Cl1i  89.78 (10)  C9—C10—H10  119.1  
N3—Ni1—Cl1i  90.22 (10)  C9—C10—H10  119.1  
N1—Ni1—Cl1i  90.31 (10)  C9—C10—H10  119.1  
N1i—Ni1—Cl1i  89.69 (10)  C9—C10—H10  119.1  
C11—Ni1—Cl1i  180.0  N3—C11—N4  112.5 (4)  
C1—N1—C3  103.7 (4)  N4—C11—H11  123.8  
C1—N1—Ni1  130.8 (3)  N4—C11—H11  123.8  
C3—N1—Ni1  125.5 (3)  C10—C11—N3  127.5  
C1—N2—C2  107.0 (4)  C10—C11—N4  127.5  
C1—N2—C4  127.1 (4)  C10—C11—N4  127.5  
C2—N2—C4  125.7 (4)  C10—C11—H13  127.5  
C11—N3—C12  104.2 (4)  N4—C13—H13  112.6 (4)  
C11—N3—Ni1  128.3 (3)  N4—C14—H14A  112.6 (4)  
C12—N3—Ni1  127.4 (3)  N4—C14—H14A  112.6 (4)  
C11—N4—C13  107.3 (4)  N4—C14—H14B  109.3  
C11—N4—C14  125.6 (4)  N4—C14—H14B  109.3  
C13—N4—C14  127.1 (4)  N4—C14—H14B  109.3  
N1—C1—N2  112.6 (4)  H14A—C14—H14B  108.0  
N1—C1—H1  123.7  C16—C15—C20  117.4 (4)  
N2—C1—H1  123.7  C16—C15—C14  123.0 (4)  
N2—C2—C3  106.0 (4)  C20—C15—C14  119.6 (4)  
N2—C2—H2  127.0  C15—C16—C17  122.0 (4)  
C3—C2—H2  127.0  C15—C16—H16  119.0  
C2—C3—N1  110.8 (4)  C17—C16—H16  119.0  
C2—C3—H3  124.6  C18—C17—C16  120.1 (4)  
N1—C3—H3  124.6  C18—C17—H17  120.0  
N2—C4—C5  110.8 (3)  C16—C17—H17  120.0
N2—C4—H4A 109.5  C19—C18—C17 118.2 (4)
C5—C4—H4A 109.5  C19—C18—C18iii 120.6 (5)
N2—C4—H4B 109.5  C17—C18—C18iii 121.2 (5)
C5—C4—H4B 109.5  C18—C19—C20 121.1 (4)
H4A—C4—H4B 108.1  C18—C19—H19 119.4
C10—C5—C6 118.0 (4)  C20—C19—H19 119.4
C10—C5—C4 120.5 (4)  C15—C20—C19 121.1 (4)
C6—C5—C4 121.4 (5)  C15—C20—H20 119.4
C5—C6—C7 120.6 (5)  C19—C20—H20 119.4
C5—C6—H6 119.7

C3—N1—C1—N2 −0.9 (5)  C12—N3—C11—N4 1.7 (5)
Ni1—N1—C1—N2 176.6 (3)  Ni1—N3—C11—N4 178.2 (3)
C2—N2—C1—N1 1.2 (5)  C13—N4—C11—N3 −1.1 (5)
C4—N2—C1—N1 175.8 (4)  C14—N4—C11—N3 179.6 (4)
C1—N2—C2—C3 −0.9 (5)  C11—N3—C12—C13 −1.7 (5)
C4—N2—C2—C3 −175.6 (4)  Ni1—N3—C12—C13 −178.2 (3)
N2—C2—C3—N1 0.4 (6)  N3—C12—C13—N4 1.1 (6)
C1—N1—C3—C2 0.3 (5)  C11—N4—C13—C12 0.0 (5)
Ni1—N1—C3—C2 −177.4 (3)  C14—N4—C13—C12 179.3 (4)
C1—N2—C4—C5 −105.6 (5)  C11—N4—C14—C15 −116.3 (5)
C2—N2—C4—C5 68.0 (6)  C13—N4—C14—C15 64.6 (6)
C6—C5—C10—C9 2.8 (7)  C16—C15—C20—C19 −0.4 (7)
C4—C5—C10—C9 −173.6 (4)  C14—C15—C20—C19 −179.5 (4)
N2—C4—C5—C6 −98.6 (5)  C18—C19—C20—C15 0.7 (7)
C10—C5—C6—C7 −2.6 (7)  C20—C15—C16—C17 0.6 (7)
C4—C5—C6—C7 173.8 (4)  C14—C15—C16—C17 179.7 (4)
C5—C6—C7—C8 0.0 (7)  C15—C16—C17—C18 −1.0 (7)
C6—C7—C8—C9 2.2 (7)  C16—C17—C18—C19 1.2 (7)
C6—C7—C8—C8iii −175.6 (3)  C16—C17—C18—C18iii −177.3 (3)
C7—C8—C9—C10 −2.0 (7)  C17—C18—C19—C20 −1.0 (7)
C8iii—C8—C9—C10 175.9 (4)  C18iii—C18—C19—C20 177.5 (3)
C6—C5—C10—C9 2.8 (7)  C16—C15—C20—C19 −0.4 (7)
C4—C5—C10—C9 −173.6 (4)  C14—C15—C20—C19 −179.5 (4)
C8—C9—C10—C5 −0.5 (8)  C18—C19—C20—C15 0.7 (7)

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

Hydrogen-bond geometry (Å, °)

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|------|-------|-------|---------|
| C11—H11···Cl1iv | 0.93 | 2.79 | 3.605 (4) | 147 |
| C14—H14B···Cl1iv | 0.97 | 2.80 | 3.686 (5) | 153 |

Symmetry code: (iv) −x+1/2, −y+3/2, −z+1.