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# [2-Formyl-4-methyl-6-({2-[2-(4-nitrobenzylamino)ethylamino]ethylimino}methyl)phenolato]nickel(II) perchlorate

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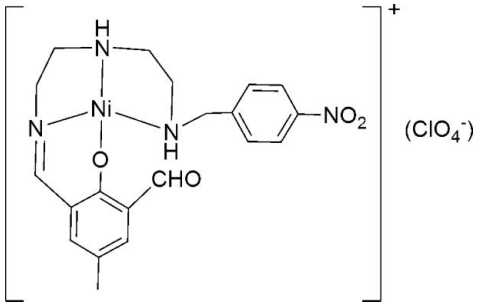
Key indicators: single-crystal X-ray study; $T$ = 291 K; mean $\sigma$(C–C) = 0.006 Å; $R$ factor = 0.060; $wR$ factor = 0.138; data-to-parameter ratio = 14.0.

In the unsymmetrical title complex, $[Ni(C_{20}H_{23}N_4O_4)]ClO_4$, the coordination geometry for the $Ni^{II}$ atom can be described as square planar. The aromatic rings in the two ligands are almost vertical, with a dihedral angle of 85.3°. In the crystal, cations and anions are linked by weak C(N)—H···O hydrogen bonding.

## Related literature

For Schiff base complexes containing polynitrogen ligands, see: Gao *et al.* (2002); Souza *et al.* (2009); Tsubomura *et al.* (2000) and for nickel–Schiff base complexes, see: Wu *et al.* (2011); Cheng *et al.* (2011); Wang *et al.* (2008). For the synthesis, see: Zhou *et al.* (2009). For the preparation of 2,6-diformyl-4-methylphenol, see: Long & Hendrickson (1983); Mandal *et al.* (1989) and for the preparation of $N^1$-(2-aminoethyl)-$N^2$-(4-nitrobenzyl)ethane-1,2-diamine, see: Hu *et al.* (2011); Jian *et al.* (2004).




## Experimental

### Crystal data

$[Ni(C_{20}H_{23}N_4O_4)]ClO_4$
$M_r$ = 541.58
Triclinic, $P\overline{1}$
$a$ = 9.5240 (14) Å
$b$ = 9.6423 (14) Å
$c$ = 14.024 (2) Å
$\alpha$ = 97.897 (2)°
$\beta$ = 109.415 (3)°
$\gamma$ = 107.453 (2)°
$V$ = 1117.7 (3) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 1.04 mm$^{-1}$
$T$ = 291 K
0.28 × 0.24 × 0.22 mm

### Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min}$ = 0.739, $T_{max}$ = 0.786
6276 measured reflections
4314 independent reflections
3004 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.028

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.138
$S$ = 1.10
4314 reflections
308 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.49 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.44 e Å$^{-3}$

**Table 1**
Selected bond lengths (Å).

| | | | |
|---|---|---|---|
| N2—Ni1 | 1.841 (3) | N4—Ni1 | 1.936 (4) |
| N3—Ni1 | 1.893 (3) | Ni1—O1 | 1.829 (3) |

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: KP2412).

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# supporting information

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# [2-Formyl-4-methyl-6-({2-[2-(4-nitrobenzylamino)ethylamino]ethylimino}-methyl)phenolato]nickel(II) perchlorate

**Yang Wang, Jia-Wei Mao, Hui-Ting Song and Hong Zhou**

## S1. Comment

For a long time Schiff base complexes containing polynitrogen ligands have been given considerable attention (Gao *et al.* 2002; Tsubomura *et al.* 2000; Souza *et al.* 2009). In this paper, we report on the synthesis and crystal structure determination of the title complex obtained by the reaction of 2,6-diformyl-4-methylphenol (*L*$^1$) and the polynitrogen ligand N$^1$-(2-aminoethyl)-N$^2$-(4-nitrobenzyl)ethane-1,2-diamine (*L*$^2$) in the presence of $Ni(ClO_4)_2.6H_2O$.

In the title complex, $[NiC_{20}H_{23}N_4O_4].ClO_4$, the Ni$^{II}$ atom is four-coordinated by three amino N atoms from the ligand *L*$^2$ and one O atom from the ligand *L*$^1$, the basal bond distances around the Ni$^{II}$ atom are in the range of 1.829–1.936 Å (Fig. 1, Table 1), and the Ni–O distance shorter than that of Ni–N. The four atoms are coplanar with mean plane deviation of 0.027 (4) Å.

## S2. Experimental

All the solvents and chemicals were of analytical grade and used without further purification. 2,6-Diformyl-4-methyl-phenol was prepared according to the literature method (Mandal *et al.* 1989; Long *et al.* 1983). N$^1$-(2-aminoethyl)- N$^2$-(4-nitrobenzyl)ethane-1,2-diamine was prepared according to the literature method (Jian *et al.* 2004; Hu *et al.* 2011). *L*$^2$ (0.119 g, 0.5 mmol) dissolved in 10 mL water was added dropwise to a solution of *L*$^1$ (0.082 g, 0.5 mmol) and $Ni(ClO_4)_2.6H_2O$ (0.183g, 0.5 mmol) in anhydrous ethanol (25 mL), the mixture was stirred at ambient temperature for 8 h and filtered. The orange block crystals suitable for the X-ray measurement were obtained by evaporation of the filtrate at room temperature for three weeks.

## S3. Refinement

All H atoms for C-H distances were placed in calculated positions and included in the refinement in the riding-model approximation, with U(H) set to -1.2Ueq of the parent atom.

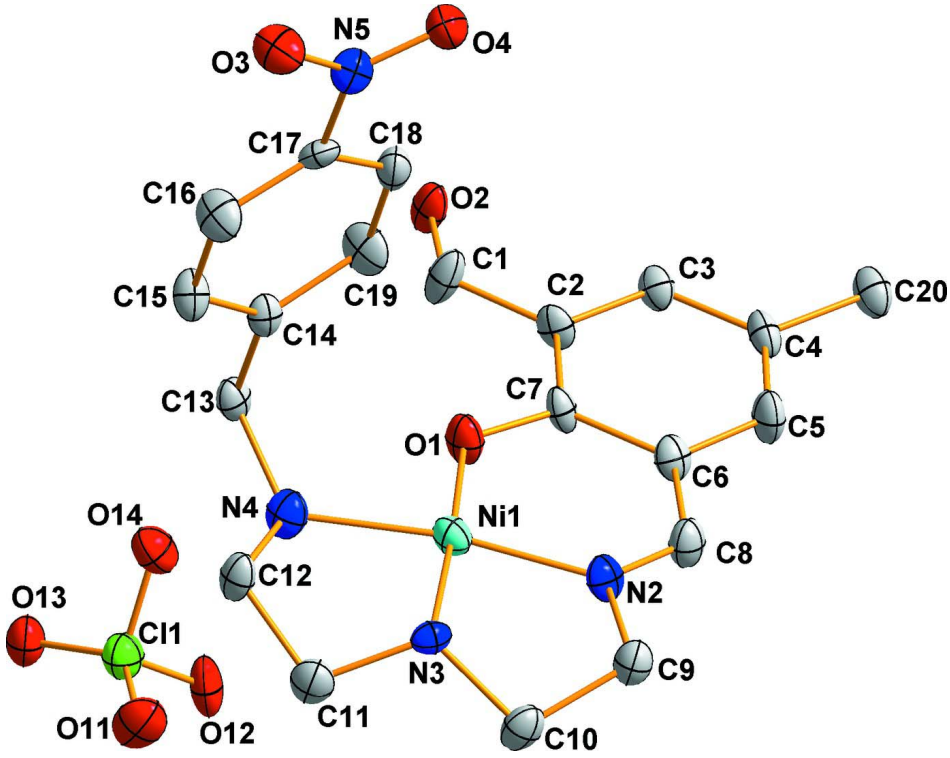


**Figure 1**

A view of the title complex with displacement ellipsoids at the 30% probability level. H atoms are excluded for clarity.

**[2-Formyl-4-methyl-6-({2-[2-(4- nitrobenzylamino)ethylamino]ethylimino}methyl)phenolato]nickel(II) perchlorate**

*Crystal data*

$[Ni(C_{20}H_{23}N_4O_4)]ClO_4$
$M_r = 541.58$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 9.5240$ (14) Å
$b = 9.6423$ (14) Å
$c = 14.024$ (2) Å
$\alpha = 97.897$ (2)°
$\beta = 109.415$ (3)°
$\gamma = 107.453$ (2)°
$V = 1117.7$ (3) Å$^3$
$Z = 2$
$F(000) = 560$
$D_x = 1.609$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3108 reflections
$\theta = 2.3$–$28.0°$
$\mu = 1.04$ mm$^{-1}$
$T = 291$ K
Block, orange
0.28 × 0.24 × 0.22 mm

*Data collection*

Bruker SMART APEX CCD diffractometer
Radiation source: sealed tube
Graphite monochromator
phi and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min} = 0.739$, $T_{max} = 0.786$
6276 measured reflections
4314 independent reflections
3004 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 26.0°$, $\theta_{min} = 2.3°$
$h = -11 \rightarrow 11$
$k = -9 \rightarrow 11$
$l = -17 \rightarrow 15$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.060$
$wR(F^2) = 0.138$
$S = 1.10$
4314 reflections
308 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
$w = 1/[\sigma^2(F_o^2) + (0.0689P)^2 + 0.0812P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.49$ e Å$^{-3}$
$\Delta\rho_{min} = -0.44$ 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**. 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 $F^2 > 2$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 | 0.2128 (6) | 0.2151 (5) | 0.6715 (3) | 0.0490 (11) |
| H1 | 0.3112 | 0.2616 | 0.7281 | 0.059* |
| C2 | 0.1781 (4) | 0.2941 (4) | 0.5895 (3) | 0.0349 (8) |
| C3 | 0.0262 (4) | 0.2311 (4) | 0.5055 (3) | 0.0361 (8) |
| H3 | −0.0489 | 0.1421 | 0.5039 | 0.043* |
| C4 | −0.0125 (4) | 0.3010 (4) | 0.4249 (3) | 0.0371 (8) |
| C5 | 0.1016 (5) | 0.4324 (5) | 0.4308 (3) | 0.0474 (10) |
| H5 | 0.0762 | 0.4798 | 0.3776 | 0.057* |
| C6 | 0.2522 (5) | 0.4988 (4) | 0.5112 (3) | 0.0370 (8) |
| C7 | 0.2919 (4) | 0.4294 (4) | 0.5956 (3) | 0.0328 (8) |
| C8 | 0.3605 (5) | 0.6361 (5) | 0.5105 (3) | 0.0444 (9) |
| H8 | 0.3318 | 0.6709 | 0.4510 | 0.053* |
| C9 | 0.5962 (5) | 0.8644 (5) | 0.5798 (3) | 0.0418 (9) |
| H9A | 0.5653 | 0.9445 | 0.6055 | 0.050* |
| H9B | 0.5828 | 0.8606 | 0.5077 | 0.050* |
| C10 | 0.7678 (5) | 0.8907 (5) | 0.6473 (3) | 0.0456 (10) |
| H10A | 0.8073 | 0.8262 | 0.6128 | 0.055* |
| H10B | 0.8365 | 0.9949 | 0.6616 | 0.055* |
| C11 | 0.9105 (5) | 0.8406 (5) | 0.8155 (3) | 0.0472 (10) |
| H11A | 0.9946 | 0.9394 | 0.8498 | 0.057* |
| H11B | 0.9477 | 0.7792 | 0.7764 | 0.057* |
| C12 | 0.8651 (5) | 0.7671 (4) | 0.8951 (3) | 0.0400 (9) |
| H12A | 0.9505 | 0.7381 | 0.9362 | 0.048* |
| H12B | 0.8471 | 0.8368 | 0.9422 | 0.048* |
| C13 | 0.6434 (4) | 0.5655 (4) | 0.9063 (3) | 0.0353 (8) |

| | | | | |
|---|---|---|---|---|
| H13A | 0.5598 | 0.4680 | 0.8666 | 0.042* |
| H13B | 0.7246 | 0.5495 | 0.9624 | 0.042* |
| C14 | 0.5743 (4) | 0.6609 (4) | 0.9526 (3) | 0.0312 (7) |
| C15 | 0.6606 (5) | 0.7570 (4) | 1.0532 (3) | 0.0390 (9) |
| H15 | 0.7632 | 0.7612 | 1.0912 | 0.047* |
| C16 | 0.5967 (5) | 0.8476 (5) | 1.0984 (3) | 0.0509 (11) |
| H16 | 0.6567 | 0.9150 | 1.1644 | 0.061* |
| C17 | 0.4424 (4) | 0.8333 (4) | 1.0419 (3) | 0.0341 (8) |
| C18 | 0.3494 (5) | 0.7342 (5) | 0.9422 (3) | 0.0424 (9) |
| H18 | 0.2446 | 0.7261 | 0.9062 | 0.051* |
| C19 | 0.4163 (5) | 0.6489 (5) | 0.8986 (3) | 0.0496 (11) |
| H19 | 0.3560 | 0.5825 | 0.8323 | 0.060* |
| C20 | −0.1760 (5) | 0.2319 (5) | 0.3341 (3) | 0.0455 (10) |
| H20A | −0.2257 | 0.3053 | 0.3274 | 0.068* |
| H20B | −0.2422 | 0.1461 | 0.3472 | 0.068* |
| H20C | −0.1634 | 0.2008 | 0.2704 | 0.068* |
| Cl1 | 0.88876 (12) | 0.32607 (11) | 0.79500 (8) | 0.0463 (3) |
| N2 | 0.4978 (4) | 0.7171 (3) | 0.5876 (3) | 0.0393 (7) |
| N3 | 0.7633 (3) | 0.8528 (3) | 0.7458 (2) | 0.0307 (6) |
| H3A | 0.7438 | 0.9259 | 0.7817 | 0.037* |
| N4 | 0.7161 (4) | 0.6317 (4) | 0.8366 (3) | 0.0463 (8) |
| H4 | 0.7513 | 0.5615 | 0.8131 | 0.056* |
| N5 | 0.3722 (4) | 0.9276 (3) | 1.0862 (3) | 0.0393 (7) |
| Ni1 | 0.58985 (5) | 0.66844 (6) | 0.70964 (4) | 0.03821 (17) |
| O1 | 0.4300 (3) | 0.4860 (3) | 0.6787 (2) | 0.0460 (7) |
| O2 | 0.1258 (3) | 0.0957 (3) | 0.6725 (2) | 0.0457 (7) |
| O3 | 0.4487 (4) | 1.0016 (4) | 1.1791 (2) | 0.0542 (8) |
| O4 | 0.2448 (3) | 0.9331 (3) | 1.0331 (2) | 0.0458 (7) |
| O11 | 0.9790 (4) | 0.4623 (4) | 0.8000 (3) | 0.0642 (10) |
| O12 | 0.8093 (4) | 0.2515 (3) | 0.6896 (2) | 0.0553 (8) |
| O13 | 0.9309 (4) | 0.2177 (3) | 0.8403 (2) | 0.0494 (7) |
| O14 | 0.7706 (4) | 0.3386 (3) | 0.8214 (3) | 0.0527 (8) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.057 (3) | 0.034 (2) | 0.0290 (19) | −0.0028 (19) | 0.0020 (18) | 0.0108 (16) |
| C2 | 0.0257 (16) | 0.0219 (16) | 0.0395 (19) | 0.0004 (13) | 0.0019 (14) | 0.0016 (14) |
| C3 | 0.0340 (19) | 0.0333 (18) | 0.0306 (18) | 0.0112 (16) | 0.0044 (15) | 0.0006 (15) |
| C4 | 0.0330 (18) | 0.041 (2) | 0.0288 (17) | 0.0206 (16) | 0.0001 (14) | −0.0013 (15) |
| C5 | 0.039 (2) | 0.048 (2) | 0.035 (2) | 0.0113 (18) | −0.0043 (17) | 0.0095 (17) |
| C6 | 0.0396 (19) | 0.0278 (17) | 0.042 (2) | 0.0162 (15) | 0.0107 (16) | 0.0097 (15) |
| C7 | 0.0268 (16) | 0.0225 (16) | 0.0392 (19) | 0.0095 (14) | 0.0007 (14) | 0.0084 (14) |
| C8 | 0.046 (2) | 0.038 (2) | 0.043 (2) | 0.0146 (18) | 0.0089 (18) | 0.0186 (17) |
| C9 | 0.045 (2) | 0.040 (2) | 0.036 (2) | 0.0135 (17) | 0.0109 (17) | 0.0121 (16) |
| C10 | 0.051 (2) | 0.037 (2) | 0.042 (2) | 0.0060 (18) | 0.0174 (19) | 0.0155 (18) |
| C11 | 0.041 (2) | 0.043 (2) | 0.047 (2) | 0.0061 (18) | 0.0127 (18) | 0.0121 (18) |
| C12 | 0.0399 (19) | 0.0326 (18) | 0.0293 (18) | 0.0033 (16) | 0.0019 (15) | 0.0061 (14) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C13 | 0.0355 (19) | 0.0280 (17) | 0.042 (2) | 0.0137 (15) | 0.0111 (16) | 0.0147 (15) |
| C14 | 0.0332 (18) | 0.0202 (15) | 0.0344 (18) | 0.0082 (14) | 0.0076 (14) | 0.0083 (13) |
| C15 | 0.046 (2) | 0.0255 (18) | 0.0349 (19) | 0.0162 (16) | 0.0035 (17) | 0.0025 (14) |
| C16 | 0.049 (2) | 0.048 (2) | 0.037 (2) | 0.014 (2) | 0.0058 (18) | −0.0099 (18) |
| C17 | 0.0376 (19) | 0.0390 (19) | 0.0334 (19) | 0.0109 (16) | 0.0239 (16) | 0.0147 (15) |
| C18 | 0.037 (2) | 0.049 (2) | 0.037 (2) | 0.0136 (18) | 0.0078 (16) | 0.0194 (17) |
| C19 | 0.039 (2) | 0.042 (2) | 0.038 (2) | 0.0032 (18) | −0.0005 (17) | −0.0135 (17) |
| C20 | 0.040 (2) | 0.046 (2) | 0.035 (2) | 0.0166 (19) | 0.0017 (17) | −0.0023 (17) |
| Cl1 | 0.0463 (6) | 0.0431 (6) | 0.0465 (6) | 0.0159 (4) | 0.0142 (4) | 0.0146 (4) |
| N2 | 0.0400 (17) | 0.0305 (16) | 0.0445 (18) | 0.0154 (14) | 0.0107 (14) | 0.0117 (13) |
| N3 | 0.0364 (16) | 0.0258 (14) | 0.0349 (16) | 0.0115 (12) | 0.0196 (13) | 0.0092 (12) |
| N4 | 0.0458 (19) | 0.0414 (18) | 0.0373 (18) | 0.0090 (16) | 0.0078 (15) | 0.0043 (14) |
| N5 | 0.0464 (18) | 0.0294 (15) | 0.0393 (18) | 0.0135 (14) | 0.0147 (15) | 0.0071 (13) |
| Ni1 | 0.0278 (3) | 0.0389 (3) | 0.0393 (3) | 0.0069 (2) | 0.0085 (2) | 0.0086 (2) |
| O1 | 0.0350 (14) | 0.0392 (15) | 0.0425 (16) | 0.0036 (12) | −0.0002 (12) | 0.0087 (12) |
| O2 | 0.0385 (15) | 0.0402 (15) | 0.0300 (13) | −0.0050 (12) | −0.0028 (11) | 0.0086 (11) |
| O3 | 0.0502 (17) | 0.0526 (18) | 0.0440 (17) | 0.0139 (14) | 0.0136 (14) | −0.0118 (14) |
| O4 | 0.0422 (15) | 0.0493 (17) | 0.0461 (16) | 0.0191 (13) | 0.0152 (13) | 0.0142 (13) |
| O11 | 0.056 (2) | 0.055 (2) | 0.0561 (19) | −0.0056 (16) | 0.0158 (16) | 0.0115 (15) |
| O12 | 0.0471 (16) | 0.0429 (16) | 0.0541 (18) | 0.0146 (13) | −0.0071 (14) | 0.0198 (14) |
| O13 | 0.0488 (16) | 0.0480 (17) | 0.0433 (15) | 0.0168 (13) | 0.0077 (13) | 0.0165 (13) |
| O14 | 0.0452 (16) | 0.0505 (17) | 0.063 (2) | 0.0222 (14) | 0.0182 (15) | 0.0161 (15) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—O2 | 1.205 (5) | C13—C14 | 1.478 (5) |
| C1—C2 | 1.466 (5) | C13—N4 | 1.483 (5) |
| C1—H1 | 0.9300 | C13—H13A | 0.9700 |
| C2—C7 | 1.394 (5) | C13—H13B | 0.9700 |
| C2—C3 | 1.411 (5) | C14—C15 | 1.392 (5) |
| C3—C4 | 1.397 (5) | C14—C19 | 1.402 (5) |
| C3—H3 | 0.9300 | C15—C16 | 1.400 (6) |
| C4—C5 | 1.370 (6) | C15—H15 | 0.9300 |
| C4—C20 | 1.522 (5) | C16—C17 | 1.372 (6) |
| C5—C6 | 1.384 (5) | C16—H16 | 0.9300 |
| C5—H5 | 0.9300 | C17—C18 | 1.398 (5) |
| C6—C8 | 1.417 (6) | C17—N5 | 1.465 (5) |
| C6—C7 | 1.437 (5) | C18—C19 | 1.376 (6) |
| C7—O1 | 1.327 (4) | C18—H18 | 0.9300 |
| C8—N2 | 1.305 (5) | C19—H19 | 0.9300 |
| C8—H8 | 0.9300 | C20—H20A | 0.9600 |
| C9—N2 | 1.489 (5) | C20—H20B | 0.9600 |
| C9—C10 | 1.513 (6) | C20—H20C | 0.9600 |
| C9—H9A | 0.9700 | Cl1—O11 | 1.314 (3) |
| C9—H9B | 0.9700 | Cl1—O14 | 1.328 (3) |
| C10—N3 | 1.487 (5) | Cl1—O12 | 1.383 (3) |
| C10—H10A | 0.9700 | Cl1—O13 | 1.386 (3) |
| C10—H10B | 0.9700 | N2—Ni1 | 1.841 (3) |

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| C11—N3 | 1.468 (5) | N3—Ni1 | 1.893 (3) |
| C11—C12 | 1.513 (6) | N3—H3A | 0.9100 |
| C11—H11A | 0.9700 | N4—Ni1 | 1.936 (4) |
| C11—H11B | 0.9700 | N4—H4 | 0.9100 |
| C12—N4 | 1.487 (5) | N5—O4 | 1.216 (4) |
| C12—H12A | 0.9700 | N5—O3 | 1.241 (4) |
| C12—H12B | 0.9700 | Ni1—O1 | 1.829 (3) |

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| O2—C1—C2 | 126.2 (4) | C15—C14—C13 | 120.8 (3) |
| O2—C1—H1 | 116.9 | C19—C14—C13 | 120.4 (3) |
| C2—C1—H1 | 116.9 | C14—C15—C16 | 121.5 (4) |
| C7—C2—C3 | 121.0 (3) | C14—C15—H15 | 119.2 |
| C7—C2—C1 | 120.5 (3) | C16—C15—H15 | 119.2 |
| C3—C2—C1 | 118.4 (3) | C17—C16—C15 | 117.5 (4) |
| C4—C3—C2 | 120.7 (3) | C17—C16—H16 | 121.2 |
| C4—C3—H3 | 119.6 | C15—C16—H16 | 121.2 |
| C2—C3—H3 | 119.6 | C16—C17—C18 | 122.8 (4) |
| C5—C4—C3 | 117.6 (3) | C16—C17—N5 | 119.0 (3) |
| C5—C4—C20 | 121.9 (3) | C18—C17—N5 | 118.1 (3) |
| C3—C4—C20 | 120.5 (4) | C19—C18—C17 | 118.5 (4) |
| C4—C5—C6 | 124.0 (4) | C19—C18—H18 | 120.7 |
| C4—C5—H5 | 118.0 | C17—C18—H18 | 120.8 |
| C6—C5—H5 | 118.0 | C18—C19—C14 | 120.9 (4) |
| C5—C6—C8 | 119.4 (4) | C18—C19—H19 | 119.6 |
| C5—C6—C7 | 118.8 (3) | C14—C19—H19 | 119.6 |
| C8—C6—C7 | 121.7 (3) | C4—C20—H20A | 109.5 |
| O1—C7—C2 | 118.2 (3) | C4—C20—H20B | 109.5 |
| O1—C7—C6 | 124.0 (3) | H20A—C20—H20B | 109.5 |
| C2—C7—C6 | 117.8 (3) | C4—C20—H20C | 109.5 |
| N2—C8—C6 | 124.6 (4) | H20A—C20—H20C | 109.5 |
| N2—C8—H8 | 117.7 | H20B—C20—H20C | 109.5 |
| C6—C8—H8 | 117.7 | O11—Cl1—O14 | 107.3 (2) |
| N2—C9—C10 | 105.6 (3) | O11—Cl1—O12 | 106.0 (2) |
| N2—C9—H9A | 110.6 | O14—Cl1—O12 | 102.5 (2) |
| C10—C9—H9A | 110.6 | O11—Cl1—O13 | 129.9 (2) |
| N2—C9—H9B | 110.6 | O14—Cl1—O13 | 105.0 (2) |
| C10—C9—H9B | 110.6 | O12—Cl1—O13 | 103.12 (18) |
| H9A—C9—H9B | 108.8 | C8—N2—C9 | 119.4 (3) |
| N3—C10—C9 | 105.2 (3) | C8—N2—Ni1 | 126.6 (3) |
| N3—C10—H10A | 110.7 | C9—N2—Ni1 | 114.0 (2) |
| C9—C10—H10A | 110.7 | C11—N3—C10 | 115.1 (3) |
| N3—C10—H10B | 110.7 | C11—N3—Ni1 | 109.1 (2) |
| C9—C10—H10B | 110.7 | C10—N3—Ni1 | 108.2 (2) |
| H10A—C10—H10B | 108.8 | C11—N3—H3A | 108.1 |
| N3—C11—C12 | 105.3 (3) | C10—N3—H3A | 108.1 |
| N3—C11—H11A | 110.7 | Ni1—N3—H3A | 108.1 |
| C12—C11—H11A | 110.7 | C13—N4—C12 | 112.6 (3) |
| N3—C11—H11B | 110.7 | C13—N4—Ni1 | 121.8 (3) |

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| C12—C11—H11B | 110.7 | C12—N4—Ni1 | 109.0 (3) |
| H11A—C11—H11B | 108.8 | C13—N4—H4 | 103.8 |
| N4—C12—C11 | 107.6 (3) | C12—N4—H4 | 103.8 |
| N4—C12—H12A | 110.2 | Ni1—N4—H4 | 103.8 |
| C11—C12—H12A | 110.2 | O4—N5—O3 | 122.0 (3) |
| N4—C12—H12B | 110.2 | O4—N5—C17 | 120.8 (3) |
| C11—C12—H12B | 110.2 | O3—N5—C17 | 117.2 (3) |
| H12A—C12—H12B | 108.5 | O1—Ni1—N2 | 96.40 (13) |
| C14—C13—N4 | 113.3 (3) | O1—Ni1—N3 | 176.88 (13) |
| C14—C13—H13A | 108.9 | N2—Ni1—N3 | 86.25 (13) |
| N4—C13—H13A | 108.9 | O1—Ni1—N4 | 90.39 (14) |
| C14—C13—H13B | 108.9 | N2—Ni1—N4 | 171.64 (15) |
| N4—C13—H13B | 108.9 | N3—Ni1—N4 | 86.84 (14) |
| H13A—C13—H13B | 107.7 | C7—O1—Ni1 | 126.2 (2) |
| C15—C14—C19 | 118.7 (4) | | |

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| O2—C1—C2—C7 | −177.4 (5) | C13—C14—C19—C18 | 178.2 (4) |
| O2—C1—C2—C3 | 3.6 (7) | C6—C8—N2—C9 | −176.1 (4) |
| C7—C2—C3—C4 | 1.6 (6) | C6—C8—N2—Ni1 | 4.6 (7) |
| C1—C2—C3—C4 | −179.4 (4) | C10—C9—N2—C8 | −153.0 (4) |
| C2—C3—C4—C5 | −0.4 (6) | C10—C9—N2—Ni1 | 26.4 (4) |
| C2—C3—C4—C20 | 179.6 (4) | C12—C11—N3—C10 | −167.1 (3) |
| C3—C4—C5—C6 | 0.5 (6) | C12—C11—N3—Ni1 | −45.2 (4) |
| C20—C4—C5—C6 | −179.4 (4) | C9—C10—N3—C11 | 168.3 (3) |
| C4—C5—C6—C8 | −179.1 (4) | C9—C10—N3—Ni1 | 46.0 (3) |
| C4—C5—C6—C7 | −1.8 (7) | C14—C13—N4—C12 | 69.5 (4) |
| C3—C2—C7—O1 | 177.3 (3) | C14—C13—N4—Ni1 | −62.8 (4) |
| C1—C2—C7—O1 | −1.7 (6) | C11—C12—N4—C13 | −169.7 (3) |
| C3—C2—C7—C6 | −2.8 (6) | C11—C12—N4—Ni1 | −31.3 (4) |
| C1—C2—C7—C6 | 178.2 (4) | C16—C17—N5—O4 | 169.9 (4) |
| C5—C6—C7—O1 | −177.2 (4) | C18—C17—N5—O4 | −8.8 (5) |
| C8—C6—C7—O1 | 0.1 (6) | C16—C17—N5—O3 | −9.5 (5) |
| C5—C6—C7—C2 | 2.9 (6) | C18—C17—N5—O3 | 171.8 (3) |
| C8—C6—C7—C2 | −179.8 (4) | C8—N2—Ni1—O1 | 0.3 (4) |
| C5—C6—C8—N2 | 171.9 (4) | C9—N2—Ni1—O1 | −179.0 (3) |
| C7—C6—C8—N2 | −5.4 (7) | C8—N2—Ni1—N3 | 178.7 (4) |
| N2—C9—C10—N3 | −45.3 (4) | C9—N2—Ni1—N3 | −0.6 (3) |
| N3—C11—C12—N4 | 49.4 (4) | C11—N3—Ni1—N2 | −152.1 (3) |
| N4—C13—C14—C15 | −97.3 (4) | C10—N3—Ni1—N2 | −26.2 (3) |
| N4—C13—C14—C19 | 86.9 (4) | C11—N3—Ni1—N4 | 23.2 (3) |
| C19—C14—C15—C16 | −3.8 (6) | C10—N3—Ni1—N4 | 149.1 (3) |
| C13—C14—C15—C16 | −179.7 (4) | C13—N4—Ni1—O1 | −42.5 (3) |
| C14—C15—C16—C17 | 3.0 (6) | C12—N4—Ni1—O1 | −176.3 (3) |
| C15—C16—C17—C18 | −0.6 (6) | C13—N4—Ni1—N3 | 138.9 (3) |
| C15—C16—C17—N5 | −179.3 (4) | C12—N4—Ni1—N3 | 5.1 (3) |
| C16—C17—C18—C19 | −0.8 (6) | C2—C7—O1—Ni1 | −174.5 (3) |
| N5—C17—C18—C19 | 177.8 (4) | C6—C7—O1—Ni1 | 5.7 (6) |
| C17—C18—C19—C14 | 0.0 (6) | N2—Ni1—O1—C7 | −5.3 (4) |

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| C15—C14—C19—C18 | 2.3 (6) | N4—Ni1—O1—C7 | 179.6 (3) |