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# [4′,4″-Dichloro-7,12-diphenyl-5,6:13,14-dibenzo-1,4,8,11-tetraazacyclopentadeca-5,7,11,13-tetraene-2,3-dione(2−)]-nickel(II)

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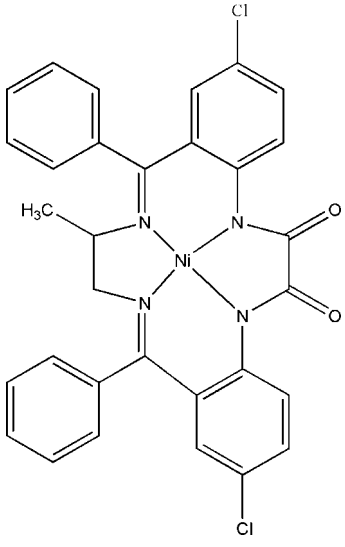
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.004 Å; disorder in main residue; $R$ factor = 0.038; $wR$ factor = 0.094; data-to-parameter ratio = 14.9.

In the crystal structure of the title compound, $[Ni(C_{31}H_{22}Cl_2N_4O_2)]$, the Ni atoms are coordinated by four N atoms of the macrocyclic ligand in a distorted square-planar configuration. Three C atoms are disordered over two positions, with site occupancy factors of 0.85 and 0.15.

## Related literature

For the synthesis of the starting material 2,2′-(oxalyldiimino)bis(chlorobenzaldehyde), see: Zhang *et al.* (2005).




## Experimental

### Crystal data

$[Ni(C_{31}H_{22}Cl_2N_4O_2)]$
$M_r$ = 612.14
Monoclinic, $P2_1/n$
$a$ = 11.7341 (17) Å
$b$ = 14.900 (2) Å
$c$ = 16.235 (2) Å
$\beta$ = 106.582 (2)°
$V$ = 2720.4 (6) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.95 mm$^{-1}$
$T$ = 293 (2) K
0.28 × 0.20 × 0.14 mm

### Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.805, $T_{max}$ = 0.891
15103 measured reflections
5556 independent reflections
3780 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.034

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.095
$S$ = 1.03
5556 reflections
373 parameters
4 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.53 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.28 e Å$^{-3}$

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

| | | | |
|---|---|---|---|
| Ni1—N2 | 1.857 (2) | Ni1—N4 | 1.870 (2) |
| Ni1—N1 | 1.867 (2) | Ni1—N3 | 1.874 (2) |
| N2—Ni1—N1 | 86.53 (9) | N2—Ni1—N3 | 92.76 (9) |
| N2—Ni1—N4 | 176.84 (10) | N1—Ni1—N3 | 171.82 (10) |
| N1—Ni1—N4 | 93.78 (9) | N4—Ni1—N3 | 87.37 (9) |

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); 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: NC2051).

## References

Bruker (1997). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2000). *SHELXTL*. Version 6.12. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Zhang, R.-H., Wang, Q.-L., Wang, Y.-F., Yang, G.-M., Liu, Z.-Q., Ding, B. & Xu, G.-F. (2005). *J. Coord. Chem.* **58**, 857–862.

# supplementary materials

*Acta Cryst.* (2007). E**63**, m2335 [ **doi:10.1107/S1600536807039451** ]

# [4',4''-Dichloro-7,12-diphenyl-5,6:13,14-dibenzo-1,4,8,11-tetraazacyclopentadeca-5,7,11,13-tetraene-2,3-dione(2-)]nickel(II)

**R.-H. Zhang and G.-M. Yang**

## Comment

The crystal structure of the title compound was determined as a part of a project on the structural properties of new nickel complexes with macrocyclic ligands. In the crystal structure of the title compound the nickel atoms are coordinated by four nitrogen atoms of the 2,3-dioxo-5,6:13,14-dichlorobenzo-7,12-diphenyl-1,4,8,11- tetraazacyclo-pentadeca-7,11-diene ligand within a slightly distorted square planar coordination (Fig 1). The nickel atom is located in the molecular plane of the ligand and deviates −0.0405 (12)Å from the plane calculated for N1, N2, N3 and N4.

## Experimental

The synthesis of the reactand 2, 2'-(oxalyldiimino)bis(chlorobenzaldehyde) was described previously (Zhang *et al.*, 2005). 2.96 mmol 2, 2'-(oxalyldiimino)bis(chlorobenzaldehyde), 5.92 mmol 1,2-diaminopropane and 2.96 mmol nickel(II)acetate were refluxed for 10 h in 50 ml of methanol and 2.80 ml of 2 *M* sodium hydroxide. The mixture was cooled down and the red precipitate of the title compound was filtered off. Crystals of the title compound were obtained by slow evaporation of the solvent from the deep red filtrate at room temperature.

## Refinement

The H atoms were positioned with idealized geometry and were refined isotropic ($U_{iso}$ (H) = 1.2 $U_{eq}$(C) (1.5 for methyl H atoms) using a riding model. The carbon atoms C16, C17 and C18 are disordered in two orientations and were refined using a split model and site occupation factors of 75:25. The carbon atoms of lower occupany were refined only isotropic.

## Figures

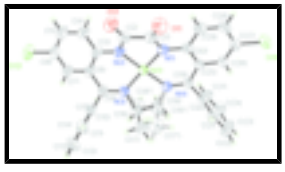


Fig. 1. Crystal structure of compound I with labelling and displacement ellipsoids drawn at the 50% probability level.

## [4',4''-Dichloro-7,12-diphenyl-5,6:13,14-dibenzo-1,4,8,11- tetraazacyclopentadeca-5,7,11,13-tetraene-2,3-dione(2-)]nickel(II)

*Crystal data*

| | |
|---|---|
| $[Ni(C_{31}H_{22}Cl_2N_4O_2)]$ | $F_{000}$ = 1256 |
| $M_r$ = 612.14 | $D_x$ = 1.495 Mg m$^{-3}$ |
| Monoclinic, $P2_1/n$ | Mo $K\alpha$ radiation |

| | |
|---|---|
| | λ = 0.71073 Å |
| *a* = 11.7341 (17) Å | Cell parameters from 4332 reflections |
| *b* = 14.900 (2) Å | θ = 2.5–26.0º |
| *c* = 16.235 (2) Å | μ = 0.95 $mm^{-1}$ |
| β = 106.582 (2)º | *T* = 293 (2) K |
| *V* = 2720.4 (6) $Å^3$ | Block, red |
| *Z* = 4 | 0.28 × 0.20 × 0.14 mm |

*Data collection*

| | |
|---|---|
| Bruker SMART CCD area-detector diffractometer | 5556 independent reflections |
| Radiation source: fine-focus sealed tube | 3780 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{int}$ = 0.034 |
| *T* = 293(2) K | $\theta_{max}$ = 26.4º |
| φ and ω scans | $\theta_{min}$ = 1.9º |
| Absorption correction: multi-scan (SADABS; Sheldrick, 1996) | *h* = −12→14 |
| $T_{min}$ = 0.805, $T_{max}$ = 0.891 | *k* = −18→18 |
| 15103 measured reflections | *l* = −20→10 |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)]$ = 0.038 | H-atom parameters constrained |
| $wR(F^2)$ = 0.095 | $w = 1/[\sigma^2(F_o^2) + (0.0349P)^2 + 1.2682P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| *S* = 1.03 | $(\Delta/\sigma)_{max}$ = 0.001 |
| 5556 reflections | $\Delta\rho_{max}$ = 0.53 e $Å^{-3}$ |
| 373 parameters | $\Delta\rho_{min}$ = −0.28 e $Å^{-3}$ |
| 4 restraints | Extinction correction: none |
| 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 $F^2 > 2\text{sigma}(F^2)$ 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* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| Ni1 | 0.60785 (3) | 0.66024 (2) | 0.08881 (2) | 0.03775 (11) | |
| Cl1 | 0.07277 (7) | 0.39951 (6) | 0.04929 (6) | 0.0701 (3) | |
| Cl2 | 1.23327 (7) | 0.75094 (7) | 0.16199 (6) | 0.0721 (3) | |
| O1 | 0.58144 (19) | 0.55245 (16) | 0.29861 (12) | 0.0666 (6) | |
| O2 | 0.81015 (19) | 0.60420 (15) | 0.31840 (12) | 0.0643 (6) | |
| N1 | 0.54278 (18) | 0.58085 (14) | 0.15208 (13) | 0.0395 (5) | |
| N2 | 0.75073 (19) | 0.64553 (15) | 0.17400 (14) | 0.0427 (6) | |
| N3 | 0.66880 (19) | 0.75272 (15) | 0.03553 (14) | 0.0435 (6) | |
| N4 | 0.46670 (19) | 0.67016 (15) | −0.00050 (14) | 0.0444 (6) | |
| C1 | 0.6104 (2) | 0.57871 (19) | 0.23642 (17) | 0.0442 (7) | |
| C2 | 0.7364 (3) | 0.61159 (18) | 0.24794 (18) | 0.0449 (7) | |
| C3 | 0.8631 (2) | 0.66991 (19) | 0.16892 (17) | 0.0449 (7) | |
| C4 | 0.9652 (3) | 0.6247 (2) | 0.2172 (2) | 0.0610 (9) | |
| H4 | 0.9570 | 0.5774 | 0.2525 | 0.073* | |
| C5 | 1.0769 (3) | 0.6482 (2) | 0.2137 (2) | 0.0661 (9) | |
| H5 | 1.1432 | 0.6167 | 0.2457 | 0.079* | |
| C6 | 1.0897 (2) | 0.7189 (2) | 0.1625 (2) | 0.0542 (8) | |
| C7 | 0.9931 (2) | 0.7629 (2) | 0.11227 (18) | 0.0489 (7) | |
| H7 | 1.0036 | 0.8099 | 0.0775 | 0.059* | |
| C8 | 0.8774 (2) | 0.73791 (18) | 0.11249 (17) | 0.0416 (6) | |
| C9 | 0.7764 (2) | 0.78221 (18) | 0.05036 (17) | 0.0414 (6) | |
| C10 | 0.8029 (2) | 0.86080 (18) | 0.00121 (17) | 0.0415 (7) | |
| C11 | 0.8030 (3) | 0.9470 (2) | 0.0326 (2) | 0.0596 (8) | |
| H11 | 0.7879 | 0.9570 | 0.0850 | 0.071* | |
| C12 | 0.8260 (3) | 1.0182 (2) | −0.0149 (3) | 0.0760 (11) | |
| H12 | 0.8246 | 1.0765 | 0.0053 | 0.091* | |
| C13 | 0.8508 (3) | 1.0040 (3) | −0.0916 (2) | 0.0700 (10) | |
| H13 | 0.8667 | 1.0524 | −0.1227 | 0.084* | |
| C14 | 0.8522 (3) | 0.9188 (2) | −0.1220 (2) | 0.0576 (8) | |
| H14 | 0.8700 | 0.9093 | −0.1736 | 0.069* | |
| C15 | 0.8275 (2) | 0.8472 (2) | −0.07685 (19) | 0.0497 (7) | |
| H15 | 0.8273 | 0.7894 | −0.0984 | 0.060* | |
| C16 | 0.5678 (3) | 0.8024 (2) | −0.0258 (2) | 0.0444 (9) | 0.85 |
| H16 | 0.5976 | 0.8347 | −0.0681 | 0.053* | 0.85 |
| C17 | 0.5088 (4) | 0.8678 (3) | 0.0195 (3) | 0.0659 (11) | 0.85 |
| H17A | 0.4463 | 0.8987 | −0.0219 | 0.099* | 0.85 |
| H17B | 0.5664 | 0.9104 | 0.0507 | 0.099* | 0.85 |
| H17C | 0.4759 | 0.8359 | 0.0588 | 0.099* | 0.85 |
| C18 | 0.4826 (3) | 0.7307 (2) | −0.0705 (2) | 0.0471 (9) | 0.85 |
| H18A | 0.5148 | 0.6975 | −0.1102 | 0.057* | 0.85 |
| H18B | 0.4071 | 0.7568 | −0.1023 | 0.057* | 0.85 |
| C16' | 0.4666 (15) | 0.7683 (9) | −0.0329 (12) | 0.038 (5)* | 0.15 |
| H16' | 0.4057 | 0.7700 | −0.0886 | 0.046* | 0.15 |
| C17' | 0.436 (3) | 0.8440 (16) | 0.0189 (17) | 0.084 (9)* | 0.15 |
| H17D | 0.3598 | 0.8329 | 0.0277 | 0.126* | 0.15 |

| | | | | | |
|---|---|---|---|---|---|
| H17E | 0.4337 | 0.8995 | −0.0115 | 0.126* | 0.15 |
| H17F | 0.4954 | 0.8477 | 0.0736 | 0.126* | 0.15 |
| C18' | 0.5836 (17) | 0.771 (2) | −0.0533 (12) | 0.076 (11)* | 0.15 |
| H18C | 0.5979 | 0.8296 | −0.0749 | 0.091* | 0.15 |
| H18D | 0.5886 | 0.7253 | −0.0944 | 0.091* | 0.15 |
| C19 | 0.3662 (2) | 0.62871 (17) | −0.01172 (17) | 0.0379 (6) | |
| C20 | 0.2648 (2) | 0.64962 (18) | −0.08949 (17) | 0.0394 (6) | |
| C21 | 0.1782 (2) | 0.7101 (2) | −0.0846 (2) | 0.0518 (8) | |
| H21 | 0.1822 | 0.7382 | −0.0327 | 0.062* | |
| C22 | 0.0852 (3) | 0.7291 (2) | −0.1571 (2) | 0.0645 (10) | |
| H22 | 0.0275 | 0.7706 | −0.1538 | 0.077* | |
| C23 | 0.0778 (3) | 0.6872 (3) | −0.2331 (2) | 0.0664 (10) | |
| H23 | 0.0146 | 0.6996 | −0.2812 | 0.080* | |
| C24 | 0.1633 (3) | 0.6271 (3) | −0.2385 (2) | 0.0805 (11) | |
| H24 | 0.1585 | 0.5987 | −0.2904 | 0.097* | |
| C25 | 0.3475 (2) | 0.56027 (17) | 0.04796 (17) | 0.0378 (6) | |
| C26 | 0.2372 (2) | 0.51519 (18) | 0.02465 (19) | 0.0444 (7) | |
| H26 | 0.1814 | 0.5290 | −0.0272 | 0.053* | |
| C27 | 0.2112 (3) | 0.45165 (19) | 0.0770 (2) | 0.0496 (7) | |
| C28 | 0.2941 (3) | 0.4282 (2) | 0.1531 (2) | 0.0590 (8) | |
| H28 | 0.2764 | 0.3841 | 0.1881 | 0.071* | |
| C29 | 0.4022 (3) | 0.4702 (2) | 0.17671 (19) | 0.0561 (8) | |
| H29 | 0.4578 | 0.4530 | 0.2275 | 0.067* | |
| C30 | 0.4322 (2) | 0.53880 (17) | 0.12656 (17) | 0.0390 (6) | |
| C31 | 0.2567 (3) | 0.6084 (2) | −0.1668 (2) | 0.0629 (9) | |
| H31 | 0.3147 | 0.5676 | −0.1708 | 0.076* | |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Ni1 | 0.03631 (19) | 0.0415 (2) | 0.03085 (19) | −0.00117 (15) | 0.00221 (14) | 0.00725 (16) |
| Cl1 | 0.0572 (5) | 0.0555 (5) | 0.0975 (7) | −0.0167 (4) | 0.0221 (5) | 0.0114 (5) |
| Cl2 | 0.0373 (4) | 0.0993 (7) | 0.0743 (6) | −0.0043 (4) | 0.0072 (4) | −0.0041 (5) |
| O1 | 0.0724 (15) | 0.0953 (18) | 0.0321 (11) | −0.0104 (13) | 0.0147 (11) | 0.0105 (11) |
| O2 | 0.0642 (14) | 0.0843 (16) | 0.0322 (11) | −0.0081 (12) | −0.0058 (10) | 0.0055 (11) |
| N1 | 0.0432 (13) | 0.0445 (13) | 0.0287 (12) | 0.0010 (10) | 0.0069 (10) | 0.0063 (10) |
| N2 | 0.0376 (12) | 0.0540 (15) | 0.0323 (12) | −0.0008 (10) | 0.0033 (10) | 0.0078 (11) |
| N3 | 0.0382 (12) | 0.0481 (14) | 0.0368 (13) | −0.0053 (10) | −0.0011 (10) | 0.0114 (11) |
| N4 | 0.0390 (13) | 0.0502 (14) | 0.0390 (13) | −0.0041 (11) | 0.0030 (10) | 0.0155 (11) |
| C1 | 0.0537 (17) | 0.0465 (17) | 0.0313 (15) | 0.0021 (13) | 0.0104 (13) | 0.0025 (13) |
| C2 | 0.0519 (17) | 0.0438 (17) | 0.0343 (16) | 0.0015 (13) | 0.0048 (14) | −0.0005 (13) |
| C3 | 0.0416 (16) | 0.0509 (18) | 0.0358 (15) | 0.0009 (13) | 0.0006 (12) | 0.0012 (13) |
| C4 | 0.0479 (18) | 0.070 (2) | 0.055 (2) | 0.0021 (16) | −0.0023 (15) | 0.0191 (17) |
| C5 | 0.0402 (17) | 0.082 (3) | 0.063 (2) | 0.0065 (17) | −0.0052 (15) | 0.0115 (19) |
| C6 | 0.0375 (16) | 0.069 (2) | 0.0510 (19) | −0.0060 (15) | 0.0039 (14) | −0.0086 (17) |
| C7 | 0.0419 (16) | 0.0561 (19) | 0.0430 (17) | −0.0045 (14) | 0.0029 (13) | 0.0002 (14) |
| C8 | 0.0364 (14) | 0.0480 (17) | 0.0350 (15) | −0.0025 (12) | 0.0013 (12) | −0.0032 (13) |
| C9 | 0.0406 (15) | 0.0449 (16) | 0.0349 (15) | −0.0071 (13) | 0.0047 (12) | −0.0016 (13) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C10 | 0.0326 (14) | 0.0478 (17) | 0.0374 (15) | −0.0080 (12) | −0.0006 (12) | 0.0057 (13) |
| C11 | 0.068 (2) | 0.057 (2) | 0.0495 (19) | −0.0166 (17) | 0.0106 (16) | −0.0070 (16) |
| C12 | 0.092 (3) | 0.045 (2) | 0.086 (3) | −0.0238 (19) | 0.018 (2) | −0.0063 (19) |
| C13 | 0.073 (2) | 0.064 (2) | 0.069 (2) | −0.0216 (18) | 0.014 (2) | 0.0187 (19) |
| C14 | 0.0483 (17) | 0.071 (2) | 0.0527 (19) | −0.0113 (16) | 0.0130 (15) | 0.0119 (17) |
| C15 | 0.0446 (16) | 0.0505 (18) | 0.0522 (18) | −0.0064 (14) | 0.0108 (14) | 0.0014 (15) |
| C16 | 0.0367 (19) | 0.046 (2) | 0.043 (2) | −0.0021 (16) | −0.0005 (16) | 0.0172 (19) |
| C17 | 0.071 (3) | 0.061 (3) | 0.058 (3) | 0.008 (2) | 0.005 (2) | 0.004 (2) |
| C18 | 0.0385 (18) | 0.056 (2) | 0.0381 (19) | −0.0064 (16) | −0.0029 (15) | 0.0164 (18) |
| C19 | 0.0352 (14) | 0.0389 (15) | 0.0378 (15) | 0.0003 (12) | 0.0075 (12) | 0.0039 (12) |
| C20 | 0.0344 (14) | 0.0401 (15) | 0.0403 (15) | −0.0041 (12) | 0.0052 (11) | 0.0093 (13) |
| C21 | 0.0471 (17) | 0.059 (2) | 0.0516 (19) | 0.0070 (15) | 0.0179 (15) | 0.0105 (15) |
| C22 | 0.0428 (17) | 0.075 (2) | 0.077 (3) | 0.0173 (16) | 0.0190 (17) | 0.034 (2) |
| C23 | 0.0477 (19) | 0.085 (3) | 0.055 (2) | −0.0020 (18) | −0.0049 (16) | 0.0267 (19) |
| C24 | 0.082 (3) | 0.087 (3) | 0.052 (2) | 0.010 (2) | −0.0128 (19) | −0.0062 (19) |
| C25 | 0.0396 (14) | 0.0337 (14) | 0.0409 (15) | 0.0003 (12) | 0.0128 (12) | 0.0033 (12) |
| C26 | 0.0424 (15) | 0.0389 (16) | 0.0501 (17) | −0.0016 (13) | 0.0105 (13) | 0.0081 (13) |
| C27 | 0.0468 (17) | 0.0426 (17) | 0.061 (2) | −0.0059 (13) | 0.0175 (15) | 0.0017 (15) |
| C28 | 0.068 (2) | 0.054 (2) | 0.057 (2) | −0.0100 (17) | 0.0218 (17) | 0.0136 (16) |
| C29 | 0.0602 (19) | 0.060 (2) | 0.0438 (18) | −0.0056 (16) | 0.0078 (15) | 0.0145 (15) |
| C30 | 0.0432 (15) | 0.0388 (15) | 0.0365 (15) | 0.0021 (12) | 0.0139 (12) | 0.0035 (12) |
| C31 | 0.066 (2) | 0.063 (2) | 0.052 (2) | 0.0161 (17) | 0.0025 (17) | −0.0050 (17) |

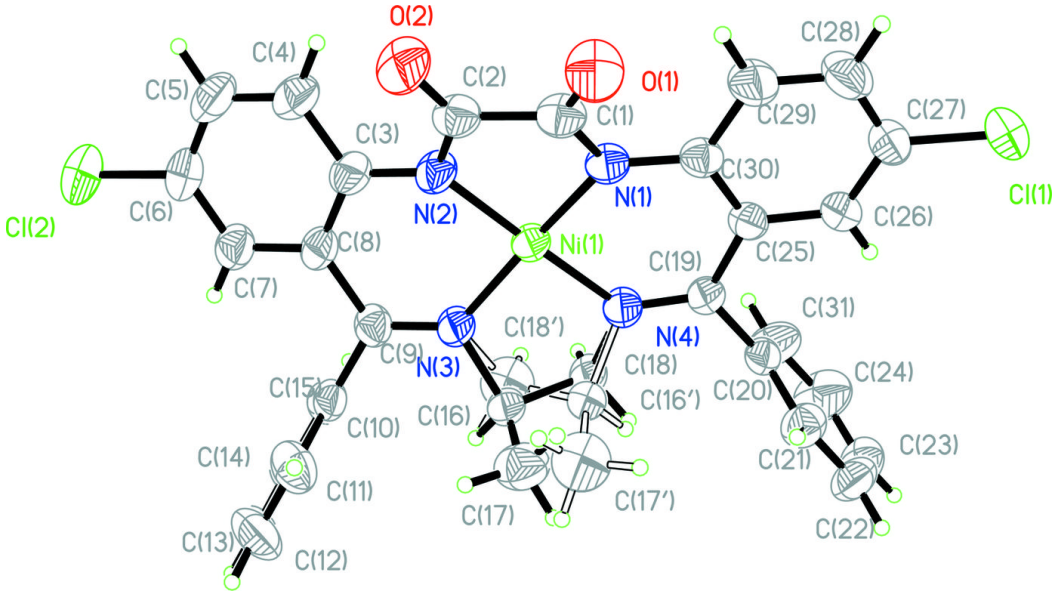
*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| Ni1—N2 | 1.857 (2) | C15—H15 | 0.9300 |
| Ni1—N1 | 1.867 (2) | C16—C18 | 1.502 (4) |
| Ni1—N4 | 1.870 (2) | C16—C17 | 1.504 (6) |
| Ni1—N3 | 1.874 (2) | C16—H16 | 0.9800 |
| Cl1—C27 | 1.740 (3) | C17—H17A | 0.9600 |
| Cl2—C6 | 1.753 (3) | C17—H17B | 0.9600 |
| O1—C1 | 1.218 (3) | C17—H17C | 0.9600 |
| O2—C2 | 1.226 (3) | C18—H18A | 0.9700 |
| N1—C1 | 1.373 (3) | C18—H18B | 0.9700 |
| N1—C30 | 1.394 (3) | C16'—C18' | 1.501 (17) |
| N2—C2 | 1.356 (3) | C16'—C17' | 1.510 (17) |
| N2—C3 | 1.392 (3) | C16'—H16' | 0.9800 |
| N3—C9 | 1.293 (3) | C17'—H17D | 0.9600 |
| N3—C16 | 1.508 (3) | C17'—H17E | 0.9600 |
| N3—C18' | 1.527 (17) | C17'—H17F | 0.9600 |
| N4—C19 | 1.297 (3) | C18'—H18C | 0.9700 |
| N4—C18 | 1.503 (4) | C18'—H18D | 0.9700 |
| N4—C16' | 1.553 (14) | C19—C25 | 1.466 (3) |
| C1—C2 | 1.518 (4) | C19—C20 | 1.500 (3) |
| C3—C4 | 1.403 (4) | C20—C31 | 1.376 (4) |
| C3—C8 | 1.408 (4) | C20—C21 | 1.377 (4) |
| C4—C5 | 1.373 (4) | C21—C22 | 1.387 (4) |
| C4—H4 | 0.9300 | C21—H21 | 0.9300 |
| C5—C6 | 1.377 (4) | C22—C23 | 1.363 (5) |

| | | | |
|---|---|---|---|
| C5—H5 | 0.9300 | C22—H22 | 0.9300 |
| C6—C7 | 1.360 (4) | C23—C24 | 1.366 (5) |
| C7—C8 | 1.409 (4) | C23—H23 | 0.9300 |
| C7—H7 | 0.9300 | C24—C31 | 1.380 (4) |
| C8—C9 | 1.475 (4) | C24—H24 | 0.9300 |
| C9—C10 | 1.499 (4) | C25—C26 | 1.411 (4) |
| C10—C11 | 1.382 (4) | C25—C30 | 1.412 (3) |
| C10—C15 | 1.393 (4) | C26—C27 | 1.364 (4) |
| C11—C12 | 1.383 (4) | C26—H26 | 0.9300 |
| C11—H11 | 0.9300 | C27—C28 | 1.381 (4) |
| C12—C13 | 1.373 (5) | C28—C29 | 1.368 (4) |
| C12—H12 | 0.9300 | C28—H28 | 0.9300 |
| C13—C14 | 1.363 (5) | C29—C30 | 1.413 (4) |
| C13—H13 | 0.9300 | C29—H29 | 0.9300 |
| C14—C15 | 1.372 (4) | C31—H31 | 0.9300 |
| C14—H14 | 0.9300 | | |
| N2—Ni1—N1 | 86.53 (9) | C17—C16—H16 | 109.4 |
| N2—Ni1—N4 | 176.84 (10) | N3—C16—H16 | 109.4 |
| N1—Ni1—N4 | 93.78 (9) | C16—C17—H17A | 109.5 |
| N2—Ni1—N3 | 92.76 (9) | C16—C17—H17B | 109.5 |
| N1—Ni1—N3 | 171.82 (10) | H17A—C17—H17B | 109.5 |
| N4—Ni1—N3 | 87.37 (9) | C16—C17—H17C | 109.5 |
| C1—N1—C30 | 120.7 (2) | H17A—C17—H17C | 109.5 |
| C1—N1—Ni1 | 110.65 (18) | H17B—C17—H17C | 109.5 |
| C30—N1—Ni1 | 127.86 (17) | C16—C18—N4 | 105.7 (3) |
| C2—N2—C3 | 120.5 (2) | C16—C18—H18A | 110.6 |
| C2—N2—Ni1 | 112.94 (18) | N4—C18—H18A | 110.6 |
| C3—N2—Ni1 | 126.44 (18) | C16—C18—H18B | 110.6 |
| C9—N3—C16 | 119.9 (2) | N4—C18—H18B | 110.6 |
| C9—N3—C18' | 117.1 (9) | H18A—C18—H18B | 108.7 |
| C9—N3—Ni1 | 130.16 (19) | C18'—C16'—C17' | 119 (2) |
| C16—N3—Ni1 | 109.55 (18) | C18'—C16'—N4 | 101.0 (15) |
| C18'—N3—Ni1 | 109.6 (11) | C17'—C16'—N4 | 119.5 (16) |
| C19—N4—C18 | 118.9 (2) | C18'—C16'—H16' | 105.5 |
| C19—N4—C16' | 119.2 (7) | C17'—C16'—H16' | 105.5 |
| C19—N4—Ni1 | 129.77 (19) | N4—C16'—H16' | 105.5 |
| C18—N4—Ni1 | 111.12 (17) | C16'—C17'—H17D | 109.5 |
| C16'—N4—Ni1 | 104.6 (7) | C16'—C17'—H17E | 109.5 |
| O1—C1—N1 | 128.0 (3) | H17D—C17'—H17E | 109.5 |
| O1—C1—C2 | 119.6 (3) | C16'—C17'—H17F | 109.5 |
| N1—C1—C2 | 112.4 (2) | H17D—C17'—H17F | 109.5 |
| O2—C2—N2 | 128.8 (3) | H17E—C17'—H17F | 109.5 |
| O2—C2—C1 | 119.3 (3) | C16'—C18'—N3 | 100.4 (14) |
| N2—C2—C1 | 111.9 (2) | C16'—C18'—H18C | 111.7 |
| N2—C3—C4 | 120.9 (3) | N3—C18'—H18C | 111.7 |
| N2—C3—C8 | 121.1 (2) | C16'—C18'—H18D | 111.7 |
| C4—C3—C8 | 117.9 (3) | N3—C18'—H18D | 111.7 |
| C5—C4—C3 | 121.9 (3) | H18C—C18'—H18D | 109.5 |
| C5—C4—H4 | 119.1 | N4—C19—C25 | 122.7 (2) |

| | | | |
|---|---|---|---|
| C3—C4—H4 | 119.1 | N4—C19—C20 | 119.4 (2) |
| C4—C5—C6 | 119.3 (3) | C25—C19—C20 | 117.9 (2) |
| C4—C5—H5 | 120.3 | C31—C20—C21 | 119.0 (3) |
| C6—C5—H5 | 120.3 | C31—C20—C19 | 120.5 (2) |
| C7—C6—C5 | 121.0 (3) | C21—C20—C19 | 120.5 (3) |
| C7—C6—Cl2 | 120.2 (3) | C20—C21—C22 | 119.9 (3) |
| C5—C6—Cl2 | 118.8 (2) | C20—C21—H21 | 120.0 |
| C6—C7—C8 | 120.6 (3) | C22—C21—H21 | 120.0 |
| C6—C7—H7 | 119.7 | C23—C22—C21 | 120.4 (3) |
| C8—C7—H7 | 119.7 | C23—C22—H22 | 119.8 |
| C3—C8—C7 | 119.1 (2) | C21—C22—H22 | 119.8 |
| C3—C8—C9 | 123.1 (2) | C22—C23—C24 | 120.0 (3) |
| C7—C8—C9 | 117.8 (2) | C22—C23—H23 | 120.0 |
| N3—C9—C8 | 121.8 (2) | C24—C23—H23 | 120.0 |
| N3—C9—C10 | 120.4 (2) | C23—C24—C31 | 119.9 (3) |
| C8—C9—C10 | 117.8 (2) | C23—C24—H24 | 120.1 |
| C11—C10—C15 | 119.5 (3) | C31—C24—H24 | 120.1 |
| C11—C10—C9 | 120.6 (3) | C26—C25—C30 | 119.3 (2) |
| C15—C10—C9 | 119.9 (3) | C26—C25—C19 | 116.9 (2) |
| C10—C11—C12 | 119.2 (3) | C30—C25—C19 | 123.8 (2) |
| C10—C11—H11 | 120.4 | C27—C26—C25 | 120.9 (3) |
| C12—C11—H11 | 120.4 | C27—C26—H26 | 119.5 |
| C13—C12—C11 | 120.8 (3) | C25—C26—H26 | 119.5 |
| C13—C12—H12 | 119.6 | C26—C27—C28 | 120.5 (3) |
| C11—C12—H12 | 119.6 | C26—C27—Cl1 | 120.4 (2) |
| C14—C13—C12 | 120.0 (3) | C28—C27—Cl1 | 119.0 (2) |
| C14—C13—H13 | 120.0 | C29—C28—C27 | 119.6 (3) |
| C12—C13—H13 | 120.0 | C29—C28—H28 | 120.2 |
| C13—C14—C15 | 120.3 (3) | C27—C28—H28 | 120.2 |
| C13—C14—H14 | 119.9 | C28—C29—C30 | 122.2 (3) |
| C15—C14—H14 | 119.9 | C28—C29—H29 | 118.9 |
| C14—C15—C10 | 120.2 (3) | C30—C29—H29 | 118.9 |
| C14—C15—H15 | 119.9 | N1—C30—C25 | 121.5 (2) |
| C10—C15—H15 | 119.9 | N1—C30—C29 | 121.2 (2) |
| C18—C16—C17 | 111.2 (3) | C25—C30—C29 | 117.3 (2) |
| C18—C16—N3 | 105.0 (3) | C20—C31—C24 | 120.8 (3) |
| C17—C16—N3 | 112.3 (3) | C20—C31—H31 | 119.6 |
| C18—C16—H16 | 109.4 | C24—C31—H31 | 119.6 |

**Fig. 1**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