17449 measured reflections

 $R_{\rm int} = 0.090$

4874 independent reflections 2682 reflections with $I > 2\sigma(I)$

Acta Crystallographica Section E **Structure Reports** Online

ISSN 1600-5368

{4,4',6,6'-Tetrachloro-2,2'-[2,2-dimethylpropane-1,3-divlbis(nitrilomethanylylidene)]{nickel(II)

Hadi Kargar,^a* Reza Kia,^{b,c} Saeideh Abbasian^a and Muhammad Nawaz Tahir^d*

^aDepartment of Chemistry, Payame Noor University, PO BOX 19395-3697 Tehran, I. R. of IRAN, ^bX-ray Crystallography Lab., Plasma Physics Research Center, Science and Research Branch, Islamic Azad University, Tehran, Iran, ^cDepartment of Chemistry, Science and Research Branch, Islamic Azad University, Tehran, Iran, and ^dDepartment of Physics, University of Sargodha, Punjab, Pakistan Correspondence e-mail: hkargar@pnu.ac.ir, dmntahir_uos@yahoo.com

Received 8 January 2012; accepted 17 January 2012

Key indicators: single-crystal X-ray study; T = 291 K; mean σ (C–C) = 0.006 Å; R factor = 0.065; wR factor = 0.127; data-to-parameter ratio = 19.1.

In the title compound, $[Ni(C_{19}H_{16}Cl_4N_2O_2)]$, the Ni^{II} ion is in a distorted square-planar environment coordinated by two N atoms and two O atoms of the tetradentate ligand. The dihedral angle between the benzene rings is 24.8 (2)°. In the crystal, molecules are linked into chains along the b axis by weak C-H···O and C-H···Cl interactions. An intermolecular Cl···Cl [3.4564 (19) Å] interaction is present which is shorter than the sum of the van der Waals radii of Cl atoms (3.50 Å).

Related literature

For applications of Schiff bases in coordination chemistry, see: Granovski et al. (1993); Blower et al. (1998). For related structures see: Ghaemi et al. (2011); Kargar et al. (2011, 2012). For standard bond lengths, see: Allen et al. (1987).



Experimental

Crystal data

| $[Ni(C_{19}H_{16}Cl_4N_2O_2)]$ | V = 2057.0 (2) Å ³ |
|--------------------------------|---|
| $M_r = 504.85$ | Z = 4 |
| Monoclinic, $P2_1/n$ | Mo Ka radiation |
| a = 12.4019 (8) Å | $\mu = 1.48 \text{ mm}^{-1}$ |
| b = 8.1883 (6) Å | $T = 291 { m K}$ |
| c = 20.3945 (13) Å | $0.25 \times 0.18 \times 0.09 \text{ mm}$ |
| $\beta = 96.680 \ (3)^{\circ}$ | |

Data collection

Bruker SMART APEXII CCD area-detector diffractometer Absorption correction: multi-scan (SADABS; Bruker, 2005) $T_{\min} = 0.694, \ T_{\max} = 0.871$

Refinement

| $R[F^2 > 2\sigma(F^2)] = 0.065$ | 255 parameters |
|---------------------------------|--|
| $wR(F^2) = 0.127$ | H-atom parameters constrained |
| S = 1.05 | $\Delta \rho_{\rm max} = 0.49 \ {\rm e} \ {\rm \AA}^{-3}$ |
| 4874 reflections | $\Delta \rho_{\rm min} = -0.41 \text{ e } \text{\AA}^{-3}$ |

Table 1

Hydrogen-bond geometry (Å, °).

 $D - H \cdot \cdot \cdot A$ D-H $H \cdots A$ $D \cdot \cdot \cdot A$ $D - H \cdot \cdot \cdot A$ $C8 - H8A \cdots O1^{i}$ 0.97 2.52 3.269 (5) 134 C12-H12A···Cl4ⁱⁱ 0.97 2.80 3.578 (5) 138

Symmetry codes: (i) -x + 1, -y + 1, -z; (ii) -x + 1, -y, -z.

Data collection: APEX2 (Bruker, 2005); cell refinement: SAINT (Bruker, 2005); 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 and PLATON (Spek, 2009).

HK and SA thank PNU for the financial support. MNT thanks GC University of Sargodha, Pakistan for the research facility.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5404).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). J. Chem. Soc. Perkin Trans. 2, pp. S1-19.

Blower, P. J. (1998). Transition Met. Chem., 23, 109-112.

Bruker (2005). APEX2, SAINT and SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.

Ghaemi, A., Rayati, S., Elahi, E., Ng, S. W. & Tiekink, E. R. T. (2011). Acta Crvst. E67. m1445-m1446.

Granovski, A. D., Nivorozhkin, A. L. & Minkin, V. I. (1993). Coord. Chem. Rev., 126, 1-69.

Kargar, H., Kia, R., Pahlavani, E. & Tahir, M. N. (2011). Acta Cryst. E67, m941. Kargar, H., Kia, R., Sharafi, Z. & Tahir, M. N. (2012). Acta Cryst. E68, m82. Sheldrick, G. M. (2008). Acta Cryst. A64, 112-122.

Spek, A. L. (2009). Acta Cryst. D65, 148-155.

Acta Cryst. (2012). E68, m193 [doi:10.1107/S1600536812002085]

{4,4',6,6'-Tetrachloro-2,2'-[2,2-dimethylpropane-1,3-diylbis(nitrilomethanylylidene)]}nickel(II)

H. Kargar, R. Kia, S. Abbasian and M. N. Tahir

Comment

Schiff base complexes are one of the most important stereochemical models in transition metal coordination chemistry, with ease of preparation and structural variation (Granovski *et al.*, 1993; Blower *et al.*, (1998). In continuation of our work on the crystal structure of Schiff base metal complexes (Kargar *et al.*, 2012; Kargar *et al.*, 2011; Ghaemi, *et al.*, (2011), we have determined the X-ray structure of the title compound.

The asymmetric unit of the title compound, Fig. 1, comprises a Schiff base complex. The bond lengths (Allen *et al.*, 1987) and angles are within the normal ranges and are comparable to the related structure (Kargar *et al.*, 2012; Kargar *et al.*, 2011; Ghaemi, *et al.*, (2011).

The geometry around the Ni^{II} ion is a distorted square-plane which is supported by the N_2O_2 donor atoms of the coordinated Schiff base ligand. The dihedral angle between the substituted benzene rings is 24.8 (2)°.

In the crystal, molecules are linked along the *b*-axis, forming one-dimensional extended chains via intermolecular C—H···O and C—H···Cl interactions (Table 1, Fig. 2). In addition, Cl1···Cl2ⁱⁱⁱ [3.4564 (19)Å; (iii) 1/2 - x, -1/2 + y, -1/2 - z] interactions are present in the crystal structure which are shorter than the sum of the van der Waals radii of Cl atoms [3.50Å]. These also link neighboring molecules along the *b*-axis (Fig. 3).

Experimental

The title compound was synthesized by adding 3,5-dichloro-salicylaldehyde-2,2-dimethyl-1, 3-propanediamine (2 mmol) to a solution of NiCl₂. $6H_2O$ (2.1 mmol) in ethanol (30 ml). The mixture was refluxed with stirring for half an hour. The resultant solution was filtered. Dark-green single crystals of the title compound suitable for *X*-ray structure determination were recrystallized from ethanol by slow evaporation of the solvents at room temperature over several days.

Refinement

The H-atoms were included in calculated positions and treated as riding atoms: C—H = 0.93, 0.96 and 0.97 Å for CH, CH₃ and CH₂ H-atoms, respectively, with U_{iso} (H) = k x U_{eq} (C), where k = 1.5 for CH₃ H-atoms, and k = 1.2 for all other H-atoms.

Figures



Fig. 1. The molecular structure of the title compound, showing 40% probability displacement ellipsoids.



Fig. 2. Part of the packing of the title compound showing molecules linked through intermolecular C—H···O and C—H···Cl intearctions (dashed lines). Only the H atoms involved in the interactions are shown.



Fig. 3. Part of the packing of the title compound viewed along the a -axis, showing molecules linked along the b-axis by intermolecular Cl···Cl intearctions(dashed lines).

{4,4',6,6'-Tetrachloro-2,2'-[2,2-dimethylpropane-1,3- diylbis(nitrilomethanylylidene)]}nickel(II)

| Crystal data | |
|--|---|
| [Ni(C ₁₉ H ₁₆ Cl ₄ N ₂ O ₂)] | F(000) = 1024 |
| $M_r = 504.85$ | $D_{\rm x} = 1.630 {\rm ~Mg~m}^{-3}$ |
| Monoclinic, $P2_1/n$ | Mo <i>K</i> α radiation, $\lambda = 0.71073$ Å |
| Hall symbol: -P 2yn | Cell parameters from 2540 reflections |
| <i>a</i> = 12.4019 (8) Å | $\theta = 2.5 - 27.4^{\circ}$ |
| <i>b</i> = 8.1883 (6) Å | $\mu = 1.48 \text{ mm}^{-1}$ |
| c = 20.3945 (13) Å | T = 291 K |
| $\beta = 96.680 \ (3)^{\circ}$ | Block, dark-red |
| $V = 2057.0 (2) \text{ Å}^3$ | $0.25\times0.18\times0.09~mm$ |
| Z = 4 | |

Data collection

| Bruker SMART APEXII CCD area-detector diffractometer | 4874 independent reflections |
|--|---|
| Radiation source: fine-focus sealed tube | 2682 reflections with $I > 2\sigma(I)$ |
| graphite | $R_{\rm int} = 0.090$ |
| φ and ω scans | $\theta_{\text{max}} = 27.9^\circ, \ \theta_{\text{min}} = 1.8^\circ$ |
| Absorption correction: multi-scan (<i>SADABS</i> ; Bruker, 2005) | $h = -16 \rightarrow 15$ |
| $T_{\min} = 0.694, \ T_{\max} = 0.871$ | $k = -10 \rightarrow 7$ |
| 17449 measured reflections | <i>l</i> = −26→26 |

Refinement

| Refinement on F^2 | Primary atom site location: structure-invariant direct methods |
|---------------------------------|--|
| Least-squares matrix: full | Secondary atom site location: difference Fourier map |
| $R[F^2 > 2\sigma(F^2)] = 0.065$ | Hydrogen site location: inferred from neighbouring sites |

| $wR(F^2) = 0.127$ | H-atom parameters constrained |
|-------------------|---|
| <i>S</i> = 1.05 | $w = 1/[\sigma^2(F_o^2) + (0.0401P)^2 + 1.6535P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| 4874 reflections | $(\Delta/\sigma)_{max} < 0.001$ |
| 255 parameters | $\Delta \rho_{max} = 0.49 \text{ e} \text{ Å}^{-3}$ |
| 0 restraints | $\Delta \rho_{min} = -0.41 \text{ e } \text{\AA}^{-3}$ |

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 $F^2 > 2 \text{sigma}(F^2)$ 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 (A^2)

| | x | У | Z | $U_{\rm iso}$ */ $U_{\rm eq}$ |
|------|------------|------------|-------------|-------------------------------|
| C1 | 0.4518 (3) | 0.3786 (5) | -0.1322 (2) | 0.0284 (10) |
| C2 | 0.3696 (3) | 0.3864 (6) | -0.1865 (2) | 0.0329 (11) |
| C3 | 0.3866 (4) | 0.4565 (6) | -0.2451 (2) | 0.0396 (12) |
| Н3 | 0.3311 | 0.4580 | -0.2799 | 0.047* |
| C4 | 0.4868 (4) | 0.5257 (6) | -0.2527 (2) | 0.0390 (12) |
| C5 | 0.5684 (4) | 0.5219 (6) | -0.2020 (2) | 0.0371 (12) |
| Н5 | 0.6350 | 0.5693 | -0.2073 | 0.044* |
| C6 | 0.5530 (3) | 0.4470 (6) | -0.1417 (2) | 0.0322 (11) |
| C7 | 0.6396 (3) | 0.4527 (5) | -0.0889 (2) | 0.0331 (11) |
| H7 | 0.7009 | 0.5123 | -0.0961 | 0.040* |
| C8 | 0.7307 (3) | 0.4234 (6) | 0.0187 (2) | 0.0371 (12) |
| H8A | 0.7009 | 0.4640 | 0.0574 | 0.044* |
| H8B | 0.7742 | 0.5098 | 0.0025 | 0.044* |
| C9 | 0.8032 (3) | 0.2782 (5) | 0.0382 (2) | 0.0338 (11) |
| C10 | 0.8628 (5) | 0.3121 (7) | 0.1068 (3) | 0.0632 (17) |
| H10A | 0.8112 | 0.3181 | 0.1383 | 0.095* |
| H10B | 0.9011 | 0.4138 | 0.1062 | 0.095* |
| H10C | 0.9135 | 0.2256 | 0.1189 | 0.095* |
| C11 | 0.8856 (4) | 0.2535 (7) | -0.0109 (3) | 0.0626 (16) |
| H11A | 0.9338 | 0.3455 | -0.0091 | 0.094* |
| H11B | 0.8481 | 0.2436 | -0.0546 | 0.094* |
| H11C | 0.9266 | 0.1559 | 0.0002 | 0.094* |
| C12 | 0.7341 (3) | 0.1245 (6) | 0.0386 (2) | 0.0382 (12) |
| H12A | 0.7265 | 0.0769 | -0.0052 | 0.046* |
| H12B | 0.7717 | 0.0459 | 0.0687 | 0.046* |
| | | | | |

| C13 | 0.6000 (4) | 0.0806 (6) | 0.1105 (2) | 0.0356 (11) |
|-----|--------------|--------------|---------------|--------------|
| H13 | 0.6551 | 0.0239 | 0.1356 | 0.043* |
| C14 | 0.4951 (3) | 0.0785 (6) | 0.1330 (2) | 0.0320 (11) |
| C15 | 0.4848 (4) | 0.0125 (6) | 0.1952 (2) | 0.0442 (13) |
| H15 | 0.5461 | -0.0254 | 0.2213 | 0.053* |
| C16 | 0.3867 (4) | 0.0033 (6) | 0.2179 (2) | 0.0447 (13) |
| C17 | 0.2938 (4) | 0.0556 (6) | 0.1791 (2) | 0.0409 (12) |
| H17 | 0.2265 | 0.0493 | 0.1949 | 0.049* |
| C18 | 0.3019 (3) | 0.1168 (6) | 0.1174 (2) | 0.0325 (11) |
| C19 | 0.4025 (3) | 0.1323 (5) | 0.0910 (2) | 0.0310 (11) |
| Cl1 | 0.24344 (10) | 0.30295 (16) | -0.17810 (7) | 0.0511 (4) |
| Cl2 | 0.50613 (11) | 0.6188 (2) | -0.32739 (6) | 0.0619 (4) |
| C13 | 0.37569 (12) | -0.0781 (2) | 0.29595 (7) | 0.0770 (5) |
| Cl4 | 0.18413 (9) | 0.18304 (15) | 0.07001 (6) | 0.0390 (3) |
| N1 | 0.6402 (3) | 0.3834 (5) | -0.03285 (17) | 0.0314 (9) |
| N2 | 0.6250 (3) | 0.1539 (5) | 0.05842 (18) | 0.0330 (9) |
| Ni1 | 0.52748 (4) | 0.26003 (7) | -0.00440 (3) | 0.02984 (17) |
| 01 | 0.4305 (2) | 0.3113 (4) | -0.07708 (14) | 0.0357 (8) |
| O2 | 0.4051 (2) | 0.1881 (4) | 0.03171 (14) | 0.0342 (7) |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|------------|--------------|-------------|--------------|
| C1 | 0.022 (2) | 0.025 (3) | 0.039 (2) | 0.0036 (19) | 0.0069 (19) | -0.005 (2) |
| C2 | 0.031 (2) | 0.027 (3) | 0.041 (3) | 0.001 (2) | 0.002 (2) | -0.003 (2) |
| C3 | 0.035 (3) | 0.038 (3) | 0.043 (3) | 0.004 (2) | -0.002 (2) | -0.006 (2) |
| C4 | 0.043 (3) | 0.036 (3) | 0.039 (3) | 0.010 (2) | 0.006 (2) | 0.000 (2) |
| C5 | 0.027 (2) | 0.045 (3) | 0.041 (3) | -0.003 (2) | 0.011 (2) | 0.000 (2) |
| C6 | 0.030 (2) | 0.029 (3) | 0.039 (3) | 0.001 (2) | 0.0066 (19) | -0.002 (2) |
| C7 | 0.028 (2) | 0.027 (3) | 0.044 (3) | -0.005 (2) | 0.005 (2) | -0.004 (2) |
| C8 | 0.031 (2) | 0.036 (3) | 0.044 (3) | -0.008 (2) | -0.001 (2) | -0.005 (2) |
| С9 | 0.028 (2) | 0.027 (3) | 0.046 (3) | 0.000 (2) | 0.003 (2) | -0.006 (2) |
| C10 | 0.061 (4) | 0.053 (4) | 0.069 (4) | 0.001 (3) | -0.024 (3) | -0.003 (3) |
| C11 | 0.041 (3) | 0.061 (4) | 0.091 (4) | -0.005 (3) | 0.025 (3) | -0.001 (3) |
| C12 | 0.031 (3) | 0.031 (3) | 0.054 (3) | 0.005 (2) | 0.009 (2) | -0.008 (2) |
| C13 | 0.030 (2) | 0.030 (3) | 0.046 (3) | 0.001 (2) | -0.001 (2) | 0.003 (2) |
| C14 | 0.027 (2) | 0.028 (3) | 0.041 (3) | 0.001 (2) | 0.004 (2) | -0.001 (2) |
| C15 | 0.035 (3) | 0.043 (4) | 0.053 (3) | -0.001 (2) | 0.001 (2) | 0.014 (3) |
| C16 | 0.041 (3) | 0.052 (4) | 0.042 (3) | 0.000 (2) | 0.007 (2) | 0.013 (2) |
| C17 | 0.034 (3) | 0.040 (3) | 0.051 (3) | -0.008 (2) | 0.015 (2) | 0.003 (2) |
| C18 | 0.032 (2) | 0.026 (3) | 0.040 (3) | 0.001 (2) | 0.006 (2) | -0.001 (2) |
| C19 | 0.028 (2) | 0.021 (3) | 0.044 (3) | -0.0025 (19) | 0.005 (2) | -0.005 (2) |
| Cl1 | 0.0323 (6) | 0.0514 (9) | 0.0667 (8) | -0.0087 (6) | -0.0056 (6) | 0.0055 (7) |
| Cl2 | 0.0578 (9) | 0.0838 (12) | 0.0461 (8) | 0.0107 (8) | 0.0139 (6) | 0.0175 (7) |
| Cl3 | 0.0539 (9) | 0.1181 (15) | 0.0603 (9) | 0.0003 (9) | 0.0124 (7) | 0.0444 (9) |
| Cl4 | 0.0269 (6) | 0.0416 (8) | 0.0489 (7) | 0.0010 (5) | 0.0061 (5) | 0.0017 (6) |
| N1 | 0.0261 (19) | 0.029 (2) | 0.039 (2) | -0.0021 (16) | 0.0024 (16) | -0.0054 (18) |
| N2 | 0.0257 (19) | 0.028 (2) | 0.046 (2) | 0.0007 (17) | 0.0076 (17) | -0.0085 (19) |

| Ni1 | 0.0238 (3) | 0.0288 (4) | 0.0374 (3) | -0.0009 (3) | 0.0056 (2) | -0.0023 (3) |
|----------------|---------------|------------|-------------|--------------|-------------|-------------|
| 01 | 0.0227 (15) | 0.041 (2) | 0.0445 (18) | -0.0029 (14) | 0.0090 (13) | 0.0015 (15) |
| 02 | 0.0277 (16) | 0.040 (2) | 0.0354 (17) | -0.0030 (14) | 0.0052 (13) | 0.0016 (14) |
| Geometric para | meters (Å, °) | | | | | |
| C101 | | 1.306 (5) | C11– | -H11A | 0.90 | 500 |
| C1—C6 | | 1.408 (6) | C11– | -H11B | 0.90 | 500 |
| C1—C2 | | 1.417 (5) | C11- | -H11C | 0.90 | 500 |
| C2—C3 | | 1.363 (6) | C12— | -N2 | 1.4 | 77 (5) |
| C2—Cl1 | | 1.734 (5) | C12- | -H12A | 0.97 | 700 |
| C3—C4 | | 1.392 (6) | C12- | -H12B | 0.97 | 700 |
| С3—Н3 | | 0.9300 | C13— | -N2 | 1.28 | 89 (5) |
| C4—C5 | | 1.360 (6) | C13— | -C14 | 1.42 | 29 (6) |
| C4—Cl2 | | 1.744 (5) | C13— | -H13 | 0.93 | 300 |
| С5—С6 | | 1.408 (6) | C14— | -C15 | 1.39 | 97 (6) |
| С5—Н5 | | 0.9300 | C14— | -C19 | 1.42 | 21 (6) |
| С6—С7 | | 1.430 (6) | C15— | -C16 | 1.3 | 54 (7) |
| C7—N1 | | 1.276 (5) | C15— | -H15 | 0.92 | 300 |
| С7—Н7 | | 0.9300 | C16— | -C17 | 1.38 | 87 (6) |
| C8—N1 | | 1.482 (5) | C16- | -Cl3 | 1.74 | 46 (5) |
| С8—С9 | | 1.516 (6) | C17— | -C18 | 1.30 | 69 (6) |
| C8—H8A | | 0.9700 | C17— | -H17 | 0.93 | 300 |
| C8—H8B | | 0.9700 | C18- | -C19 | 1.42 | 21 (6) |
| C9—C12 | | 1.523 (6) | C18— | -Cl4 | 1.74 | 42 (4) |
| C9—C11 | | 1.524 (7) | C19– | -02 | 1.29 | 97 (5) |
| C9—C10 | | 1.529 (6) | N1—1 | Ni1 | 1.87 | 71 (4) |
| C10—H10A | | 0.9600 | N2—1 | Ni1 | 1.87 | 70 (4) |
| C10—H10B | | 0.9600 | Ni1— | -01 | 1.84 | 46 (3) |
| C10—H10C | | 0.9600 | Ni1— | -02 | 1.83 | 58 (3) |
| O1—C1—C6 | | 124.1 (4) | H11A | —С11—Н11С | 109 | .5 |
| O1—C1—C2 | | 119.5 (4) | H11B | —C11—H11C | 109 | .5 |
| C6—C1—C2 | | 116.5 (4) | N2 | С12—С9 | 113 | .6 (4) |
| C3—C2—C1 | | 122.3 (4) | N2— | C12—H12A | 108 | .8 |
| C3—C2—Cl1 | | 119.0 (3) | С9—(| C12—H12A | 108 | .8 |
| C1—C2—Cl1 | | 118.7 (3) | N2— | C12—H12B | 108 | .8 |
| C2—C3—C4 | | 120.0 (4) | С9—(| C12—H12B | 108 | .8 |
| С2—С3—Н3 | | 120.0 | H12A | | 107 | .7 |
| С4—С3—Н3 | | 120.0 | N2—4 | C13—C14 | 125 | .9 (4) |
| C5—C4—C3 | | 120.1 (4) | N2 | С13—Н13 | 117 | .1 |
| C5—C4—Cl2 | | 120.5 (4) | C14— | -C13—H13 | 117 | .1 |
| C3—C4—Cl2 | | 119.4 (4) | C15- | -C14—C19 | 120 | .9 (4) |
| C4—C5—C6 | | 120.6 (4) | C15— | -C14—C13 | 118 | .7 (4) |
| C4—C5—H5 | | 119.7 | C19– | -C14—C13 | 120 | .3 (4) |
| С6—С5—Н5 | | 119.7 | C16- | -C15—C14 | 120 | .8 (4) |
| C5—C6—C1 | | 120.5 (4) | C16- | -C15—H15 | 119 | .6 |
| C5—C6—C7 | | 118.5 (4) | C14— | -C15—H15 | 119 | .6 |
| C1—C6—C7 | | 120.8 (4) | C15— | -C16—C17 | 120 | .5 (5) |
| N1—C7—C6 | | 125.9 (4) | C15- | -C16—Cl3 | 120 | .1 (4) |

| N1—C7—H7 | 117.1 | C17—C16—Cl3 | 119.3 (4) |
|----------------|------------|-----------------|-------------|
| С6—С7—Н7 | 117.1 | C18—C17—C16 | 119.5 (4) |
| N1—C8—C9 | 113.0 (4) | С18—С17—Н17 | 120.3 |
| N1—C8—H8A | 109.0 | С16—С17—Н17 | 120.3 |
| С9—С8—Н8А | 109.0 | C17—C18—C19 | 122.9 (4) |
| N1—C8—H8B | 109.0 | C17—C18—Cl4 | 118.5 (4) |
| С9—С8—Н8В | 109.0 | C19—C18—Cl4 | 118.6 (3) |
| H8A—C8—H8B | 107.8 | O2—C19—C18 | 120.2 (4) |
| C8—C9—C12 | 109.4 (4) | O2—C19—C14 | 124.5 (4) |
| C8—C9—C11 | 110.7 (4) | C18—C19—C14 | 115.3 (4) |
| C12—C9—C11 | 108.3 (4) | C7—N1—C8 | 117.6 (4) |
| C8—C9—C10 | 107.9 (4) | C7—N1—Ni1 | 126.2 (3) |
| C12—C9—C10 | 110.9 (4) | C8—N1—Ni1 | 115.5 (3) |
| C11—C9—C10 | 109.6 (4) | C13—N2—C12 | 117.8 (4) |
| C9—C10—H10A | 109.5 | C13—N2—Ni1 | 125.6 (3) |
| C9—C10—H10B | 109.5 | C12—N2—Ni1 | 115.4 (3) |
| H10A—C10—H10B | 109.5 | O1—Ni1—O2 | 84.50 (12) |
| С9—С10—Н10С | 109.5 | O1—Ni1—N2 | 164.78 (14) |
| H10A—C10—H10C | 109.5 | O2—Ni1—N2 | 94.25 (15) |
| H10B-C10-H10C | 109.5 | O1—Ni1—N1 | 93.97 (14) |
| C9—C11—H11A | 109.5 | O2—Ni1—N1 | 165.55 (14) |
| C9—C11—H11B | 109.5 | N2—Ni1—N1 | 90.93 (16) |
| H11A—C11—H11B | 109.5 | C1—O1—Ni1 | 127.5 (3) |
| C9—C11—H11C | 109.5 | C19—O2—Ni1 | 126.2 (3) |
| O1—C1—C2—C3 | 179.3 (4) | C17—C18—C19—C14 | 0.5 (7) |
| C6—C1—C2—C3 | -0.2 (7) | Cl4—C18—C19—C14 | 178.9 (3) |
| O1—C1—C2—Cl1 | -0.9 (6) | C15—C14—C19—O2 | -176.9 (4) |
| C6—C1—C2—Cl1 | 179.6 (3) | C13—C14—C19—O2 | -1.2 (7) |
| C1—C2—C3—C4 | -1.0(7) | C15-C14-C19-C18 | 1.2 (6) |
| Cl1—C2—C3—C4 | 179.3 (4) | C13—C14—C19—C18 | 176.8 (4) |
| C2—C3—C4—C5 | 0.7 (7) | C6—C7—N1—C8 | -171.3 (4) |
| C2—C3—C4—Cl2 | -178.6 (4) | C6—C7—N1—Ni1 | -1.3 (7) |
| C3—C4—C5—C6 | 0.6 (7) | C9—C8—N1—C7 | -115.1 (5) |
| Cl2—C4—C5—C6 | 179.9 (4) | C9—C8—N1—Ni1 | 73.8 (4) |
| C4—C5—C6—C1 | -1.7 (7) | C14—C13—N2—C12 | -172.4 (4) |
| C4—C5—C6—C7 | -177.2 (4) | C14—C13—N2—Ni1 | -5.8 (7) |
| O1—C1—C6—C5 | -178.0 (4) | C9—C12—N2—C13 | -118.9 (4) |
| C2—C1—C6—C5 | 1.5 (6) | C9—C12—N2—Ni1 | 73.2 (4) |
| O1—C1—C6—C7 | -2.6 (7) | C13—N2—Ni1—O1 | -92.3 (7) |
| C2—C1—C6—C7 | 176.8 (4) | C12—N2—Ni1—O1 | 74.5 (7) |
| C5—C6—C7—N1 | -176.0 (4) | C13—N2—Ni1—O2 | -7.7 (4) |
| C1—C6—C7—N1 | 8.6 (7) | C12—N2—Ni1—O2 | 159.2 (3) |
| N1—C8—C9—C12 | -37.5 (5) | C13—N2—Ni1—N1 | 158.8 (4) |
| N1-C8-C9-C11 | 81.8 (5) | C12—N2—Ni1—N1 | -34.4 (3) |
| N1—C8—C9—C10 | -158.3 (4) | C7—N1—Ni1—O1 | -7.7 (4) |
| C8—C9—C12—N2 | -33.1 (5) | C8—N1—Ni1—O1 | 162.5 (3) |
| C11—C9—C12—N2 | -153.9 (4) | C7—N1—Ni1—O2 | -91.0 (7) |
| C10—C9—C12—N2 | 85.8 (5) | C8—N1—Ni1—O2 | 79.2 (6) |
| N2-C13-C14-C15 | -171.2 (5) | C7—N1—Ni1—N2 | 157.9 (4) |

| | | ~ | |
|-----------------|------------|----------------|------------|
| N2—C13—C14—C19 | 13.0 (7) | C8-N1-N1-N2 | -31.9 (3) |
| C19—C14—C15—C16 | -2.2 (7) | C6-C1-O1-Ni1 | -10.1 (6) |
| C13-C14-C15-C16 | -177.9 (5) | C2-C1-O1-Ni1 | 170.4 (3) |
| C14-C15-C16-C17 | 1.5 (8) | O2-Ni1-O1-C1 | 178.9 (4) |
| C14—C15—C16—Cl3 | -179.5 (4) | N2-Ni1-O1-C1 | -95.1 (7) |
| C15-C16-C17-C18 | 0.1 (8) | N1-Ni1-O1-C1 | 13.3 (4) |
| Cl3—C16—C17—C18 | -178.8 (4) | C18—C19—O2—Ni1 | 165.5 (3) |
| C16—C17—C18—C19 | -1.2 (7) | C14—C19—O2—Ni1 | -16.6 (6) |
| C16-C17-C18-Cl4 | -179.5 (4) | O1-Ni1-O2-C19 | -176.6 (4) |
| C17—C18—C19—O2 | 178.6 (4) | N2-Ni1-O2-C19 | 18.6 (4) |
| Cl4—C18—C19—O2 | -3.0 (6) | N1—Ni1—O2—C19 | -92.1 (6) |
| | | | |

Hydrogen-bond geometry (Å, °)

| D—H···A | <i>D</i> —Н | $H \cdots A$ | $D \cdots A$ | D—H···A |
|------------------------------|-------------|--------------|--------------|---------|
| C8—H8A···O1 ⁱ | 0.97 | 2.52 | 3.269 (5) | 134 |
| C12—H12A···Cl4 ⁱⁱ | 0.97 | 2.80 | 3.578 (5) | 138 |
| | | | | |

Symmetry codes: (i) -x+1, -y+1, -z; (ii) -x+1, -y, -z.



Fig. 1



Fig. 2

Fig. 3