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Di- μ -chlorido-bis{chlorido[2-(2-furyl-methyliminomethyl)pyridine- κ^2 N,N']-nickel(II)}

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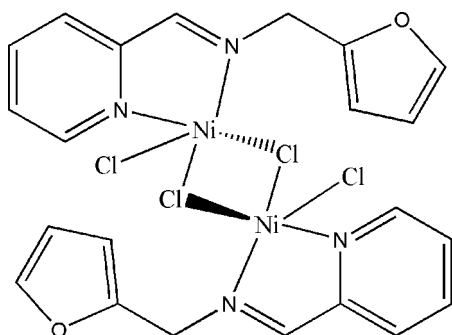
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.006$ Å; R factor = 0.041; wR factor = 0.099; data-to-parameter ratio = 15.6.

The title dinuclear nickel(II) complex, $[\text{Ni}_2\text{Cl}_4(\text{C}_{11}\text{H}_{10}\text{N}_2\text{O})_2]$, lies on a centre of symmetry located at the centroid of the four-membered ring formed by the two Ni atoms and the bridging chloride ions. The Ni^{II} atom is five-coordinated in a square-pyramidal geometry by the imine and pyridine N atoms of the Schiff base ligand, and by one terminal and two bridging Cl atoms. The Ni...Ni distance is 3.506 (2) Å. The O atom of the furan substituent in the ligand unit is not involved in coordination to the Ni atom.

Related literature

For related structures, see: Cheng *et al.* (2007); Li *et al.* (2007); Qiu *et al.* (2006); Shi *et al.* (2007); Wang *et al.* (2005); Zhu *et al.* (2003).



Experimental

Crystal data

| | |
|--|-----------------------------------|
| $[\text{Ni}_2\text{Cl}_4(\text{C}_{11}\text{H}_{10}\text{N}_2\text{O})_2]$ | $\gamma = 70.132$ (8) $^\circ$ |
| $M_r = 631.64$ | $V = 615.39$ (10) Å ³ |
| Triclinic, $P\bar{1}$ | $Z = 1$ |
| $a = 8.0439$ (8) Å | Mo $K\alpha$ radiation |
| $b = 8.5659$ (8) Å | $\mu = 1.99$ mm ⁻¹ |
| $c = 10.0610$ (9) Å | $T = 298$ (2) K |
| $\alpha = 77.522$ (8) $^\circ$ | $0.30 \times 0.30 \times 0.28$ mm |
| $\beta = 72.040$ (7) $^\circ$ | |

Data collection

| | |
|---|--|
| Bruker SMART CCD area-detector diffractometer | 2585 measured reflections |
| Absorption correction: multi-scan (SADABS; Sheldrick, 1996) | 2408 independent reflections |
| $T_{\min} = 0.554$, $T_{\max} = 0.572$ | 1971 reflections with $I > 2\sigma(I)$ |
| | $R_{\text{int}} = 0.020$ |

Refinement

| | |
|---------------------------------|---|
| $R[F^2 > 2\sigma(F^2)] = 0.040$ | 154 parameters |
| $wR(F^2) = 0.099$ | H-atom parameters constrained |
| $S = 1.07$ | $\Delta\rho_{\text{max}} = 0.51$ e Å ⁻³ |
| 2408 reflections | $\Delta\rho_{\text{min}} = -0.58$ e Å ⁻³ |

Data collection: SMART (Bruker, 1998); cell refinement: SAINT (Bruker, 1998); data reduction: SAINT; 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.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2503).

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supporting information

Acta Cryst. (2008). E64, m836 [doi:10.1107/S1600536808015183]

Di- μ -chlorido-bis{chlorido[2-(2-furylmethyliminomethyl)pyridine- κ^2N,N']nickel(II)}**Dong-Sheng Xia, Wu Chen, Xin-Long Tang and Qing-Fu Zeng****S1. Comment**

As part of our ongoing interest in the structure of nickel(II) complexes (Zhu *et al.*, 2003), we report herein the crystal structure of the title compound, a new centrosymmetric dinuclear nickel(II) complex, (I), Fig. 1, derived from the Schiff base ligand furan-2-ylmethyl-(1-pyridin-2-ylmethylidene)amine.

The Ni^{II} atom in (I) is five-coordinate in a square pyramidal geometry, binding to the imine and pyridine N atoms of the Schiff base ligand, and to one terminal Cl and two bridging Cl atoms. The Ni...Ni distance is 3.506 (2) Å. The dihedral angle between the benzene ring and the furan ring is 73.3 (3) °. The O atom of the furan substituent in the ligand lies well away from the coordination sphere of the Ni atom. The coordinate bond values (Table 1) are comparable to values observed in other similar nickel(II) complexes (Shi *et al.*, 2007; Li *et al.*, 2007; Cheng *et al.*, 2007; Qiu *et al.*, 2006; Wang *et al.*, 2005).

S2. Experimental

Pyridine-2-carbaldehyde (10.7 mg, 0.1 mmol), furan-2-ylmethylamine (9.7 mg, 0.1 mmol), and NiCl₂·6H₂O (23.8 mg, 0.1 mmol) were dissolved in methanol (30 ml). The mixture was stirred for 30 min at room temperature. The resulting solution was left in air for a few days, yielding green crystals.

S3. Refinement

H atoms were placed in idealized positions and constrained to ride on their parent atoms with C–H distances in the range 0.93–0.97 Å, and with $U_{\text{iso}}(\text{H})$ set at $1.2U_{\text{eq}}(\text{C})$.

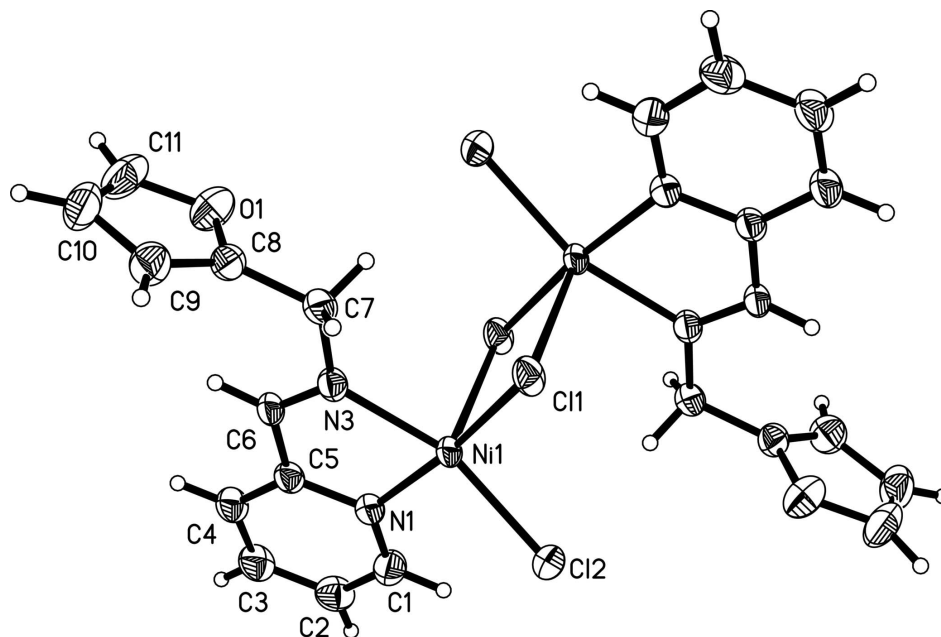


Figure 1

The structure of (I) showing 30% probability displacement ellipsoids and the atom-numbering scheme. Numbered atoms are related to un-numbered atoms by the symmetry code 1-x, 2-y, 1-z.

Di- μ -chlorido-bis[chlorido[2-(2-furlymethyliminomethyl)pyridine- κ^2 N,N']]nickel(II)}

Crystal data

[Ni₂Cl₄(C₁₁H₁₀N₂O)₂]

$M_r = 631.64$

Triclinic, *P*1

Hall symbol: -P 1

$a = 8.0439$ (8) Å

$b = 8.5659$ (8) Å

$c = 10.0610$ (9) Å

$\alpha = 77.522$ (8)°

$\beta = 72.040$ (7)°

$\gamma = 70.132$ (8)°

$V = 615.39$ (10) Å³

$Z = 1$

$F(000) = 320$

$D_x = 1.704$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 1237 reflections

$\theta = 2.4$ – 25.3 °

$\mu = 1.99$ mm⁻¹

$T = 298$ K

Block, green

$0.30 \times 0.30 \times 0.28$ mm

Data collection

Bruker SMART CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

ω scans

Absorption correction: multi-scan

(*SADABS*; Sheldrick, 1996)

$T_{\min} = 0.554$, $T_{\max} = 0.572$

2585 measured reflections

2408 independent reflections

1971 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.020$

$\theta_{\max} = 26.0$ °, $\theta_{\min} = 2.6$ °

$h = 0 \rightarrow 9$

$k = -9 \rightarrow 10$

$l = -11 \rightarrow 12$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.040$

$wR(F^2) = 0.099$

$S = 1.07$

2408 reflections

154 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

$w = 1/[\sigma^2(F_o^2) + (0.0486P)^2 + 0.2142P]$

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

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

$\Delta\rho_{\max} = 0.51 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.58 \text{ e } \text{\AA}^{-3}$

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)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|--------------|--------------|----------------------------------|
| Ni1 | 0.72650 (5) | 1.00555 (5) | 0.45738 (4) | 0.03767 (16) |
| Cl1 | 0.46003 (12) | 1.19011 (10) | 0.55654 (10) | 0.0481 (2) |
| Cl2 | 0.76194 (14) | 1.16826 (13) | 0.24879 (11) | 0.0608 (3) |
| O1 | 0.6666 (5) | 0.6517 (4) | 0.9351 (3) | 0.0729 (9) |
| N1 | 0.9716 (4) | 0.8340 (4) | 0.3931 (3) | 0.0444 (7) |
| C11 | 0.7243 (7) | 0.5754 (6) | 1.0541 (5) | 0.0848 (16) |
| H11 | 0.7256 | 0.4670 | 1.0955 | 0.102* |
| N3 | 0.7676 (4) | 0.8807 (3) | 0.6475 (3) | 0.0418 (7) |
| C1 | 1.0714 (5) | 0.8105 (5) | 0.2615 (4) | 0.0576 (10) |
| H1 | 1.0313 | 0.8837 | 0.1871 | 0.069* |
| C2 | 1.2312 (6) | 0.6821 (5) | 0.2320 (5) | 0.0617 (11) |
| H2 | 1.2962 | 0.6683 | 0.1392 | 0.074* |
| C3 | 1.2941 (5) | 0.5746 (5) | 0.3406 (5) | 0.0608 (11) |
| H3 | 1.4016 | 0.4870 | 0.3227 | 0.073* |
| C4 | 1.1937 (5) | 0.5998 (5) | 0.4771 (4) | 0.0533 (10) |
| H4 | 1.2343 | 0.5305 | 0.5528 | 0.064* |
| C5 | 1.0333 (5) | 0.7283 (4) | 0.4998 (4) | 0.0429 (8) |
| C6 | 0.9147 (4) | 0.7611 (4) | 0.6387 (4) | 0.0428 (8) |
| H6 | 0.9458 | 0.6960 | 0.7193 | 0.051* |
| C7 | 0.6364 (5) | 0.9170 (5) | 0.7850 (4) | 0.0477 (9) |
| H7A | 0.6191 | 1.0315 | 0.7966 | 0.057* |
| H7B | 0.5196 | 0.9099 | 0.7827 | 0.057* |
| C8 | 0.6882 (5) | 0.8072 (5) | 0.9094 (4) | 0.0479 (9) |
| C9 | 0.7552 (6) | 0.8277 (6) | 1.0092 (4) | 0.0658 (11) |
| H9 | 0.7815 | 0.9228 | 1.0160 | 0.079* |

| | | | | |
|-----|------------|------------|------------|-------------|
| C10 | 0.7779 (7) | 0.6769 (7) | 1.1019 (5) | 0.0820 (16) |
| H10 | 0.8223 | 0.6537 | 1.1813 | 0.098* |

Atomic displacement parameters (Å²)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|---------------|---------------|--------------|
| Ni1 | 0.0292 (2) | 0.0323 (2) | 0.0470 (3) | -0.00427 (17) | -0.01482 (18) | 0.00379 (17) |
| Cl1 | 0.0407 (5) | 0.0327 (4) | 0.0685 (6) | -0.0016 (4) | -0.0209 (4) | -0.0061 (4) |
| Cl2 | 0.0525 (6) | 0.0567 (6) | 0.0665 (6) | -0.0179 (5) | -0.0227 (5) | 0.0201 (5) |
| O1 | 0.094 (2) | 0.0557 (18) | 0.0644 (19) | -0.0237 (17) | -0.0199 (17) | 0.0038 (14) |
| N1 | 0.0373 (16) | 0.0441 (16) | 0.0497 (17) | -0.0115 (13) | -0.0144 (13) | 0.0024 (13) |
| C11 | 0.093 (4) | 0.062 (3) | 0.058 (3) | 0.002 (3) | -0.005 (3) | 0.015 (2) |
| N3 | 0.0380 (15) | 0.0382 (15) | 0.0498 (17) | -0.0091 (13) | -0.0171 (13) | -0.0013 (13) |
| C1 | 0.052 (2) | 0.061 (2) | 0.052 (2) | -0.010 (2) | -0.0134 (19) | 0.0004 (19) |
| C2 | 0.050 (2) | 0.063 (3) | 0.062 (3) | -0.011 (2) | 0.000 (2) | -0.017 (2) |
| C3 | 0.044 (2) | 0.048 (2) | 0.078 (3) | -0.0007 (18) | -0.012 (2) | -0.010 (2) |
| C4 | 0.041 (2) | 0.043 (2) | 0.067 (3) | -0.0028 (16) | -0.0165 (19) | -0.0015 (18) |
| C5 | 0.0357 (18) | 0.0364 (18) | 0.055 (2) | -0.0091 (14) | -0.0146 (16) | -0.0008 (15) |
| C6 | 0.0351 (18) | 0.0396 (18) | 0.052 (2) | -0.0077 (15) | -0.0197 (16) | 0.0043 (15) |
| C7 | 0.0371 (19) | 0.045 (2) | 0.054 (2) | -0.0053 (16) | -0.0105 (16) | -0.0041 (16) |
| C8 | 0.042 (2) | 0.045 (2) | 0.048 (2) | -0.0045 (16) | -0.0093 (16) | -0.0051 (16) |
| C9 | 0.066 (3) | 0.076 (3) | 0.054 (2) | -0.019 (2) | -0.016 (2) | -0.009 (2) |
| C10 | 0.074 (3) | 0.099 (4) | 0.049 (3) | 0.000 (3) | -0.019 (2) | 0.004 (3) |

Geometric parameters (Å, °)

| | | | |
|----------------------|-------------|----------|-----------|
| Ni1—N1 | 2.030 (3) | C2—C3 | 1.374 (6) |
| Ni1—N3 | 2.044 (3) | C2—H2 | 0.9300 |
| Ni1—Cl2 | 2.2506 (10) | C3—C4 | 1.382 (5) |
| Ni1—Cl1 | 2.2690 (10) | C3—H3 | 0.9300 |
| Ni1—Cl1 ⁱ | 2.6496 (10) | C4—C5 | 1.375 (5) |
| Cl1—Ni1 ⁱ | 2.6496 (10) | C4—H4 | 0.9300 |
| O1—C8 | 1.361 (5) | C5—C6 | 1.452 (5) |
| O1—C11 | 1.369 (5) | C6—H6 | 0.9300 |
| N1—C1 | 1.337 (5) | C7—C8 | 1.473 (5) |
| N1—C5 | 1.350 (4) | C7—H7A | 0.9700 |
| C11—C10 | 1.317 (7) | C7—H7B | 0.9700 |
| C11—H11 | 0.9300 | C8—C9 | 1.343 (5) |
| N3—C6 | 1.268 (4) | C9—C10 | 1.412 (6) |
| N3—C7 | 1.479 (4) | C9—H9 | 0.9300 |
| C1—C2 | 1.377 (5) | C10—H10 | 0.9300 |
| C1—H1 | 0.9300 | | |
| N1—Ni1—N3 | 79.68 (11) | C2—C3—C4 | 118.4 (4) |
| N1—Ni1—Cl2 | 92.50 (9) | C2—C3—H3 | 120.8 |
| N3—Ni1—Cl2 | 160.74 (8) | C4—C3—H3 | 120.8 |
| N1—Ni1—Cl1 | 172.70 (9) | C5—C4—C3 | 119.3 (4) |
| N3—Ni1—Cl1 | 93.38 (8) | C5—C4—H4 | 120.4 |

| | | | |
|--|------------|---------------|------------|
| C12—Ni1—C11 | 93.42 (4) | C3—C4—H4 | 120.4 |
| N1—Ni1—C11 ⁱ | 93.04 (8) | N1—C5—C4 | 122.3 (3) |
| N3—Ni1—C11 ⁱ | 91.84 (8) | N1—C5—C6 | 114.2 (3) |
| C12—Ni1—C11 ⁱ | 106.23 (4) | C4—C5—C6 | 123.5 (3) |
| C11—Ni1—C11 ⁱ | 89.40 (3) | N3—C6—C5 | 118.3 (3) |
| Ni1—C11—Ni1 ⁱ | 90.60 (3) | N3—C6—H6 | 120.8 |
| C8—O1—C11 | 106.2 (4) | C5—C6—H6 | 120.8 |
| C1—N1—C5 | 118.0 (3) | C8—C7—N3 | 116.0 (3) |
| C1—N1—Ni1 | 128.3 (3) | C8—C7—H7A | 108.3 |
| C5—N1—Ni1 | 113.7 (2) | N3—C7—H7A | 108.3 |
| C10—C11—O1 | 110.6 (4) | C8—C7—H7B | 108.3 |
| C10—C11—H11 | 124.7 | N3—C7—H7B | 108.3 |
| O1—C11—H11 | 124.7 | H7A—C7—H7B | 107.4 |
| C6—N3—C7 | 121.3 (3) | C9—C8—O1 | 109.5 (4) |
| C6—N3—Ni1 | 114.1 (2) | C9—C8—C7 | 133.0 (4) |
| C7—N3—Ni1 | 124.5 (2) | O1—C8—C7 | 117.5 (3) |
| N1—C1—C2 | 122.4 (4) | C8—C9—C10 | 106.9 (5) |
| N1—C1—H1 | 118.8 | C8—C9—H9 | 126.6 |
| C2—C1—H1 | 118.8 | C10—C9—H9 | 126.6 |
| C3—C2—C1 | 119.6 (4) | C11—C10—C9 | 106.8 (4) |
| C3—C2—H2 | 120.2 | C11—C10—H10 | 126.6 |
| C1—C2—H2 | 120.2 | C9—C10—H10 | 126.6 |
| | | | |
| N1—Ni1—C11—Ni1 ⁱ | -109.6 (7) | N1—C1—C2—C3 | -1.1 (7) |
| N3—Ni1—C11—Ni1 ⁱ | -91.81 (8) | C1—C2—C3—C4 | -0.2 (6) |
| C12—Ni1—C11—Ni1 ⁱ | 106.22 (4) | C2—C3—C4—C5 | 1.4 (6) |
| C11 ⁱ —Ni1—C11—Ni1 ⁱ | 0.0 | C1—N1—C5—C4 | 0.2 (5) |
| N3—Ni1—N1—C1 | 178.4 (3) | Ni1—N1—C5—C4 | 177.7 (3) |
| C12—Ni1—N1—C1 | -19.4 (3) | C1—N1—C5—C6 | -178.9 (3) |
| C11—Ni1—N1—C1 | -163.6 (5) | Ni1—N1—C5—C6 | -1.4 (4) |
| C11 ⁱ —Ni1—N1—C1 | 87.0 (3) | C3—C4—C5—N1 | -1.5 (6) |
| N3—Ni1—N1—C5 | 1.3 (2) | C3—C4—C5—C6 | 177.5 (4) |
| C12—Ni1—N1—C5 | 163.5 (2) | C7—N3—C6—C5 | 177.4 (3) |
| C11—Ni1—N1—C5 | 19.3 (8) | Ni1—N3—C6—C5 | 0.4 (4) |
| C11 ⁱ —Ni1—N1—C5 | -90.1 (2) | N1—C5—C6—N3 | 0.7 (5) |
| C8—O1—C11—C10 | 0.5 (5) | C4—C5—C6—N3 | -178.4 (3) |
| N1—Ni1—N3—C6 | -0.9 (2) | C6—N3—C7—C8 | 0.5 (5) |
| C12—Ni1—N3—C6 | -68.1 (4) | Ni1—N3—C7—C8 | 177.2 (2) |
| C11—Ni1—N3—C6 | -178.6 (2) | C11—O1—C8—C9 | -0.6 (5) |
| C11 ⁱ —Ni1—N3—C6 | 91.9 (2) | C11—O1—C8—C7 | 179.7 (3) |
| N1—Ni1—N3—C7 | -177.8 (3) | N3—C7—C8—C9 | 103.2 (5) |
| C12—Ni1—N3—C7 | 114.9 (3) | N3—C7—C8—O1 | -77.1 (4) |
| C11—Ni1—N3—C7 | 4.4 (3) | O1—C8—C9—C10 | 0.4 (5) |
| C11 ⁱ —Ni1—N3—C7 | -85.1 (3) | C7—C8—C9—C10 | -179.9 (4) |
| C5—N1—C1—C2 | 1.0 (6) | O1—C11—C10—C9 | -0.3 (6) |
| Ni1—N1—C1—C2 | -175.9 (3) | C8—C9—C10—C11 | -0.1 (6) |

Symmetry code: (i) $-x+1, -y+2, -z+1$.