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Bis[2,4-dichloro-6-(ethyliminomethyl)-phenolato-κ^2N,O]nickel(II)

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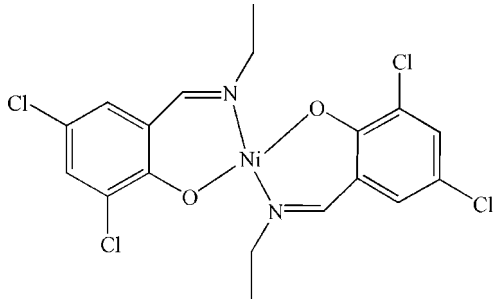
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.031; wR factor = 0.055; data-to-parameter ratio = 13.6.

In the title compound, $[Ni(C_9H_8Cl_2NO)_2]$, the Ni^{II} ion lies on an inversion centre and is coordinated in a slightly distorted square-planar geometry by an N and an O atom from two symmetry-related bidentate 2,4-dichloro-6-(ethyliminomethyl)phenolate ligands. In the crystal structure, there are short Cl···Cl distances of 3.506 (1) and 3.350 (1) Å.

Related literature

For halogen–halogen interactions in supramolecular chemistry and crystal engineering, see: Cohen *et al.* (1964); Desiraju (1989); Xiao & Zhang (2008); Aakeröy *et al.* (2011).



Experimental

Crystal data

$[Ni(C_9H_8Cl_2NO)_2]$
M_r = 492.84
Monoclinic, $P2_1/c$
a = 7.5004 (6) Å
b = 9.3155 (7) Å
c = 14.1498 (12) Å
β = 103.841 (1)°
V = 959.94 (13) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.58 mm^{-1}
T = 293 K
0.32 × 0.28 × 0.26 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.612, T_{max} = 0.667
4890 measured reflections
1685 independent reflections
1267 reflections with $I > 2\sigma(I)$
R_{int} = 0.060

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.055
S = 0.97
1685 reflections
124 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

Data collection: *SMART* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); 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: LH5353).

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supplementary materials

Acta Cryst. (2011). E**67**, m1611 [**doi:10.1107/S160053681104325X**]

Bis[2,4-dichloro-6-(ethyliminomethyl)phenolato-κ^2N,O]nickel(II)

Q. P. Huang, S. H. Zhang, J. J. Guo, C. Feng and F. S. Tang

Comment

Halogens have a ubiquitous presence in both inorganic and organic chemistry. Schiff bases of chloro substituents on aromatic systems have aroused interest in recent years because these halogenated compounds are an attractive target for use in supramolecular chemistry and crystal engineering wherein the halogen atoms are directly involved in forming intermolecular interactions (Cohen *et al.*, 1964; Desiraju, 1989; Xiao & Zhang, 2008; Aakeröy *et al.* 2011). The title compound, (I), contains a deprotonated 2,4-dichloro-2-ethyliminomethyl-phenol ligand, with two Cl atoms accesible for Cl···Cl interactions.

In (I), the Ni^{II} ion lies on an inversion center and is coordinated by two O and two N atoms from two symmetry related bidentate 2,4-diChloro-*N*-ethylsalicylaldimino ligands, forming a slightly distorted square-planar geometry (Fig. 1). In the crystal, there are short Cl···Cl contacts ($Cl1\cdots Cl2^{i}$ 3.506 (1) Å, $Cl2\cdots Cl2^{ii}$ 3.350 (1) Å symmetry code:(i) 1 - *x*, 1/2 + *y*, 1/2 - *z*, (ii) -*x*, -*y*, -*z*) (Fig. 2).

Experimental

A solution of (0.191 g, 1.0 mmol) 3,5-dichloro-2-hydroxy-benzaldehyde and (0.044 g, 1 mmol) ethylamine and (0.040 g, 1 mmol) sodium hydroxide in 20 ml absolute methanol was added slowly a solution of nickel nitrate hexahydrate (0.145 g, 0.5 mmol) in methanol. The mixture was stirred for 3 h at room temperature to give a green solution which was filtered and the filtrate was left to stand at room temperature. Green block-shaped crystals suitable for X-ray diffraction were obtained by slow evaporation. yield: 78.2% (Based on Nickel). Elemental analysis calculated: C 43.83, H 3.75, N 5.68%; Found: C 43.79, H,3.78, N 5.71%.

Refinement

H atoms were positioned geometrically and refined with a riding model, with C—H distances = 0.93–0.97 Å and with $U_{iso}(H) = 1.2U_{eq}(C)$ or $U_{iso}(H) = 1.5U_{eq}(C_{methyl})$.

Figures

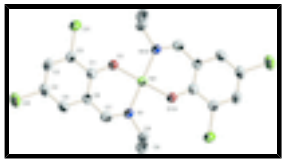

Fig. 1. The molecular structure of (I), showing 30% probability displacement ellipsoids. H atoms are omitted.

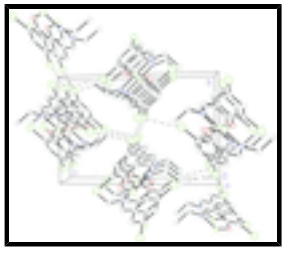

Fig. 2. Part of the crystal structure showing short Cl···Cl contacts as dashed lines.

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Crystal data

$[Ni(C_9H_8Cl_2NO)_2]$	$F(000) = 500$
$M_r = 492.84$	$D_x = 1.705$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 1685 reflections
$a = 7.5004$ (6) Å	$\theta = 2.6–25.0°$
$b = 9.3155$ (7) Å	$\mu = 1.58$ mm^{-1}
$c = 14.1498$ (12) Å	$T = 293$ K
$\beta = 103.841$ (1)°	Block, green
$V = 959.94$ (13) Å^3	0.32 × 0.28 × 0.26 mm
$Z = 2$	

Data collection

Bruker SMART CCD diffractometer	1685 independent reflections
Radiation source: fine-focus sealed tube	1267 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.060$
φ and ω scans	$\theta_{max} = 25.0°$, $\theta_{min} = 2.6°$
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)	$h = -8\rightarrow7$
$T_{min} = 0.612$, $T_{max} = 0.667$	$k = -11\rightarrow8$
4890 measured reflections	$l = -16\rightarrow16$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.031$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.055$	H-atom parameters constrained
$S = 0.97$	$w = 1/[\sigma^2(F_o^2) + (0.0012P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
1685 reflections	$(\Delta/\sigma)_{max} < 0.001$
124 parameters	$\Delta\rho_{max} = 0.28$ e Å^{-3}

0 restraints $\Delta\rho_{min} = -0.35$ e Å^{-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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.6897 (3)	0.3911 (3)	0.06190 (18)	0.0321 (6)
C2	0.5743 (3)	0.3976 (3)	0.12798 (17)	0.0344 (7)
C3	0.4233 (3)	0.3110 (3)	0.11860 (18)	0.0383 (7)
H3A	0.3502	0.3176	0.1631	0.046*
C4	0.3801 (3)	0.2139 (3)	0.0429 (2)	0.0384 (7)
C5	0.4853 (3)	0.2045 (3)	−0.02361 (18)	0.0393 (7)
H5A	0.4546	0.1393	−0.0747	0.047*
C6	0.6402 (3)	0.2937 (3)	−0.01460 (18)	0.0324 (6)
Cl1	0.62781 (9)	0.51726 (8)	0.22421 (4)	0.0454 (2)
Cl2	0.18645 (10)	0.10465 (8)	0.03097 (5)	0.0538 (2)
Ni1	1.0000	0.5000	0.0000	0.03205 (15)
O1	0.8324 (2)	0.47509 (19)	0.07451 (12)	0.0393 (5)
C7	0.7422 (3)	0.2847 (3)	−0.08801 (18)	0.0374 (7)
H7A	0.7023	0.2171	−0.1369	0.045*
C8	0.9509 (4)	0.3268 (3)	−0.18214 (19)	0.0465 (8)
H8A	0.9135	0.2303	−0.2040	0.056*
H8B	1.0840	0.3302	−0.1659	0.056*
C9	0.8770 (4)	0.4311 (4)	−0.26300 (19)	0.0654 (10)
H9A	0.9229	0.4067	−0.3187	0.098*
H9B	0.9155	0.5265	−0.2419	0.098*
H9C	0.7453	0.4266	−0.2800	0.098*
N1	0.8836 (3)	0.3602 (2)	−0.09375 (14)	0.0337 (5)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0305 (15)	0.0322 (17)	0.0333 (15)	0.0026 (13)	0.0070 (13)	0.0053 (13)
C2	0.0343 (16)	0.0373 (17)	0.0315 (15)	0.0041 (13)	0.0078 (13)	0.0053 (13)
C3	0.0341 (16)	0.0456 (19)	0.0376 (16)	0.0033 (14)	0.0132 (14)	0.0094 (15)
C4	0.0303 (16)	0.0388 (17)	0.0449 (17)	−0.0045 (14)	0.0068 (14)	0.0099 (15)
C5	0.0387 (16)	0.0399 (18)	0.0376 (17)	−0.0064 (14)	0.0055 (15)	−0.0018 (13)

C6	0.0329 (15)	0.0315 (16)	0.0332 (15)	−0.0005 (13)	0.0084 (13)	0.0012 (13)
Cl1	0.0483 (4)	0.0536 (5)	0.0372 (4)	−0.0018 (4)	0.0162 (4)	−0.0060 (4)
Cl2	0.0394 (4)	0.0634 (6)	0.0583 (5)	−0.0150 (4)	0.0110 (4)	0.0083 (4)
Ni1	0.0334 (3)	0.0326 (3)	0.0319 (3)	−0.0019 (2)	0.0112 (2)	−0.0032 (2)
O1	0.0403 (11)	0.0445 (13)	0.0372 (10)	−0.0116 (10)	0.0173 (9)	−0.0094 (9)
C7	0.0427 (17)	0.0348 (17)	0.0341 (16)	−0.0010 (14)	0.0077 (14)	−0.0038 (13)
C8	0.0481 (18)	0.051 (2)	0.0465 (18)	−0.0117 (15)	0.0238 (15)	−0.0196 (16)
C9	0.055 (2)	0.105 (3)	0.0394 (18)	−0.013 (2)	0.0178 (17)	0.002 (2)
N1	0.0374 (13)	0.0342 (13)	0.0326 (12)	−0.0020 (11)	0.0143 (11)	−0.0027 (11)

Geometric parameters (Å, °)

C1—O1	1.303 (3)	Ni1—O1^{i}	1.8382 (16)
C1—C6	1.393 (3)	Ni1—N1^{i}	1.914 (2)
C1—C2	1.419 (3)	Ni1—N1	1.914 (2)
C2—C3	1.372 (3)	C7—N1	1.291 (3)
C2—Cl1	1.731 (3)	C7—H7A	0.9300
C3—C4	1.380 (3)	C8—N1	1.489 (3)
C3—H3A	0.9300	C8—C9	1.502 (4)
C4—C5	1.368 (3)	C8—H8A	0.9700
C4—Cl2	1.749 (3)	C8—H8B	0.9700
C5—C6	1.410 (3)	C9—H9A	0.9600
C5—H5A	0.9300	C9—H9B	0.9600
C6—C7	1.432 (3)	C9—H9C	0.9600
Ni1—O1	1.8382 (16)		
O1—C1—C6	123.7 (2)	O1^{i}—Ni1—N1	87.10 (8)
O1—C1—C2	119.6 (2)	N1^{i}—Ni1—N1	180.0
C6—C1—C2	116.7 (2)	C1—O1—Ni1	130.49 (17)
C3—C2—C1	122.1 (3)	N1—C7—C6	127.1 (3)
C3—C2—Cl1	119.1 (2)	N1—C7—H7A	116.5
C1—C2—Cl1	118.9 (2)	C6—C7—H7A	116.5
C2—C3—C4	119.7 (3)	N1—C8—C9	111.6 (2)
C2—C3—H3A	120.1	N1—C8—H8A	109.3
C4—C3—H3A	120.1	C9—C8—H8A	109.3
C5—C4—C3	120.6 (2)	N1—C8—H8B	109.3
C5—C4—Cl2	119.9 (2)	C9—C8—H8B	109.3
C3—C4—Cl2	119.4 (2)	H8A—C8—H8B	108.0
C4—C5—C6	119.9 (3)	C8—C9—H9A	109.5
C4—C5—H5A	120.1	C8—C9—H9B	109.5
C6—C5—H5A	120.1	H9A—C9—H9B	109.5
C1—C6—C5	121.0 (2)	C8—C9—H9C	109.5
C1—C6—C7	120.8 (2)	H9A—C9—H9C	109.5
C5—C6—C7	118.2 (2)	H9B—C9—H9C	109.5
O1—Ni1—O1^{i}	180.00 (13)	C7—N1—C8	112.8 (2)
O1—Ni1—N1^{i}	87.10 (8)	C7—N1—Ni1	124.90 (19)
O1^{i}—Ni1—N1^{i}	92.90 (8)	C8—N1—Ni1	122.30 (17)
O1—Ni1—N1	92.90 (8)		

Symmetry codes: (i) −*x*+2, −*y*+1, −*z*.

Fig. 1

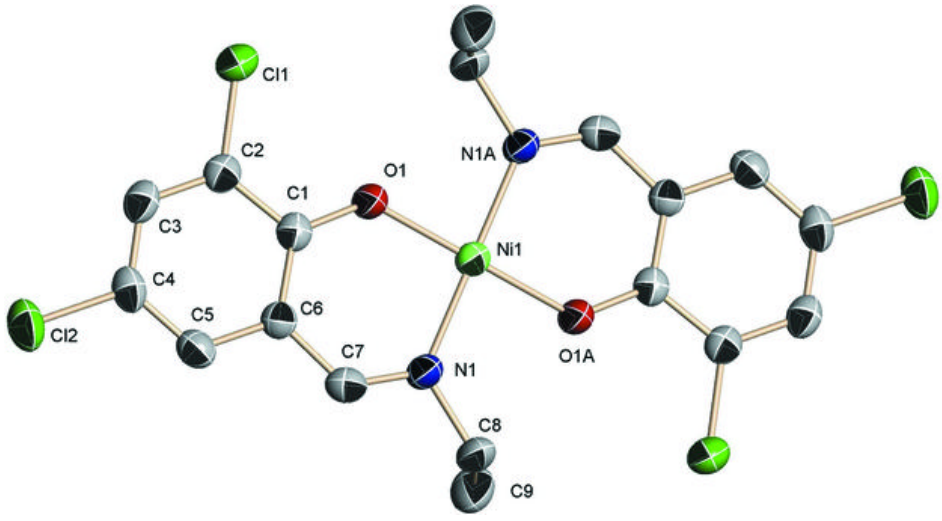

Fig. 2