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Dichlorobis[1-(2-ethoxyethyl)-1*H*-benzimidazole-κN^3]nickel(II)

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.003 Å
R factor = 0.027
wR factor = 0.069
Data-to-parameter ratio = 18.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $[NiCl_2(C_{11}H_{14}N_2O)_2]$, was synthesized from 1-(2-ethoxyethyl)benzimidazole and nickel dichloride in ethanol and is isostructural with its cobalt analogue. It shows a distorted tetrahedral geometry about Ni^{II}, involving two Cl atoms and two benzimidazole N atoms. The crystal structure is stabilized by C—H···Cl and C—H···O interactions.

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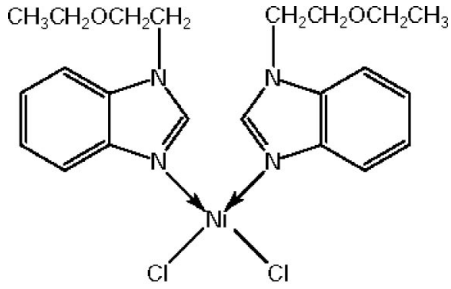
Comment

The title compound, (I) (Fig. 1), was prepared and characterized as part of our ongoing studies (Türktekin *et al.*, 2004, and references therein) of benzimidazole complexes of transition metals. As shown in Fig. 1, the Ni atom in (I) is tetrahedrally coordinated by two Cl atoms and two benzimidazole N atoms. The Cl_2N_2 donor set defines a distorted tetrahedron (Table 1), with bond angles ranging from 102.59 (5) to 107.99 (5)°.



(I)

The average Ni—N bond length of 1.978 (2) Å in (I) is similar to the average value of 2.003 (3) Å in *rac*-[2,2′-bis(1-ethylbenzimidazol-2-yl-κN^3)biphenyl]dichloronickel(II)

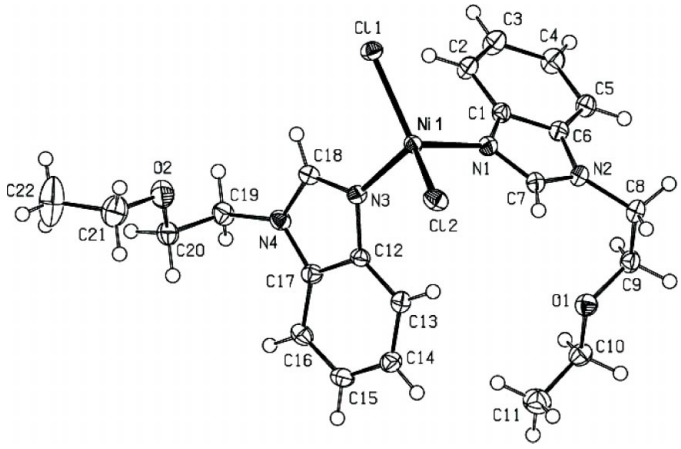


Figure 1
View of (I) with the atom-numbering scheme and 50% probability displacement ellipsoids (arbitrary spheres for the H atoms).

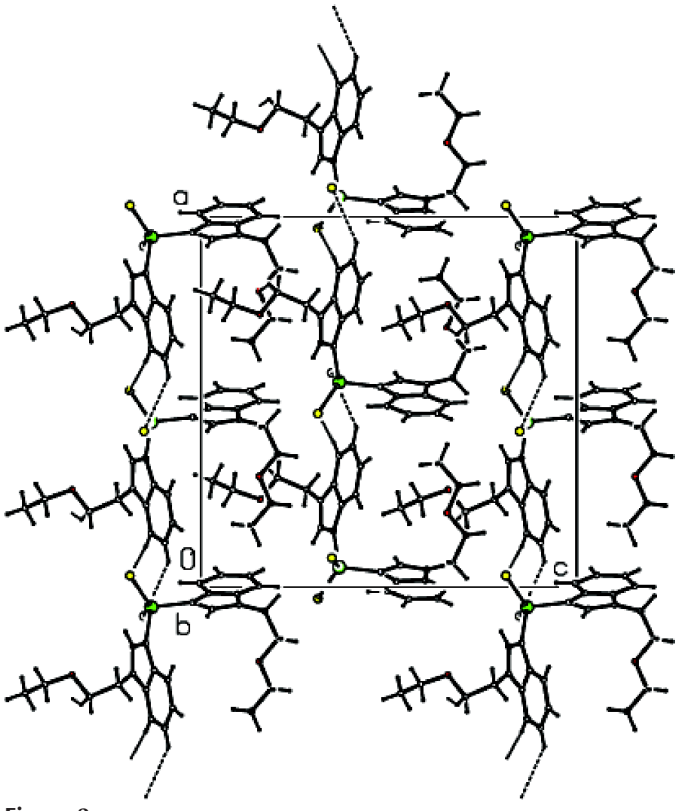


Figure 2
A view of the packing and hydrogen bonds (dashed lines) in (I) (colour key: Ni atoms green and Cl atoms yellow).

(Stibrany *et al.*, 2004), 2.103 (4) Å in bis[bis(benzimidazol-2-ylmethyl-κN^3)amine-κN]nickel(II) dichloride (Thangarasu *et al.*, 1997) and 2.0775 (17) Å in diaquabis{2-[(5-methylpyrazol-3-yl)methyl]benzimidazole}nickel(II) dichloride dihydrate (Sbai *et al.*, 2002). The Ni—Cl bond lengths in (I) [2.2316 (16) and 2.2435 (16) Å] are nearly equal to the corresponding lengths of 2.2310 (15) and 2.2405 (15) Å in *rac*-[2,2′-bis(1-ethylbenzimidazol-2-yl-κN^3)biphenyl]dichloronickel(II) (Stibrany *et al.*, 2004). The dihedral angle between the least-squares planes through the benzimidazole rings is 85.22 (7)°.

The molecules of (I) are linked through C—H···Cl and C—H···O interactions (Fig. 2 and Table 2); this was also observed for the isostructural cobalt(II) analogue of (I) (Türktekin *et al.*, 2004).

Experimental

1-(2-Ethoxyethyl)benzimidazole was synthesized from benzimidazole and 2-chloroethyl ethyl ether according to a literature procedure (Küçükbay *et al.*, 2001). A mixture of 1-(2-ethoxyethyl)benzimidazole (0.5 g; 2.63 mmol) and nickel dichloride (0.34 g; 2.63 mmol) in ethanol (25 ml) was heated under reflux for 4 h. All volatiles were then removed *in vacuo* (0.02 mmHg). The crude product was crystallized from ethanol/2-propanol (3:1) mixture upon cooling to 243 K (yield: 1.03 g, 77%; m.p. 451–452 K). ^{1}H NMR ($CDCl_3$): δ 1.1 (*t*, CH_3CH_2O—, 6H), 3.2 (*q*, CH_3CH_2O—, 4H), 3.6 (*t*, NCH_2CH_2O—, 4H), 3.7 (*t*, NCH_2CH_2O—, 4H), 7.6–8.0 (*m*, Ar-H, 8H), 8.5 (*s*, CH, 2H). Analysis calculated for $C_{13}H_{17}N_3O$: C 51.66, H 5.48, N 10.96%; found: C 51.17, H 5.43, N 10.91%.

Crystal data

$[NiCl_2(C_{11}H_{14}N_2O)_2]$
M_r = 510.07
Orthorhombic, $Pca2_1$
a = 17.416 (5) Å
b = 7.624 (5) Å
c = 17.667 (5) Å
V = 2345.8 (18) Å^3
Z = 4
D_x = 1.444 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 40494 reflections
θ = 1.6–27.3°
μ = 1.08 mm^{-1}
T = 100 K
Prism, violet
0.32 × 0.31 × 0.30 mm

Data collection

Stoe IPDS-II diffractometer
ω scans
Absorption correction: none
25872 measured reflections
5171 independent reflections
4987 reflections with $I > 2\sigma(I$
R_{int} = 0.056
θ_{max} = 27.2°
$h = -22 \rightarrow 22$
$k = -9 \rightarrow 9$
$l = -22 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.069
S = 1.07
5171 reflections
280 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0495P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.77 e Å^{-3}
Absolute structure: Flack (1983), 2480 Friedel pairs
Flack parameter = 0.079 (9)

Table 1
Selected geometric parameters (Å, °).

Ni1—Cl1	2.2316 (16)	Ni1—N1	1.981 (2)
Ni1—Cl2	2.2435 (16)	Ni1—N3	1.974 (2)
Cl1—Ni1—Cl2	129.75 (2)	Cl2—Ni1—N1	105.72 (5)
Cl1—Ni1—N1	107.99 (5)	Cl2—Ni1—N3	106.66 (5)
Cl1—Ni1—N3	102.59 (5)	N1—Ni1—N3	100.48 (7)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···Cl1	0.93	2.80	3.514 (3)	134
C15—H15···Cl2^{i}	0.93	2.82	3.612 (3)	143
C16—H16···Cl1^{ii}	0.93	2.78	3.699 (3)	169
C20—H20A···O1^{iii}	0.97	2.52	3.372 (3)	146

Symmetry codes: (i) $x-\frac{1}{2}, 2-y, z$; (ii) $x-\frac{1}{2}, 1-y, z$; (iii) $\frac{1}{2}-x, y-1, z-\frac{1}{2}$.

H atoms were positioned geometrically, with C—H = 0.93–0.97 Å, and refined using a riding model, with U_{iso}(H) = 1.5U_{eq}(carrier) for methyl H atoms and 1.2U_{eq}(carrier) for all other atoms.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED*32 (Stoe & Cie, 2002); program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*III (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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