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

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean *σ*(C–C) = 0.004 Å
R factor = 0.030
wR factor = 0.073
Data-to-parameter ratio = 20.6

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

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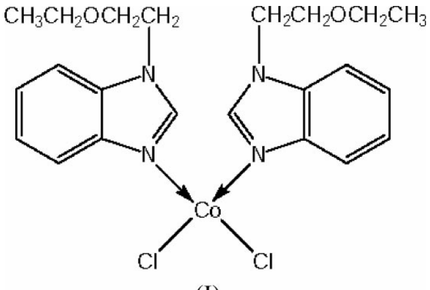
The title compound, [$CoCl_2(C_{11}H_{14}N_2O)_2$], was synthesized from 1-(2-ethoxyethyl)benzimidazole and cobalt dichloride in ethanol. The Co^{II} atom is coordinated in a distorted tetrahedral environment by two Cl atoms and two N atoms. The crystal structure is stabilized by intermolecular C—H···Cl and C—H···O interactions.

Comment

Benzimidazole and its derivatives have been found to have a broad range of pharmacological activity (Küçükbay *et al.*, 2001, 2004; Küçükbay, Durmaz, Orhan & Günal, 2003; Küçükbay, Durmaz, Okuyucu & Günal, 2003; Tavman, 2003). Transition metal complexes of benzimidazoles are increasingly used to model important bioinorganic systems (Khalil *et al.*, 2001). Furthermore, benzimidazole complexes have been evaluated as antifungal (Küçükbay & Durmaz, 1997), antibacterial (Çetinkaya *et al.*, 1996) and antitumour agents (Lukevics *et al.*, 2001). We have also synthesized a number of rhodium and ruthenium complexes of benzimidazole derivatives and determined their antimicrobial properties (Küçükbay & Durmaz, 1997; Durmaz *et al.*, 1997; Çetinkaya *et al.*, 1996), while results for related compounds containing the benzimidazole moiety have been reported in our earlier papers (Öztürk *et al.*, 2004; Akkurt, Öztürk, Küçükbay *et al.*, 2004*a*,*b*; Akkurt, Öztürk, Şireci *et al.*, 2004; Türktekin *et al.*, 2004). The aim of the present study was to elucidate the crystal structure of the title compound, (I).



(I)

The Co atom in (I) is coordinated tetrahedrally by two Cl atoms and two N atoms (Fig. 1 and Table 1). The Cl_2N_2 donor set defines a distorted tetrahedron, with angles ranging from 104.61 (5) to 119.84 (2)°.

The average Co—N bond length of 2.008 (2) Å may be compared with the reported average values of 2.1653 (16) for *trans*-tetraaquabis(*p*-nitrobenzoxasulfamato)cobalt(II) (Kazak *et al.*, 2004), 2.0944 (14) in diaquabis(1*H*-imidazole-*κN*^{3})bis(4-nitrobenzoato-*κO*)cobalt(II) (Xu & Xu, 2004) and

2.159 (2) Å in diaquadiformatodipyridinecobalt(II) (Zhu *et al.*, 2004). The Co—N bond lengths are almost equal to the value of 2.008 (3) Å observed in *trans*-diaquabis(pyridine-4-carboxylato)cobalt(II)dihydrate (Yan, 2004).

The Co—Cl bond lengths of 2.2423 (6) and 2.5616 (6) Å in (I) are nearly equal to the corresponding lengths reported for other compounds, *viz*. 2.2525 (8) Å in quinolinium trichloro(quinoline-κ*N*)cobaltate(II) (Pan & Xu, 2004) and 2.236 (1) Å in dichlorobis(1-propylimidazolidine-2-thione-κ*S*)cobalt(II) (Castro *et al.*, 2002), but shorter than the value of 2.391 (1) Å observed in aquachlorobis(1,10-phenanthroline)-cobalt(II)chloride dimethyl formamide solvate (Liu *et al.*, 2004).

The dihedral angle between the least-squares planes through the two benzimidazole ring systems is 81.78 (8)°. In the crystal structure of (I), individual molecules are loosely associated into pairs *via* C—H···Cl and C—H···O hydrogen-bonding interactions (Fig. 2 and Table 2).

Experimental

1-(2-Ethoxyethyl)benzimidazole was synthesized from benzimidazole and 2-chloroethyl ethyl ether according to the literature procedure of Küçükbay *et al.* (2001). A mixture of 1-(2-ethoxyethyl)benzimidazole (0.4 g, 2.10 mmol) and cobalt dichloride (0.27 g, 2.10 mmol) in ethanol (25 ml) was heated under reflux for 4 h. All volatiles were removed *in vacuo* (0.02 mm Hg; 1 mm Hg = 133.322 Pa). The crude product, (I), was crystallized from an ethanol–propan-2-ol (3:1) mixture upon cooling to 243 K (yield 0.93 g, 86%; m.p. 434–435 K). ^{1}H NMR (300 MHz, CDCl$_3$, p.p.m.): 1.1 (*t*, CH_3CH_2—O, 6H), 3.3 (*q*, CH_3CH_2—O, 4H), 3.7 (*s*, NCH_2CH_2—O, 4H), 3.9 (*t*, NCH_2CH_2—O, 4H), 7.8–8.2 (*m*, Ar-H, 8H), 8.3 (*s*, 2-CH, 2H). Analysis, calculated for $C_{22}H_{28}N_4O_2CoCl_2$: C 51.66, H 5.48, N 10.96%; found: C 51.17, H 5.43, N 10.91%.

Crystal data

$[CoCl_2(C_{11}H_{14}N_2O)_2]$
$M_r = 510.31$
Orthorhombic, $Pca2_1$
$a = 17.5842\ (17)$ Å
$b = 7.6235\ (8)$ Å
$c = 17.8892\ (13)$ Å
$V = 2398.1\ (4)$ Å^3
$Z = 4$
$D_x = 1.413$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 5762 reflections
$\theta = 1.6$–$28.4°$
$\mu = 0.96$ mm^{-1}
$T = 150$ K
Prism, violet
0.64 × 0.50 × 0.41 mm

Data collection

Stoe IPDS II diffractometer
ω scans
Absorption correction: by integration (*X-RED*32; Stoe & Cie, 2002)
$T_{min} = 0.577$, $T_{max} = 0.693$
15 539 measured reflections
5762 independent reflections
5101 reflections with $I > 2\sigma(I)$
$R_{int} = 0.031$
$\theta_{max} = 28.3°$
$h = -23 \rightarrow 23$
$k = -10 \rightarrow 10$
$l = -23 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.073$
$S = 0.98$
5762 reflections
280 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0489P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.50$ e Å^{-3}
$\Delta\rho_{min} = -0.32$ e Å^{-3}
Absolute structure: Flack (1983), with 2688 Friedel pairs
Flack parameter = 0.000 (1)

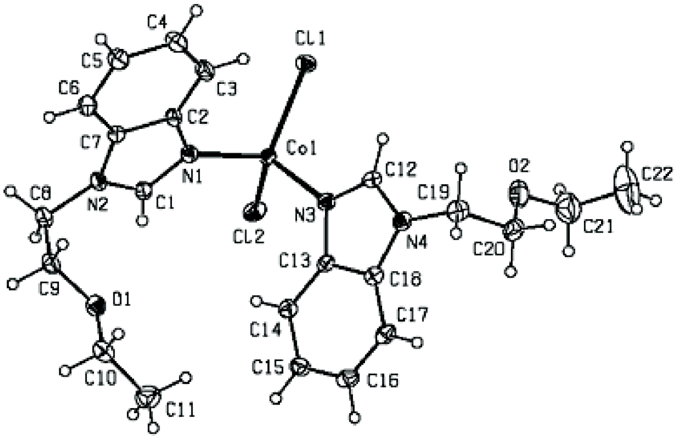


Figure 1
A view of the molecule of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 20% probability level and H atoms are shown as small spheres of arbitrary radii.

Table 1
Selected geometric parameters (Å, °).

Co1—Cl1	2.2423 (6)	N1—C2	1.392 (3)
Co1—Cl2	2.2516 (6)	N2—C1	1.343 (3)
Co1—N1	2.014 (2)	N2—C7	1.383 (3)
Co1—N3	2.0021 (16)	N2—C8	1.456 (3)
O1—C9	1.417 (3)	N3—C12	1.307 (3)
O1—C10	1.433 (3)	N3—C13	1.394 (2)
O2—C20	1.395 (4)	N4—C12	1.345 (3)
O2—C21	1.447 (4)	N4—C18	1.387 (3)
N1—C1	1.315 (3)	N4—C19	1.455 (3)
Cl1—Co1—Cl2	119.84 (2)	C12—N4—C19	125.62 (19)
Cl1—Co1—N1	108.89 (5)	C18—N4—C19	127.26 (19)
Cl1—Co1—N3	104.61 (5)	N1—C1—N2	113.1 (2)
Cl2—Co1—N1	108.86 (5)	N1—C2—C3	131.1 (2)
Cl2—Co1—N3	107.27 (5)	N1—C2—C7	108.79 (19)
N1—Co1—N3	106.57 (7)	N2—C7—C2	105.5 (2)
C9—O1—C10	111.09 (18)	N2—C7—C6	131.7 (2)
C20—O2—C21	116.5 (3)	N2—C8—C9	111.8 (2)
Co1—N1—C1	125.78 (15)	O1—C9—C8	108.63 (19)
Co1—N1—C2	128.56 (14)	O1—C10—C11	108.8 (2)
C1—N1—C2	105.45 (19)	N3—C12—N4	113.03 (19)
C1—N2—C7	107.2 (2)	N3—C13—C14	130.84 (19)
C1—N2—C8	126.5 (2)	N3—C13—C18	108.27 (19)
C7—N2—C8	126.1 (2)	N4—C18—C13	105.75 (18)
Co1—N3—C12	127.09 (14)	N4—C18—C17	132.3 (2)
Co1—N3—C13	126.85 (14)	N4—C19—C20	112.2 (2)
C12—N3—C13	106.04 (17)	O2—C20—C19	108.9 (2)
C12—N4—C18	106.91 (17)	O2—C21—C22	116.3 (4)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C16—H16···Cl2^{i}	0.93	2.80	3.591 (3)	144
C17—H17···Cl1^{ii}	0.93	2.80	3.717 (2)	168
C20—H20*A*···O1^{iii}	0.97	2.51	3.356 (3)	145

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

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

Acta Cryst. (2004). E**60**, m1220–m1222 Sevim Türktekin *et al.* • $[CoCl_2(C_{11}H_{14}N_2O)_2]$ **m1221**

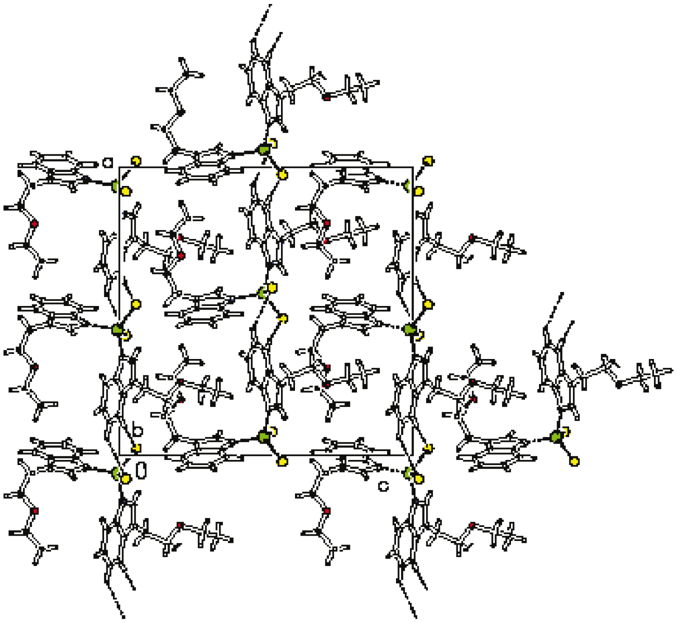


Figure 2
A view of the packing and hydrogen bonds (dashed lines) of (I).

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: *SHELXS*97 (Sheldrick, 1997); 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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