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Clemizoledichlorocobalt(II)

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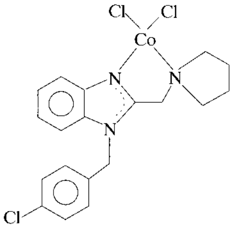
The structure of dichloro[1-(*p*-chlorobenzyl)-2-(1-pyrrolidinylmethyl-*N*)-1,3-benzimidazole-N^3]cobalt(II), [$CoCl_2(C_{19}H_{20}ClN_3)$], contains a molecule of clemizole bound in a bidentate manner to cobalt through its imidazole and pyrrolidinyl N atoms, with significantly different Co—N distances of 1.976 (5) and 2.126 (5) Å, respectively. The geometry around cobalt is distorted tetrahedral, with significantly different Co—Cl distances of 2.217 (2) and 2.233 (2) Å, and the pyrrolidinyl ring is disordered.

Comment

The crystal structures of clemizole hydrochloride (Parvez, 1996), clemizoledichlorozinc(II) (Parvez & Sabir, 1996*a*), clemizole as a free base (Parvez & Sabir, 1996*b*), clemizole tetrachlorocuprate(II) and clemizole tetrachlorocobaltate(II) (Parvez & Sabir, 1997) have been reported from our laboratory. This H_1 antihistamine has now been incorporated into a cobalt complex to investigate further the effects such complex formation may have on the conformation of the clemizole moiety. In this paper, we report the structure of clemizoledichlorocobalt(II), (I).



(I)

In (I) (Fig. 1), clemizole is coordinated in a bidentate manner to cobalt *via* imidazole and pyrrolidinyl N atoms with significantly different Co—N distances of 1.976 (5) and 2.126 (5) Å, respectively. The Co atom is also bound to two Cl atoms with Co—Cl distances of 2.217 (2) and 2.233 (2) Å. The geometry around cobalt is distorted tetrahedral. Similar distances and geometry have been reported for a number of closely related cobalt complexes, *e.g.* dichlorobis(2-methoxypyridine)cobalt(II) (Allan *et al.*, 1981), bis(1,3-benzothiazole)dichlorocobalt(II) (Oughtred *et al.*, 1982), dichlorobis[2-(4-thiazolyl)-1*H*-benzimidazole-*N*,*N*′]cobalt(II) monohydrate (Umadevi *et al.*, 1995), dichloro[phenylbis(2-pyridyl)phosphine-*N*,*N*′]cobalt(II) ethanol solvate (Ehrlich *et al.*, 1984) and bis(*N*-2-propylsalicylideneamino-*N*,*O*)cobalt(II) (Elerman *et al.*, 1996).

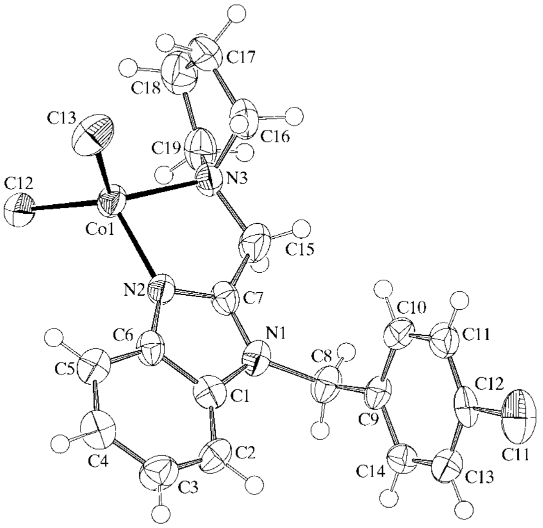


Figure 1
*ORTEP*II (Johnson, 1976) drawing of (I) with displacement ellipsoids plotted at the 50% probability level. The minor fraction of the disordered pyrrolidinyl ring atoms are not shown.

The molecular dimensions in the benzimidazole part of the ligand are normal and the pyrrolidinyl ring is disordered. The important bond distances are: Csp^2—Cl = 1.747 (7) Å and mean Csp^3—Csp^2 = 1.51 (2), C—$C_{aromatic}$ = 1.383 (11), N—Csp^2 = 1.390 (4) and N—Csp^3 = 1.482 (3) Å, not including the disordered pyrrolidinyl ring. It is interesting to note that the N1—C7 bond [1.340 (7) Å] is much shorter than expected for a single bond and that the N2—C7 bond [1.337 (7) Å] is much longer than a double bond; these bonds must represent a delocalized system with a mean N—C distance of 1.339 (2) Å; the corresponding distances in the structure of clemizoledichlorozinc(II) (Parvez & Sabir, 1996*a*) were 1.357 (5) and 1.321 (5) Å, respectively. The pyrrolidinyl ring in (I) is disordered over two sites with inequivalent site-occupancy factors and its N—C and C—C distances were therefore constrained at 1.48 (1) and 1.50 (1) Å, respectively, during refinement. The separations between pairs of C atoms of the pyrrolidinyl ring are in the range 0.47–0.77 Å.

The benzimidazole and phenyl rings are essentially planar, with the maximum deviations of atoms from the least-squares planes being 0.013 (6) and 0.021 (4) Å, respectively. The dihedral angle between these planes is 86.2 (2)°; the corresponding angle in the structures of clemizoledichlorozinc, clemizole hydrochloride, clemizole as a free base, clemizole tetrachlorocuperate(II) and clemizole tetrachlorocobaltate(II) are 81.1 (4) (Parvez & Sabir, 1996*a*), 82.8 (9) (Parvez, 1996), 68.8 (6) (Parvez & Sabir, 1996*b*), 70.5 (6) and 82.7 (9)° (Parvez & Sabir, 1997), respectively. An examination of the N1—C8—C9—C14 torsion angle in these complex molecules shows that the orientation of the phenyl ring with respect to the imidazole ring in (I) [torsion angle 115.0 (16)°] is somewhat similar to the orientation of these rings in the free base (torsion angle 125°) and clemizole tetrachlorocobaltate(II) (torsion angle 116°); the rest of the structures have torsion angles in the range of 133–153°. The major and minor conformers of the disordered pyrrolidinyl ring have an N3-envelope conformation with N3 0.62 (5) and 0.64 (5) Å out of the planes composed of the C16–C19 and C16′–C19′ atoms, respectively; a similar conformation of the five-membered ring has been observed in the crystal structures mentioned above.

The structure of (I) is composed of independent complex molecules separated by normal van der Waals distances and is devoid of any intermolecular interactions. The imidazole moieties of the molecules, which lie about inversion centers, are stacked parallel to each other.

Table 1
Selected geometric parameters (Å, °).

Co1—N2	1.976 (5)	N2—C7	1.337 (7)
Co1—N3	2.126 (5)	N2—C6	1.386 (7)
Co1—Cl3	2.217 (2)	N3—C19′	1.482 (10)
Co1—Cl2	2.233 (2)	N3—C16′	1.485 (10)
Cl1—C12	1.747 (7)	N3—C15	1.484 (8)
N1—C7	1.340 (7)	N3—C19	1.489 (7)
N1—C1	1.394 (7)	N3—C16	1.503 (7)
N1—C8	1.479 (7)		
N2—Co1—N3	83.34 (18)	C7—N2—C6	105.6 (5)
N2—Co1—Cl3	109.35 (16)	C7—N2—Co1	114.7 (4)
N3—Co1—Cl3	115.01 (15)	C6—N2—Co1	139.7 (4)
N2—Co1—Cl2	118.05 (16)	C15—N3—C19	114.5 (6)
N3—Co1—Cl2	111.27 (15)	C15—N3—C16	109.5 (6)
Cl3—Co1—Cl2	115.79 (8)	C19—N3—C16	101.7 (6)
C7—N1—C1	107.2 (5)	C15—N3—Co1	108.9 (3)
C7—N1—C8	126.7 (5)	C19—N3—Co1	115.0 (5)
C1—N1—C8	126.2 (5)	C16—N3—Co1	106.7 (5)

Experimental

The title compound was synthesized by adding 1.0 mmol of $CoCl_2\cdot 6H_2O$ to 2.0 mmol of clemizole hydrochloride in 20 ml of ethanol. NH_4OH was added until the pH was 8–9. The solution was evaporated slowly at room temperature and blue prismatic crystals separated after a few days.

Crystal data

$[CoCl_2(C_{19}H_{20}ClN_3)]$
$M_r = 455.66$
Monoclinic, $P2_1/n$
$a = 9.647$ (6) Å
$b = 12.675$ (6) Å
$c = 16.359$ (5) Å
$\beta = 93.77$ (4)°
$V = 1996$ (2) Å^3
$Z = 4$
$D_x = 1.516$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 10$–25°
$\mu = 1.27$ mm^{-1}
$T = 170$ (2) K
Prismatic, blue
0.40 × 0.22 × 0.16 mm

Data collection

Rigaku AFC-6*S* diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical ψ scan (3 reflections; North *et al.*, 1968)
$T_{min} = 0.63$, $T_{max} = 0.82$
3754 measured reflections
3533 independent reflections
1425 reflections with $I > 2\sigma(I)$
$R_{int} = 0.097$
$\theta_{max} = 25°$
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 15$
$l = -19 \rightarrow 19$
3 standard reflections every 200 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.136$
$S = 0.979$
3533 reflections
251 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0384P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.40$ e Å^{-3}
$\Delta\rho_{min} = -0.44$ e Å^{-3}

The C atoms of the pyrrolidinyl ring were disordered with inequivalent site-occupancy factors of 0.76 and 0.24 which were established in the early refinement cycles. In the final rounds of calculations, the site-occupancy factors of these C atoms were fixed and the atoms of the minor conformer were refined with isotropic displacement parameters; the N—C and C—C distances in the pyrrolidinyl ring were fixed at 1.48 (1) and 1.50 (1) Å, respectively, using the command *DFIX* in *SHELXL*97 (Sheldrick, 1997). H atoms were included in the refinement at idealized positions with aromatic and methylene C—H distances of 0.95 and 0.99 Å, respectively. The isotropic displacement parameters of the H atoms were set equal to $1.2U_{eq}$ of the C atoms to which they were bonded.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1994); program(s) used to solve structure: *SAPI*91 (Fan, 1991); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *TEXSAN*; software used to prepare material for publication: *SHELXL*97.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FR1273). Services for accessing these data are described at the back of the journal.

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