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Trichlorido(1-ethylpiperazin-1-ium)-cobalt(II)

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Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.003 Å;
R factor = 0.022; wR factor = 0.050; data-to-parameter ratio = 20.3.

In the title complex, [Co(C6H15N2)Cl3], the Co2+ ion is coordinated in a distorted tetrahedral fashion by three chloride ions and one N atom of the piperazine ring; the ring adopts a chair conformation with the N—Co and N—C Et bonds in equatorial orientations. In the crystal, molecules are connected by N—H···Cl hydrogen bonds, generating (10T) sheets.

Related literature

For related structures, see: Ciccarese et al. (1998); Clemente et al. (1999); Marzotto et al. (2000).

Experimental

Crystal data

[Co(C6H15N2)Cl3]

M = 280.49

Monoclinic, P21/n

a = 7.421 (3) Å

b = 18.160 (7) Å

β = 90.524 (7)°

V = 1171.1 (8) Å³

Z = 4

Mo Ka radiation

μ = 2.10 mm⁻¹

T = 173 K

0.32 × 0.13 × 0.13 mm

Data collection

Rigaku XtaLAB mini diffractometer

2399 independent reflections

Absorption correction: multi-scan

2099 reflections with F² > 2σ(F²)

Tmin = 0.529, Tmax = 0.761

wR[F³] = 0.050

H atoms treated by a mixture of independent and constrained refinement

Δρmax = 0.27 e Å⁻³

Δρmin = −0.27 e Å⁻³

Table 1

Selected bond lengths (Å).

Co1—Cl1 2.2720 (8) Co1—C12 2.2419 (10)

Co1—Cl2 2.2419 (10) Co1—N1 2.0686 (15)

Co1—Cl3 2.2691 (8) N1—H1 0.83 (2)

Table 2

Hydrogen-bond geometry (Å, °).

N2···H2 — Cl1i 0.854 (19) 2.347 (18) 3.1794 (16) 165.4 (15)

N1—H1···Cl3ii 0.83 (2) 2.49 (2) 3.3192 (17) 176.0 (17)

Symmetry codes: (i) x, y + 1, z; (ii) −x + 1, −y, −z.

Data collection: CrystalClear-SM Expert (Rigaku, 2011); cell refinement: CrystalClear-SM Expert; data reduction: CrystalClear-SM Expert; program(s) used to solve structure: SIR2004 (Burla et al., 2005); program(s) used to refine structure: SHELXL97 (Sheldrick, 1998); molecular graphics: ORTEP-3 for Windows (Farrugia, 2012); software used to prepare material for publication: CrystalStructure (Rigaku, 2010).

We acknowledge the NSF—MRI grant No. 1125975 "MRI Consortium Acquisition of a Single Crystal X-ray Diffractometer for a Regional PUI Molecular Structure Facility".

Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7215).

References


supplementary materials

Supplementary  materials


Trichlorido(1-ethylpiperazin-1-ium)cobalt(II)

Abdelhamid Chiheb Dhieb, Daron E. Janzen, Mohamed Rzaigui and Wajda Smirani Sta

1. Comment
Piperazine (H₂ppz) and its derivatives, are cyclic diamines possessing a non-planar six-membered ring analogous to cyclohexane with two basic nitrogen atoms in the 1,4 positions. As these nitrogen atoms are basic, piperazine (H₂ppz) and its derivatives may coordinate metal ions as monodentate, bidentate or bidentate–chelate ligands. Several 1-methylpiperazine and 1,4-di- methylpiperazine platinum(II) complexes have been synthesized and characterized through X-ray diffraction (Ciccarese et al., 1998). The cobalt complexes [CoCl₃(HMe₂ppz)] and [CoCl₃(H₂Meppz)] have shown interesting cytotoxic activity on human colon and carcinoma cells (Marzotto et al., 2000). In order to explore possible biological applications and to gather further chemical and structural information on metal complexes capable of interacting selectively with nitrogen donors of DNA nucleobases, we have extended to cobalt(II) the study on the behaviour and coordinating properties of piperazine derivatives. This study includes the synthesis and structural characterization of the new complex, (C₆H₁₅N₂)CoCl₃ (I).

Complex I is present as a neutral zwitterionic (amphiionic) species. The negative charge of the –CoCl₃ group is balanced by the positive charge at the ethylated N₂ nitrogen atom of the 1-ethylpiperazin-1-ium ring. Zwitterionic complexes having the formula MX₃LH (where M = Mn(II), Fe(II), Co(II), Ni(II), Cu(II), Zn(II); X = Cl, Br, I; LH = monoprotonated diamine), although rare, are already known. The six-membered piperazine ring in complex I possesses the characteristic chair conformation. The average of the Co(II)–Cl bond lengths, 2.261 Å, falls in the expected range. It is noteworthy that this Co(II)–Cl distance increases on increasing the number of chloride ions bonded to cobalt(II) atom, but also increases when the chloride ions are involved in strong hydrogen bonding. In the structure of I, the Co–Cl₁, 2.2720 (8) Å, and Co–Cl₃, 2.2691 (8) Å, distances are significantly longer than the Co–Cl₂ distance, 2.2419 (10) Å, as the former are involved in strong hydrogen bonding while the latter is not. The Co(II)–N₁ distance, 2.0686 (15) Å, is typical (Clemente et al., 1999).

The structure consists of a neutral Co(II) complex with a 4-Etppz ligand bonded through the secondary nitrogen atom and three chloride ligands. The tertiary nitrogen atom is protonated balancing the overall charge (Fig. 1). Each complex undergoes two hydrogen bonding interactions as donors (N₁—H₁ and N₂—H₂) and two interactions as acceptors (Cl₁ and Cl₃). Hydrogen bonding occurs in two-dimensional sheets (Fig. 2). Dimer donor-acceptor hydrogen-bonding interactions (related by inversion) dominate the hydrogen bonding motif (R₂:8) with additional single donor-acceptor hydrogen bonding interaction joining the dimer interactions. The same hydrogen-bonding pattern is found in the structure of the related [CoCl₃(H₂Meppz)] complex (Clemente et al., 1999).

2. Experimental
The complex was synthesized by adding dropwise under stirring a blue-violet solution, previously prepared at 40°C, of anhydrous CoCl₂ in 10 ml EtOH, to a 1-ethylpiperazine (HEtppz) solution dissolved in 5 ml EtOH in a molar ratio 1:1. After mixing, an aqueous hydrochloric acid solution was added to the blue powder compound and was stirred for 2 h.
After filtration, the filtrate was allowed to stand at room temperature. Blue crystals were obtained by slow evaporation.

3. Refinement

Many hydrogen atoms were treated in calculated positions and refined in the model as riding with distances of C—H = 0.98 and 0.99 Å for the methyl and methylene groups, respectively, and with $U_{iso}(H) = k \times U_{eq}(C)$, $k = 1.2$. Hydrogen atoms H1 and H2 were located in the electron density map, and their positions were refined with $U_{iso}(H) = k \times U_{eq}(C)$, $k = 1.2$.

Figure 1

*ORTEP*-3 view of (C$_6$H$_{15}$N$_2$)CoCl$_3$ with displacement ellipsoids for non-H atoms drawn at the 30% probability level.
Figure 2
Perspective view of the crystal packing of the title complex. Hydrogen bonds are shown as dashed lines.

Trichlorido(1-ethylpiperazin-1-ium)cobalt(II)

Crystal data

\[\text{[Co(C}_6\text{H}_{15}\text{N}_2\text{)Cl}_3]\]

\[F(000) = 572.00\]

\[M_r = 280.49\]

Monoclinic, \(P 2_1/n\)

Hall symbol: -\(P 2yn\)

\(a = 7.421 (3) \text{ Å}\)

\(b = 18.160 (7) \text{ Å}\)

\(c = 8.691 (4) \text{ Å}\)

\(\beta = 90.524 (7)^\circ\)

\(V = 1171.1 (8) \text{ Å}^3\)

\(Z = 4\)

Data collection

Rigaku XtaLAB mini
diffractometer

Detector resolution: 6.849 pixels mm\(^{-1}\)

\(\omega\) scans

Absorption correction: multi-scan

\(\text{(REQAB; Rigaku, 1998)}\)

\(T_{\text{min}} = 0.529, T_{\text{max}} = 0.761\)

11015 measured reflections

2399 independent reflections

2099 reflections with \(F^2 > 2\sigma(F^2)\)

\(R_{\text{int}} = 0.025\)

\(\theta_{\text{max}} = 26.4^\circ\)

\(h = -9 \rightarrow 9\)

\(k = -22 \rightarrow 22\)

\(l = -10 \rightarrow 10\)
supplementary materials

Refinement

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.022$
$wR(F^2) = 0.050$
$S = 1.08$
2399 reflections
118 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 atoms treated by a mixture of independent and constrained refinement

$w = \frac{1}{\sigma^2(F_o^2) + (0.0195P)^2 + 0.4P}$

$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.27 \text{ e Å}^{-3}$
$\Delta\rho_{min} = -0.27 \text{ e Å}^{-3}$

Special details

Geometry. ENTER SPECIAL DETAILS OF THE MOLECULAR GEOMETRY

Refinement. Refinement was performed using all reflections. The weighted $R$-factor ($wR$) and goodness of fit ($S$) are based on $F^2$. $R$-factor (gt) are based on $F$. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating $R$-factor (gt).

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{Å}^2$)

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Atomic displacement parameters (Å²)

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Geometric parameters (Å, °)

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<td>N1—C4—H4A</td>
<td>108.972</td>
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<tr>
<td>N1—C1—C2</td>
<td>112.39 (14)</td>
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<td>108.974</td>
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<td>N2—C2—C1</td>
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<td>N2—C3—C4</td>
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<td>N1—C4—C3</td>
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<td>N2—C5—H5A</td>
<td>108.969</td>
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Co1—N1—H1 107.3 (13)  N2—C5—H5B 108.974
C1—N1—H1 105.7 (14)  C6—C5—H5A 108.963
C4—N1—H1 106.6 (14)  C6—C5—H5B 108.971
C2—N2—H2 107.2 (12)  H5A—C5—H5B 107.768
C3—N2—H2 108.2 (12)  C5—C6—H6A 109.474
C5—N2—H2 107.0 (11)  C5—C6—H6B 109.466
N1—C1—H1A 109.127  C5—C6—H6C 109.477
N1—C1—H1B 109.120  H6A—C6—H6B 109.472
C2—C1—H1A 109.128  H6A—C6—H6C 109.470
C2—C1—H1B 109.129  H6B—C6—H6C 109.468

C1—Co1—N1—C1 55.30 (9)  C4—N1—C1—C2 −55.55 (16)
C1—Co1—N1—C4 −70.78 (9)  C2—N2—C3—C4 54.82 (16)
C2—Co1—N1—C1 −65.66 (8)  C3—N2—C2—C1 −55.64 (16)
C2—Co1—N1—C4 168.27 (7)  C3—N2—C5—C6 −174.02 (12)
C3—Co1—N1—C1 173.39 (7)  C5—N2—C6—H6C 179.64 (12)
C3—Co1—N1—C4 47.31 (9)  C3—N2—C6—H6B 56.87 (17)
Co1—N1—C1—C2 176.93 (8)  C5—N2—C6—H6A −55.84 (18)
Co1—N1—C4—C3 −176.03 (9)  N1—C1—C2—N2 56.87 (17)
C1—N1—C4—C3 55.22 (17)

$\text{Hydrogen-bond geometry (Å, °)}$

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<td>N1—H1···Cl3ii</td>
<td>0.83 (2)</td>
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<td>176.0 (17)</td>
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Symmetry codes: (i) $x+1/2, -y+1/2, z+1/2$; (ii) $-x+1, -y, -z$. 

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