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Astemizole tetrachlorocobaltate(II)

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Key indicators

Single-crystal X-ray study
T = 170 K
Mean σ(C–C) = 0.009 Å
Disorder in main residue
R factor = 0.065
wR factor = 0.157
Data-to-parameter ratio = 14.3

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

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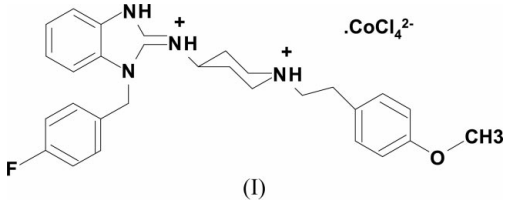
The structure of {3-[(4-fluorophenyl)methyl]-1*H*-benzimidazol-2-ylidene}{1-[2-(4-methoxyphenyl)ethyl]-4-piperidin-1-io}ammonium tetrachlorocobaltate(II), $(C_{28}H_{33}FN_4O)[CoCl_4]$, is isomorphous with its $CuCl_4^{2-}$ analogue. It contains diprotonated cations of astemizole hydrogen-bonded to three Cl atoms in two different $CoCl_4^{2-}$ anions, with Cl···N distances in the range 3.109 (5)–3.212 (5) Å. One of the Cl atoms in the $CoCl_4^{2-}$ anion is disordered and the geometry around cobalt is distorted tetrahedral. The phenylethyl C atoms of the (4-methoxyphenyl)ethyl group attached to the piperidine ring are disordered, indicating the presence of two conformers in the crystal.

Comment

Astemizole is a potent antihistaminic drug with long-lasting effectiveness and very little or no drowsiness effect (Casy, 1991). Continued interest in the effects of anionic transition metal complexes on the conformation of antihistamines effective on H_1 receptors led to the preparation and crystal structure determination of astemizole tetrachlorocuprate(II) (Parvez & Braitenbach, 2000). In this paper, the structure of astemizole tetrachlorocobaltate(II), (I), is reported; it is isomorphous with the tetrachlorocuprate salt.



(I)

The structure is composed of diprotonated astemizole hydrogen-bonded to the tetrachlorocobaltate(II) anion (Fig. 1). The molecular dimensions in the dication are normal and agree well with the corresponding dimensions reported for astemizole tetrachlorocuprate(II) (Parvez & Braitenbach, 2000) and astemizole (Peeters *et al.*, 1995). As in the case of astemizole tetrachlorocuprate(II) (Parvez & Braitenbach, 2000), the phenylethyl C atoms are disordered over C20–C27 and C20′–C27′, indicating the presence of two conformers. The methoxy and the piperidinyl groups are not affected by this disorder. The mean plane of the methoxy group is inclined at 25.5 (4) and 7.6 (6)° to the benzene rings C22–C27 and C22′–C27′, respectively. The dihedral angle between the mean planes of these benzene rings is 30.8 (3)°. The benzimidazole and fluorophenyl rings are essentially planar and the maximum deviations of atoms from the least-squares planes are 0.026 (4) and 0.013 (4) Å, respectively. The dihedral angle

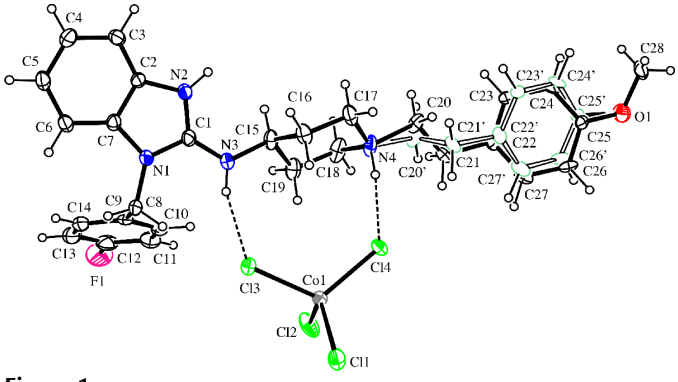


Figure 1
*ORTEP*II (Johnson, 1976) drawing of the two conformers of (I), with displacement ellipsoids plotted at the 30% probability level; atoms C20′–C27′ are shown in green.

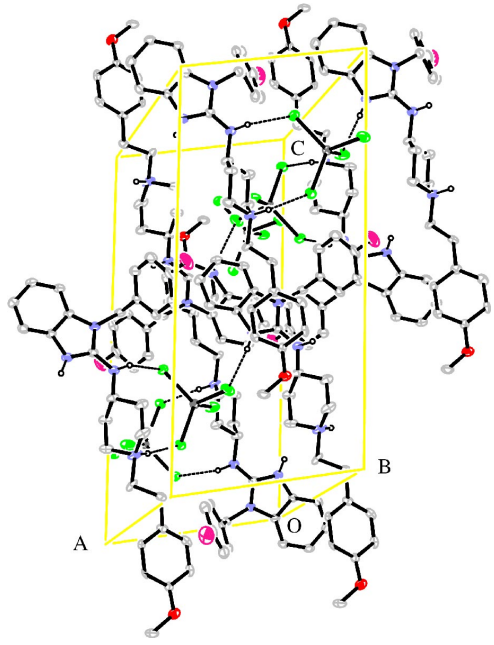


Figure 2
*ORTEP*II (Johnson, 1976) drawing of the contents of a unit cell, showing the hydrogen bonding in (I).

between these planes is 68.04 (11)°. The piperidinyl ring adopts a classical chair conformation with puckering parameters (Cremer & Pople, 1975) Q = 0.582 (6) Å, θ = 179.2 (6)° and φ = 220 (22)°.

The $CoCl_4^{2-}$ anion exhibits a distorted tetrahedral geometry wherein Cl1 is disordered over two sites Cl1 and Cl1′ with unequal site-occupancy factors. The hydrogen bonding in (I) is essentially identical to that observed in the isomorphous structure of astemizole tetrachlorocuprate(II) (Parvez & Braitenbach, 2000). The details of the hydrogen-bonding geometry in (I) are given in Table 2.

Experimental

The preparation and crystallization of (I) was achieved following the procedure reported for astemizole tetrachlorocuprate(II) (Parvez & Braitenbach, 2000), resulting in green crystals suitable for crystallographic studies.

Crystal data

$(C_{28}H_{33}FN_4O)[CoCl_4]$	D_x = 1.465 Mg m^{-3}
M_r = 661.31	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 9625 reflections
a = 9.327 (3) Å	θ = 2.5–25.0°
b = 15.836 (6) Å	μ = 0.96 mm^{-1}
c = 20.691 (9) Å	T = 170 (2) K
β = 101.186 (14)°	Prism, blue
V = 2998 (2) Å^3	0.10 × 0.08 × 0.06 mm
Z = 4	

Data collection

Nonius KappaCCD diffractometer	2899 reflections with $I > 2\sigma(I)$
ω and φ scans	R_{int} = 0.084
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)	θ_{max} = 25.0°
T_{min} = 0.91, T_{max} = 0.94	$h = -11 \rightarrow 11$
9625 measured reflections	$k = -17 \rightarrow 18$
5205 independent reflections	$l = -24 \rightarrow 24$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.062P)^2 + 2.13P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.065	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.157	$\Delta\rho_{max}$ = 0.44 e Å^{-3}
S = 1.02	$\Delta\rho_{min}$ = −0.55 e Å^{-3}
5205 reflections	
363 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Co1—Cl1′	2.232 (14)	N1—C7	1.397 (7)
Co1—Cl1	2.240 (8)	N1—C8	1.473 (7)
Co1—Cl2	2.257 (2)	N2—C1	1.333 (7)
Co1—Cl4	2.268 (2)	N2—C2	1.397 (6)
Co1—Cl3	2.300 (2)	N3—C1	1.329 (6)
F1—C12	1.367 (8)	N3—C15	1.459 (7)
O1—C25	1.344 (6)	N4—C20′	1.507 (8)
O1—C25′	1.409 (6)	N4—C17	1.489 (7)
O1—C28	1.418 (7)	N4—C18	1.509 (7)
N1—C1	1.353 (6)	N4—C20	1.515 (8)
Cl1′—Co1—Cl2	126.6 (11)	C1—N1—C7	108.2 (4)
Cl1—Co1—Cl2	109.3 (9)	C1—N1—C8	126.2 (5)
Cl1′—Co1—Cl4	102.9 (9)	C7—N1—C8	125.5 (4)
Cl1—Co1—Cl4	112.0 (3)	C1—N2—C2	109.3 (4)
Cl2—Co1—Cl4	105.68 (7)	C1—N3—C15	124.1 (5)
Cl1′—Co1—Cl3	102.7 (4)	C20′—N4—C17	124.4 (8)
Cl1—Co1—Cl3	109.7 (5)	C20′—N4—C18	103.1 (9)
Cl2—Co1—Cl3	104.21 (7)	C17—N4—C18	110.3 (4)
Cl4—Co1—Cl3	115.42 (7)	C17—N4—C20	101.3 (7)
C25—O1—C28	121.6 (7)	C18—N4—C20	119.1 (10)
C25′—O1—C28	113.8 (7)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···Cl2^{i}	0.88	2.28	3.154 (5)	170
N3—H3···Cl3	0.88	2.42	3.212 (5)	150
N4—H4···Cl4	0.93	2.19	3.109 (5)	171

Symmetry code: (i) $x-1, y, z$.

The phenylethyl C atoms of the (4-methoxyphenyl)ethyl group attached to the piperidine ring are disordered with essentially equal site-occupancy factors. The benzene ring was restrained as a regular hexagon, with C—C distances of 1.39 (1) Å. Atom Cl1 of the anion is

also disordered over two sites Cl1 and Cl1′ with site-occupancy factors 0.65 (6) and 0.35 (6), respectively. Most of the H atoms were located in difference Fourier syntheses and all were included in the refinements at geometrically idealized positions with C—H = 0.95, 0.98 and 0.99 Å, and N—H = 0.88 and 0.93 Å, and with U_{iso}(H) = 1.5 (methyl) and 1.2 (the rest) times the U_{eq} of the atoms to which they were bonded. The final difference map was free of any chemically significant features.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *HKL DENZO* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SAPI*91 (Fan, 1991); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

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