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Acetonitriledichloro(1,4,7-triazacyclodecane)cobalt(III) perchlorate

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In the title compound, $[CoCl_2(C_7H_{17}N_3)(C_2H_3N)]ClO_4$, the Co^{3+} ion is six-coordinated by three N atoms from the 1,4,7-triazacyclodecane (tacd) ligand, two chloride anions and one N atom of the acetonitrile group, giving a distorted octahedral geometry. The cation and anion both have crystallographic mirror symmetry. Intermolecular hydrogen bonds link the ions into a three-dimensional network.

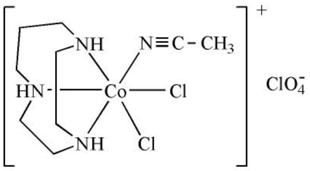
Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.005 Å
R factor = 0.041
wR factor = 0.118
Data-to-parameter ratio = 19.3

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

Comment

Macrocyclic triamines have attracted considerable interest in the synthesis of metal complexes of biological significance. In the past decades, a large number of metal complexes containing this kind of tripodal ligand have been prepared and investigated extensively (Wieghardt *et al.*, 1985; Hotzelmann *et al.*, 1992; Kimura *et al.*, 1990). For those metal complexes, most studies focused on stability and electronic spectra (Yang & Zompa, 1976; Zompa, 1978; McAuley *et al.*, 1984), and the structural data are limited (Hambley & Searle, 1995; Jackson *et al.*, 2004; Anderson *et al.*, 2004). In this paper, we report a new Co^{3+} complex with 1,4,7-triazacyclodecane (tacd), acetonitrile and Cl^- as ligands. Both the cation and the anion are located on a crystallographic mirror plane.



(I)

The structure of the complex consists of $[Co(tacd)(CH_3CN)Cl_2]^+$ cations and ClO_4^- anions (Fig. 1). The Co^{3+} ion is six-coordinate. The *cis* angles around Co^{3+} are in the range 86.89 (10)–93.67 (10)°, indicating a distorted octahedral geometry (Table 1). The Co^{3+} ion lies in the equatorial plane composed of atoms N2, N2^{i}, Cl1 and Cl1^{i} (symmetry codes as in Table 1). The axial bond lengths are significantly shorter than the equatorial bonds lengths (Table 1). It is noteworthy that the Co1—N3 bond length is obviously shorter than those in most Co^{3+} amine complexes (Xie *et al.*, 2005; Amadei *et al.*, 1999; Anderson *et al.*, 2004). The shorter Co—N3 bond distance means a strong coordination of acetonitrile to the Co^{3+} ion. The Co—Cl1 bond distance is unexceptional. In the cation, there is an intramolecular hydrogen bond (N2—H2···Cl1; Table 2). Another two intermolecular hydrogen bonds (N2—H2···O3 and N1—H1···O2) between cations and the perchlorate anions link these ions into a three-dimensional network.

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Experimental

The triamine 1,4,7-triazacyclodecane trihydrochloride (tacd·3HCl) was prepared as described previously (Richman & Atkins, 1974; Koyama & Yoshino, 1972). The other reagents were of analytical grade from commercial sources and were used without any further purification. To an acetonitrile/water (7:3, *v*/*v*) (10 ml) mixture were added $Co(ClO_4)_2 \cdot 6H_2O$ (0.366 g, 1.0 mmol), $NaBF_4$ (0.109 g, 1.0 mmol) and tacd·3HCl (0.248 g, 1 mmol). The mixture was adjusted to pH 7 with a solution of NaOH (2 mol l^{-1}). The solution was filtered and allowed to stand in air at room temperature for several days, giving purple crystals.

Crystal data

$[CoCl_2(C_7H_{17}N_3)(C_2H_3N)]ClO_4$
$M_r = 413.57$
Orthorhombic, *Pnma*
$a = 20.863$ (4) Å
$b = 10.7899$ (19) Å
$c = 7.3630$ (13) Å
$V = 1657.5$ (5) Å^3
$Z = 4$
$D_x = 1.657$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1445 reflections
$\theta = 2.7$–23.6°
$\mu = 1.54$ mm^{-1}
$T = 273$ (2) K
Block, purple
0.21 × 0.10 × 0.03 mm

Data collection

Bruker SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.735$, $T_{max} = 0.955$
10 135 measured reflections
2199 independent reflections
1526 reflections with $I > 2\sigma(I)$
$R_{int} = 0.044$
$\theta_{max} = 28.7°$
$h = -28 \rightarrow 27$
$k = -14 \rightarrow 14$
$l = -9 \rightarrow 7$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.118$
$S = 1.06$
2199 reflections
114 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0583P)^2 + 0.6755P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.80$ e Å^{-3}
$\Delta\rho_{min} = -0.44$ e Å^{-3}

Table 1

Selected geometric parameters (Å, °).

Co1—N3	1.917 (4)	Co1—N2	1.963 (2)
Co1—N1	1.919 (3)	Co1—Cl1	2.2648 (9)
N3—Co1—N1	179.20 (15)	N3—Co1—Cl1	89.22 (8)
N3—Co1—N2	93.67 (10)	N1—Co1—Cl1	90.22 (8)
N1—Co1—N2	86.89 (10)	N2—Co1—Cl1	88.09 (8)
N2^i—Co1—N2	91.41 (14)	N2—Co1—Cl1^i	177.09 (7)

Symmetry code: (i) $x, -y + \frac{3}{2}, z$.

Table 2

Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···Cl1	0.90 (6)	2.45 (7)	2.947 (3)	115 (5)
N2—H2···O3^{ii}	0.90 (6)	2.27 (6)	2.961 (4)	133 (6)
N1—H1···O2^{iii}	0.87 (8)	2.13 (9)	2.893 (6)	145 (8)

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

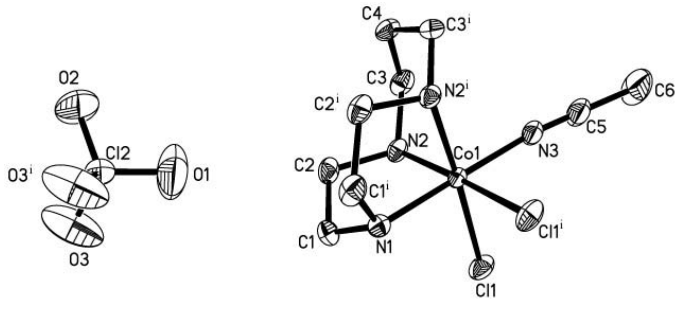

Figure 1
View of the title complex, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. All H atoms have been omitted for clarity. [Symmetry code: (i) $x, -y + \frac{3}{2}, z$].

H atoms on C atoms were treated as riding, with C—H = 0.96 or 0.97 Å, and $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}(C)$. H atoms on N atoms were refined with $U_{iso}(H) = 0.16$ Å^2 (see Table 2 for N—H distances).

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

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