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Bis(2-aminoethylaminium-κN^2)bis(dimethylglyoximato-$\kappa^2 N,N'$)cobalt(III) dichloro(dimethylglyoximato-$\kappa^2 N,N'$)(dimethylglyoxime-$\kappa^2 N,N'$)-cobaltate(III) dichloride 0.75-hydrate

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Key indicators

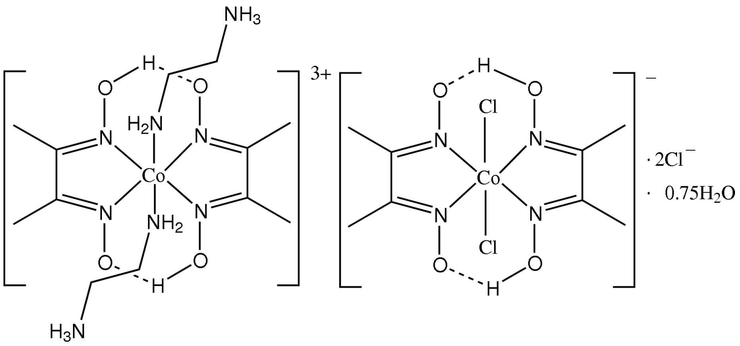
Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.014 Å
Disorder in main residue
R factor = 0.048
wR factor = 0.144
Data-to-parameter ratio = 18.7

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

The title compound, $[Co(C_4H_7N_2O_2)_2(C_2H_9N_2)_2][Co(C_4H_6N_2O_2)Cl_2(C_4H_8N_2O_2)]Cl_2\cdot 0.75H_2O$, contains a Co-centered cation and a Co-centered anion, together with two uncoordinated Cl^- anions and a total of 0.75 uncoordinated water molecules in the asymmetric unit. Both Co atoms are six-coordinated in distorted octahedral configurations, with two dimethylglyoximate and two protonated ethylenediamine ligands attached to the Co atom in the cation; a dimethylglyoximate and a dimethylglyoxime ligand with two Cl^- anions are bonded to the Co atom in the anion. The Co atom belonging to the cation lies on a inversion center, while the anion centered on the other Co atom lies on a mirror plane, as does one of the disordered water molecules.

Comment

In recent years, considerable effort has been dedicated to the design and synthesis of supramolecular architectures of coordination complexes (Lehn, 1995, 1999; Khlobystov *et al.*, 2001). The primary reason for the interest in such complexes is their new and versatile topologies and potential applications in functional materials (Desiraju, 1995; Seo *et al.*, 2000). Dimethylglyoximate is a good ligand with excellent coordination capability to generate mono-, bi- or trinuclear complexes, which are commonly used as precursors for the formation of supramolecular architectures (Chaudhuri *et al.*, 1991; Kubiak *et al.*, 1995; Cervera *et al.*, 1997). As a bidentate flexible ligand, ethylenediamine is also a familiar candidate for the construction of supramolecular complexes with versatile binding modes (You & Zhu, 2004). In the present contribution, we report a complex involving dimethylglyoximate and ethylenediamine ligands, (I).



(I)

As shown in Fig. 1, the title compound, (I), consists of five components, namely a Co-centered cation and a Co-centered anion, together with two uncoordinated chloride anions and

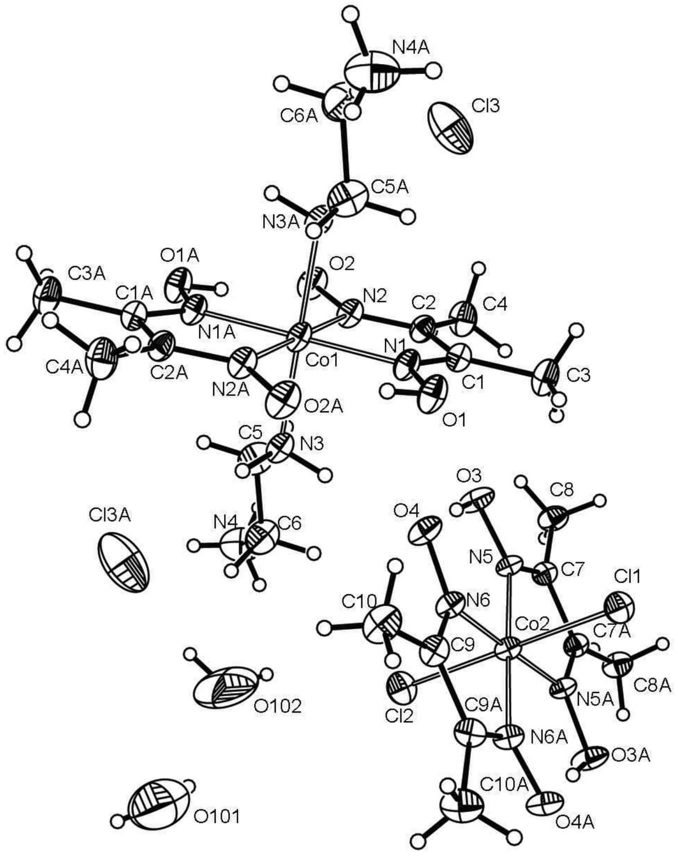


Figure 1

View of the title compound. Displacement ellipsoids are drawn at the 40% probability level. For the cation, as for Cl3, atoms with the suffix A are generated by symmetry code ($\frac{3}{2} - x$, $\frac{3}{2} - y$, $2 - z$). For the anion, atoms with the suffix A are generated by symmetry code (x, $2 - y$, z). Minor disordered components for Cl3 and 2-ammonioethylamine have been omitted for clarity.

0.75 uncoordinated water molecules. Each of the two Co atoms is hexacoordinated with distorted octahedral geometry (Table 1), the Co atom and the four N atoms of dimethylglyoximate or dimethylglyoxime ligands being approximately coplanar. Each ethylenediamine ligand coordinates Co1 through one terminal amino group, while the other uncoordinated amine group is protonated. Besides the dimethylglyoximate and dimethylglyoxime ligands, atom Co2 is coordinated by two chloride anions in a *trans* arrangement. There are strong intramolecular O—H···O hydrogen bonds between dimethylglyoximate and dimethylglyoxime ligands in both cation and anion (Table 2). Both complex ions lie on special positions: the cation on an inversion center and the anion on a mirror plane, as does disordered water molecule O101.

Fig. 2 shows the packing arrangement for the crystal structure of (I).

Experimental

To a 95% ethanol solution (25 ml) containing [$CoCl_2$(dimethylglyoxime)$_2$]H [0.36 g, 1 mmol, synthesized following a known procedure (Jolly, 1968)], an aqueous solution (1 ml) of 1 mol l^{-1} ethylenediamine (1 mmol) was added. After stirring for 1 h, the resulting dark-red solution was filtered and allowed to evaporate at 298 K. After 7 d, dark-red crystals of the title complex suitable for X-ray analysis were obtained [yield: 1.70 g (*ca* 40% based on the cobalt starting material)].

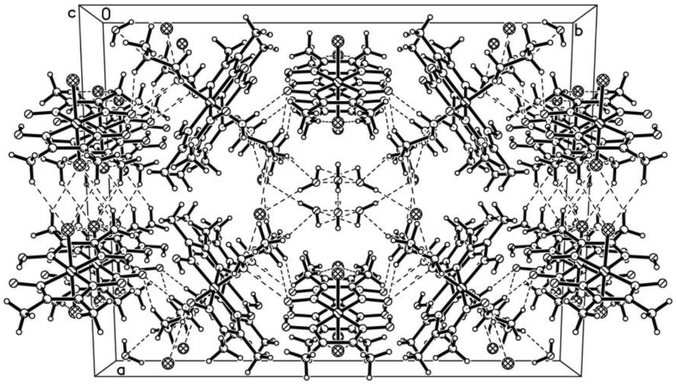


Figure 2

A view of the packing of (I), viewed along the [001] axis. Dashed lines indicate hydrogen bonds.

Crystal data

$[Co(C_4H_7N_2O_2)_2(C_2H_9N_2)_2]$-
$[Co(C_4H_6N_2O_2)Cl_2$-
$(C_4H_8N_2O_2)]Cl_2 \cdot 0.75H_2O$
$M_r = 855.86$
Monoclinic, $C2/m$
$a = 19.531$ (2) Å
$b = 25.500$ (2) Å
$c = 7.8777$ (7) Å
$\beta = 104.847$ (2)°
$V = 3792.4$ (6) Å^3

$Z = 4$
$D_x = 1.499$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4776 reflections
$\theta = 1.6$–$28.3°$
$\mu = 1.21$ mm^{-1}
$T = 295$ (2) K
Block, dark-red
0.33 × 0.25 × 0.21 mm

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{min} = 0.692$, $T_{max} = 0.771$
13650 measured reflections

4774 independent reflections
2557 reflections with $I > 2\sigma(I)$
$R_{int} = 0.067$
$\theta_{max} = 28.3°$
$h = -24 \rightarrow 25$
$k = -33 \rightarrow 30$
$l = -9 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.144$
$S = 1.04$
4774 reflections
255 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0575P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.47$ e Å^{-3}
$\Delta\rho_{min} = -0.43$ e Å^{-3}

Table 1

Selected geometric parameters (Å, °).

Co1—N1	1.892 (7)	Co2—N5	1.906 (7)
Co1—N2	1.900 (7)	Co2—Cl1	2.230 (4)
Co1—N3	1.993 (8)	Co2—Cl2	2.258 (4)
Co2—N6	1.879 (7)		
N1—Co1—N2	81.0 (3)	N5—Co2—Cl1	89.5 (3)
N1—Co1—N3	90.2 (3)	N6—Co2—Cl2	89.8 (3)
N2—Co1—N3	91.6 (3)	N5—Co2—Cl2	90.8 (3)
N6—Co2—N5	99.5 (3)	Cl1—Co2—Cl2	179.59 (16)
N6—Co2—Cl1	89.9 (3)		

m1186 Shen *et al.* • $C_{12}H_{32}CoN_8O_4^{3+} \cdot C_8H_{14}Cl_2CoN_4O_4^{-} \cdot 2Cl^{-}$ *Acta Cryst.* (2005). E**61**, m1185–m1187

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O1—H1$\cdots$O2^{i}	0.82	1.71	2.518 (10)	168
O3—H3$\cdots$O4	0.82	1.70	2.512 (9)	172

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

The water H atoms were located in difference maps and refined with O—H distances restrained to 0.82 (1) Å and fixed isotropic displacement parameters $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(O). In the final cycles, these H atoms were refined using a riding model, with O—H distances constrained to those found in previous cycles. Other H atoms were positioned geometrically and treated as riding on their parent atoms, with $U_{\rm iso}$(H) fixed to $xU_{\rm eq}$(carrier atom). Constrained C—H or N—H distances and x values: methyl CH_3 0.96 Å and x = 1.5; methylene CH_2 0.97 Å and x = 1.2; ammonium NH_3: 0.89 Å and x = 1.5; amine NH_2: 0.90 Å and x = 1.2. The terminal NH_3 group of the 2-ammonioethylamine ligand coordinated to Co1 is disordered over two sites. Corresponding site-occupation factors were refined with their sum constrained to 1, and converged to 0.656 (19) for N4 and 0.344 (19) for N4′. Uncoordinated chloride anion is also disordered over two sites and was refined in the same way; site-occupation factors: 0.58 (4) for Cl3 and 0.42 (4) for Cl3′. The lattice water O atom is disordered over two sites and the sum of the occupancies was fixed and refined.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2002); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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