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trans-Dichloro(1,4,8,11-tetraazacyclotetradecane)-cobalt(III) chloride

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In the structure of the title compound, $[CoCl_2(C_{10}H_{24}N_4)]Cl$, the Co^{III} atom has a six-coordinate arrangement displaying distorted octahedral coordination geometry with the four N atoms from the 1,4,8,11-tetraazacyclotetradecane ring in the equatorial plane and two chloride anions in axial positions. The Co atom is located on a centre of inversion; the two Cl ions not bonded to Co are located on special positions of site symmetry $\bar{4}$.

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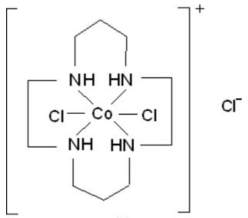
Key indicators

Single-crystal X-ray study
T = 95 K
Mean σ(C–C) = 0.002 Å
R factor = 0.020
wR factor = 0.056
Data-to-parameter ratio = 12.9

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

Comment

We have been interested in the preparation of new polynuclear transition metal complexes with different ligands. Recently, some cobalt(III) complexes with similar ligands have been reported (Tahirov *et al.*, 1994; Chen *et al.*, 1996). Cyclam is a neutral cyclic N_4-donor ligand and therefore it is able to occupy four coordination sites around the central metal atom. This ligand is flexible so that the remaining positions of the central metal atom can be occupied by other ligands in *cis* or *trans* positions (Sosa-Torres & Toscano, 1997; Panneerselvam *et al.*, 1998; Burgess *et al.*, 1999; Funston *et al.*, 2002). The flexibility of cyclam and the ability to block four coordination sites has motivated us to use this ligand in our project on the preparation and study of polynuclear complexes with bridging ligands. The title compound was prepared as a precursor for the follow-up preparation of a dinuclear complex with chloride as a bridging ligand and it is interesting from a structural point of view.



(I)

The molecular structure of the title complex consists of discrete monomeric units with the Co^{III} atom in a distorted octahedral environment coordinated by the neutral cyclam ligand and two chloride anions. The Co atom is located on a centre of inversion; the two Cl ions not bonded to Co are located on special positions of site symmetry $\bar{4}$. These two ions form N—H···Cl hydrogen bonds (Table 2).

Experimental

The reaction of $CoCl_2·6H_2O$ with cyclam in the ratio 1:1 in methanol under 1 h reflux led to the precipitation of $[Co(cyclam)Cl_2]Cl$. In the preparative sequence oxidation of Co^{II} to Co^{III} takes place. Bright-green single crystals were obtained by slow evaporation of a methanol solution in a refridgerator.

Crystal data

$[CoCl_2(C_{10}H_{24}N_4)]Cl$
$M_r = 365.61$
Tetragonal, $P4_2/n$
$a = 10.610$ (1) Å
$c = 13.335$ (1) Å
$V = 1501.1$ (2) Å^3
$Z = 4$

$D_x = 1.618$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 1.67$ mm^{-1}
$T = 95$ (2) K
Prism, green
0.36 × 0.28 × 0.14 mm

Data collection

Oxford Diffraction Xcalibur diffractometer
ω and φ scans
Absorption correction: analytical (*CrysAlis RED*; Oxford Diffraction, 2004)
$T_{min} = 0.585$, $T_{max} = 0.800$

10515 measured reflections
1535 independent reflections
1156 reflections with $I > 2\sigma(I)$
$R_{int} = 0.021$
$\theta_{max} = 26.4°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.020$
$wR(F^2) = 0.057$
$S = 1.05$
1535 reflections
119 parameters

Only H-atom coordinates refined
$w = 1/[\sigma^2(F_o^2) + (0.0348P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.31$ e Å^{-3}
$\Delta\rho_{min} = -0.26$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—Co1	1.9697 (12)	Cl1—Co1	2.2533 (4)
N2—Co1	1.9785 (12)		
N1—Co1—N2	86.23 (5)	N2—Co1—Cl1	91.60 (4)
N1—Co1—Cl1	88.43 (3)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1C···Cl2	0.848 (15)	2.693 (14)	3.3251 (12)	132.5 (12)
N2—H2C···Cl3	0.807 (15)	2.757 (15)	3.3638 (12)	133.6 (13)

The coordinates of the H atoms were refined with U_{iso}(H) values set at $1.2U_{eq}$ of the parent atom [C—H = 0.922 (15)–1.015 (15) Å; N—H as in Table 2]. The structure of the title compound shows pseudo-symmetry. The Co and Cl atoms could be refined in space group $P4_2/m$, transforming the cell using the matrix $(\overline{1/2}\ \overline{1/2}\ 0,\ \overline{1/2}\ 1/2\ 0,\ 0\ 0\ \overline{1})$. The C and N atoms, however, would be disordered in this setting. Therefore, the current cell was chosen.

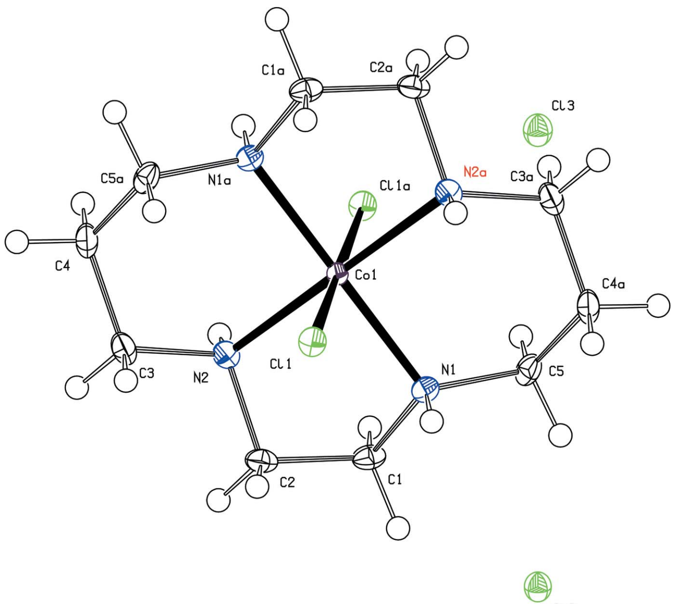


Figure 1
Molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level. Atoms with suffix 'a' are generated by the symmetry operation ($1 - x, -y, 1 - z$).

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2003); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2004; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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