

**[*N*-(2-Aminoethyl)-*N,N*-bis(3-aminopropyl)amine]-dichlorocobalt(III) chloride, [Co(abap)Cl<sub>2</sub>]Cl****Rachel L. Fanshawe, Charles R. Clark† and Allan G. Blackman\***Department of Chemistry, University of Otago,  
PO Box 56, Dunedin, New Zealand† Additional correspondence author:  
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blackman@alkali.otago.ac.nz**Key indicators**

Single-crystal X-ray study

*T* = 173 KMean  $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$ *R* factor = 0.023*wR* factor = 0.051

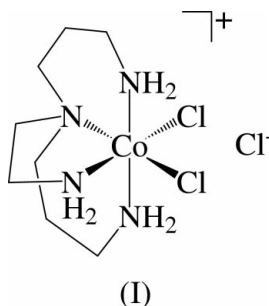
Data-to-parameter ratio = 19.7

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

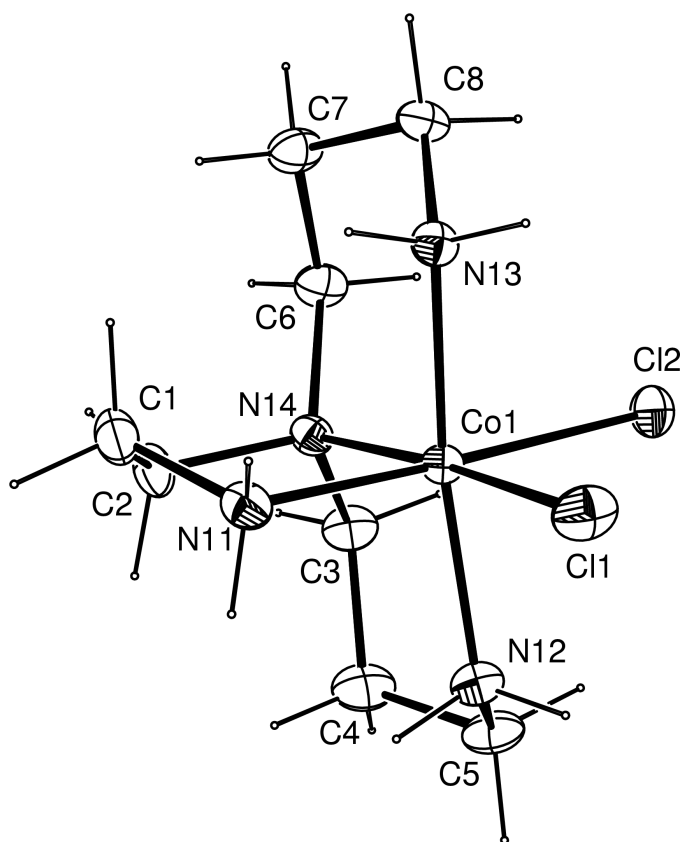
Structural analysis of the title compound, [Co(abap)Cl<sub>2</sub>]Cl, where abap is *N*-(2-aminoethyl)-*N,N*-bis(3-aminopropyl)amine (C<sub>8</sub>H<sub>22</sub>N<sub>4</sub>), confirms that the asymmetric amine ligand is oriented such that the N atoms of the five-membered chelate ring are in the same plane as the chloride ligands.

**Comment**

The asymmetric tetraamine ligand abap [abap is *N*-(2-aminoethyl)-*N,N*-bis(3-aminopropyl)amine] can coordinate to four mutually *cis* sites of a six-coordinate metal complex in two ways. Coordination such that the N atoms of a six-membered chelate ring lie in the same plane as the remaining ligand(s) gives the '6' isomer, as found in the structurally characterized complex [Co(abap)O<sub>2</sub>NO](ClO<sub>4</sub>)<sub>2</sub> (Fanshawe & Blackman, 1995), while the '5' isomer results on coordination of the ligand so that the five-membered chelate ring lies in the same plane as the ancillary ligand(s). The structure of [Co(abap)Cl<sub>2</sub>]Cl, (I), described herein confirms assignment of this complex as the '5' isomer.



The structure consists of a Co<sup>III</sup> ion coordinated to four N atoms of the abap ligand, with coordination completed by two chloride ligands. A single chloride counter-ion balances the charge. Co–N bond lengths are typical of Co<sup>III</sup> complexes, with the bond to the tertiary N atom of the abap ligand being the longest [2.030 (2) Å]. Similarly lengthened Co–N (tertiary) bonds have been observed in [Co(abap)(NO<sub>2</sub>)<sub>2</sub>]X (*X* = Cl<sup>−</sup>, ClO<sub>4</sub><sup>−</sup>·H<sub>2</sub>O, PF<sub>6</sub><sup>−</sup>·H<sub>2</sub>O, I<sup>−</sup>·H<sub>2</sub>O), all of which crystallize as the '5' isomer (Bernal *et al.*, 1996). The six-membered chelate rings adopt chair conformations, while the conformation of the five-membered ring is λ. The bond angles involving the primary N atoms of the six-membered chelate rings (C5–N12–Co1 and C8–N13–Co1) are 122.10 (16) and 121.92 (15)°, respectively. The expansion of these angles, when compared to the corresponding angle in the five-membered chelate ring [C1–N11–Co1 109.89 (15)°] presumably reflects the inherently greater strain present in a six-membered ring having one angle significantly larger than 90°.



**Figure 1**  
The cation of (I) with displacement ellipsoids at the 50% probability level.

## Experimental

[Co(abap)(O<sub>2</sub>NO)](ClO<sub>4</sub>)<sub>2</sub> (1.0 g) was dissolved in water and loaded onto a Dowex 50 W × 2 cation-exchange column. The column was washed with water and 1 M HCl before the complex was eluted with 2 M HCl. The purple eluate was collected and taken to dryness by rotary evaporation to give a blue solid which was crystallized from dilute HCl. X-ray quality crystals were obtained on slow evaporation of a concentrated solution of the product in 5 M HCl.

### Crystal data

[CoCl<sub>2</sub>(C<sub>8</sub>H<sub>22</sub>N<sub>4</sub>)Cl]  
*M<sub>r</sub>* = 339.58  
 Orthorhombic, *P*2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>  
*a* = 8.405 (2) Å  
*b* = 10.2633 (14) Å  
*c* = 15.9652 (15) Å  
*V* = 1377.2 (5) Å<sup>3</sup>  
*Z* = 4  
*D<sub>x</sub>* = 1.638 Mg m<sup>-3</sup>

Mo *K*α radiation  
 Cell parameters from 16 reflections  
 $\theta$  = 5–12.5°  
 $\mu$  = 1.81 mm<sup>-1</sup>  
*T* = 173 (2) K  
 Block, blue  
 0.42 × 0.20 × 0.18 mm

### Data collection

Siemens P4 diffractometer  
 $\omega$  scans  
 Absorption correction:  $\psi$  scan (SHELXTL-Plus; Sheldrick, 1990)  
 $T_{\min}$  = 0.662,  $T_{\max}$  = 0.772  
 3058 measured reflections  
 2850 independent reflections  
 2717 reflections with  $I > 2\sigma(I)$

$R_{\text{int}}$  = 0.017  
 $\theta_{\text{max}}$  = 27°  
 $h$  = -10 → 8  
 $k$  = 0 → 13  
 $l$  = 0 → 20  
 3 standard reflections every 997 reflections  
 intensity decay: <2%

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)]$  = 0.023  
 $wR(F^2)$  = 0.051  
 $S$  = 1.05  
 2850 reflections  
 145 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0258P)^2 + 0.2974P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}}$  = 0.001  
 $\Delta\rho_{\text{max}}$  = 0.25 e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}}$  = -0.18 e Å<sup>-3</sup>  
 Absolute structure: Flack (1983);  
 1110 Friedel pairs  
 Flack parameter = 0.004 (13)

**Table 1**

Selected geometric parameters (Å, °).

Co1–N11	1.9528 (19)	N14–C2	1.508 (3)
Co1–N13	1.9728 (19)	N14–C3	1.516 (3)
Co1–N12	1.982 (2)	N14–C6	1.518 (3)
Co1–N14	2.030 (2)	C1–C2	1.501 (3)
Co1–Cl2	2.2510 (6)	C3–C4	1.520 (3)
Co1–Cl1	2.2835 (8)	C4–C5	1.505 (4)
N11–C1	1.481 (3)	C6–C7	1.510 (3)
N12–C5	1.487 (3)	C7–C8	1.510 (3)
N13–C8	1.482 (3)		
N11–Co1–N13	88.88 (8)	N14–Co1–Cl2	93.33 (6)
N11–Co1–N12	92.78 (8)	N11–Co1–Cl1	87.85 (6)
N13–Co1–N12	169.68 (9)	N13–Co1–Cl1	85.17 (6)
N11–Co1–N14	86.23 (8)	N12–Co1–Cl1	84.71 (6)
N13–Co1–N14	96.15 (8)	N14–Co1–Cl1	173.90 (6)
N12–Co1–N14	94.13 (8)	Cl2–Co1–Cl1	92.64 (3)
N11–Co1–Cl2	178.09 (6)	C1–N11–Co1	109.89 (15)
N13–Co1–Cl2	89.32 (6)	C5–N12–Co1	122.10 (16)
N12–Co1–Cl2	89.11 (6)	C8–N13–Co1	121.92 (15)

Data collection: XSCANS (Siemens, 1991); cell refinement: XSCANS; data reduction: XSCANS; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: WinGX (Farrugia, 1999); software used to prepare material for publication: WinGX.

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