Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

trans-Bis(1,4-butanediamine)dichlorocobalt(III) chloride monohydrate

Kazuya Fujimoto, Kiyonori Nagata, Honoh Suzuki* and Kan Kanamori

Department of Chemistry, Toyama University, 3190 Gofuku, Toyama 930-8555, Japan

Correspondence e-mail:
honoh@sci.toyama-u.ac.jp

Received 12 September 2001
Accepted 17 September 2001
Online 29 September 2001

Key indicators

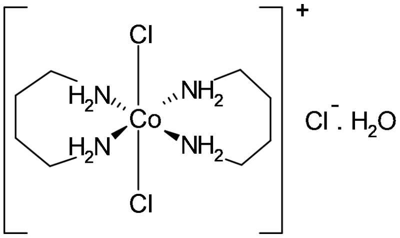
Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.014 Å
Disorder in main residue
R factor = 0.033
wR factor = 0.082
Data-to-parameter ratio = 12.7

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

The title compound, $[CoCl_2(C_4H_{12}N_2)_2]Cl\cdot H_2O$, (I), has an octahedral coordination geometry. The conformations of the seven-membered chelate rings are disordered between the twist-chair and twist-boat forms. The space group of (I) is *Cc*, although the structure has a pseudo-center of symmetry.

Comment

1,4-Butanediamine, also known as putrescine or tetramethylenediamine (tmd), is a biogenic diamine. The interaction between transition metals and tmd is of interest from the viewpoint of bioinorganic chemistry (Gasowska *et al.*, 2000). However, only a few structures have been reported for transition metal complexes of tmd (Sato *et al.*, 1974; Shimoi *et al.*, 1988; Kurachi & Ohba, 1992). We report here the structure of the title compound, *trans*-$[Co^{III}Cl_2(tmd)_2]Cl\cdot H_2O$, (I).



(I)

The complex cation (Fig. 1) has a pseudo-center of symmetry of inversion and thus the conformations of the seven-membered chelate rings are almost identical. One of the C atoms in each of the two tmd chelates (C3 and C7, respectively) is disordered with equal occupancy factors. The C3b and C7a positions correspond to the twist-chair conformation of the cycloheptane ring, and the C3a and C7b positions to the twist-boat conformation (Fig. 2), though the observed conformations deviate from the ideal conformations to a considerable extent (Hendrickson, 1961).

The Co—N bond distances range from 1.978 (4) to 2.003 (4) Å and are comparable with those found in $[Co(NO_2)_2(tmd)_2]NO_3$ (Shimoi *et al.*, 1988). The bite angles of the seven-membered chelate rings, 91.4 (2) and 89.59 (18)°, are also normal.

Experimental

The title compound was prepared by adapting the procedures described by Nagata *et al.* (1985) and Nagata & Kanamori (2001). Crystals of (I) were obtained by a spontaneous evaporation of an aqueous solution at room temperature.

DOI: 10.1107/S1600536801015227

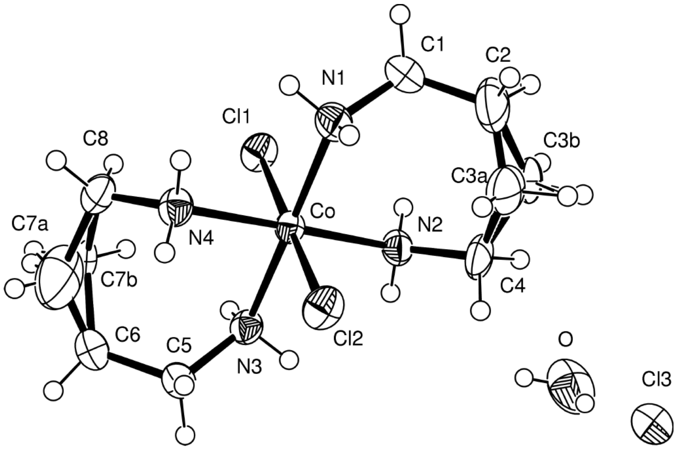


Figure 1
The molecular structure of (I) showing the labeling of the non-H atoms. Displacement ellipsoids are shown at 50% probability levels.

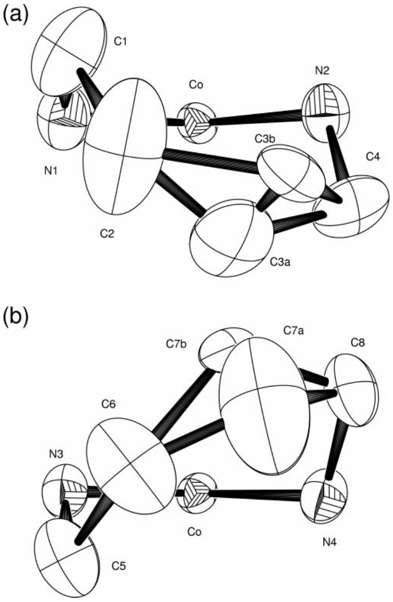


Figure 2
The conformations of the chelate rings projected along the bisector of the N—Co—N angle.

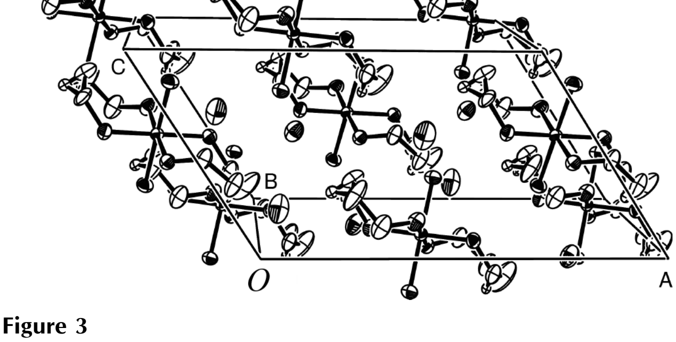


Figure 3
A packing diagram of (I). H atoms have been omitted for clarity.

Crystal data

$[CoCl_2(C_4H_{12}N_2)_2]Cl\cdot H_2O$
$M_r = 359.61$
Monoclinic, Cc
$a = 14.914\ (5)$ Å
$b = 13.560\ (6)$ Å
$c = 8.994\ (4)$ Å
$\beta = 122.75\ (2)^\circ$
$V = 1529.8\ (11)$ Å^3
$Z = 4$
$D_x = 1.561$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 14.2$–14.9°
$\mu = 1.64$ mm^{-1}
$T = 296\ (2)$ K
Prism, green
$0.20 \times 0.20 \times 0.20$ mm

Data collection

Rigaku AFC-7*R* diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.735$, $T_{\max} = 0.735$
2259 measured reflections
2259 independent reflections
2097 reflections with $I > 2\sigma(I)$
$\theta_{\max} = 30.0^\circ$
$h = 0 \rightarrow 20$
$k = 0 \rightarrow 19$
$l = -12 \rightarrow 10$
3 standard reflections
frequency: 60 min
intensity decay: 2.8%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.082$
$S = 1.05$
2259 reflections
178 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0335P)^2 + 2.6779P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 1.00$ e Å^{-3}
$\Delta\rho_{\min} = -0.55$ e Å^{-3}
Absolute structure: (Flack, 1983), 29 Friedel pairs
Flack parameter = 0.13 (3)

Table 1
Selected geometric parameters (Å, °).

Co—Cl1	2.2582 (19)	Co—N2	1.978 (4)
Co—Cl2	2.2571 (19)	Co—N3	1.989 (4)
Co—N1	2.002 (5)	Co—N4	2.003 (4)
Cl1—Co—Cl2	178.83 (8)	Cl2—Co—N4	86.68 (14)
Cl1—Co—N1	89.22 (16)	N1—Co—N2	91.4 (2)
Cl1—Co—N2	88.11 (15)	N1—Co—N3	178.4 (3)
Cl1—Co—N3	91.62 (14)	N1—Co—N4	89.0 (2)
Cl1—Co—N4	92.61 (15)	N2—Co—N3	89.94 (18)
Cl2—Co—N1	89.85 (17)	N2—Co—N4	179.1 (3)
Cl2—Co—N2	92.61 (15)	N3—Co—N4	89.59 (18)
Cl2—Co—N3	89.30 (14)		

Systematic absences showed the space group to be either $C2/c$ or Cc. In $C2/c$, the non-coordinating anions and water molecules could not be located, whereas in Cc, they could ($C2/c$, R = 8.0%; Cc, R = 3.3%). Comparison of the two solutions revealed that the $[CoCl_2(tmd)_2]^+$ moiety has a pseudo-center of symmetry of inversion but that the non-coordinating Cl^- and water lower the lattice symmetry to Cc (Fig. 3). The Flack (1983) parameter [0.13 (3)] is also consistent with the absence of inversion symmetry. Disorder of the two C atoms (C3 and C7) was suggested by their highly anisotropic displacement parameters, so that two positions for each atom were calculated by moving along the direction of the largest eigenvalue of the displacement tensor, with a fixed site occupation factor of 0.5. H atoms bonded to C and N atoms were placed geometrically and refined using a riding model *via* the *SHELXL*97 *HFIX*/*AFIX* 23 facility. The displacement parameter was set as 1.2 times that of the parent atom. H atoms of water were refined with a fixed O—H distance and H—O—H angle *via the SHELXL*97 *DFIX* facility.

Data collection: *AFC-7R Diffractometer Control Software* (Rigaku, 1999); cell refinement: *AFC-7R Diffractometer Control Software*; data reduction: *AFC-7R Diffractometer Control Software*;

program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

We thank the Center for Instrumental Analysis at Toyama University for their support with the X-ray diffraction measurements.

References

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.

Gasowska, A., Lomozik, L. & Jastrzab, R. (2000). *J. Inorg. Biochem.* **78**, 139–147.

Hendrickson, J. B. (1961). *J. Am. Chem. Soc.* **83**, 4537–4547.

Kurachi, S. & Ohba, S. (1992). *Bull. Chem. Soc. Jpn*, **65**, 3033–3041.

Nagata, K. & Kanamori, K. (2001). *J. Coord. Chem.* In the press.

Nagata, K., Kanamori, K., Kawai, K. & Ogino, H. (1985). *Chem. Lett.* pp. 1507–1510.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

Rigaku (1999). *AFC-7R Diffractometer Control Software*. Rigaku Corporation, Tokyo, Japan.

Sato, S., Saito, Y., Fujita, J. & Ogino, H. (1974). *Inorg. Nucl. Chem. Lett.* **10**, 669.

Sheldrick, G. M. (1997). *SHELXL*97 and *SHELXS*97. University of Göttingen, Germany.

Shimoi, M., Fujinawa, Y., Ogino, H. Kanamori, K. & Kawai, K. (1988). *Bull. Chem. Soc. Jpn*, **61**, 3491–3496.