(N,N'-Bis{[1-(2-oxidophenyl)ethylene]aminoethyl}-ethylenediamine)cobalt(III) chloride methanol solvate hydrate

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The title complex, $[Co(C_{22}H_{28}N_4O_2)]Cl\cdot CH_4O\cdot H_2O$, consists of one ligand L^{2-} ($L = (N,N'$-bis((1-(2-hydroxyphenyl)ethylene)amino)ethyl)aminoethylamine), one cobalt ion, one chloride ion, one methanol molecule and one water molecule. The cobalt(III) exhibits a distorted octahedral CoN_4O_2 coordination. There are four unique O—H···O, O—H···Cl and N—H···Cl hydrogen bonds between the hexadentate ligand L^{2-}, the solvent molecules and the chloride ions. The hydrogen bonds link the residues of two asymmetric units into a discrete cluster, located about an inversion centre.

Comment

In recent years, there has been considerable interest in metal complexes supported by tripodal ligands due to their chemical and biological activities (Moreno *et al.*, 2005; Deroche *et al.*, 1996). However, only a few cobalt tripodal complexes have been synthesized, such as $[Co(C_{21}H_{48}N_8O_3)](BF_4)_2$(Myunghyun *et al.*, 1997), $[Co(C_{18}H_{25}N_5)(H_2O)][(ClO_4)_3]H_2O$ (McLachlan *et al.*, 1995) $[Co(C_{12}H_{21}N_5ClO)][(ClO_4)_2]H_2O$ (McLachlan *et al.*, 1995) and $[[Co(C_{24}H_{27}N_7O_3)][(PF_6)_2]CH_3CN$ (Gou *et al.*, 1993). Recently, we have synthesized the title compound, (I), and we report here its crystal structure.

(I)
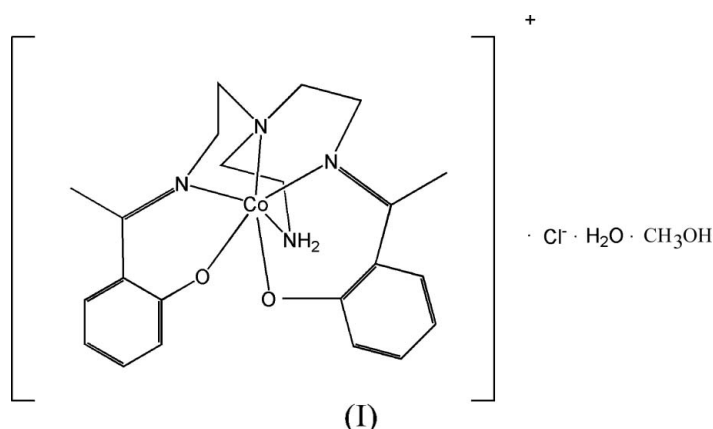
The asymmetric unit (Fig. 1) consists of one ligand L^{2-}, one cobalt ion, one chloride ion, one methanol molecule and one water molecule. The geometry around the cobalt center (Table 1) is a slightly distorted octahedron with composition CoN_4O_2. The average Co—N distance is 1.945 (3) Å and the average Co—O distance is 1.900 (3) Å. The coordinating N and O atoms are all supplied by the ligand L^{2-}, which forms two six-membered rings and three five-membered rings with the Co atom.

The finite hydrogen-bonded cluster (illustrated in Fig. 2) displays four unique hydrogen bonds of types O—H···O, O—
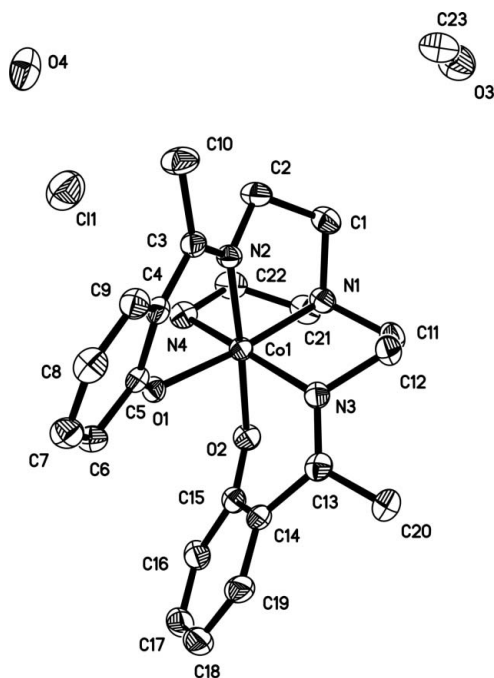
Figure 1
The molecular structure of (I). Displacement ellipsoids are drawn at 30% probability level and H atoms have been omitted for clarity.
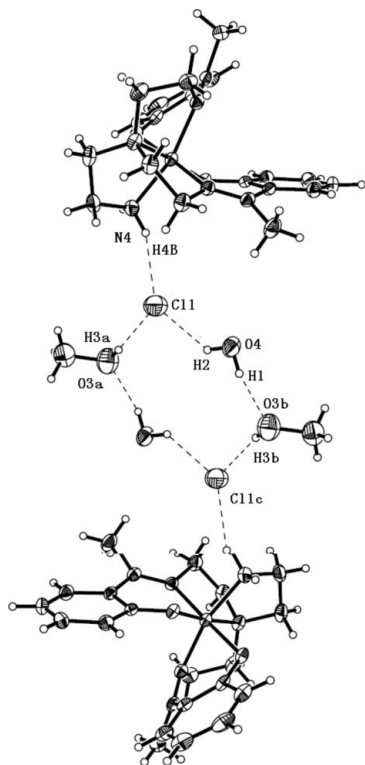

Figure 2
The six-membered hydrogen-bond donor/acceptor ring. Dashed lines indicate hydrogen bonds. [Symmetry codes: (a) $1 - x, 1 - y, 1 - z$; (b) x, $y, z - 1$; (c) $1 - x, 1 - y, -z$.]

$H\cdots Cl$ and $N-H\cdots Cl$. The hydrogen bonds interact, forming a six-membered donor/acceptor ring located about a crystallographic inversion centre. The six-membered rings are linked to two neighbouring molecules.

Experimental

Ligand L (1 mmol, 0.382 g), $N(CH_2CH_3)_3$ (3 ml) and $CoCl_2$ (1 mmol, 0.13 g) were dissolved in a mixture of methanol, acetonitrile and 1,2-dichloroethane (1:1:1 $v/v/v$). The solution was refluxed for about 4 h with stirring and a red solution was formed. About a week later, red crystal were isolated from the solution [m.p. 430–431 K (decomposition)].

Crystal data

$[Co(C_{22}H_{28}N_4O_2)]Cl \cdot CH_4O \cdot H_2O$	$Z = 4$
$M_r = 524.92$	$D_x = 1.437$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 14.875 (5)$ Å	$\mu = 0.85$ mm^{-1}
$b = 11.708 (4)$ Å	$T = 298 (2)$ K
$c = 14.040 (5)$ Å	Block, red
$\beta = 97.110 (5)°$	$0.46 \times 0.43 \times 0.40$ mm
$V = 2426.3 (14)$ Å^3	

Data collection

Bruker CCD area-detector diffractometer	12440 measured reflections
	4270 independent reflections
φ and ω scans	3170 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan *SADABS* (Sheldrick, 1996)	$R_{int} = 0.032$
	$\theta_{max} = 25.0°$
$T_{min} = 0.695$, $T_{max} = 0.726$	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.082P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.051$	$+ 3.7562P]$
$wR(F^2) = 0.158$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.02$	$(\Delta/\sigma)_{max} = 0.001$
4270 reflections	$\Delta\rho_{max} = 0.71$ e Å^{-3}
302 parameters	$\Delta\rho_{min} = -0.82$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Co1—O1	1.884 (3)	Co1—N3	1.929 (3)
Co1—N2	1.910 (3)	Co1—N1	1.965 (3)
Co1—O2	1.916 (3)	Co1—N4	1.975 (3)
O1—Co1—N2	90.51 (12)	N2—Co1—N4	93.75 (14)
O1—Co1—O2	87.43 (11)	O2—Co1—N3	85.10 (13)
O1—Co1—N3	98.29 (12)	O2—Co1—N1	95.88 (13)
O1—Co1—N1	174.55 (13)	O2—Co1—N4	84.02 (13)
O1—Co1—N4	91.12 (13)	N3—Co1—N1	86.32 (14)
N2—Co1—O2	176.94 (13)	N3—Co1—N4	165.27 (14)
N2—Co1—N3	97.45 (13)	N1—Co1—N4	84.95 (14)
N2—Co1—N1	86.00 (13)		

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N4—H4B$\cdots$Cl1	0.90	2.33	3.190 (4)	159
O3—H3$\cdots$Cl1^i	0.82	2.22	3.011 (6)	163
O4—H1$\cdots$O3^{ii}	0.88	2.26	2.909 (6)	131
O4—H2$\cdots$Cl1	0.83	2.31	2.976 (4)	137

Symmetry codes: (i) $-x + 1, -y + 1, -z + 1$; (ii) $x, y, z - 1$.

The water H atoms were located in a difference Fourier map and included in the refinement riding on their parent atoms. All other H

atoms were positioned geometrically and treated as riding on their parent atoms, with benzene C—H distances of 0.93 Å, methylene C—H distances of 0.97 Å, methyl C—H distances of 0.96 Å, methanol hydroxyl O—H distances of 0.82 Å, amino N—H distances of 0.90 Å. The $U_{iso}(H)$ values were set at $1.2U_{eq}(C,N)$ or $1.5U_{eq}(O)$.

Data collection: Bruker *SMART* (Siemens, 1996); cell refinement: Bruker *SAINT* (Siemens, 1996); data reduction: Bruker *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: Bruker *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: Bruker *SHELXTL*.

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