trans-Diaquatetrakis(4,4'-methylenediphenyl-amine-κN)cobalt(II) dinitrate dihydrate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.041
wR factor = 0.081
Data-to-parameter ratio = 13.7

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

The hydrothermally prepared title compound, $[Co(C_{13}H_{14}-N_2)_4(H_2O)_2](NO_3)_2 \cdot 2H_2O$, consists of mononuclear cationic complexes of six-coordinate cobalt(II), together with nitrate counter-ions and uncoordinated water molecules. Four monodentate 4,4'-methylenediphenylamine (dapm) ligands and two water molecules comprise the octahedral coordination of the Co^{II} ion, which lies on an inversion center. The compound is isostructural with the previously reported Cd and Ni analogs.

Comment

The structure of the title compound, (I), consists of a pseudo-octahedral Co^{2+} complex with four monodentate dapm ligands [dapm is 4,4'-methylenediphenylamine] coordinated through one amine N and two water molecules, as shown in Fig. 1, with two nitrate ions and two uncoordinated water molecules. The Co^{2+} ion lies on a crystallographic inversion center. Hydrothermally prepared (I) is isostructural with the Cd and Ni analogs obtained from room temperature evaporation of water–alcohol mixtures of the $M(NO_3)_2$ salt (M = Cd, Ni) and dapm (Wang et al., 2001; Zhang et al., 2001).

(I)
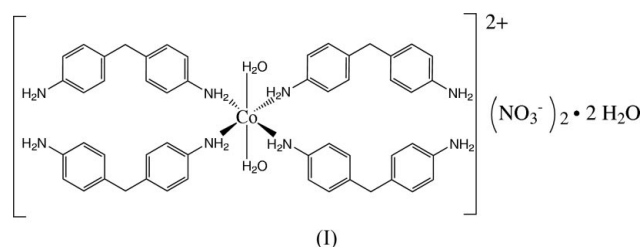
Experimental

The title compound was prepared by hydrothermal reaction of 4,4'-methylenedianiline (dapm) (0.0240 g, 0.12 mmol) with $Co(NO_3)_2 \cdot 6H_2O$ (0.0386 g, 0.11 mmol) in water (0.80 ml) in an evacuated sealed Pyrex tube. The tube was heated to 358 K at 1 K
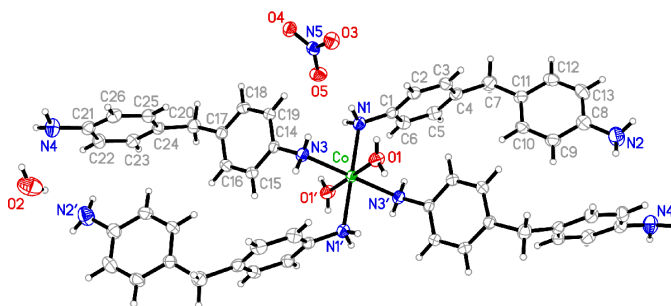
Figure 1
The structure of (I). Displacement ellipsoids are drawn at the 30% probability level. Symmetry code: (') $1 - x$, $-y$, $1 - z$. The symmetry-related nitrate ion and uncoordinated water molecule are not shown.

min^{-1}, and held at that temperature for 8 h before slowly cooling (0.2 K min^{-1}) to 303 K. Pale-orange block-shaped crystals were formed along with some pink powder. Analysis calculated for $C_{52}H_{64}CoN_{10}O_{10}$: C 59.59, H 6.16, N 13.36%; found: C 59.25, H 5.87, N 13.22%.

Crystal data

$[Co(C_{13}H_{14}N_2)_4(H_2O)_2]$- $(NO_3)_2 \cdot 2H_2O$	$Z = 1$
	$D_x = 1.370$ Mg m^{-3}
$M_r = 1048.06$	Mo $K\alpha$ radiation
Triclinic, $P\bar{1}$	Cell parameters from 6696
$a = 9.2265 (5)$ Å	reflections
$b = 11.6587 (6)$ Å	$\theta = 2.3–26.3°$
$c = 11.9719 (7)$ Å	$\mu = 0.41$ mm^{-1}
$\alpha = 80.920 (1)°$	$T = 293 (2)$ K
$\beta = 87.804 (1)°$	Block, pale orange
$\gamma = 89.488 (1)°$	$0.42 \times 0.24 \times 0.18$ mm
$V = 1270.73 (12)$ Å^3	

Data collection

Bruker SMART APEX CCD diffractometer	5200 independent reflections
	3875 reflections with $I > 2\sigma(I)$
ω and φ scans	$R_{int} = 0.030$
Absorption correction: multi-scan (SADABS; Bruker, 1999)	$\theta_{max} = 26.4°$
	$h = -11 \rightarrow 11$
$T_{min} = 0.619$, $T_{max} = 0.928$	$k = -14 \rightarrow 14$
11803 measured reflections	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.041$	
$wR(F^2) = 0.081$	
$S = 0.99$	$w = 1/[\sigma^2(F_o^2) + (0.03P)^2]$
5200 reflections	where $P = (F_o^2 + 2F_c^2)/3$
379 parameters	$(\Delta/\sigma)_{max} < 0.001$
	$\Delta\rho_{max} = 0.45$ e Å^{-3}
	$\Delta\rho_{min} = -0.32$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Co—O1	2.0526 (15)	Co—N1	2.2448 (17)
Co—N3	2.2005 (16)		
O1—Co—N3	86.26 (7)	N3^i—Co—N1^i	86.15 (7)
O1^i—Co—N1^i	88.95 (7)		

Symmetry code: (i) $1 - x, -y, 1 - z$.

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

$D—H \cdots A$	$D—H$	$H \cdots A$	$D \cdots A$	$D—H \cdots A$
N1—H1A$\cdots$O3^i	0.90 (2)	2.22 (2)	3.114 (2)	173.2 (18)
N3—H3A$\cdots$O3^{ii}	0.82 (2)	2.52 (2)	3.263 (3)	150.0 (19)
O1—H1WA$\cdots$O4^{ii}	0.79 (2)	1.99 (3)	2.779 (2)	175 (3)
N4—H4B$\cdots$O5^{iii}	0.93 (3)	2.29 (3)	3.107 (3)	147 (2)
O2—H2WA$\cdots$O5^{iii}	0.76 (4)	2.56 (4)	3.193 (4)	142 (4)
N2—H2B$\cdots$O2^{iv}	0.86 (3)	2.46 (3)	3.258 (4)	156 (2)
N4—H4A$\cdots$O4^v	0.96 (2)	2.14 (3)	3.048 (3)	157 (2)
O1—H1WB$\cdots$O2^{vi}	0.83 (2)	1.87 (3)	2.694 (3)	170 (2)
O2—H2WB$\cdots$N2^i	0.72 (4)	2.26 (4)	2.958 (5)	165 (5)

Symmetry codes: (i) $1 - x, -y, 1 - z$; (ii) $-x, -y, 1 - z$; (iii) $x, y, z - 1$; (iv) $x, y - 1, 2 + z$; (v) $-x, 1 - y, -z$; (vi) $1 - x, -y, -z$.

H atoms attached to C atoms were geometrically idealized, with C—H = 0.93 Å and $U_{iso}(H) = 1.2U_{eq}(C)$. H atoms on N atoms and water O were located and refined freely with isotropic displacement parameters.

Data collection: *SMART-NT* (Bruker, 1999); cell refinement: *SAINT-Plus-NT* (Bruker, 1999); data reduction: *SAINT-Plus-NT* ; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997*a*); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

References

Bruker (1999). *SMART-NT* (Version 5.624), *SAINT-Plus-NT* (Version 6.02*a*) and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

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

Sheldrick, G. M. (1997*b*). *SHELXTL* (Version 5.10). Bruker AXS Inc., Madison, Wisconsin, USA.

Wang, R., Hong, M., Weng, J., Cao, R., Liang, Y. & Zhao, Y. (2001). *Acta Cryst.* E**57**, m344–m345.

Zhang, Y., Lei, Z., Jianmin, L., Nishiura, M. & Imamoto, T. (2001) *J. Mol. Struct.* **559**, 55–58.