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{2,6-Bis[bis(2-pyridylmethyl)aminomethyl]-pyridine}cobalt(II) bis(perchlorate) 0.365-hydrate

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.002 Å
Disorder in solvent or counterion
R factor = 0.031
wR factor = 0.034
Data-to-parameter ratio = 17.1

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

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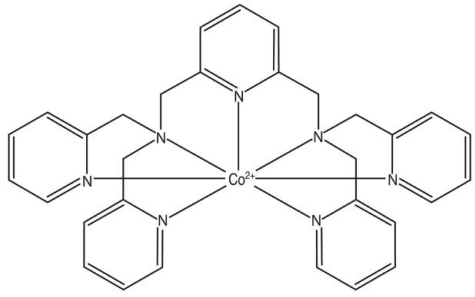
The title compound, $[Co(C_{31}H_{31}N_7)](ClO_4)_2.0.365H_2O$, contains a high-spin Co^{II} atom which is coordinated by seven N atoms at the corners of a distorted pentagonal bipyramid.

Comment

The ligand {2,6-bis[bis(2-pyridylmethyl)amino]methyl}-pyridine (BPATPA) has the potential to form seven-coordinate complexes. We have determined the structures of the isostructural Co^{II} and Mn^{II} complexes of BPATPA. The title compound, $[Co(BPATPA)](ClO_4)_2.0.365H_2O$, (I), is described here, while $[Mn(BPATPA)](ClO_4)_2.0.435H_2O$ is described in our following report (Hazell & Toftlund, 2007). The complexes are not isostructural with the previously reported compound $[Fe(BPATPA)](ClO_4)_2{\cdot}H_2O$ (Lonnon *et al.*, 2002).



2 (ClO_4^-) • 0.365 H_2O

(I)

The Co^{II} atom in (I) is coordinated by seven N atoms which form a distorted pentagonal bipyramid (Fig. 1 and Table 1), with atoms N21 and N31 in the axial positions. The axial Co—Nsp^2 bonds are longer than the equatorial Co—Nsp^2 bonds and are similar in length to the Co—Nsp^3 bonds (N1 and N9). The mean Co—N bond length is 2.274 Å, compared with the mean Fe—N bond length of 2.313 Å in $[Fe(BPATPA)](ClO_4)_2{\cdot}H_2O$ (Lonnon *et al.*, 2002).

The water molecule is included with fractional site occupancy and is hydrogen bonded to two O atoms of two perchlorate anions (Table 2).

Experimental

The BPATPA ligand (0.25 g, 0.5 mmol) was dissolved in methanol (10 ml) and the solution was flushed with dinitrogen for 5 min. $Co(ClO_4)_2{\cdot}6H_2O$ (0.18 g, 0.5 mmol) was added and the solution was stirred with continuous bubbling of dinitrogen for another 15 min. The solution was then left to stand in a closed vessel and crystals of (I) formed overnight.

Crystal data

$[Co(C_{31}H_{31}N_7)](ClO_4)_2\cdot0.365H_2O$
M_r = 766.08
Monoclinic, $P2_1/c$
a = 14.8754 (7) Å
b = 12.3883 (6) Å
c = 18.4518 (9) Å
β = 107.875 (1)°
V = 3236.2 (3) Å^3
Z = 4
D_x = 1.572 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.76 mm^{-1}
T = 120 K
Block, orange
0.48 × 0.36 × 0.28 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: integration (*XPREP*; Siemens, 1995)
T_{min} = 0.736, T_{max} = 0.830
42706 measured reflections
9261 independent reflections
7712 reflections with $I > 3\sigma(I)$
R_{int} = 0.032
θ_{max} = 29.8°

Refinement

Refinement on F
$R[F^2 > 3\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.034
S = 1.02
7712 reflections
452 parameters
H-atom parameters constrained
$w = 1/\{[\sigma_{cs}(F^2 + B) + (1 + A)F^2]^{1/2} - |F|\}^2$, where A = 0.03 and B = 2.0
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.52 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 e Å^{-3}

Table 1
Selected bond lengths (Å).

Co1—N1	2.303 (1)	Co1—N21	2.360 (1)
Co1—N9	2.325 (1)	Co1—N31	2.290 (1)
Co1—N10	2.181 (1)	Co1—N41	2.188 (1)
Co1—N11	2.273 (1)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O9—H9A···O5	0.85	1.89	2.740 (7)	180
O9—H9B···O4	0.85	2.245	3.097 (7)	180

H atoms bound to C atoms were placed in calculated positions, with C—H = 0.95 Å, and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C). The H atoms of the water molecule were placed along the vector from O9 to the nearest hydrogen-bond acceptor O atom, with O9—H = 0.85 Å. They were allowed to ride during subsequent refinement, with U_{iso}(H) = 1.2U_{eq}(O). The refined site occupancy factor of atom O9 is 0.365 (5).

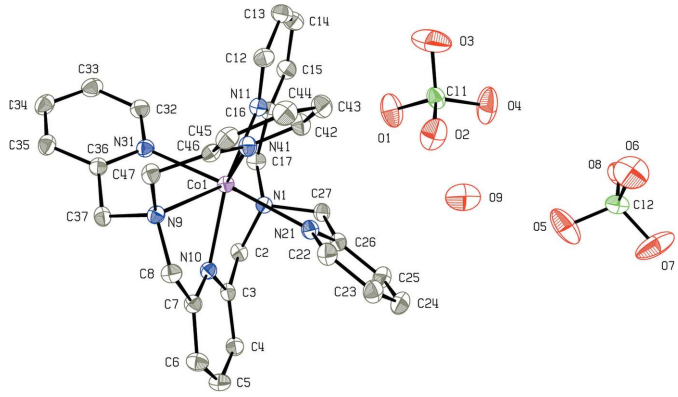


Figure 1
The molecular structure of (I), showing displacement ellipsoids at the 50% probability level. H atoms have been omitted.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SAINT* (Siemens, 1995); data reduction: *SAINT*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1997); program(s) used to refine structure: *KRYSTAL* (Hazell, 1995); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *KRYSTAL*.

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