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{2,6-Bis[bis(2-pyridylmethyl)aminomethyl]pyridine}-manganese(II) bis(perchlorate) 0.435-hydrate

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Key indicators

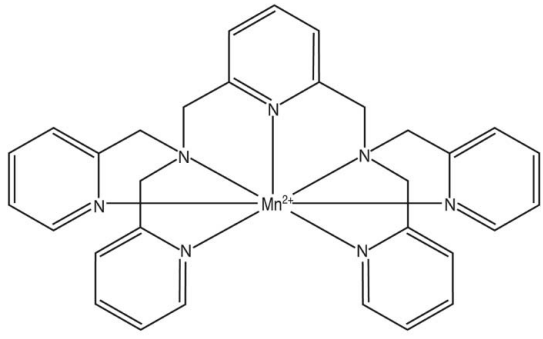
Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
Disorder in solvent or counterion
R factor = 0.035
wR factor = 0.037
Data-to-parameter ratio = 17.5

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

The title compound, $[Mn(C_{31}H_{31}N_7)](ClO_4)_2.0.435H_2O$, contains a high-spin Mn^{II} atom which is coordinated by seven N atoms at the corners of a distorted pentagonal bipyramid.

Comment

The title compound, $[Mn(BPATPA)](ClO_4)_2.0.435H_2O$, (I), is isostructural with $[Co(BPATPA)](ClO_4)_2.0.365H_2O$, described in our preceding report (Hazell & Toftlund, 2007). In (I), the Mn^{II} atom 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 Mn—Nsp^2 bonds are longer than the equatorial Mn—Nsp^2 bonds and are similar in length to the Mn—Nsp^3 bonds (atoms N1 and N9). The mean Mn—N bond length of 2.351 Å compares with the mean Fe—N bond length of 2.313 Å in $[Fe(BPATPA)](ClO_4)_2\cdot H_2O$ (Lonnon *et al.*, 2002) and the mean Co—N bond length of 2.274 Å in $[Co(BPATPA)](ClO_4)_2.0.365H_2O$ (Hazell & Toftlund, 2007). Thus, the mean M—N bond length decreases regularly with increasing number of unpaired d electrons on M in these high-spin complexes.



2 (ClO_4^-) · 0.435 H_2O

(I)

As is the case for the Co^{II} complex, 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. $Mn(ClO_4)_2\cdot 6H_2O$ (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

$[Mn(C_{31}H_{31}N_7)](ClO_4)_2\cdot0.435H_2O$	$Z = 4$
$M_r = 763.34$	$D_x = 1.556$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 14.9038$ (9) Å	$\mu = 0.63$ mm^{-1}
$b = 12.3825$ (8) Å	$T = 100$ K
$c = 18.647$ (1) Å	Block, colourless
$\beta = 108.733$ (1)°	0.42 × 0.34 × 0.32 mm
$V = 3258.9$ (4) Å^3	

Data collection

Siemens SMART CCD area-detector diffractometer	35942 measured reflections
	9357 independent reflections
ω scans	7918 reflections with $I > 3\sigma(I)$
Absorption correction: integration (*XPREP*; Siemens, 1995)	$R_{int} = 0.038$
	$\theta_{max} = 29.8°$
$T_{min} = 0.757$, $T_{max} = 0.844$	

Refinement

Refinement on F	$w = 1/\{[\sigma_{cs}(F^2 + B) + (1 + A)F^2]^{1/2} - \lvert F\rvert\}^2$, where A = 0.03 and B = 2.0
$R[F^2 > 3\sigma(F^2)] = 0.035$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.037$	$\Delta\rho_{max} = 0.60$ e Å^{-3}
$S = 1.04$	$\Delta\rho_{min} = -0.39$ e Å^{-3}
7918 reflections	
452 parameters	
H-atom parameters constrained	

Table 1
Selected bond lengths (Å).

Mn1—N1	2.397 (1)	Mn1—N21	2.431 (1)
Mn1—N9	2.409 (1)	Mn1—N31	2.350 (1)
Mn1—N10	2.290 (1)	Mn1—N41	2.279 (1)
Mn1—N11	2.306 (1)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O9—H9A···O5	0.85	1.92	2.768 (4)	180
O9—H9B···O4	0.85	2.31	3.165 (4)	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 0.435 (5).

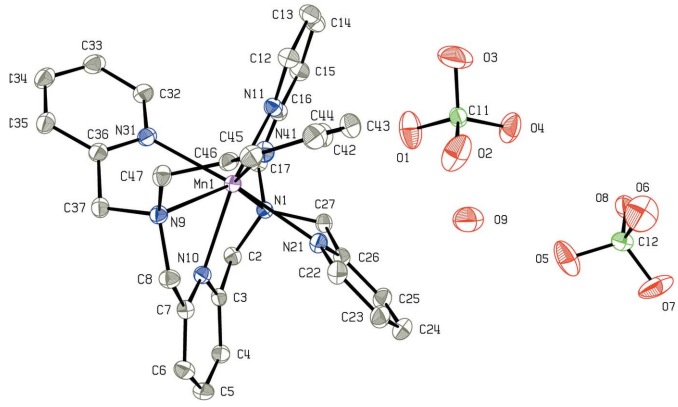


Figure 1
The molecular structure of (I), showing displacement ellipsoids at the 50% probability level. H atoms have been omitted.

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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