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Bis(2,2′:6′,2″-terpyridyl-κ^3N)manganese(II) dinitrate dihydrate

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Key indicators

Single-crystal X-ray study
T = 180 K
Mean σ(C–C) = 0.004 Å
R factor = 0.049
wR factor = 0.153
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.

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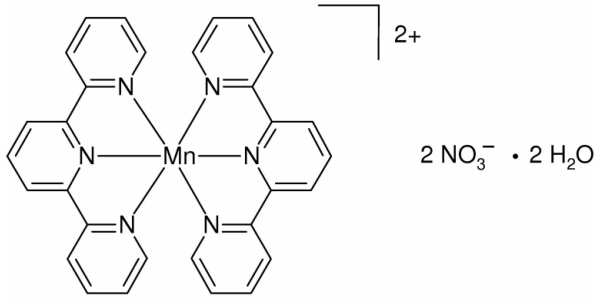
In the crystal structure of the title compound, $[Mn(C_{15}H_{11}N_3)_2](NO_3)_2\cdot 2H_2O$, at 180 K, the complex cation lies on a twofold axis.

Comment

The comproportion reaction between $KMnO_4$ and manganese(II) nitrate in the presence of bis(2,2′:6′,2″-terpyridine) (terpy) gives the oxo-bridged Mn^{III}–Mn^{IV} complex $[Mn_2O_2(terpy)_2(H_2O)_2](NO_3)_3\cdot 6H_2O$ (Collomb *et al.*, 1999; Limburg *et al.*, 1999). Preparation of this compound from a reaction mixture in acetonitrile/water also yields a significant proportion of the title complex, bis(2,2′:6′,2″-terpyridyl)-manganese(II) dinitrate dihydrate, (I). The two crystalline components are readily distinguished by their colours: the oxo-bridged complex is dark green, while the title complex is orange.



(I)

In its crystal structure at 180 K, the cation of the title complex is sited on a twofold axis at $(0,\frac{1}{4},z)$, which passes through atoms Mn1, N2, N4, C8 and C16. The nitrate counter-anions and water molecules form hydrogen-bonded ribbons that run in perpendicular directions parallel to [100] and [010] [$H2W\cdots O52^{ii}$ = 2.157 (13) Å and $O1W-H2W\cdots O52^{ii}$ = 171 (4)°; $H1W\cdots O53^{ii}$ = 2.212 (14) Å and $O1W-H1W\cdots O53^{iii}$ = 169 (4)°, symmetry codes: (ii) $\frac{1}{4}+y, \frac{3}{4}-x, \frac{3}{4}-z$; (iii) $\frac{1}{2}+y, \frac{1}{4}-x, \frac{1}{4}+z$].

Experimental

$Mn(NO_3)_2\cdot 4H_2O$ (273 mg, 1.08 mmol) dissolved in water (2 ml) was mixed with 2,2′:6′,2″-terpyridine (358 mg, 1.54 mmol) dissolved in acetonitrile (2 ml) to give a yellow solution. $KMnO_4$ (74 mg, 0.47 mmol) in water (2 ml) was then added, causing the solution to turn green and then brown–red. After 48 h, the title complex crystallized as orange plates, together with green needles of $[Mn_2O_2(terpy)_2(H_2O)_2](NO_3)_3\cdot 6H_2O$ in an approximate 2:1 ratio.

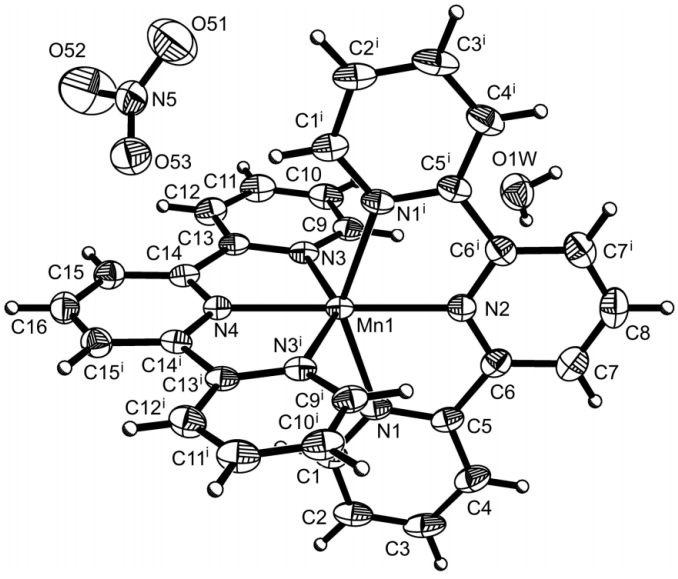


Figure 1
The components of (I), showing displacement ellipsoids at the 50% probability level for non-H atoms. H atoms are shown as spheres of arbitrary radius. [Symmetry code: (i) $-x, \frac{1}{2}-y, z$.]

Crystal data

$[Mn(C_{15}H_{11}N_3)_2](NO_3)_2\cdot 2H_2O$
$M_r = 681.53$
Tetragonal, $I4_1/a$
$a = 12.3571\ (1)$ Å
$c = 38.6211\ (6)$ Å
$V = 5897.36\ (11)$ Å^3
$Z = 8$
$D_x = 1.535$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 9582 reflections
$\theta = 2.6$–$26.0°$
$\mu = 0.52$ mm^{-1}
$T = 180\ (2)$ K
Block, orange
0.20 × 0.20 × 0.12 mm

Data collection

Bruker–Nonius X8APEX2 CCD diffractometer
Thin-slice ω and φ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{min} = 0.837$, $T_{max} = 0.941$
51440 measured reflections
3027 independent reflections
2265 reflections with $I > 2\sigma(I)$
$R_{int} = 0.046$
$\theta_{max} = 26.4°$
$h = -15 \rightarrow 15$
$k = -15 \rightarrow 15$
$l = -48 \rightarrow 48$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.153$
$S = 1.03$
3027 reflections
221 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0805P)^2 + 14.4081P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 1.14$ e Å^{-3}
$\Delta\rho_{min} = -0.44$ e Å^{-3}

H atoms bound to carbon were placed geometrically and allowed to ride during subsequent refinement, with C—H = 0.95 Å and $U_{iso}(H) = 1.2U_{eq}(C)$. H atoms of the water molecule were located in difference Fourier maps, and refined with O—H distances restrained to 0.85 (1) Å, the H···H distance restrained to 1.36 (2) Å, and $U_{iso}(H) = 1.5U_{eq}(O)$. The largest residual peak lies in the vicinity of the nitrate anion.

Data collection: *APEX*2 (Bruker–Nonius, 2004); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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