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Hexaaquamanganese(II) dinitrate bis(hexamethylenetetramine) tetrahydrate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(O–N) = 0.003 Å
R factor = 0.046
wR factor = 0.118
Data-to-parameter ratio = 12.4

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

In the title compound, $[Mn(H_2O)_6](NO_3)_2{\cdot}2C_6H_{12}N_4{\cdot}4H_2O$, the hexamethyleneteramine molecules have no direct coordination to the Mn^{II} atom. O—H···O and O—H···N intermolecular hydrogen bonds form a three-dimensional network.

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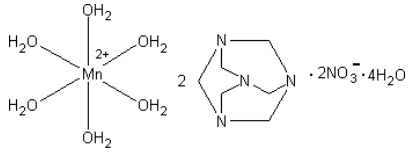
Comment

Hexamethylenetetramine, a versatile ligand (Blazevic & Kolbah, 1979) with three fused rings in a chair conformation and four bridgehead N atoms, is known to form coordination compounds with metal salts (Zheng *et al.*, 2001). One such compound, with dichromate as the counter-ion, $[Ni(H_2O)_6][Cr_2O_7]{\cdot}2C_6H_{12}N_4{\cdot}H_2O$, has recently been reported (Dagur *et al.*, 2003).



(I)

Fig. 1 shows the components of the asymmetric unit of the title compound, (I). The packing diagram (Fig. 2) shows both the organic molecules and the inorganic counter-ions, packed together in the crystal structure. A set of strong O—H···O hydrogen bonds (Table 1) links the solvent water molecules with the coordinated water molecules. There are also O—H···O hydrogen bonds between the nitrate ion and both the free and the coordinated water molecules. There also exist

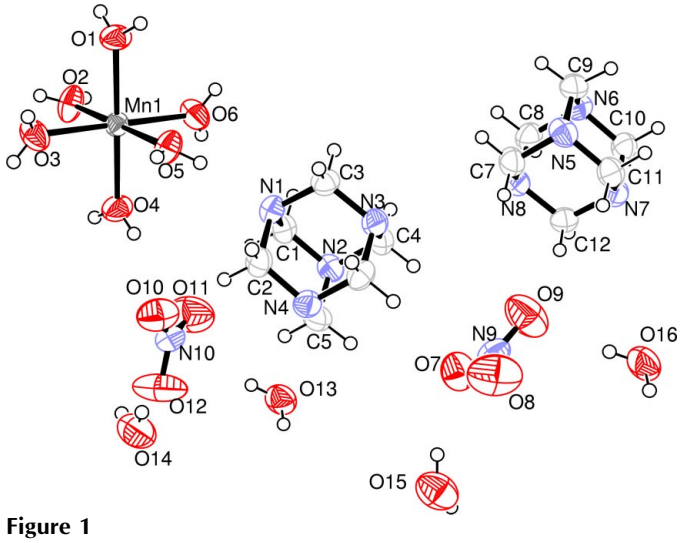


Figure 1
The asymmetric unit of (I), with displacement ellipsoids at the 50% probability level.

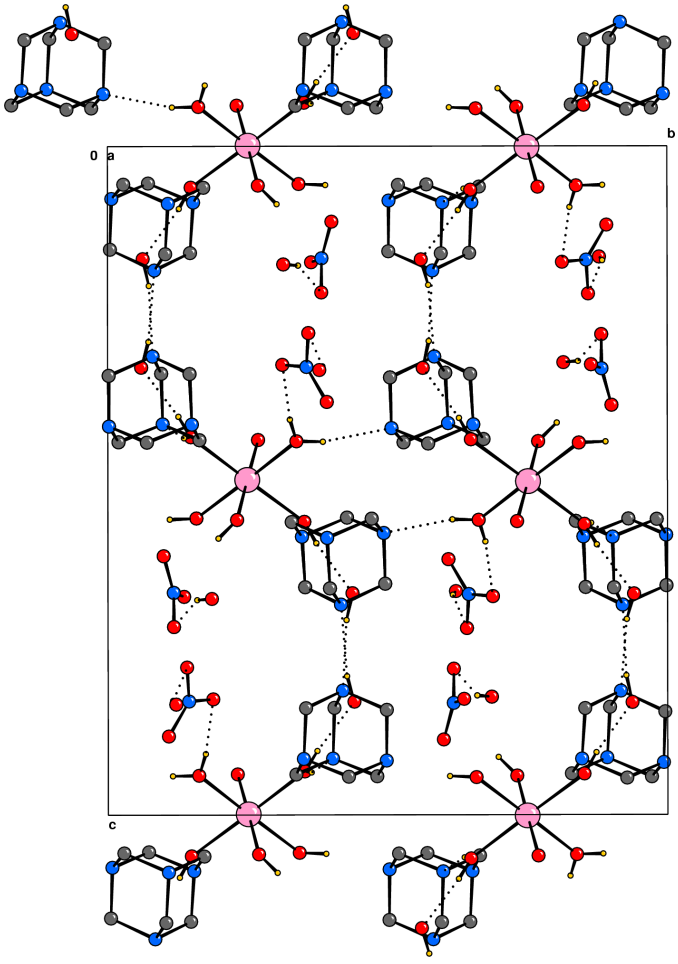


Figure 2
Packing diagram of (I), viewed down the *a* axis. The dotted lines indicate hydrogen bonds.

O—H···N hydrogen bonds between the hexamethylenetetramine unit and both the coordinated and the free water molecules. These strong intermolecular hydrogen bonds form a three-dimensional network.

Experimental

Compound (I) was prepared by mixing aqueous solutions of manganese nitrate hexahydrate and hexamethylenetetramine in a 1:2 molar ratio. A pale-pink precipitate was obtained. Crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an aqueous solution of the product.

Crystal data

$[Mn(H_2O)_6](NO_3)_2\cdot 2C_6H_{12}N_4\cdot 4H_2O$
$M_r = 639.51$
Monoclinic, $P2_1/n$
$a = 9.511\ (3)$ Å
$b = 16.232\ (4)$ Å
$c = 19.426\ (5)$ Å
$\beta = 90.600\ (4)^\circ$
$V = 2998.9\ (14)$ Å^3
$Z = 4$
$D_x = 1.416$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 180 reflections
$\theta = 1.6$–26.4°
$\mu = 0.52$ mm^{-1}
$T = 293\ (2)$ K
Prism, pale pink
0.42 × 0.30 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)
$T_{min} = 0.811$, $T_{max} = 0.903$
24 304 measured reflections
6539 independent reflections
4834 reflections with $I > 2\sigma(I)$
$R_{int} = 0.045$
$\theta_{max} = 27.8^\circ$
$h = -11 \rightarrow 11$
$k = -17 \rightarrow 20$
$l = -25 \rightarrow 25$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.118$
$S = 1.06$
6539 reflections
528 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0529P)^2 + 0.5875P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.37$ e Å^{-3}
$\Delta\rho_{min} = -0.30$ e Å^{-3}

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O2—H21···N7^{i}	0.77 (3)	2.05 (3)	2.808 (3)	168 (3)
O2—H22···O7^{ii}	0.83 (3)	1.97 (3)	2.792 (3)	173 (3)
O3—H31···O16^{i}	0.80 (3)	1.94 (3)	2.730 (3)	172 (3)
O4—H42···O10	0.77 (3)	2.07 (3)	2.818 (3)	163 (3)
O5—H51···N1	0.83 (3)	1.99 (3)	2.813 (3)	169 (3)
O6—H61···N2^{ii}	0.76 (3)	2.11 (3)	2.855 (3)	166 (3)
O6—H62···O13^{ii}	0.79 (3)	1.97 (3)	2.753 (3)	174 (3)
O13—H131···N6^{iii}	0.78 (4)	2.10 (4)	2.868 (3)	168 (3)
O14—H142···O8^{iv}	0.78 (4)	2.22 (4)	2.942 (4)	155 (4)
O15—H151···O12^{v}	0.84 (5)	2.11 (5)	2.909 (4)	158 (4)
O16—H161···N4^{vi}	0.83 (4)	2.04 (4)	2.854 (3)	166 (4)

Symmetry codes: (i) $x, 1+y, z$; (ii) $2-x, 2-y, 1-z$; (iii) $x-\frac{1}{2}, \frac{3}{2}-y, \frac{1}{2}+z$; (iv) $\frac{5}{2}-x, \frac{1}{2}+y, \frac{3}{2}-z$; (v) $\frac{3}{2}-x, y-\frac{1}{2}, \frac{3}{2}-z$; (vi) $\frac{5}{2}-x, y-\frac{1}{2}, \frac{3}{2}-z$.

All H atoms were located in difference maps and refined isotropically. The C—H and O—H bond lengths are in the ranges 0.90 (2)–1.01 (2) and 0.74 (3)–0.90 (6) Å, respectively.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1998); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997) and *CAMERON* (Watkin *et al.*, 1993); software used to prepare material for publication: *PLATON* (Spek, 2003).

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