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#### Key indicators

Single-crystal X-ray study

$T = 298\text{ K}$

Mean  $\sigma(\text{O}-\text{N}) = 0.005\text{ \AA}$

$R$  factor = 0.043

$wR$  factor = 0.117

Data-to-parameter ratio = 9.2

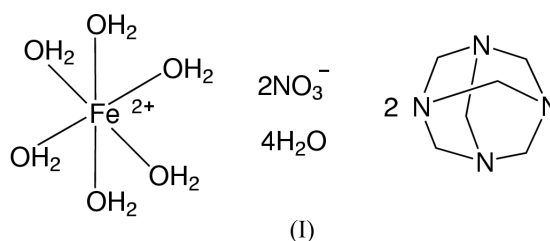
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

## Hexaquaairon(II) dinitrate bis(dihexamethylenetetramine) tetrahydrate

In the title compound,  $[\text{Fe}(\text{H}_2\text{O})_6](\text{NO}_3)_2 \cdot 2\text{C}_6\text{H}_{12}\text{N}_4 \cdot 4\text{H}_2\text{O}$ , the ferrous ion is coordinated by six O atoms from six water molecules and is in a slightly distorted octahedral geometry, lying on an inversion center. All the N atoms of the hexamethylenetetramine molecules, all the O atoms in the nitrate anions and all the water molecules contribute to the formation of hydrogen bonds. This leads to the formation of a three-dimensional network.

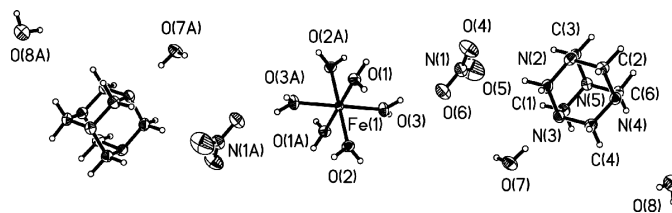
#### Comment

Ferrous solutions are normally unstable to air and are easily oxidized to produce ferric compounds, especially in neutral or alkaline solution. In an attempt to prepare an aquaferrous compound, the title complex was isolated and found to contain a hexaquaferrous dication, which is very stable in solution.



The formula unit of the title complex, (I), consists of a hexaquaairon(II) dication, two nitrate anions, two hexamethylenetetramine molecules, and four uncoordinated water molecules, as shown in Fig. 1. The iron atom is situated on an inversion center and is coordinated by six water molecules, displaying an octahedral  $\text{FeO}_6$  geometry. The  $\text{Fe}-\text{O}$  bond lengths are in the range 2.024 (2)–2.065 (2)  $\text{\AA}$ , which is normal for ferrous complexes with water. The three trans angles of the  $\text{FeO}_6$  octahedron are all  $180^\circ$  by symmetry. The other angles around atom Fe1 vary from 87.60 (8) to 92.40 (8)°, indicating a slightly distorted octahedral environment.

All the N atoms in the hexamethylenetetramine molecules, all the water molecules and the O atoms of the nitrate anions



**Figure 1**

The structure of the formula unit (twice the asymmetric unit) of the title compound (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme; the suffix A denotes an atom generated by the inversion center.

contribute to the formation of hydrogen bonds, leading to the formation of a three-dimensional network (see Table 1 and Fig. 2).

## Experimental

$\text{Fe}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  (1.0 mmol, 288 mg) and hexamethylenetetramine (2.0 mmol, 280 mg) were dissolved in water (10 ml) and stirred for *ca* 10 min to obtain a clear purple solution. After keeping the resulting solution in air for 3 d large violet crystals were formed. The crystals were isolated, washed with water three times and dried in a vacuum desiccator using  $\text{CaCl}_2$  (yield 75%). Elemental analysis found: C 22.59, H 6.95, N 21.80%; calculated for  $\text{C}_{12}\text{H}_{44}\text{FeN}_{10}\text{O}_{16}$ : C 22.51, H 6.93, N 21.87%.

### Crystal data

$[\text{Fe}(\text{H}_2\text{O})_6](\text{NO}_3)_2 \cdot 2\text{C}_6\text{H}_{12}\text{N}_4 \cdot 4\text{H}_2\text{O}$	$Z = 1$
$M_r = 640.42$	$D_x = 1.523 \text{ Mg m}^{-3}$
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 9.104 (4) \text{ \AA}$	Cell parameters from 2715 reflections
$b = 9.349 (4) \text{ \AA}$	$\theta = 3.0\text{--}26.4^\circ$
$c = 9.700 (4) \text{ \AA}$	$\mu = 0.63 \text{ mm}^{-1}$
$\alpha = 87.743 (5)^\circ$	$T = 298 (2) \text{ K}$
$\beta = 75.674 (4)^\circ$	Prism, violet
$\gamma = 61.248 (5)^\circ$	$0.33 \times 0.25 \times 0.18 \text{ mm}$
$V = 698.3 (5) \text{ \AA}^3$	

### Data collection

Siemens SMART CCD area-detector diffractometer	2436 independent reflections
$\varphi$ and $\omega$ scans	2261 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$R_{\text{int}} = 0.015$
$T_{\text{min}} = 0.820$ , $T_{\text{max}} = 0.896$	$\theta_{\text{max}} = 25.0^\circ$
3689 measured reflections	$h = -10 \rightarrow 9$
	$k = -10 \rightarrow 11$
	$l = -11 \rightarrow 11$

### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0803P)^2 + 0.1663P]$
$R[F^2 > 2\sigma(F^2)] = 0.043$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.117$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.09$	$\Delta\rho_{\text{max}} = 0.63 \text{ e \AA}^{-3}$
2436 reflections	$\Delta\rho_{\text{min}} = -0.46 \text{ e \AA}^{-3}$
266 parameters	
All H-atom parameters refined	

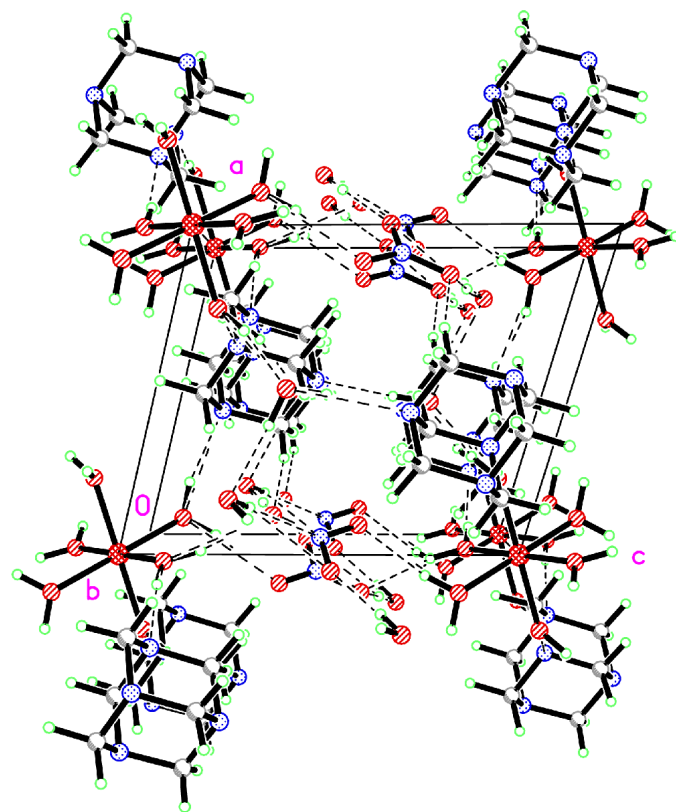
**Table 1**

Hydrogen-bonding geometry ( $\text{\AA}$ ,  $^\circ$ ).

$D\text{--H}\cdots A$	$D\text{--H}$	$\text{H}\cdots A$	$D\cdots A$	$D\text{--H}\cdots A$
$\text{O1--H1}\cdots\text{O4}^{\text{i}}$	0.907 (10)	1.982 (13)	2.874 (4)	167 (3)
$\text{O1--H2}\cdots\text{N4}^{\text{ii}}$	0.904 (10)	1.997 (15)	2.879 (3)	165 (3)
$\text{O2--H3}\cdots\text{O7}^{\text{iii}}$	0.905 (10)	1.785 (15)	2.674 (3)	167 (4)
$\text{O2--H4}\cdots\text{N2}^{\text{iv}}$	0.903 (10)	1.908 (13)	2.803 (3)	171 (4)
$\text{O3--H5}\cdots\text{O6}$	0.911 (10)	1.965 (15)	2.850 (3)	163 (3)
$\text{O3--H6}\cdots\text{N5}^{\text{v}}$	0.913 (10)	1.931 (13)	2.830 (3)	168 (4)
$\text{O7--H7}\cdots\text{N3}$	0.906 (10)	1.924 (11)	2.828 (3)	175 (3)
$\text{O7--H8}\cdots\text{O8}^{\text{vi}}$	0.907 (10)	1.917 (12)	2.819 (4)	174 (4)
$\text{O8--H9}\cdots\text{O5}^{\text{vii}}$	0.912 (10)	1.887 (15)	2.791 (5)	171 (5)
$\text{O8--H10}\cdots\text{O6}^{\text{vi}}$	0.906 (11)	2.02 (2)	2.899 (5)	162 (6)

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

All the H atoms were located in Fourier difference maps and refined. The C—H H atoms were refined isotropically. The water O—



**Figure 2**

Crystal packing of (I), showing the hydrogen-bonding interactions as dashed lines.

H distances were restrained to 0.91 (1)  $\text{\AA}$ , but  $U_{\text{iso}}(\text{H})$  values were allowed to refine. The displacement parameters for atom O5 are quite large; however, no attempt was made to introduce a disorder model.

Data collection: SMART (Siemens, 1996); cell refinement: SMART; data reduction: SAINT (Siemens, 1996); program(s) used to solve structure: SHELXTL (Sheldrick, 1997); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

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