

## New iron(III) nitrate hydrates: $\text{Fe}(\text{NO}_3)_3 \cdot x\text{H}_2\text{O}$ with $x = 4, 5$ and $6$

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Received 8 March 2012  
Accepted 11 April 2012  
Online 12 May 2012

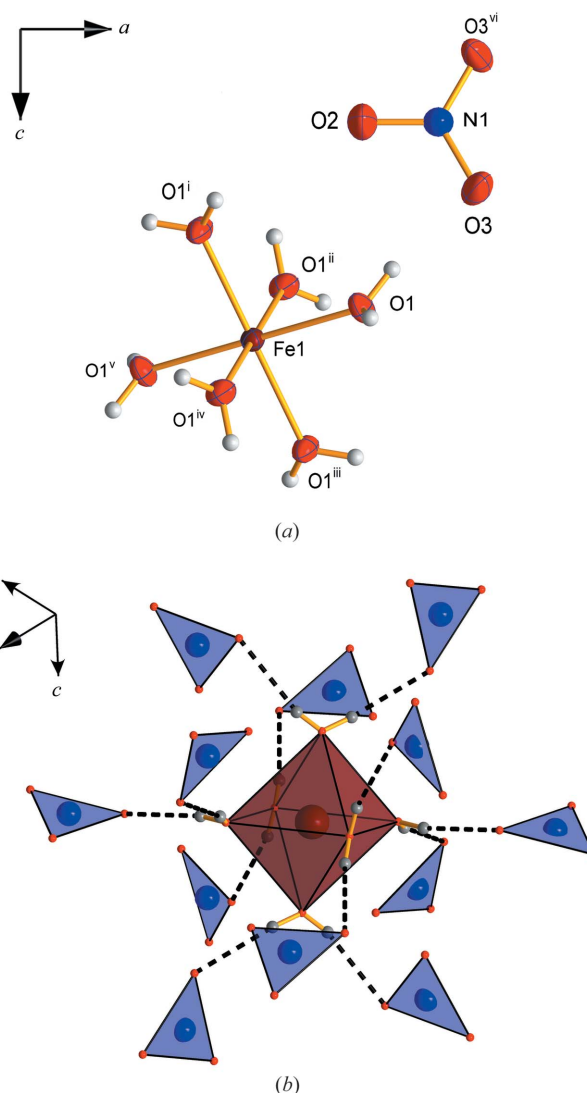
Crystals of the title compounds were grown from their hydrous melts or solutions. The crystal structure of iron(III) trinitrate hexahydrate {hexaaquairon(III) trinitrate,  $[\text{Fe}(\text{H}_2\text{O})_6](\text{NO}_3)_3$ } is built up from  $[\text{Fe}(\text{H}_2\text{O})_6]^{2+}$  octahedra and nitrate anions connected *via* hydrogen bonds. In iron(III) trinitrate pentahydrate {pentaquanitratoiron(III) dinitrate,  $[\text{Fe}(\text{NO}_3)(\text{H}_2\text{O})_5](\text{NO}_3)_2$ }, one water molecule in the coordination octahedron of the  $\text{Fe}^{\text{III}}$  atom is substituted by an O atom of a nitrate group. Iron(III) trinitrate tetrahydrate {triaquadinitratoiron(III) nitrate monohydrate,  $[\text{Fe}(\text{NO}_3)_2(\text{H}_2\text{O})_3]\text{NO}_3 \cdot \text{H}_2\text{O}$ } represents the first example of a simple iron(III) nitrate with pentagonal–bipyramidal coordination geometry, where two bidentate nitrate anions and one water molecule form a pentagonal plane.

### Comment

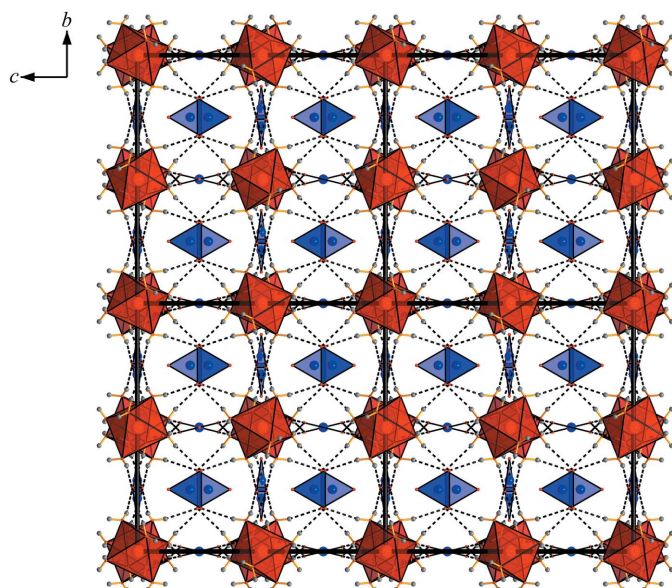
The nonahydrate  $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  crystallizes from aqueous solutions of iron(III) nitrate. Its crystal structure was reported by Hair & Beattie (1977). Because of its strong hygroscopicity and the tendency of iron(III) nitrate to hydrolyze and precipitate basic salts, neutral hydrates containing less than 9 mol water per mol salt do not crystallize from simple aqueous solutions. To prevent hydrolysis, lower hydrates can be crystallized only in the presence of high concentrations of nitric acid. Reports on the existence and stability of lower hydrates are contradictory. According to Cameron & Robinson (1909), the hexahydrate crystallizes from a solution of nonahydrate in nitric acid. Malquori (1929*a,b*) proposed the addition of  $\text{N}_2\text{O}_5$ . Hathaway & Underhill (1960) claimed to have synthesized a dihydrate by means of a reaction of anhydrous iron(III) chloride in pure nitric acid. Rodenko & Panov (1994) performed solubility determinations at temperatures in the range 283–313 K in the system  $\text{Fe}(\text{NO}_3)_3\text{–HNO}_3\text{–H}_2\text{O}$ . Graphically presented solubility isotherms were interpreted as branches from hexa- and tetrahydrate at nitric acid concentrations of 56–77%  $\text{HNO}_3$  and 77–85%  $\text{HNO}_3$ , respectively.

EL Goundali & Kaddami (2006, 2007, 2008) claimed the formation of an iron(III) nitrate hexahydrate within the system  $\text{Fe}(\text{NO}_3)_3\text{–Co}(\text{NO}_3)_2\text{–HNO}_3$  at 303 K. Addison (1980) and Addison & Chapman (1965) thoroughly investigated reactions of liquid  $\text{N}_2\text{O}_4$  with hydrated nitrates and metals. Addison stated that iron(III) nitrate nonahydrate reacted with  $\text{N}_2\text{O}_4$  to yield a yellow–brown pentahydrate (Addison & Chapman, 1965). The same authors supposed that the reaction product of metallic iron with an  $\text{N}_2\text{O}_4/\text{HNO}_3$  mixture was a dihydrate after two weeks evacuation. For all these phases, no X-ray diffraction pattern was recorded. The chemical formulae were derived from chemical analyses of the wet solid residues.

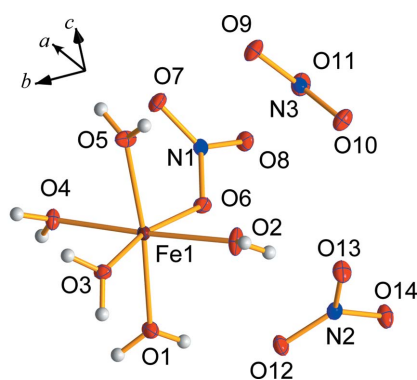
Crystals of the title compounds were grown from their hydrous melts or solutions in highly concentrated nitric acid.



**Figure 1**  
(a) The asymmetric unit and symmetry-related atoms of  $\text{Fe}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ . Displacement ellipsoids are drawn at the 50% probability level. [Symmetry codes: (i)  $-y + 1, -z + 1, -x + 1$ ; (ii)  $-z + 1, -x + 1, -y + 1$ ; (iii)  $y, z, x$ ; (iv)  $z, x, y$ ; (v)  $-x + 1, -y + 1, -z + 1$ ; (vi)  $x, -y + 1, -z + \frac{1}{2}$ ] (b) The  $[\text{Fe}(\text{H}_2\text{O})_6]^{2+}$  octahedron with hydrogen bonds (dashed lines) to nitrate anions (triangles) in iron(III) nitrate hexahydrate.



**Figure 2**  
 $2 \times 2 \times 2$  unit cells of  $\text{Fe}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ , viewed in the  $a$ -axis direction.

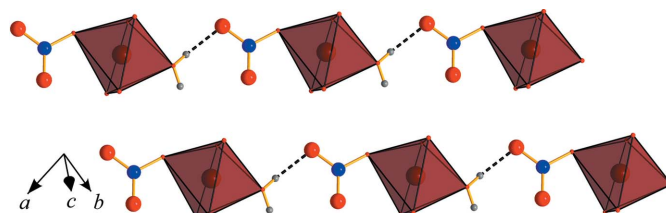


**Figure 3**  
 The asymmetric unit of  $\text{Fe}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$ . Displacement ellipsoids are drawn at the 50% probability level.

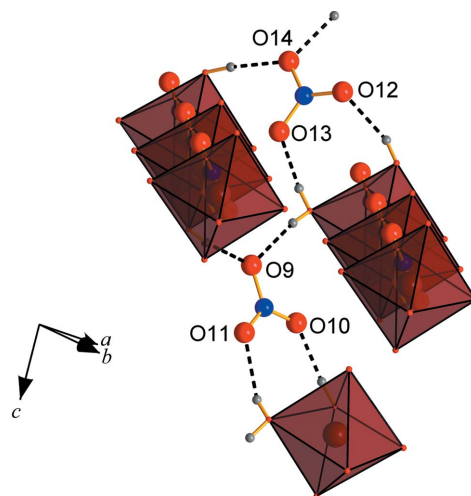
Iron(III) nitrate pentahydrate, (II), was prepared through the reaction of liquid  $\text{N}_2\text{O}_4$  with iron(III) nitrate nonahydrate. The hexahydrate, (I), and tetrahydrate, (III), were obtained by dissolving the pentahydrate in highly concentrated nitric acid and subsequent evaporation at reduced pressure. With the successful preparation of single crystals and their structure determination in this work, the existence and stoichiometry of at least three lower hydrates can be confirmed unequivocally.

As expected, the crystal structure of the hexahydrate consists of discrete  $[\text{Fe}(\text{H}_2\text{O})_6]^{2+}$  octahedra and nitrate anions (see Fig. 1*a*). The  $[\text{Fe}(\text{H}_2\text{O})_6]^{2+}$  octahedra are connected *via* hydrogen bonds to nitrate ions, where every H atom of the six water molecules is in contact with a different nitrate anion, with a  $\text{H} \cdots \text{O}$  distance of 1.89 (1) Å (Table 1), thus arranging 12 nitrate anions around an octahedral unit (Fig. 1*b*). The result is a complex but highly symmetric hydrogen-bond network, as shown in Fig. 2.

With a further decrease of the water content, nitrate ions enter the coordination sphere of iron. In  $\text{Fe}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$ , the



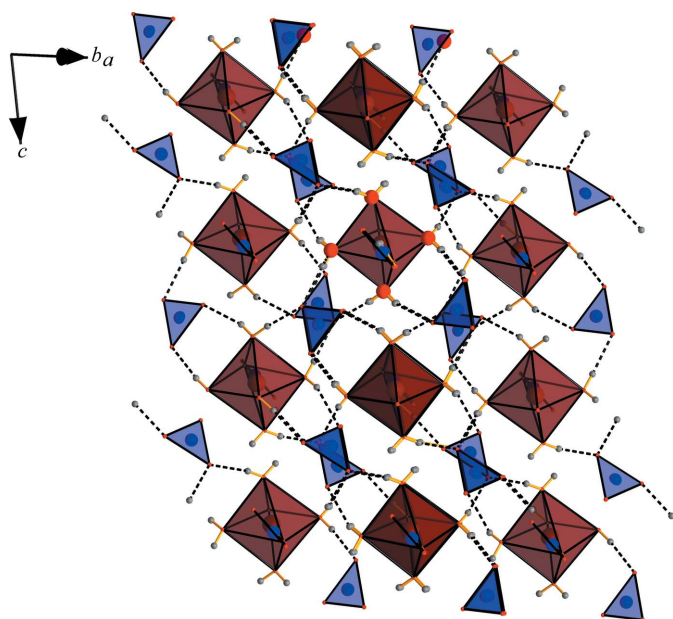
**Figure 4**  
 The chain structure in  $\text{Fe}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$ , based on hydrogen bonds of the monodentate nitrate anions.



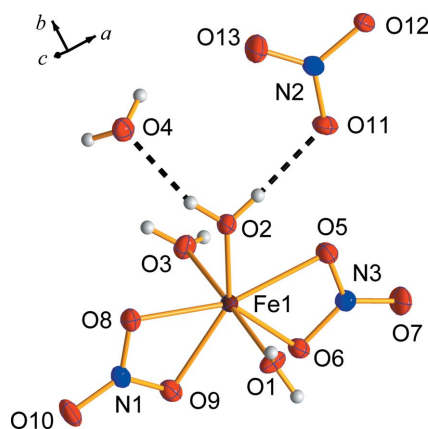
**Figure 5**  
 Noncoordinated nitrate anions connecting chains *via* hydrogen bonds in  $\text{Fe}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$ . Atoms O10/O11 and O12/O13 are bound to water molecules from the same cation, thereby spanning an edge of an octahedron, whereas O9 and O14 join different chains *via* the corners of adjacent octahedra.

$\text{Fe}^{\text{III}}$  atom is still coordinated by six ligands (see Fig. 3) completing the coordination sphere with a monodentate nitrate ligand. One of the other O atoms of the coordinated nitrate ion forms a hydrogen bond to a water molecule of an adjacent octahedral unit (Fig. 4 and Table 2). This yields two nitrate octahedral chains running approximately in the [110] direction (Fig. 4). These chains are connected by the noncoordinated nitrate ions *via* hydrogen bonds, as shown in Fig. 5. Thus, two pairs of O atoms (O10/O11 and O12/O13) each span an edge of an octahedron and the third O atom (O9 and O14) combines with different chains. The result is a three-dimensional network (Fig. 6).

Surprisingly, in  $\text{Fe}(\text{NO}_3)_3 \cdot 4\text{H}_2\text{O}$ , the coordination number of iron(III) is enhanced to seven in the form of a pentagonal bipyramid (Fig. 7). Two bidentate nitrate ions and one water molecule (O2) are arranged nearly exactly in a pentagonal plane. The other two coordinated water molecules in axial positions are nearly perpendicular to this plane. The noncoordinated water molecule is hydrogen bonded to an axial water molecule of one complex cation and to the equatorial water molecule of another complex cation (Table 3), thus forming binary complex units with the equatorial water molecules directed towards each other (Fig. 8). These double



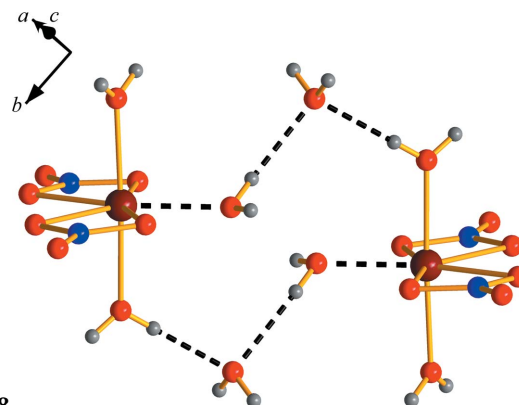
**Figure 6**  
Three-dimensional hydrogen-bond network in  $\text{Fe}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$ .



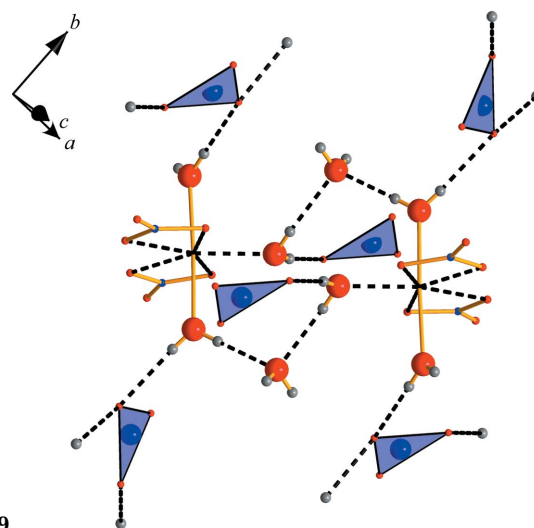
**Figure 7**  
The asymmetric unit of  $\text{Fe}(\text{NO}_3)_3 \cdot 4\text{H}_2\text{O}$ . Displacement ellipsoids are drawn at the 50% probability level.

units are interconnected *via* hydrogen bonds involving the noncoordinated nitrate ions. As illustrated in Fig. 9, every water molecule of the complex unit is connected to a nitrate ion. Thus, one O atom of the nitrate (O12) ion connects two complex units through their axial water molecules approximately in the direction of the *b* axis. The second O atom (O11) is connected with the equatorial water molecule of the complex. Altogether, a hydrogen-bond network arises with dominating bonds along the *b* and *a* axes, as shown in Fig. 10.

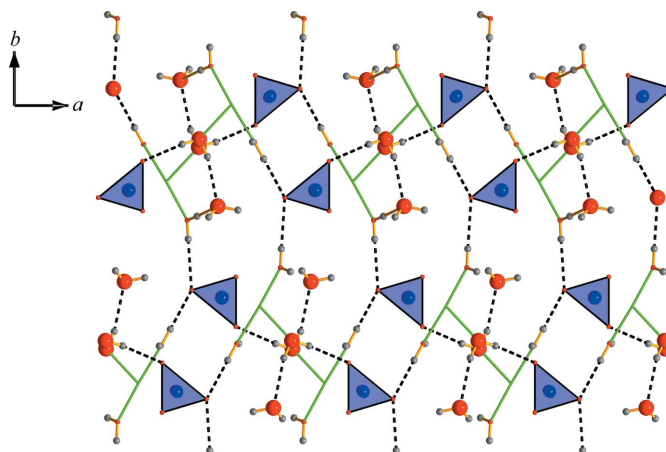
There are examples in the literature of salts with tetranitratoferrate anions,  $[\text{Fe}(\text{NO}_3)_4]^-$ , where the nitrate groups coordinate in a bidentate fashion, resulting in a coordination number of eight for  $\text{Fe}^{\text{III}}$  (Addison & Chapman, 1965; Tikhomirov *et al.*, 2002; Blackwell *et al.*, 1975; Fedorova *et al.*, 2002). However, the pentagonal-bipyramidal coordination of  $\text{Fe}^{\text{III}}$  has only been observed before for metal-organic  $\text{Fe}^{\text{III}}$  complexes (Andjelkovic *et al.*, 2002; Bonardi *et al.*, 1991). The



**Figure 8**  
Noncoordinated water molecules in  $\text{Fe}(\text{NO}_3)_3 \cdot 4\text{H}_2\text{O}$  connecting two pentagonal-bipyramidal  $[\text{Fe}(\text{NO}_3)_2(\text{H}_2\text{O})_3]^+$  units *via* hydrogen bonds (black lines).



**Figure 9**  
Interconnection of the structure units of  $\text{Fe}(\text{NO}_3)_3 \cdot 4\text{H}_2\text{O}$ , illustrated in Fig. 8 by hydrogen bonds (black lines) involving noncoordinated nitrate ions.



**Figure 10**  
Hydrogen-bond network in  $\text{Fe}(\text{NO}_3)_3 \cdot 4\text{H}_2\text{O}$ , with dominating bonds along the *b* axis. For clarity, only the  $\text{Fe}-\text{OH}_2$  bonds (thick lines, green in the electronic version of the paper) of the complex units are shown.

reported structure for the iron(III) nitrate tetrahydrate represents the first simple iron(III) salt with such a coordination geometry.

## Experimental

Iron(III) nitrate hexahydrate, (I), was prepared by recrystallizing  $\text{Fe}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$  (2 g) from pure nitric acid (1 ml of 96.2%  $\text{HNO}_3$ ) and concentrating the liquid by evaporation at reduced pressure. Colourless cubic crystals of 2–4 mm size were obtained after 14 d. The selected crystal was cut and embedded in perfluorinated ether for single-crystal diffraction experiments.

Iron(III) nitrate pentahydrate, (II), was prepared according to the method of Addison & Chapman (1965).  $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  (10 g) was reacted under vigorous stirring in an excess of liquid  $\text{N}_2\text{O}_4$  (30 ml) for 3–5 d at room temperature to form a pale-yellow powder of the pentahydrate. To obtain bigger crystals, the  $\text{N}_2\text{O}_4$  was removed under vacuum and the remaining mixture was recrystallized in sealed glass tubes by warming and subsequent cooling to room temperature. A piece of the 1 mm-sized crystal was cut and embedded in perfluoroether to prevent contact with the air humidity before being mounted on the single-crystal diffractometer.

Iron(III) nitrate tetrahydrate, (III), was prepared using a method analogous to that used for the hexahydrate using  $\text{Fe}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$  (2 g) in pure nitric acid (1 ml of 96.2%  $\text{HNO}_3$ ). Brown needle-shaped crystals with a size of approximately  $5 \times 0.2$  mm were obtained after 14 d. One piece of a crystal was cut and embedded in perfluoroether before being mounted on the single-crystal diffractometer.

## Compound (I)

### Crystal data

$[\text{Fe}(\text{H}_2\text{O})_6](\text{NO}_3)_3$	$Z = 8$
$M_r = 349.98$	Mo $K\alpha$ radiation
Cubic, $Ia\bar{3}$	$\mu = 1.23 \text{ mm}^{-1}$
$a = 13.7962$ (2) Å	$T = 190 \text{ K}$
$V = 2625.90$ (7) Å <sup>3</sup>	$0.43 \times 0.35 \times 0.29 \text{ mm}$

### Data collection

Bruker X8 Kappa APEXII diffractometer	9913 measured reflections
Absorption correction: numerical (APEX2; Bruker, 2005)	505 independent reflections
$T_{\min} = 0.623$ , $T_{\max} = 0.715$	432 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.027$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.028$	3 restraints
$wR(F^2) = 0.081$	All H-atom coordinates refined
$S = 1.09$	$\Delta\rho_{\text{max}} = 0.58 \text{ e \AA}^{-3}$
505 reflections	$\Delta\rho_{\text{min}} = -0.38 \text{ e \AA}^{-3}$
39 parameters	

## Compound (II)

### Crystal data

$[\text{Fe}(\text{NO}_3)(\text{H}_2\text{O})_5](\text{NO}_3)_2$	$\gamma = 70.279$ (2)°
$M_r = 331.96$	$V = 518.72$ (5) Å <sup>3</sup>
Triclinic, $P\bar{1}$	$Z = 2$
$a = 6.7693$ (4) Å	Mo $K\alpha$ radiation
$b = 7.0692$ (4) Å	$\mu = 1.55 \text{ mm}^{-1}$
$c = 11.6734$ (6) Å	$T = 100 \text{ K}$
$\alpha = 85.568$ (2)°	$0.47 \times 0.35 \times 0.23 \text{ mm}$
$\beta = 80.655$ (1)°	

**Table 1**  
Hydrogen-bond geometry (Å, °) for (I).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{O1}-\text{H2} \cdots \text{O3}$	0.82 (1)	1.89 (1)	2.695 (2)	169 (3)
$\text{O1}-\text{H1} \cdots \text{O3}^i$	0.82 (1)	1.89 (1)	2.6946 (19)	169 (3)

Symmetry code: (i)  $-z + 1, -x + \frac{1}{2}, y$ .

### Data collection

Bruker X8 Kappa APEXII diffractometer	9108 measured reflections
Absorption correction: multi-scan (SADABS; Bruker, 2006)	2373 independent reflections
$T_{\min} = 0.528$ , $T_{\max} = 0.716$	2293 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.017$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.019$	14 restraints
$wR(F^2) = 0.059$	All H-atom coordinates refined
$S = 1.02$	$\Delta\rho_{\text{max}} = 0.46 \text{ e \AA}^{-3}$
2373 reflections	$\Delta\rho_{\text{min}} = -0.30 \text{ e \AA}^{-3}$
203 parameters	

## Compound (III)

### Crystal data

$[\text{Fe}(\text{NO}_3)_2(\text{H}_2\text{O})_3]\text{NO}_3 \cdot \text{H}_2\text{O}$	$V = 1005.86$ (16) Å <sup>3</sup>
$M_r = 313.94$	$Z = 4$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
$a = 7.0696$ (7) Å	$\mu = 1.58 \text{ mm}^{-1}$
$b = 15.1917$ (16) Å	$T = 100 \text{ K}$
$c = 9.4264$ (7) Å	$0.5 \times 0.4 \times 0.3 \text{ mm}$
$\beta = 96.508$ (3)°	

### Data collection

Bruker X8 Kappa APEXII diffractometer	11328 measured reflections
Absorption correction: multi-scan (SADABS; Bruker, 2006)	2303 independent reflections
$T_{\min} = 0.451$ , $T_{\max} = 0.602$	1985 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.031$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.022$	12 restraints
$wR(F^2) = 0.055$	All H-atom coordinates refined
$S = 1.07$	$\Delta\rho_{\text{max}} = 0.26 \text{ e \AA}^{-3}$
2303 reflections	$\Delta\rho_{\text{min}} = -0.26 \text{ e \AA}^{-3}$
186 parameters	

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

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{O2}-\text{H1} \cdots \text{O9}^i$	0.82 (1)	1.93 (1)	2.7339 (14)	169 (2)
$\text{O2}-\text{H2} \cdots \text{O13}$	0.78 (2)	1.93 (2)	2.6933 (14)	169 (2)
$\text{O1}-\text{H3} \cdots \text{O14}^{ii}$	0.82 (1)	1.89 (1)	2.7027 (13)	171 (2)
$\text{O1}-\text{H4} \cdots \text{O12}$	0.82 (1)	2.02 (1)	2.8069 (13)	161 (2)
$\text{O3}-\text{H5} \cdots \text{O8}^{iii}$	0.82 (1)	1.90 (1)	2.7062 (13)	168 (2)
$\text{O3}-\text{H5} \cdots \text{N1}^{iii}$	0.82 (1)	2.54 (1)	3.2543 (14)	147 (2)
$\text{O3}-\text{H5} \cdots \text{O7}^{iii}$	0.82 (1)	2.57 (2)	3.0736 (13)	121 (2)
$\text{O3}-\text{H6} \cdots \text{O10}^{ii}$	0.82 (1)	1.84 (1)	2.6571 (13)	174 (2)
$\text{O4}-\text{H7} \cdots \text{O14}^{iv}$	0.82 (1)	1.82 (1)	2.6338 (13)	175 (2)
$\text{O4}-\text{H8} \cdots \text{O11}^{ii}$	0.82 (1)	2.01 (1)	2.7844 (13)	159 (2)
$\text{O5}-\text{H9} \cdots \text{O9}^v$	0.82 (1)	1.89 (1)	2.6934 (14)	168 (2)
$\text{O5}-\text{H10} \cdots \text{O10}^i$	0.82 (1)	2.19 (1)	2.8691 (13)	142 (2)
$\text{O5}-\text{H10} \cdots \text{O9}$	0.82 (1)	2.55 (2)	3.0377 (14)	120 (2)

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

All water H atoms were initially located in difference Fourier maps and then their positions were refined with O—H and H···H distance restraints of 0.820 (5) and 1.30 (2) Å, respectively.

For all compounds, data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *pubCIF* (Westrip, 2010).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: YP3012). Services for accessing these data are described at the back of the journal.

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**Table 3**

Hydrogen-bond geometry (Å, °) for (III).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
O1—H1···O12 <sup>i</sup>	0.82 (1)	1.87 (1)	2.6858 (16)	177 (2)
O1—H2···O4 <sup>ii</sup>	0.81 (1)	1.82 (1)	2.6304 (17)	173 (2)
O3—H3···O11 <sup>iii</sup>	0.81 (1)	2.09 (1)	2.8627 (17)	158 (2)
O3—H3···O5 <sup>iii</sup>	0.81 (1)	2.44 (2)	2.9572 (16)	122 (2)
O3—H4···O12 <sup>iv</sup>	0.82 (1)	1.87 (1)	2.6852 (17)	174 (2)
O2—H5···O4	0.82 (1)	1.91 (1)	2.7036 (16)	163 (2)
O2—H6···O11	0.82 (1)	1.90 (1)	2.7117 (16)	172 (2)
O4—H7···O9 <sup>v</sup>	0.82 (1)	2.44 (2)	3.0518 (17)	133 (2)
O4—H7···O7 <sup>iii</sup>	0.82 (1)	2.48 (2)	3.0896 (17)	132 (2)
O4—H7···O13	0.82 (1)	2.51 (2)	3.0140 (17)	121 (2)
O4—H8···O12 <sup>iv</sup>	0.82 (1)	2.31 (2)	2.9587 (17)	137 (2)
O4—H8···O6 <sup>v</sup>	0.82 (1)	2.55 (2)	3.0163 (16)	118 (2)

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

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