

# Bis(propane-1,3-diaminium) hexafluoridoferrate(III) fluoride trihydrate

Aymen Zouaghi,<sup>a</sup> Amor BenAli,<sup>a\*</sup> Vincent Maisonneuve<sup>b</sup> and Marc Leblanc<sup>b</sup>

<sup>a</sup>Unité de Recherche 99/UR12-30, Faculté des Sciences de Bizerte, 7021 Jarzouna, Tunisia, and <sup>b</sup>Laboratoire des Oxydes et Fluorures - UMR 6010 CNRS, Institut de Recherche en Ingénierie Moléculaire et Matériaux, Fonctionnels, IRIM2F FR CNRS 2775, Faculté des Sciences et Techniques, Université du Maine, Avenue Olivier Messiaen, 72085 LE MANS Cedex 9, France  
Correspondence e-mail: amor.benali@fsb.rnu.tn

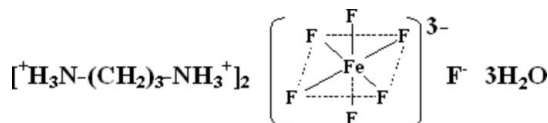
Received 6 April 2010; accepted 15 May 2010

Key indicators: single-crystal X-ray study;  $T = 295$  K; mean  $\sigma(C-C) = 0.004$  Å;  $R$  factor = 0.031;  $wR$  factor = 0.093; data-to-parameter ratio = 13.3.

The asymmetric unit of the title iron hybrid fluoride,  $(C_3H_{12}N_2)_2[FeF_6]F \cdot 3H_2O$ , contains two propane-1,3-diaminium  $[(H_2dap)^{2+}]$  cations, an octahedral  $[FeF_6]^{3-}$  anion, an isolated  $F^-$  anion and three water molecules of solvation. Each  $[FeF_6]^{3-}$  anion is surrounded by four separate hydrogen-bonded water molecules in the equatorial sites and by five separate aminium cation donor groups. The axial F atoms are only involved in  $N-H \cdots F$  hydrogen bonds, resulting in a three-dimensional structure.

## Related literature

For general background to hybrid fluorides, their synthesis and their applications, see: Ben Ali *et al.* (2007, 2009); Adil *et al.* (2007); Latroche *et al.* (2006); Rother *et al.* (1998), Bentrup *et al.* (1998). For  $F \cdots N$  interactions, see: Steiner (1998). For bond-valence sum (BVS) calculations, see: Brese & O'Keeffe (1991).



## Experimental

### Crystal data

$(C_3H_{12}N_2)_2[FeF_6]F \cdot 3H_2O$   
 $M_r = 395.18$   
Triclinic,  $P\bar{1}$   
 $a = 9.844$  (1) Å  
 $b = 9.847$  (1) Å  
 $c = 10.7740$  (8) Å  
 $\alpha = 106.959$  (7)°  
 $\beta = 95.379$  (6)°

$\gamma = 118.914$  (9)°  
 $V = 839.35$  (17) Å<sup>3</sup>  
 $Z = 2$   
Mo  $K\alpha$  radiation  
 $\mu = 0.98$  mm<sup>-1</sup>  
 $T = 295$  K  
 $0.32 \times 0.07 \times 0.07$  mm

### Data collection

SIEMENS AED2 diffractometer  
2920 measured reflections  
2920 independent reflections

2599 reflections with  $I > 2\sigma(I)$   
3 standard reflections every 120 min  
intensity decay: 4%

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.031$   
 $wR(F^2) = 0.093$   
 $S = 1.14$   
2920 reflections  
219 parameters

H atoms treated by a mixture of independent and constrained refinement  
 $\Delta\rho_{\max} = 0.42$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.35$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$N1-H1B \cdots F2^i$	0.89	2.03	2.826 (3)	148
$N1-H1B \cdots F5^i$	0.89	2.22	2.839 (3)	127
$N2-H2A \cdots F4^{ii}$	0.89	1.82	2.672 (3)	161
$N2-H2B \cdots F7^{ii}$	0.89	1.85	2.735 (3)	172
$N2-H2C \cdots O1W^{iii}$	0.89	2.22	2.926 (3)	136
$N2-H2C \cdots F6^{iii}$	0.89	2.47	3.139 (3)	132
$N3-H3A \cdots F3^{iii}$	0.89	1.95	2.777 (3)	155
$N3-H3A \cdots F4^{ii}$	0.89	2.47	3.135 (3)	132
$N3-H3B \cdots F3^{iv}$	0.89	1.93	2.762 (3)	156
$N4-H4A \cdots F7^v$	0.89	1.86	2.728 (3)	164
$N4-H4B \cdots F6^{vi}$	0.89	2.09	2.886 (3)	149
$N4-H4B \cdots F1^{vi}$	0.89	2.33	3.029 (3)	135
$O1W-H12 \cdots O3W^i$	0.81 (4)	1.99 (4)	2.787 (4)	173 (4)
$O2W-H21 \cdots F1^{vi}$	0.76 (4)	1.91 (4)	2.606 (3)	153 (4)
$O2W-H22 \cdots F6^i$	0.76 (4)	1.99 (4)	2.747 (3)	171 (4)
$O3W-H32 \cdots O2W^{vii}$	0.73 (4)	2.04 (4)	2.766 (4)	173 (4)

Symmetry codes: (i)  $-x+2, -y+1, -z+1$ ; (ii)  $-x+1, -y, -z+1$ ; (iii)  $x-1, y, z$ ; (iv)  $x, y, z+1$ ; (v)  $-x+2, -y+1, -z+2$ ; (vi)  $x, y+1, z+1$ ; (vii)  $-x+1, -y+1, -z+1$ .

Data collection: *STADIA* (Stoe & Cie, 1998); cell refinement: *STADIA*; data reduction: *X-RED* (Stoe & Cie, 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008) within *WinGX* (Farrugia, 1999); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2004) and *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *enCIFer* (Allen *et al.*, 2004).

The authors thank Professor L. S. Smiri for helpful discussions.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZS2036).

## References

- Adil, K. M., Ali Saada, M., Ben Ali, A., Body, M., Dang, M. T., Hémon-Ribaud, A., Leblanc, M. & Maisonneuve, V. (2007). *J. Fluorine Chem.* **128**, 404–412.  
Allen, F. H., Johnson, O., Shields, G. P., Smith, B. R. & Towler, M. (2004). *J. Appl. Cryst.* **37**, 335–338.  
Ben Ali, A., Dang, M. T., Grenèche, J.-M., Hémon-Ribaud, A., Leblanc, M. & Maisonneuve, V. (2007). *J. Solid State Chem.* **180**, 1911–1917.  
Ben Ali, A., Grenèche, J.-M., Leblanc, M. & Maisonneuve, V. (2009). *Solid State Sci.* **11**, 1631–1638.  
Bentrup, U., Ahmadi, A., Kang, H. C. & Massa, W. (1998). *Z. Anorg. Allg. Chem.* **624**, 1465–1470.  
Brandenburg, K. & Putz, H. (2004). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.  
Brese, N. E. & O'Keeffe, M. (1991). *Acta Cryst.* **B47**, 192–197.  
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.  
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.

- Latroche, M., Surble, S., Serre, C., Mellot-Draznieks, C., Llewellyn, P., Lee, J.-H., Jung, J.-S. & Férey, G. (2006). *Angew. Chem. Int. Ed.* **45**, 8227–8231.
- Rother, G., Worzala, H. & Bentrup, U. (1998). *Z. Kristallogr. New Cryst. Struct.* **213**, 119–120.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Steiner, T. (1998). *Acta Cryst.* **B54**, 456–463.
- Stoe & Cie (1998). *STADIA* and *X-RED*. Stoe & Cie GmbH, Darmstadt, Germany.

## supporting information

*Acta Cryst.* (2010). E66, m702–m703 [https://doi.org/10.1107/S1600536810018039]

**Bis(propane-1,3-diaminium) hexafluoridoferrate(III) fluoride trihydrate****Aymen Zouaghi, Amor BenAli, Vincent Maisonneuve and Marc Leblanc****S1. Comment**

The structure of the title compound  $(\text{H}_2\text{dap})_2[\text{FeF}_6](\text{F})\cdot 3\text{H}_2\text{O}$  (I) consists of isolated  $\text{FeF}_6$  octahedra, diprotonated 1,3-diaminopropane  $(\text{H}_2\text{dap})^{2+}$  cations and three water molecules of solvation connected by a three-dimensional framework of hydrogen bonds in which isolated fluoride anions are located (Figure 1 and Figure 2). In (I) the  $[\text{FeF}_6]$  complex anion adopts a slightly distorted octahedral environment, the Fe—F bond distance range [1.897 (2)–1.947 (2) Å] being typical of an octahedral iron<sup>III</sup> environment. Each octahedral  $\text{FeF}_6^{3-}$  anion is surrounded by four separate hydrogen-bonded water molecules in the equatorial sites and by seven separate aminium cation donor groups (Figure 3). The axial F atoms (F2, F4) are involved only in N—H $\cdots$ F interactions (Table 1). One of the equatorial F atom (F3), which has the longest Fe—F bond distance [1.947 (2)] Å, establishes three hydrogen bonds and consequently presents a low valence (0.47) with Fe<sup>III</sup>.

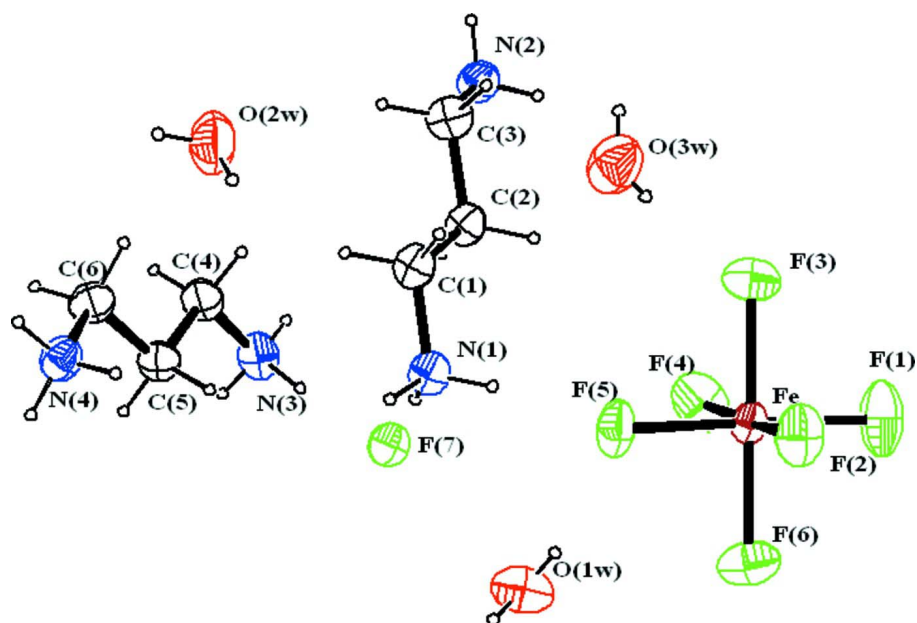
In fluoride metallates, "free" fluoride ions, are always surrounded by amine groups and their coordination number varies from 3 to 6. Also F $\cdots$ N distances increase with the coordination number (Steiner, 1998). In the title compound, "free" F ions adopt a tetrahedral coordination with four hydrogen atoms from four  $\text{H}_2\text{dap}$  cations (Figure 4). The three hydrogen-bonded water molecules form trimer clusters, presenting various triangular environments with F acceptor atoms of the  $\text{FeF}_6$  octahedra and H donor atoms of the cation aminium groups (Figure 5). The infrared absorption spectrum of the title compound gives information on the organic moiety (C—C, C—N) and on the oxidation state of the iron atom, the presence of a vibrational band in the neighbourhood of 487  $\text{cm}^{-1}$  being consistent with iron(III).

**S2. Experimental**

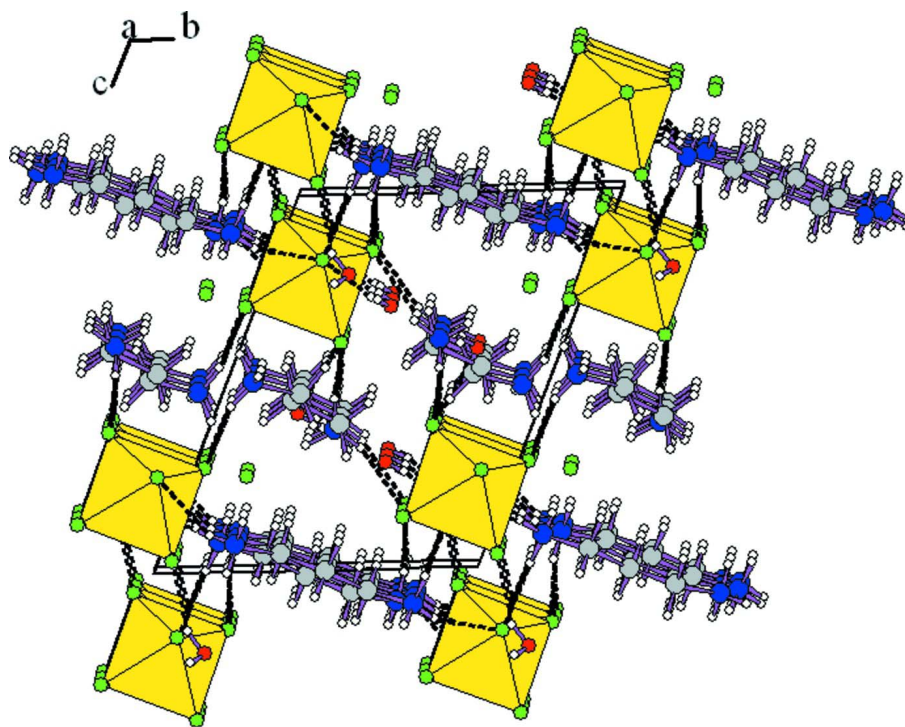
The title compound was prepared from a starting mixture of  $\text{FeF}_3$  (0.5 g) in 40% HF (3.0 ml) and ethanol (5 ml). 1,3-diaminopropane (2.7 ml) was added and mild hydrothermal conditions (463 K) were applied in a Teflon lined autoclave (25 ml). The resulting product was washed with ethanol and dried in air giving colourless single crystals.

**S3. Refinement**

All non-hydrogen atoms were refined with anisotropic displacement parameters. The H atoms of the water molecules were located using difference methods and their positional and isotropic displacement parameters were refined. Other H atoms including those on the aminium groups were included in the refinement at calculated positions and refined with a common isotropic thermal parameter.

**Figure 1**

An view the  $(\text{H}_2\text{dap})^{2+}$  cation, the  $[\text{FeF}_6]^{3-}$  and  $\text{F}^-$  anions and the water molecules of solvation (I). Thermal displacement ellipsoids are shown at the 50% probability level.

**Figure 2**

The three-dimensional H bonding network in the structure of (I).

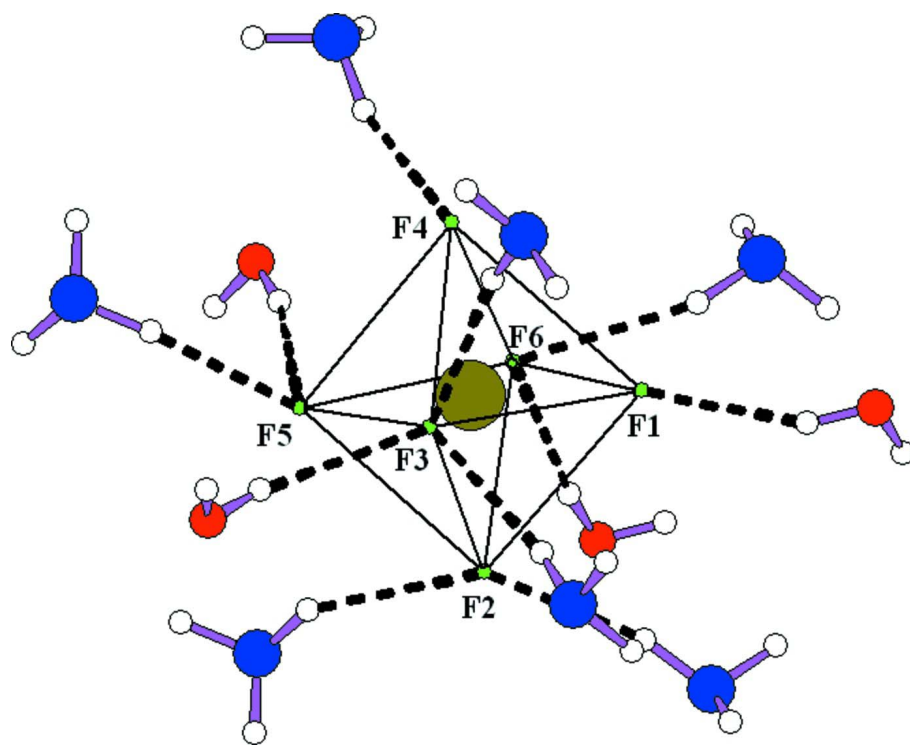


Figure 3

The environment of the  $\text{FeF}_6$  octahedron.

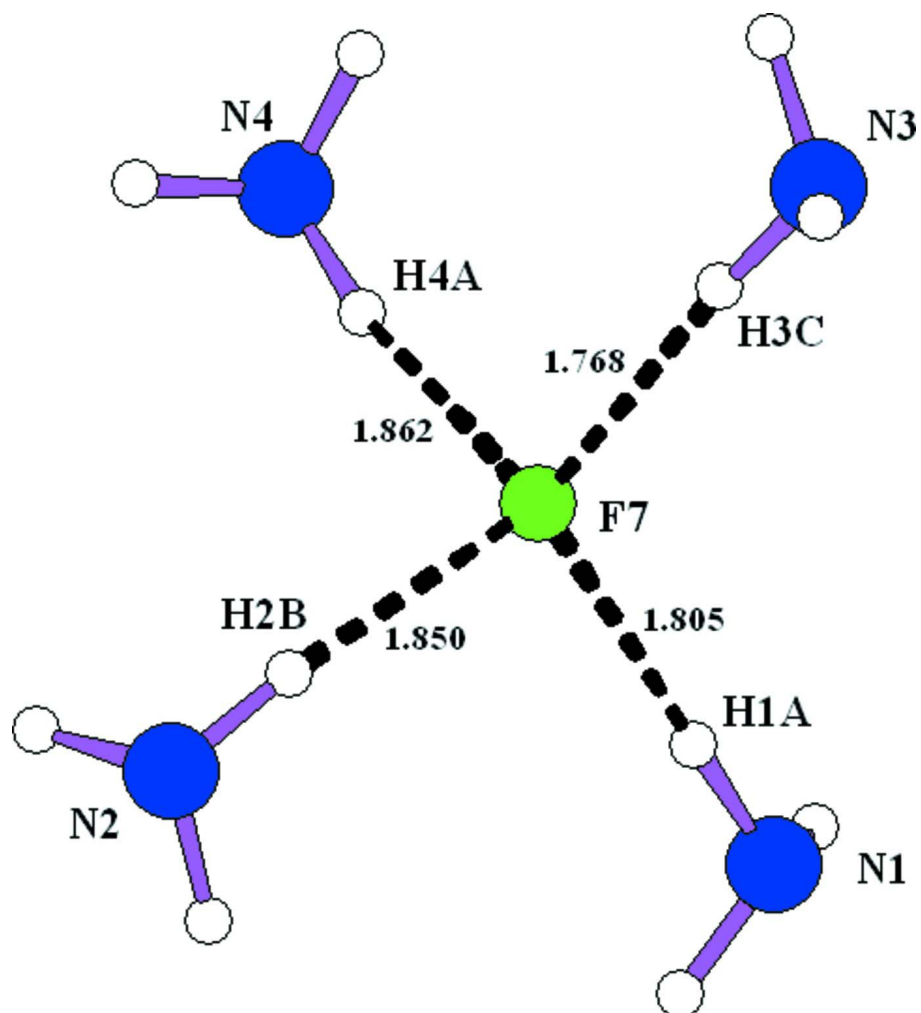


Figure 4

The environment of the isolated fluoride anion.

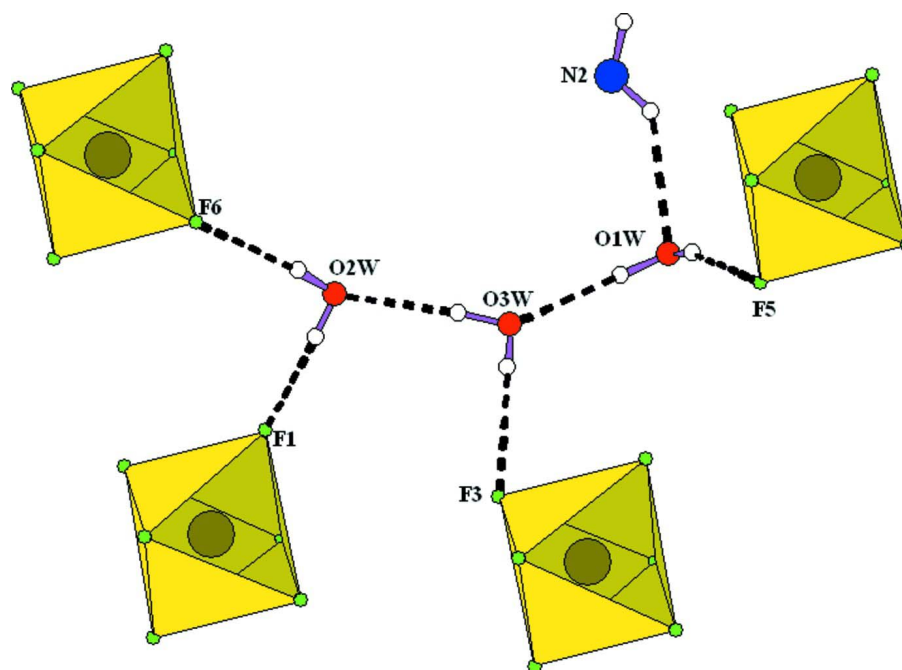


Figure 5

The environment of the water molecules

### Bis(propane-1,3-diaminium) hexafluoridoferrate(III) fluoride trihydrate

#### Crystal data

$(C_3H_{12}N_2)_2[FeF_6]F \cdot 3H_2O$

$M_r = 395.18$

Triclinic,  $P\bar{1}$

Hall symbol:  $-P\ 1$

$a = 9.844$  (1) Å

$b = 9.847$  (1) Å

$c = 10.7740$  (8) Å

$\alpha = 106.959$  (7)°

$\beta = 95.379$  (6)°

$\gamma = 118.914$  (9)°

$V = 839.35$  (17) Å<sup>3</sup>

$Z = 2$

$F(000) = 414$

$D_x = 1.564$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 24 reflections

$\theta = 5\text{--}20^\circ$

$\mu = 0.98$  mm<sup>-1</sup>

$T = 295$  K

Parallelepiped, colorless

$0.32 \times 0.07 \times 0.07$  mm

#### Data collection

SIEMENS AED2

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$2\theta/\omega$  scans

2920 measured reflections

2920 independent reflections

2599 reflections with  $I > 2\sigma(I)$

$R_{int} = 0.000$

$\theta_{max} = 25.0^\circ$ ,  $\theta_{min} = 2.1^\circ$

$h = -11 \rightarrow 11$

$k = -10 \rightarrow 11$

$l = -12 \rightarrow 0$

3 standard reflections every 120 min

intensity decay: 4%

Refinement

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.031$   
 $wR(F^2) = 0.093$   
 $S = 1.14$   
 2920 reflections  
 219 parameters  
 0 restraints  
 Primary atom site location: structure-invariant  
 direct methods

Secondary atom site location: difference Fourier  
 map  
 Hydrogen site location: inferred from  
 neighbouring sites  
 H atoms treated by a mixture of independent  
 and constrained refinement  
 $w = 1/[\sigma^2(F_o^2) + (0.0485P)^2 + 0.4679P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 0.42 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.35 \text{ e } \text{\AA}^{-3}$

Special details

**Geometry.** All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

**Refinement.** Refinement of  $F^2$  against ALL reflections. The weighted  $R$ -factor  $wR$  and goodness of fit  $S$  are based on  $F^2$ , conventional  $R$ -factors  $R$  are based on  $F$ , with  $F$  set to zero for negative  $F^2$ . The threshold expression of  $F^2 > \sigma(F^2)$  is used only for calculating  $R$ -factors(gt) etc. and is not relevant to the choice of reflections for refinement.  $R$ -factors based on  $F^2$  are statistically about twice as large as those based on  $F$ , and  $R$ -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Fe	0.84562 (4)	0.12759 (4)	0.22030 (3)	0.02722 (13)
F1	0.7789 (3)	-0.0409 (2)	0.04636 (19)	0.0734 (6)
F2	0.9740 (2)	0.29714 (19)	0.15238 (16)	0.0467 (4)
F3	0.66528 (18)	0.1495 (2)	0.16599 (17)	0.0479 (4)
F4	0.7093 (2)	-0.0330 (2)	0.28815 (18)	0.0582 (5)
F5	0.91998 (19)	0.30634 (19)	0.39249 (15)	0.0460 (4)
F6	1.0227 (2)	0.1036 (2)	0.2722 (2)	0.0582 (5)
F7	0.79472 (17)	0.17081 (17)	0.74186 (14)	0.0363 (3)
C1	0.6954 (3)	0.3991 (3)	0.6071 (2)	0.0330 (5)
H1D	0.7029	0.4645	0.5530	0.0476 (17)*
H1E	0.6899	0.4560	0.6948	0.0476 (17)*
C2	0.5420 (3)	0.2254 (3)	0.5389 (3)	0.0327 (5)
H2D	0.5468	0.1674	0.4512	0.0476 (17)*
H2E	0.5322	0.1600	0.5935	0.0476 (17)*
C3	0.3974 (3)	0.2417 (3)	0.5219 (3)	0.0371 (6)
H3D	0.4063	0.3203	0.6061	0.0476 (17)*
H3E	0.3969	0.2869	0.4531	0.0476 (17)*
C4	0.6160 (3)	0.3197 (3)	0.9521 (3)	0.0371 (6)
H4D	0.5557	0.3222	1.0181	0.0476 (17)*
H4E	0.5624	0.3215	0.8729	0.0476 (17)*
C5	0.7874 (3)	0.4733 (3)	1.0111 (3)	0.0336 (5)
H5D	0.8430	0.4677	1.0868	0.0476 (17)*
H5E	0.8456	0.4743	0.9431	0.0476 (17)*
C6	0.7863 (3)	0.6330 (3)	1.0579 (3)	0.0368 (6)



H6D	0.7111	0.6271	0.9884	0.0476 (17)*
H6E	0.7498	0.6438	1.1388	0.0476 (17)*
N1	0.8414 (2)	0.3911 (2)	0.6246 (2)	0.0318 (4)
H1A	0.8289	0.3195	0.6639	0.0476 (17)*
H1B	0.9268	0.4928	0.6762	0.0476 (17)*
H1C	0.8567	0.3562	0.5442	0.0476 (17)*
N2	0.2431 (2)	0.0780 (3)	0.4826 (2)	0.0368 (5)
H2A	0.2398	0.0399	0.5484	0.0476 (17)*
H2B	0.2363	0.0043	0.4069	0.0476 (17)*
H2C	0.1607	0.0915	0.4689	0.0476 (17)*
N3	0.6179 (3)	0.1637 (3)	0.9144 (2)	0.0393 (5)
H3A	0.5179	0.0751	0.8720	0.0476 (17)*
H3B	0.6561	0.1562	0.9887	0.0476 (17)*
H3C	0.6809	0.1661	0.8599	0.0476 (17)*
N4	0.9502 (3)	0.7830 (2)	1.0873 (2)	0.0347 (5)
H4A	1.0194	0.7879	1.1504	0.0476 (17)*
H4B	0.9471	0.8756	1.1168	0.0476 (17)*
H4C	0.9820	0.7753	1.0123	0.0476 (17)*
O1W	1.0878 (3)	0.2596 (3)	0.5849 (3)	0.0568 (6)
O2W	0.6945 (3)	0.7508 (4)	0.7980 (3)	0.0660 (7)
O3W	0.6382 (3)	0.4155 (3)	0.2944 (3)	0.0590 (6)
H11	1.046 (6)	0.265 (6)	0.526 (5)	0.095 (18)*
H12	1.168 (5)	0.352 (5)	0.625 (4)	0.067 (12)*
H21	0.717 (5)	0.829 (5)	0.858 (4)	0.063 (12)*
H22	0.772 (5)	0.782 (5)	0.776 (4)	0.064 (12)*
H31	0.649 (5)	0.330 (4)	0.265 (4)	0.075 (12)*
H32	0.550 (5)	0.373 (5)	0.277 (4)	0.054 (11)*

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Fe	0.02654 (19)	0.02225 (19)	0.02768 (19)	0.00942 (14)	0.01073 (14)	0.00871 (14)
F1	0.0992 (16)	0.0461 (10)	0.0417 (10)	0.0295 (11)	0.0150 (10)	-0.0069 (8)
F2	0.0495 (9)	0.0373 (8)	0.0437 (9)	0.0118 (7)	0.0234 (7)	0.0211 (7)
F3	0.0338 (8)	0.0473 (9)	0.0622 (10)	0.0206 (7)	0.0079 (7)	0.0244 (8)
F4	0.0472 (10)	0.0500 (10)	0.0602 (10)	0.0053 (8)	0.0147 (8)	0.0373 (9)
F5	0.0473 (9)	0.0379 (8)	0.0333 (8)	0.0151 (7)	0.0146 (7)	0.0019 (6)
F6	0.0478 (10)	0.0597 (11)	0.0797 (13)	0.0361 (9)	0.0190 (9)	0.0295 (10)
F7	0.0372 (8)	0.0356 (7)	0.0377 (8)	0.0180 (6)	0.0152 (6)	0.0176 (6)
C1	0.0341 (13)	0.0253 (11)	0.0363 (13)	0.0133 (10)	0.0121 (10)	0.0119 (10)
C2	0.0322 (13)	0.0263 (12)	0.0356 (13)	0.0135 (10)	0.0113 (10)	0.0103 (10)
C3	0.0329 (13)	0.0285 (12)	0.0463 (15)	0.0130 (11)	0.0113 (11)	0.0161 (11)
C4	0.0283 (12)	0.0379 (14)	0.0374 (13)	0.0118 (11)	0.0116 (10)	0.0150 (11)
C5	0.0281 (12)	0.0323 (13)	0.0375 (13)	0.0134 (11)	0.0099 (10)	0.0146 (11)
C6	0.0327 (13)	0.0388 (14)	0.0419 (14)	0.0190 (11)	0.0154 (11)	0.0181 (11)
N1	0.0287 (10)	0.0243 (10)	0.0363 (11)	0.0094 (8)	0.0116 (8)	0.0123 (8)
N2	0.0306 (11)	0.0338 (11)	0.0416 (12)	0.0155 (9)	0.0113 (9)	0.0122 (9)
N3	0.0316 (11)	0.0329 (11)	0.0376 (11)	0.0054 (9)	0.0138 (9)	0.0137 (9)

N4	0.0370 (11)	0.0281 (10)	0.0380 (11)	0.0173 (9)	0.0120 (9)	0.0112 (9)
O1W	0.0420 (13)	0.0576 (15)	0.0708 (16)	0.0241 (12)	0.0092 (11)	0.0318 (13)
O2W	0.0440 (14)	0.0710 (17)	0.0466 (13)	0.0213 (12)	0.0098 (11)	-0.0055 (13)
O3W	0.0513 (15)	0.0470 (13)	0.0641 (15)	0.0233 (12)	0.0159 (12)	0.0085 (11)

*Geometric parameters (Å, °)*

Fe—F1	1.8968 (17)	C5—H5E	0.9700
Fe—F4	1.9083 (15)	C6—N4	1.487 (3)
Fe—F5	1.9157 (14)	C6—H6D	0.9700
Fe—F6	1.9234 (17)	C6—H6E	0.9700
Fe—F2	1.9405 (14)	N1—H1A	0.8900
Fe—F3	1.9468 (15)	N1—H1B	0.8900
C1—N1	1.475 (3)	N1—H1C	0.8900
C1—C2	1.519 (3)	N2—H2A	0.8900
C1—H1D	0.9700	N2—H2B	0.8900
C1—H1E	0.9700	N2—H2C	0.8900
C2—C3	1.508 (3)	N3—H3A	0.8900
C2—H2D	0.9700	N3—H3B	0.8900
C2—H2E	0.9700	N3—H3C	0.8900
C3—N2	1.483 (3)	N4—H4A	0.8900
C3—H3D	0.9700	N4—H4B	0.8900
C3—H3E	0.9700	N4—H4C	0.8900
C4—N3	1.480 (3)	O1W—H11	0.75 (5)
C4—C5	1.520 (3)	O1W—H12	0.81 (4)
C4—H4D	0.9700	O2W—H21	0.76 (4)
C4—H4E	0.9700	O2W—H22	0.76 (4)
C5—C6	1.511 (3)	O3W—H31	0.87 (4)
C5—H5D	0.9700	O3W—H32	0.73 (4)
F1—Fe—F4	92.30 (9)	C6—C5—C4	110.7 (2)
F1—Fe—F5	177.01 (8)	C6—C5—H5D	109.5
F4—Fe—F5	90.69 (8)	C4—C5—H5D	109.5
F1—Fe—F6	89.93 (10)	C6—C5—H5E	109.5
F4—Fe—F6	91.86 (8)	C4—C5—H5E	109.5
F5—Fe—F6	90.04 (8)	H5D—C5—H5E	108.1
F1—Fe—F2	89.21 (8)	N4—C6—C5	111.1 (2)
F4—Fe—F2	175.81 (8)	N4—C6—H6D	109.4
F5—Fe—F2	87.80 (7)	C5—C6—H6D	109.4
F6—Fe—F2	92.04 (8)	N4—C6—H6E	109.4
F1—Fe—F3	89.45 (9)	C5—C6—H6E	109.4
F4—Fe—F3	88.01 (8)	H6D—C6—H6E	108.0
F5—Fe—F3	90.59 (7)	C1—N1—H1A	109.5
F6—Fe—F3	179.36 (8)	C1—N1—H1B	109.5
F2—Fe—F3	88.10 (7)	H1A—N1—H1B	109.5
N1—C1—C2	112.05 (19)	C1—N1—H1C	109.5
N1—C1—H1D	109.2	H1A—N1—H1C	109.5
C2—C1—H1D	109.2	H1B—N1—H1C	109.5

N1—C1—H1E	109.2	C3—N2—H2A	109.5
C2—C1—H1E	109.2	C3—N2—H2B	109.5
H1D—C1—H1E	107.9	H2A—N2—H2B	109.5
C3—C2—C1	109.6 (2)	C3—N2—H2C	109.5
C3—C2—H2D	109.7	H2A—N2—H2C	109.5
C1—C2—H2D	109.7	H2B—N2—H2C	109.5
C3—C2—H2E	109.7	C4—N3—H3A	109.5
C1—C2—H2E	109.7	C4—N3—H3B	109.5
H2D—C2—H2E	108.2	H3A—N3—H3B	109.5
N2—C3—C2	112.1 (2)	C4—N3—H3C	109.5
N2—C3—H3D	109.2	H3A—N3—H3C	109.5
C2—C3—H3D	109.2	H3B—N3—H3C	109.5
N2—C3—H3E	109.2	C6—N4—H4A	109.5
C2—C3—H3E	109.2	C6—N4—H4B	109.5
H3D—C3—H3E	107.9	H4A—N4—H4B	109.5
N3—C4—C5	110.4 (2)	C6—N4—H4C	109.5
N3—C4—H4D	109.6	H4A—N4—H4C	109.5
C5—C4—H4D	109.6	H4B—N4—H4C	109.5
N3—C4—H4E	109.6	H11—O1W—H12	106 (5)
C5—C4—H4E	109.6	H21—O2W—H22	101 (4)
H4D—C4—H4E	108.1	H31—O3W—H32	100 (4)

Hydrogen-bond geometry (Å, °)

<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>
N1—H1 <i>B</i> ...F2 <sup>i</sup>	0.89	2.03	2.826 (3)	148
N1—H1 <i>B</i> ...F5 <sup>i</sup>	0.89	2.22	2.839 (3)	127
N2—H2 <i>A</i> ...F4 <sup>ii</sup>	0.89	1.82	2.672 (3)	161
N2—H2 <i>B</i> ...F7 <sup>ii</sup>	0.89	1.85	2.735 (3)	172
N2—H2 <i>C</i> ...O1 <i>W</i> <sup>iii</sup>	0.89	2.22	2.926 (3)	136
N2—H2 <i>C</i> ...F6 <sup>iii</sup>	0.89	2.47	3.139 (3)	132
N3—H3 <i>A</i> ...F3 <sup>ii</sup>	0.89	1.95	2.777 (3)	155
N3—H3 <i>A</i> ...F4 <sup>ii</sup>	0.89	2.47	3.135 (3)	132
N3—H3 <i>B</i> ...F3 <sup>iv</sup>	0.89	1.93	2.762 (3)	156
N4—H4 <i>A</i> ...F7 <sup>v</sup>	0.89	1.86	2.728 (3)	164
N4—H4 <i>B</i> ...F6 <sup>vi</sup>	0.89	2.09	2.886 (3)	149
N4—H4 <i>B</i> ...F1 <sup>vi</sup>	0.89	2.33	3.029 (3)	135
O1 <i>W</i> —H12...O3 <i>W</i> <sup>i</sup>	0.81 (4)	1.99 (4)	2.787 (4)	173 (4)
O2 <i>W</i> —H21...F1 <sup>vi</sup>	0.76 (4)	1.91 (4)	2.606 (3)	153 (4)
O2 <i>W</i> —H22...F6 <sup>i</sup>	0.76 (4)	1.99 (4)	2.747 (3)	171 (4)
O3 <i>W</i> —H32...O2 <i>W</i> <sup>ii</sup>	0.73 (4)	2.04 (4)	2.766 (4)	173 (4)

Symmetry codes: (i)  $-x+2, -y+1, -z+1$ ; (ii)  $-x+1, -y, -z+1$ ; (iii)  $x-1, y, z$ ; (iv)  $x, y, z+1$ ; (v)  $-x+2, -y+1, -z+2$ ; (vi)  $x, y+1, z+1$ ; (vii)  $-x+1, -y+1, -z+1$ .