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2,4,6-Triamino-1,3,5-triazine-1,3-diium aquapentafluoridoaluminate

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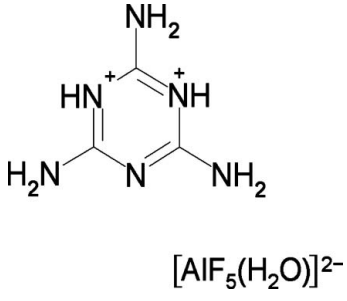
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(N–C) = 0.004 Å; R factor = 0.054; wR factor = 0.133; data-to-parameter ratio = 16.4.

The title compound, $(C_3H_8N_6)[AlF_5(H_2O)]$, was obtained by solvothermal synthesis from the reaction of aluminium hydroxide, 1,3,5-triazine-2,4,6-triamine (melamine), aqueous HF and water at 323 K for 48 h. The structure consists of $[AlF_5(H_2O)]^{2-}$ octahedra and diprotonated melaminium cations. Cohesion is ensured by a three-dimensional network of hydrogen bonds.

Related literature

For related literature, see: Adil, Ben Ali *et al.* (2006); Adil, Leblanc & Maisonneuve (2006); Farrugia (1999); Goreshnik *et al.* (2002, 2003); Rother *et al.* (1996, 1998); Schroder *et al.* (1993); Tang *et al.* (2001).



Experimental

Crystal data

$(C_3H_8N_6)[AlF_5(H2O)]$
M_r = 268.13
Monoclinic, $P2_1/c$
a = 7.571 (2) Å
b = 8.823 (2) Å
c = 13.484 (5) Å
β = 105.53 (3)°
V = 867.8 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.31 mm^{-1}
T = 298 (2) K
0.20 × 0.13 × 0.08 mm

Data collection

Siemens AED2 diffractometer
Absorption correction: none
2500 measured reflections
2500 independent reflections
1441 reflections with $I > 2\sigma(I)$
3 standard reflections
frequency: 120 min
intensity decay: 4%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.133
S = 1.04
2500 reflections
152 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1W—H1W···N1^{i}	0.90 (4)	1.94 (5)	2.789 (3)	156 (5)
O1W—H2W···F1^{ii}	0.90 (4)	1.63 (4)	2.515 (3)	169 (5)
N2—H2···F4^{iii}	0.86	1.75	2.601 (3)	169
N3—H3···F2^{iv}	0.86	2.11	2.871 (3)	148
N3—H3···F3^{iv}	0.86	2.28	2.990 (3)	140
N3—H3···F1^{iv}	0.86	2.52	2.953 (4)	112
N4—H4A···F5^{v}	0.86	2.07	2.763 (3)	138
N4—H4B···F2^{iii}	0.86	1.89	2.739 (3)	168
N5—H5A···F3	0.86	2.06	2.837 (3)	151
N5—H5B···F3^{iv}	0.86	2.02	2.804 (4)	151
N5—H5B···F4	0.86	2.39	2.863 (3)	115
N6—H6A···F1^{vi}	0.86	2.04	2.836 (4)	154
N6—H6B···F2^{iv}	0.86	2.08	2.860 (4)	150

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

Data collection: *STADI4* (Stoe & Cie, 1998); cell refinement: *STADI4*; 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); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 2005); software used to prepare material for publication: *SHELXL97*, *enCIFer* (Version 1.2; Allen *et al.*, 2004) and *WinGX* (Farrugia, 1999).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2317).

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supplementary materials

Acta Cryst. (2008). E**64**, m523-m524 [**doi:10.1107/S1600536808004091**]

2,4,6-Triamino-1,3,5-triazine-1,3-diium aquapentafluoridoaluminate

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Comment

Numerous hybrid fluoroaluminates with linear or branched amines are reported; during the last five years more than 20 compounds were evidenced (Goreshnik *et al.*, 2002; Adil, Ben Ali *et al.*, 2006; Adil, Leblanc & Maisonneuve, 2006). At the opposite, only few hybrid fluoroaluminates with cyclic amines are known (Schroder *et al.*, 1993; Rother *et al.*, 1996; Rother *et al.*, 1998; Tang *et al.*, 2001; Goreshnik *et al.*, 2003). 1,3,5-triazine-2,4,6-triamine (melamine) with three primary amines, three tertiary amines and a conjugated planar configuration was selected. $(C_3H_8N_6)\cdot[AlF_5(H_2O)]$ is synthesized and constitutes the first melamine templated fluoroaluminate.

The structure is built up from isolated $[AlF_5(H_2O)]^{2-}$ anions and diprotonated $(C_3H_8N_6)^{2+}$ cations (Fig. 1). A distortion of the aluminium coordination octahedron results from the presence of the water molecule: Al—F distances range from 1.758 (2) to 1.829 (2) Å and Al—O distance is 1.929 (3) Å. Melaminium cations are planar and two tertiary amines are protonated. C, N, H atomic positions are related by a pseudo two fold symmetry axis along the N1—C2—N5 direction. Hydrogen bonded octahedra form infinite inorganic chains along *b* axis (Fig. 2); the O1W—H2W···F1 hydrogen bonds (2.51 Å) are short. Every melaminium cation is surrounded by five $[AlF_5(H_2O)]$ octahedra (Fig. 3).

Experimental

The title compound was prepared under hydrothermal conditions at 323 K for 48 h using Teflon-lined autoclaves from a started mixture of $Al(OH)_3$ (Sochal), 1,3,5-triazine-2,4,6-triamine named melamine (Janssen chimica), HF aqueous solution (40%, Prolabo) and deionized water in the molar ratio 1:0.5:8.5:55.5. The resulting crystalline product was washed with water and dried in air. Needle crystals suitable for single-crystal X-ray diffraction were selected using an optical microscope.

Refinement

The structure was solved by direct methods (*SHELXS86*) and refined with *SHELXL97*; these programs are included in *WinGX* package (Farrugia, 1999). Hydrogen atoms of amine cations were located applying geometrical constraints which imply equal distances and angles to the central atom (AFIX option). Hydrogen atoms of water molecules were found in difference Fourier maps and the O—H distances were constrained to be equal to 0.9 Å (*DFIX* option). H atoms were refined with an isotropic thermal parameters and non-hydrogen atoms were refined with anisotropic thermal factors. The maximum residual electron density peak is located at 0.46 Å from Al. Protonation takes place on the two over three tertiary amine groups.

Figures

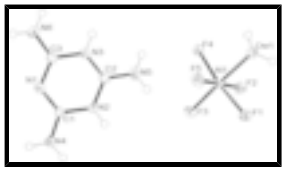

Fig. 1. View of the melaminium cation and $[AlF_5(H_2O)]^{2-}$ anion with the atom-labellin scheme. Displacement ellipsoids are drawn at the 50% probability level.

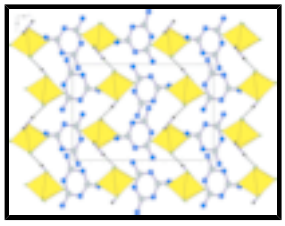

Fig. 2. (100) projection of $(C_3H_8N_6)\cdot[AlF_5(H_2O)]$ structure.

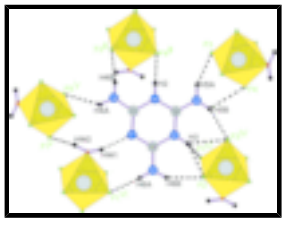

Fig. 3. Network of hydrogen bonds between melaminium cations and $[AlF_5(H_2O)]$ octahedra. [Symmetry codes: (iii) $1-x, y-1/2, 1/2-z$; (iv) $1-x, 1/2+y, 1/2-z$; (v) $1-x, -y, -z$; (vi) $1+x, 1/2-y, z-1/2$]

2,4,6-Triamino-1,3,5-triazine-1,3-diium aquapentafluoridoaluminate

Crystal data

$(C_3H_8N_6)[AlF_5(H2O)]$	$F_{000} = 544$
$M_r = 268.13$	$D_x = 2.052$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation $\lambda = 0.71069$ Å
Hall symbol: -P 2ybc	Cell parameters from 30 reflections
$a = 7.571$ (2) Å	$\theta = 28$–32^o
$b = 8.823$ (2) Å	$\mu = 0.31$ mm^{-1}
$c = 13.484$ (5) Å	$T = 298$ (2) K
$\beta = 105.53$ (3)^o	Parallepiped, colourless
$V = 867.8$ (5) Å^3	0.20 × 0.13 × 0.08 mm
$Z = 4$	

Data collection

Siemens AED2 diffractometer	$R_{int} = 0.0000$
Radiation source: fine-focus sealed tube	$\theta_{max} = 30.0^o$
Monochromator: graphite	$\theta_{min} = 2.8^o$
$T = 298(2)$ K	$h = -10\rightarrow10$
$2\theta/\omega$ scans	$k = 0\rightarrow12$
Absorption correction: none	$l = 0\rightarrow18$
2500 measured reflections	3 standard reflections
2500 independent reflections	every 120 min

1441 reflections with $I > 2\sigma(I)$

intensity decay: 4%

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.054$

$wR(F^2) = 0.133$

$S = 1.04$

2500 reflections

152 parameters

2 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

$w = 1/[\sigma^2(F_o^2) + (0.0437P)^2 + 0.9389P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.46$ e Å^{-3}

$\Delta\rho_{min} = -0.48$ e Å^{-3}

Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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 $F^2 > 2$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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Al1	0.2088 (1)	0.2232 (1)	0.30136 (7)	0.0163 (2)
F1	0.0457 (3)	0.0750 (2)	0.30467 (16)	0.0288 (5)
F2	0.2911 (3)	0.1978 (2)	0.44074 (14)	0.0251 (4)
F3	0.3778 (3)	0.0880 (2)	0.28702 (17)	0.0299 (5)
F4	0.3610 (3)	0.3839 (2)	0.31272 (16)	0.0267 (5)
F5	0.1237 (3)	0.2429 (2)	0.16732 (14)	0.0304 (5)
O1W	0.0310 (3)	0.3648 (3)	0.32584 (18)	0.0214 (5)
H1W	−0.062 (5)	0.324 (5)	0.346 (4)	0.062 (12)*
H2W	−0.007 (7)	0.444 (4)	0.284 (3)	0.062 (12)*
N1	0.7845 (4)	0.2143 (3)	−0.0619 (2)	0.0202 (6)
N2	0.6746 (4)	0.1144 (3)	0.0739 (2)	0.0195 (6)
H2	0.6499	0.0359	0.1056	0.023*
N3	0.6895 (4)	0.3729 (3)	0.0557 (2)	0.0203 (6)
H3	0.6714	0.4633	0.0748	0.024*
N4	0.7594 (4)	−0.0423 (3)	−0.0409 (2)	0.0232 (6)

H4A	0.7996	−0.0586	−0.0938	0.028*
H4B	0.7310	−0.1173	−0.0074	0.028*
N5	0.5822 (4)	0.2735 (3)	0.1861 (2)	0.0271 (6)
H5A	0.5543	0.1966	0.2181	0.033*
H5B	0.5664	0.3638	0.2063	0.033*
N6	0.8013 (4)	0.4736 (3)	−0.0717 (2)	0.0293 (7)
H6A	0.8446	0.4652	−0.1242	0.035*
H6B	0.7850	0.5619	−0.0486	0.035*
C1	0.7414 (4)	0.0960 (3)	−0.0113 (2)	0.0179 (6)
C2	0.6481 (4)	0.2534 (4)	0.1076 (2)	0.0191 (6)
C3	0.7602 (4)	0.3525 (4)	−0.0271 (2)	0.0199 (6)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Al1	0.0209 (5)	0.0127 (4)	0.0176 (4)	−0.0002 (4)	0.0090 (3)	−0.0014 (4)
F1	0.0359 (13)	0.0199 (10)	0.0351 (11)	−0.0115 (9)	0.0175 (10)	−0.0075 (9)
F2	0.0330 (11)	0.0214 (10)	0.0204 (9)	0.0027 (8)	0.0063 (8)	0.0020 (8)
F3	0.0355 (13)	0.0191 (10)	0.0413 (12)	0.0089 (9)	0.0207 (10)	0.0013 (9)
F4	0.0292 (12)	0.0191 (9)	0.0379 (11)	−0.0069 (8)	0.0197 (10)	−0.0054 (9)
F5	0.0435 (13)	0.0312 (11)	0.0188 (9)	0.0044 (10)	0.0122 (9)	−0.0009 (8)
O1W	0.0240 (13)	0.0179 (11)	0.0251 (12)	0.0055 (10)	0.0113 (10)	0.0066 (9)
N1	0.0263 (14)	0.0171 (13)	0.0202 (13)	−0.0005 (11)	0.0114 (11)	0.0003 (11)
N2	0.0256 (15)	0.0133 (12)	0.0236 (13)	−0.0001 (11)	0.0134 (12)	0.0016 (10)
N3	0.0260 (15)	0.0125 (12)	0.0240 (13)	−0.0006 (10)	0.0097 (12)	−0.0026 (10)
N4	0.0354 (18)	0.0180 (13)	0.0204 (13)	0.0015 (12)	0.0152 (12)	−0.0003 (11)
N5	0.0340 (17)	0.0241 (14)	0.0288 (15)	−0.0016 (13)	0.0181 (13)	−0.0034 (13)
N6	0.0402 (19)	0.0190 (14)	0.0328 (16)	−0.0052 (13)	0.0166 (15)	0.0020 (12)
C1	0.0200 (16)	0.0181 (15)	0.0166 (14)	0.0008 (12)	0.0065 (12)	−0.0002 (12)
C2	0.0168 (15)	0.0205 (15)	0.0204 (14)	−0.0022 (12)	0.0056 (12)	−0.0025 (12)
C3	0.0192 (16)	0.0177 (15)	0.0228 (15)	−0.0005 (12)	0.0056 (13)	0.0006 (13)

Geometric parameters (Å, °)

Al1—F5	1.757 (2)	N3—C2	1.347 (4)
Al1—F3	1.797 (2)	N3—C3	1.374 (4)
Al1—F4	1.807 (2)	N3—H3	0.8600
Al1—F1	1.807 (2)	N4—C1	1.302 (4)
Al1—F2	1.829 (2)	N4—H4A	0.8600
Al1—O1W	1.929 (2)	N4—H4B	0.8600
O1W—H1W	0.90 (4)	N5—C2	1.299 (4)
O1W—H2W	0.90 (4)	N5—H5A	0.8600
N1—C1	1.334 (4)	N5—H5B	0.8600
N1—C3	1.337 (4)	N6—C3	1.304 (4)
N2—C2	1.342 (4)	N6—H6A	0.8600
N2—C1	1.383 (4)	N6—H6B	0.8600
N2—H2	0.8600		
F5—Al1—F3	91.76 (11)	C2—N3—H3	119.5

F5—Al1—F4	93.42 (11)	C3—N3—H3	119.5
F3—Al1—F4	94.23 (10)	C1—N4—H4A	120.0
F5—Al1—F1	91.84 (11)	C1—N4—H4B	120.0
F3—Al1—F1	91.90 (10)	H4A—N4—H4B	120.0
F4—Al1—F1	171.80 (10)	C2—N5—H5A	120.0
F5—Al1—F2	177.98 (12)	C2—N5—H5B	120.0
F3—Al1—F2	88.45 (11)	H5A—N5—H5B	120.0
F4—Al1—F2	88.57 (10)	C3—N6—H6A	120.0
F1—Al1—F2	86.15 (10)	C3—N6—H6B	120.0
F5—Al1—O1W	91.80 (11)	H6A—N6—H6B	120.0
F3—Al1—O1W	176.37 (11)	N4—C1—N1	121.1 (3)
F4—Al1—O1W	86.32 (10)	N4—C1—N2	117.1 (3)
F1—Al1—O1W	87.23 (10)	N1—C1—N2	121.8 (3)
F2—Al1—O1W	87.97 (10)	N5—C2—N2	121.7 (3)
H1W—O1W—H2W	111 (4)	N5—C2—N3	120.7 (3)
C1—N1—C3	117.3 (3)	N2—C2—N3	117.6 (3)
C2—N2—C1	120.6 (3)	N6—C3—N1	120.9 (3)
C2—N2—H2	119.7	N6—C3—N3	117.4 (3)
C1—N2—H2	119.7	N1—C3—N3	121.7 (3)
C2—N3—C3	121.0 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1W—H1W···N1^{i}	0.90 (4)	1.94 (5)	2.789 (3)	156 (5)
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Symmetry codes: (i) *x*−1, −*y*+1/2, *z*+1/2; (ii) −*x*, *y*+1/2, −*z*+1/2; (iii) −*x*+1, *y*−1/2, −*z*+1/2; (iv) −*x*+1, *y*+1/2, −*z*+1/2; (v) −*x*+1, −*y*, −*z*; (vi) *x*+1, −*y*+1/2, *z*−1/2.

Fig. 1

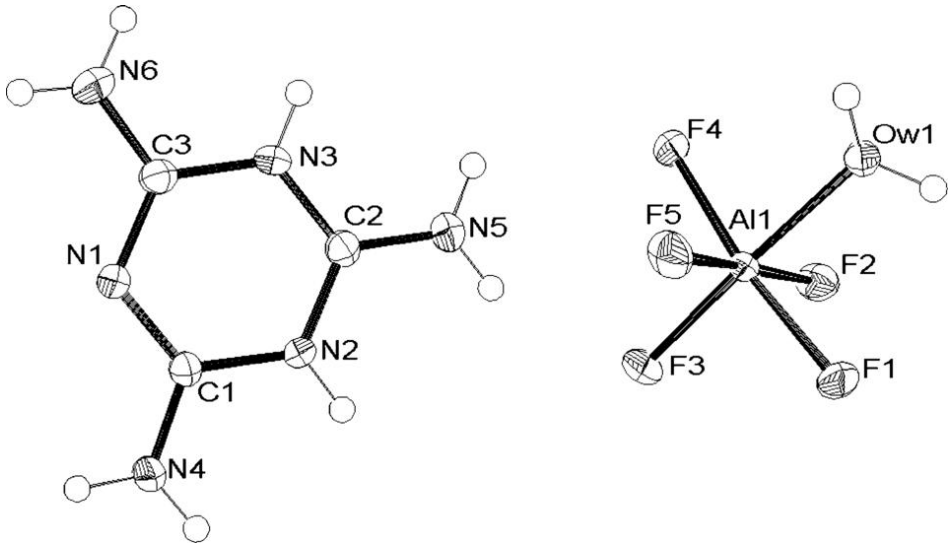


Fig. 2

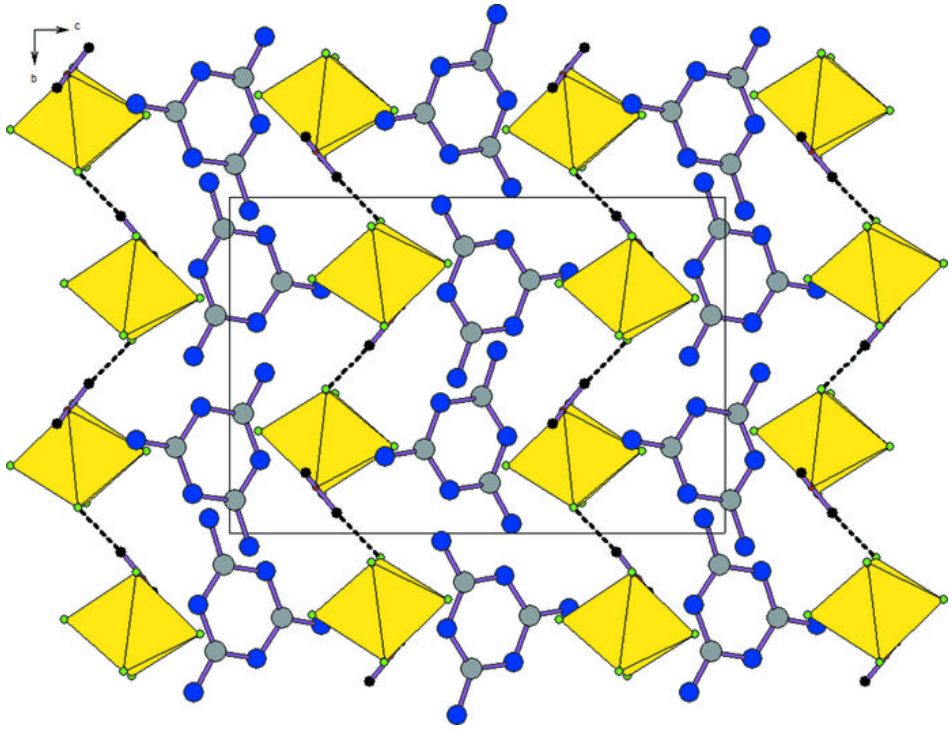
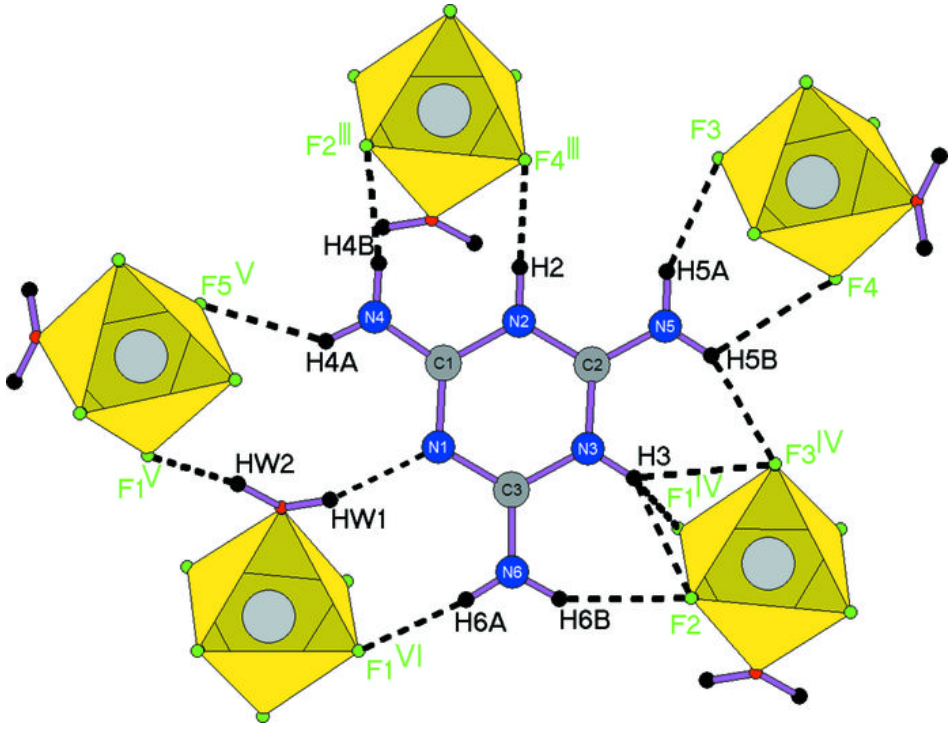


Fig. 3


F2III
F4III
F3
H4B
H2
H5A
F5V
F4
N4
N2
N5
H4A
C1
C2
H5B
N1
N3
H3
F3IV
F1V
HW2
C3
F1IV
HW1
N6
H6A
H6B
F2
F1VI