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3,3′-Dimethyl-1,1′-[2,2′-bipyridine-5,5′-diylbis(methylene)]diimidazol-3-ium bis(hexafluorophosphate)

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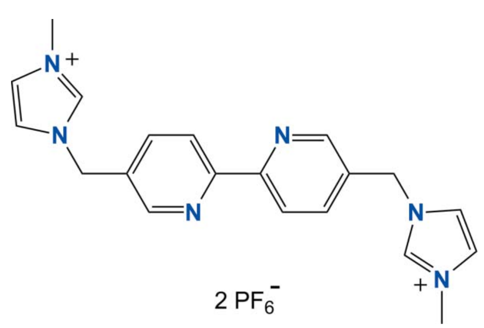
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.003 Å; R factor = 0.040; wR factor = 0.117; data-to-parameter ratio = 15.0.

The title compound, $C_{20}H_{22}N_6^{2+}\cdot 2PF_6^-$, was prepared by the reaction of 5,5′-bis(bromomethyl)-2,2′-bipyridine with 1-methylimidazole. The main molecule lies on an inversion center located at the mid-point of the C—C bond joining the two pyridine rings. The asymmetric unit therefore contains one half-molecule and one hexafluorophosphate anion. The dihedral angle between the pyridine and imidazole rings is 76.93 (7)°. In the crystal, weak intermolecular C—H···F hydrogen bonds contribute to the stabilization of the packing.

Related literature

For related syntheses, see: Sambrook *et al.* (2006); Zang *et al.* (2010). For related structures, see: Moon *et al.* (2011); Zang *et al.* (2010). For reference bond lengths, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{20}H_{22}N_6^{2+}\cdot 2PF_6^-$
M_r = 636.38
Monoclinic, $P2_1/c$
a = 7.5323 (4) Å
b = 10.7169 (6) Å
c = 15.4602 (9) Å
β = 93.922 (1)°
V = 1245.07 (12) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.29 mm^{-1}
T = 173 K
0.40 × 0.40 × 0.10 mm

Data collection

Bruker APEXII CCD diffractometer
7489 measured reflections
2717 independent reflections
1788 reflections with $I > 2\sigma(I)$
R_{int} = 0.049

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.117
S = 1.02
2717 reflections
181 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7···F1	0.95	2.23	3.111 (3)	154
C7—H7···F4	0.95	2.39	3.230 (3)	147
C8—H8···F1^{i}	0.95	2.50	3.163 (3)	127
C8—H8···F2^{i}	0.95	2.50	3.446 (3)	176
C9—H9···F2^{ii}	0.95	2.52	3.240 (3)	133

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

Data collection: *APEX2* (Bruker, 2006); cell refinement: *SAINT* (Bruker, 2006); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL* and *DIAMOND* (Brandenburg, 1998); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LX2201).

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supplementary materials

Acta Cryst. (2011). E**67**, o2392 [doi:10.1107/S160053681103340X]

3,3'-Dimethyl-1,1'-[2,2'-bipyridine-5,5'-diylbis(methylene)]diimidazol-3-ium bis(hexafluorophosphate)

S. Park, S.-H. Moon, T. H. Kim and K.-M. Park

Comment

The title compound was prepared for use as a *N*-heterocyclic carbene ligand in the formation of coordination polymers in line with similar previously reported compounds (Sambrook *et al.*, 2006; Zang *et al.*, 2010).

In the title compound (Scheme 1, Fig. 1), two pyridine rings are coplanar because the title compound lies on a crystallographic inversion center. The geomeries of the title compound are very similar with those of the previously reported compound (Moon *et al.*, 2011) The dihedral angle between the pyridine and imidazole rings is 76.93 (7)°. All the bond lengths are within normal values (Allen *et al.*, 1987).

The crystal packing (Fig. 2) is stabilized by weak intermolecular C—H···F hydrogen bonds (see, Table 1)

Experimental

A mixture of 1-methylimidazole (0.150 g, 1.83 mmol) and 5,5'-bis(bromomethyl)-2,2'-bipyridine (0.30 g, 0.88 mmol) in 1,4-dioxane (15 ml) was stirred for 10 min and then heated at reflux for 6 h. After cooling to room temperature, Et_2O (15 ml) was added and 5,5'-bis((*N*-methylimidazolium-1-yl)methyl)-2,2'-bipyridine bis(chloride) obtained as a white precipitate was separated by filtration and washed with Et_2O. For the anion exchange, an excess of KPF_6 was added to the aqueous solution of the chloride salts. After stirring for 1 hr, the title compound as a white precipitate was obtained. X–ray quality single crystals were obtained by slow evaporation of a solution of the title compound in acetonitrile at room temperature.

Refinement

All H-atoms were positioned geometrically and refined using a riding model with d(C—H) = 0.95 Å, U_{iso} = 1.2U_{eq}(C) for aromatic, d(C—H) = 0.99 Å, U_{iso} = 1.2<U>$_{eq}$(C) for methylene, and d(C—H) = 0.98 Å, U_{iso}(H) = 1.5U_{eq}(C) for methyl protons.

Figures

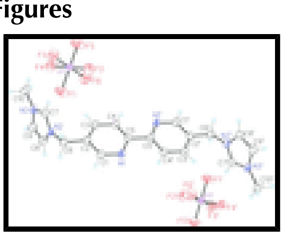


Fig. 1. The molecular structure of the title compound, showing displacement ellipsoids drawn at the 50% probability level. Hydrogen bonds are shown as dashed lines. (Symmetry code: i) -*x* + 1, -*y* + 2, -*z* + 1)

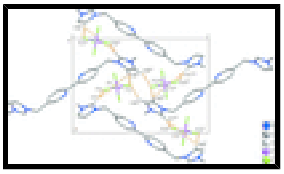


Fig. 2. Crystal packing of the title compound with intermolecular C—H···F hydrogen bonds shown as dashed lines. (Symmetry codes: i) -*x* + 1, -*y* + 2, -*z* + 1; ii) *x*, -*y* + 3/2, *z* + 1/2; iii) -*x* + 1, -*y* + 1, -*z* + 1; iv) -*x* + 1, *y* - 1/2, -*z* + 1/2; v) *x*, *y* - 1, *z*).

3,3'-Dimethyl-1,1'-[2,2'-bipyridine-5,5'-diylbis(methylene)]diimidazol-3-ium bis(hexafluorophosphate)

Crystal data

$C_{20}H_{22}N_6^{2+}\cdot 2PF_6^-$	$F(000) = 644$
$M_r = 636.38$	$D_x = 1.697$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 2214 reflections
$a = 7.5323$ (4) Å	$\theta = 2.3$–$27.5°$
$b = 10.7169$ (6) Å	$\mu = 0.29$ mm^{-1}
$c = 15.4602$ (9) Å	$T = 173$ K
$\beta = 93.922$ (1)°	Plate, colorless
$V = 1245.07$ (12) Å^3	0.40 × 0.40 × 0.10 mm
$Z = 2$	

Data collection

Bruker APEXII CCD diffractometer	1788 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.049$
graphite	$\theta_{max} = 27.0°$, $\theta_{min} = 2.6°$
φ and ω scans	$h = -9\rightarrow 9$
7489 measured reflections	$k = -13\rightarrow 11$
2717 independent reflections	$l = -19\rightarrow 17$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.040$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.117$	H-atom parameters constrained
$S = 1.02$	$w = 1/[\sigma^2(F_o^2) + (0.0536P)^2 + 0.3234P]$ where $P = (F_o^2 + 2F_c^2)/3$
2717 reflections	$(\Delta/\sigma)_{max} < 0.001$
181 parameters	$\Delta\rho_{max} = 0.30$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.29$ e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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}
N1	0.6669 (2)	0.90672 (19)	0.45131 (12)	0.0314 (5)
N2	0.4666 (2)	0.75139 (19)	0.16354 (12)	0.0292 (5)
N3	0.2383 (3)	0.7410 (2)	0.07124 (12)	0.0327 (5)
C1	0.6823 (3)	0.8369 (3)	0.38058 (15)	0.0335 (6)
H1	0.7937	0.7979	0.3739	0.040*
C2	0.5479 (3)	0.8172 (2)	0.31630 (14)	0.0272 (5)
C3	0.3880 (3)	0.8772 (2)	0.32510 (14)	0.0292 (5)
H3	0.2924	0.8675	0.2823	0.035*
C4	0.3687 (3)	0.9517 (2)	0.39729 (14)	0.0272 (5)
H4	0.2605	0.9952	0.4037	0.033*
C5	0.5085 (3)	0.9623 (2)	0.45999 (13)	0.0232 (5)
C6	0.5823 (3)	0.7291 (2)	0.24282 (15)	0.0328 (6)
H6A	0.7080	0.7377	0.2288	0.039*
H6B	0.5647	0.6423	0.2624	0.039*
C7	0.3074 (3)	0.7000 (2)	0.14706 (15)	0.0327 (6)
H7	0.2519	0.6430	0.1838	0.039*
C8	0.5004 (3)	0.8271 (3)	0.09592 (16)	0.0365 (6)
H8	0.6048	0.8755	0.0905	0.044*
C9	0.3580 (3)	0.8208 (3)	0.03775 (16)	0.0393 (6)
H9	0.3437	0.8636	−0.0161	0.047*
C10	0.0635 (3)	0.7051 (3)	0.03147 (18)	0.0451 (7)
H10A	0.0059	0.6473	0.0699	0.068*
H10B	0.0783	0.6643	−0.0243	0.068*
H10C	−0.0106	0.7797	0.0222	0.068*
P1	0.04237 (8)	0.51966 (6)	0.32208 (4)	0.03076 (19)
F1	0.24565 (19)	0.51442 (16)	0.29682 (10)	0.0478 (4)
F2	0.1084 (2)	0.48970 (16)	0.42008 (9)	0.0504 (4)
F3	0.0232 (2)	0.37450 (15)	0.30333 (11)	0.0547 (5)
F4	−0.0194 (2)	0.54954 (16)	0.22308 (10)	0.0536 (5)
F5	−0.1585 (2)	0.52519 (18)	0.34678 (11)	0.0563 (5)
F6	0.0658 (2)	0.66508 (15)	0.33975 (12)	0.0586 (5)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0241 (10)	0.0412 (13)	0.0278 (11)	0.0060 (9)	−0.0061 (8)	−0.0057 (9)
N2	0.0271 (10)	0.0340 (12)	0.0258 (11)	0.0016 (8)	−0.0040 (8)	−0.0073 (8)
N3	0.0268 (11)	0.0425 (13)	0.0279 (11)	0.0007 (9)	−0.0040 (8)	−0.0065 (9)
C1	0.0228 (12)	0.0455 (16)	0.0314 (13)	0.0080 (11)	−0.0035 (10)	−0.0055 (11)
C2	0.0273 (12)	0.0298 (13)	0.0238 (12)	0.0005 (10)	−0.0024 (9)	0.0013 (10)
C3	0.0263 (12)	0.0341 (14)	0.0256 (12)	0.0019 (10)	−0.0084 (10)	−0.0010 (10)
C4	0.0217 (11)	0.0335 (14)	0.0259 (12)	0.0044 (10)	−0.0024 (9)	0.0021 (10)
C5	0.0247 (11)	0.0234 (13)	0.0210 (11)	−0.0002 (9)	−0.0021 (9)	0.0041 (9)
C6	0.0274 (13)	0.0389 (15)	0.0305 (13)	0.0065 (11)	−0.0088 (10)	−0.0061 (11)
C7	0.0319 (13)	0.0380 (15)	0.0279 (13)	−0.0030 (11)	0.0006 (10)	−0.0031 (11)
C8	0.0315 (13)	0.0407 (16)	0.0369 (14)	−0.0113 (11)	−0.0010 (11)	−0.0016 (11)
C9	0.0456 (16)	0.0418 (17)	0.0294 (14)	−0.0044 (12)	−0.0044 (11)	0.0009 (11)
C10	0.0281 (14)	0.063 (2)	0.0428 (16)	−0.0043 (13)	−0.0095 (12)	−0.0115 (14)
P1	0.0257 (3)	0.0360 (4)	0.0301 (4)	0.0002 (3)	−0.0017 (3)	0.0030 (3)
F1	0.0296 (8)	0.0637 (11)	0.0504 (10)	0.0063 (7)	0.0055 (7)	0.0140 (8)
F2	0.0494 (10)	0.0695 (12)	0.0314 (9)	−0.0041 (8)	−0.0042 (7)	0.0066 (7)
F3	0.0684 (11)	0.0394 (10)	0.0561 (10)	−0.0069 (8)	0.0020 (9)	−0.0042 (7)
F4	0.0424 (9)	0.0776 (13)	0.0390 (10)	−0.0098 (8)	−0.0112 (7)	0.0184 (8)
F5	0.0312 (9)	0.0749 (13)	0.0637 (12)	0.0039 (8)	0.0106 (8)	0.0159 (9)
F6	0.0639 (11)	0.0363 (10)	0.0760 (12)	−0.0022 (8)	0.0072 (9)	−0.0034 (8)

Geometric parameters (Å, °)

N1—C1	1.336 (3)	C5—C5^{i}	1.490 (4)
N1—C5	1.348 (3)	C6—H6A	0.9900
N2—C7	1.329 (3)	C6—H6B	0.9900
N2—C8	1.361 (3)	C7—H7	0.9500
N2—C6	1.474 (3)	C8—C9	1.354 (3)
N3—C7	1.324 (3)	C8—H8	0.9500
N3—C9	1.370 (3)	C9—H9	0.9500
N3—C10	1.466 (3)	C10—H10A	0.9800
C1—C2	1.385 (3)	C10—H10B	0.9800
C1—H1	0.9500	C10—H10C	0.9800
C2—C3	1.380 (3)	P1—F5	1.5869 (16)
C2—C6	1.513 (3)	P1—F3	1.5872 (17)
C3—C4	1.388 (3)	P1—F6	1.5899 (18)
C3—H3	0.9500	P1—F2	1.5947 (16)
C4—C5	1.386 (3)	P1—F4	1.6014 (16)
C4—H4	0.9500	P1—F1	1.6069 (15)
C1—N1—C5	117.10 (19)	N3—C7—H7	125.4
C7—N2—C8	108.3 (2)	N2—C7—H7	125.4
C7—N2—C6	124.6 (2)	C9—C8—N2	107.4 (2)
C8—N2—C6	127.2 (2)	C9—C8—H8	126.3
C7—N3—C9	108.2 (2)	N2—C8—H8	126.3

C7—N3—C10	124.8 (2)	C8—C9—N3	107.0 (2)
C9—N3—C10	126.9 (2)	C8—C9—H9	126.5
N1—C1—C2	124.9 (2)	N3—C9—H9	126.5
N1—C1—H1	117.5	N3—C10—H10A	109.5
C2—C1—H1	117.5	N3—C10—H10B	109.5
C3—C2—C1	117.3 (2)	H10A—C10—H10B	109.5
C3—C2—C6	124.1 (2)	N3—C10—H10C	109.5
C1—C2—C6	118.6 (2)	H10A—C10—H10C	109.5
C2—C3—C4	119.2 (2)	H10B—C10—H10C	109.5
C2—C3—H3	120.4	F5—P1—F3	90.25 (10)
C4—C3—H3	120.4	F5—P1—F6	91.05 (10)
C5—C4—C3	119.5 (2)	F3—P1—F6	178.66 (10)
C5—C4—H4	120.2	F5—P1—F2	91.12 (9)
C3—C4—H4	120.2	F3—P1—F2	89.70 (9)
N1—C5—C4	121.95 (19)	F6—P1—F2	90.60 (9)
N1—C5—C5^{i}	116.7 (2)	F5—P1—F4	90.18 (9)
C4—C5—C5^{i}	121.4 (2)	F3—P1—F4	90.27 (9)
N2—C6—C2	113.67 (19)	F6—P1—F4	89.41 (10)
N2—C6—H6A	108.8	F2—P1—F4	178.70 (9)
C2—C6—H6A	108.8	F5—P1—F1	179.80 (10)
N2—C6—H6B	108.8	F3—P1—F1	89.85 (9)
C2—C6—H6B	108.8	F6—P1—F1	88.84 (9)
H6A—C6—H6B	107.7	F2—P1—F1	89.04 (9)
N3—C7—N2	109.1 (2)	F4—P1—F1	89.66 (8)

C5—N1—C1—C2	−0.4 (4)	C3—C2—C6—N2	24.8 (3)
N1—C1—C2—C3	1.9 (4)	C1—C2—C6—N2	−157.2 (2)
N1—C1—C2—C6	−176.2 (2)	C9—N3—C7—N2	0.4 (3)
C1—C2—C3—C4	−0.9 (3)	C10—N3—C7—N2	−179.8 (2)
C6—C2—C3—C4	177.2 (2)	C8—N2—C7—N3	−0.2 (3)
C2—C3—C4—C5	−1.5 (3)	C6—N2—C7—N3	179.07 (19)
C1—N1—C5—C4	−2.2 (3)	C7—N2—C8—C9	0.0 (3)
C1—N1—C5—C5^{i}	178.7 (3)	C6—N2—C8—C9	−179.3 (2)
C3—C4—C5—N1	3.1 (3)	N2—C8—C9—N3	0.2 (3)
C3—C4—C5—C5^{i}	−177.8 (3)	C7—N3—C9—C8	−0.3 (3)
C7—N2—C6—C2	−88.2 (3)	C10—N3—C9—C8	179.8 (2)
C8—N2—C6—C2	91.0 (3)		

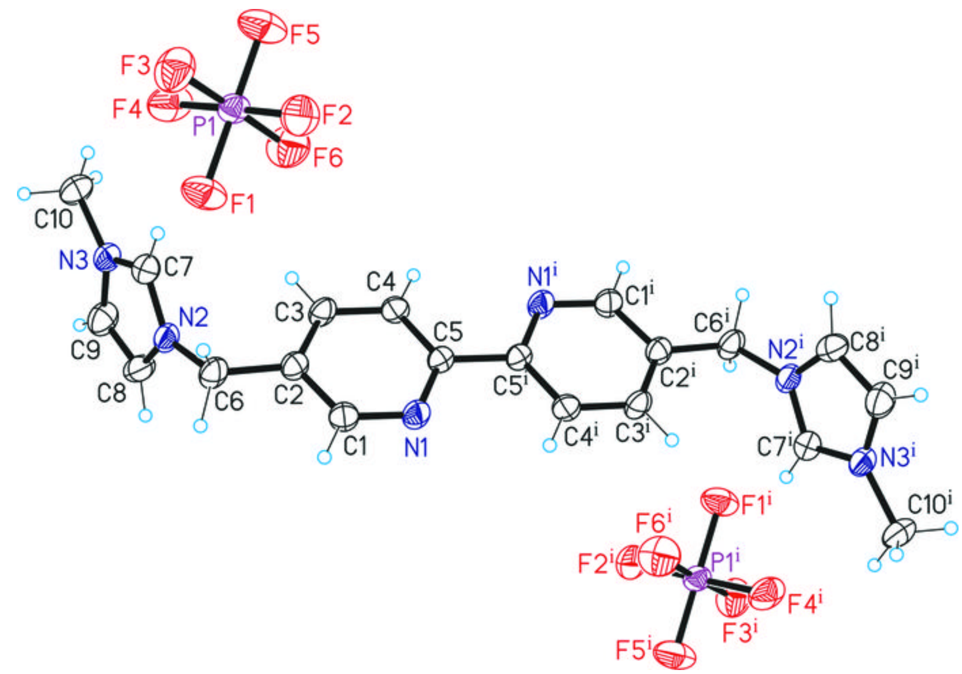
Symmetry codes: (i) −*x*+1, −*y*+2, −*z*+1.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7···F1	0.95	2.23	3.111 (3)	154.
C7—H7···F4	0.95	2.39	3.230 (3)	147.
C8—H8···F1^{ii}	0.95	2.50	3.163 (3)	127.
C8—H8···F2^{ii}	0.95	2.50	3.446 (3)	176.
C9—H9···F2^{iii}	0.95	2.52	3.240 (3)	133.

Symmetry codes: (ii) −*x*+1, *y*+1/2, −*z*+1/2; (iii) *x*, −*y*+3/2, *z*−1/2.

Fig. 1


F5
F3
F4
P1
F2
F6
F1
C10
N3
C7
N2
C9
C8
C6
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C2
C1
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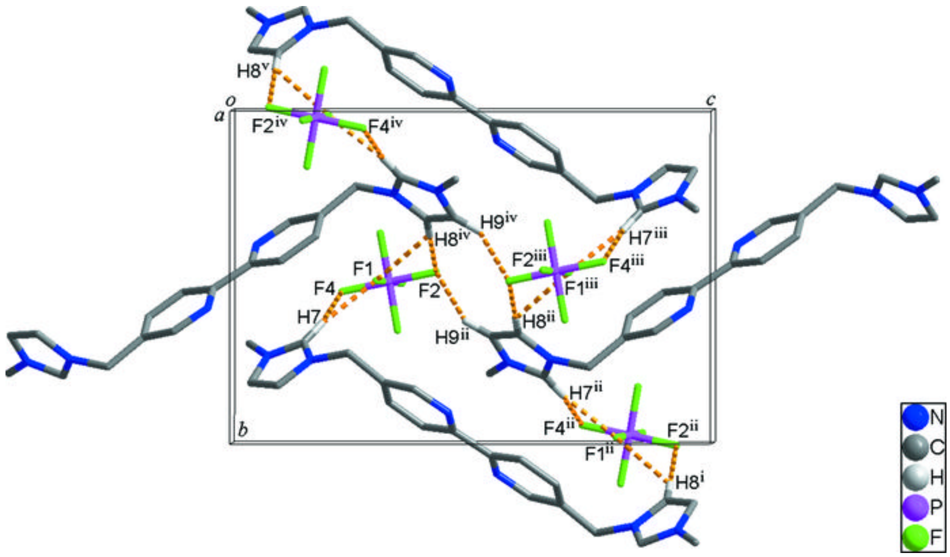
Fig. 2


H8v
F2iv
F4iv
a
o
c
H9iv
H8iv
H7iii
F2iii
F4iii
F1
F1iii
F4
F2
H7
H8ii
H9ii
H7ii
F4ii
F2ii
F1ii
H8i
b
N
C
H
P
F