organic compounds

1-Methyl-3-(4-vinylbenzyl)imidazolium hexafluorophosphate

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In the title compound, $C_{13}H_{15}N_2{}^+ \cdot PF_6{}^-$, the dihedral angle between the two aromatic rings is 85.48 (7)°. In the crystal, C—H⋯F hydrogen bonds connect the imidazolium and hexafluorophosphate ions.

Related literature

For N-heterocyclic carbenes, see: Herrmann (2002). For the synthesis of the title compound, see: Kim *et al.* (2005). For a silver compound with 1-methyl-3-(4-vinylbenzyl)imidazol-2-ylidene, see: Lu *et al.* (2009).
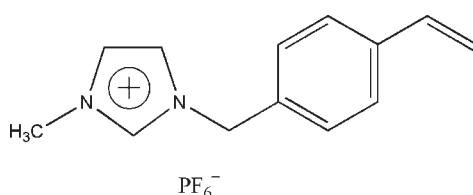
Experimental

Crystal data

$C_{13}H_{15}N_2{}^+ \cdot PF_6{}^-$
$M_r = 344.24$

Orthorhombic, $P2_12_12_1$
$a = 10.482$ (2) Å

$b = 11.272$ (3) Å
$c = 12.556$ (3) Å
$V = 1483.4$ (6) Å^3
$Z = 4$

Mo $K\alpha$ radiation
$\mu = 0.25$ mm^{-1}
$T = 298$ K
$0.32 \times 0.29 \times 0.26$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min} = 0.925$, $T_{max} = 0.939$

9185 measured reflections
3542 independent reflections
3407 reflections with $I > 2\sigma(I)$
$R_{int} = 0.085$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.127$
$S = 1.03$
3542 reflections
200 parameters
H-atom parameters constrained

$\Delta\rho_{max} = 0.65$ e Å^{-3}
$\Delta\rho_{min} = -0.89$ e Å^{-3}
Absolute structure: Flack (1983), 1484 Friedel pairs
Flack parameter: 0.05 (13)

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C4—H4⋯F1[i]	0.93	2.36	3.266 (3)	164
C3—H3⋯F6[ii]	0.93	2.25	3.044 (3)	143

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

Data collection: *SMART* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

References

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supporting information

1-Methyl-3-(4-vinylbenzyl)imidazolium hexafluorophosphate

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S1. Comment

N-heterocyclic carbenes have attracted great attention as they can act as efficient supporting ligands in organometallic chemistry and homogeneous catalysis (Herrmann, 2002). Imidazolium salts are the useful precursors of the N-heterocyclic carbenes. The silver compound with 1-methyl-3-(4-vinylbenzyl)imidazol-2-ylidene has been synthesized (Lu *et al.*, 2009).

The structure of the title compound is shown in Fig. 1. The two aromatic rings enclose a dihedral angle of 85.48 (7)°. The hexafluorophosphate anions are linked to 1-methyl-3-(4-vinylbenzyl)imidazolium cations *via* C—H···F hydrogen bonds (Table 1 and Fig. 2). These interacions stabilize the crystal structure.

S2. Experimental

1-Methyl-3-(4-vinylbenzyl)imidazolium chloride was synthesized according to the literature method (Kim *et al.*, 2005). The resulting white solid (0.70 g, 3.00 mmol) was dissolved in acetone (30 ml) and then potassium hexafluorophosphate (1.38 g, 7.50 mmol) was added. The mixture was stirred at room temperature for 26 h and the solvent was removed under reduced pressure. The residue was then dissolved in distilled water and extracted with CH_2Cl_2. The white solid was abtained after the removal of solvent. Yield: 0.78 g (76%). Anal. Calcd for $C_{13}H_{15}F_6N_2P$: C,45.36; H, 4.39; N, 8.14. Found: C, 45.13; H, 4.03; N, 8.35. The elemental analyses were performed with Vario MICRO elemental analyzer.

S3. Refinement

The H-atoms were included in the riding-model approximation with C—H = 0.93 Å and C—H = 0.96 Å, and with $U_{iso}(H)$ = $1.2U_{eq}$(C-aromatic) and $U_{iso}(H)$ = $1.5U_{eq}$(*C*-methyl).
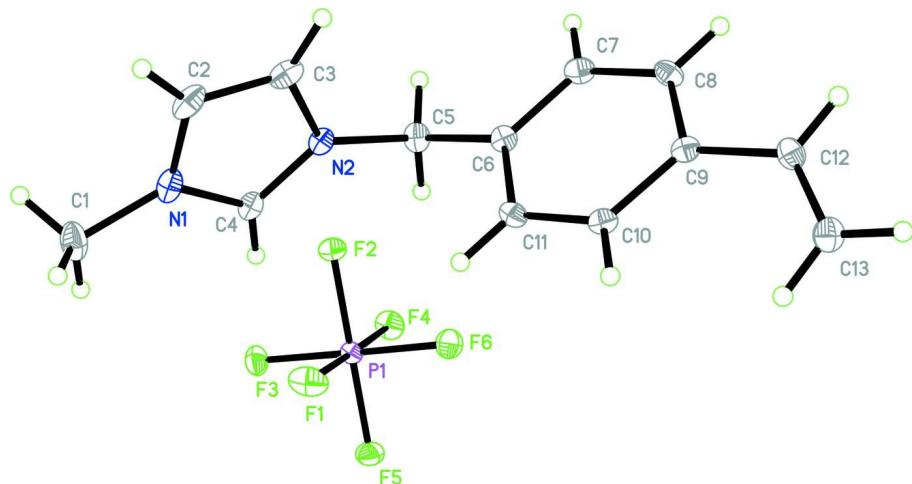
Figure 1

The molecular structure of title compound with atom labels and 30% probability displacement ellipsoids for non-H atoms.
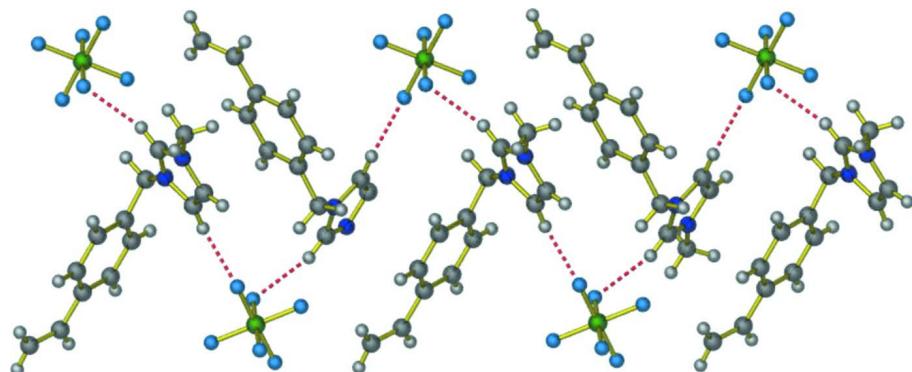
Figure 2

A packing diagram of the title compound. Hydrogen bonds are shown as dashed lines.

1-Methyl-3-(4-vinylbenzyl)imidazolium hexafluorophosphate

Crystal data

$C_{13}H_{15}N_2{}^+ \cdot PF_6{}^-$	$F(000) = 704$
$M_r = 344.24$	$D_x = 1.541$ Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: P 2ac 2ab	Cell parameters from 6513 reflections
$a = 10.482$ (2) Å	$\theta = 2.4$–28.3°
$b = 11.272$ (3) Å	$\mu = 0.25$ mm^{-1}
$c = 12.556$ (3) Å	$T = 298$ K
$V = 1483.4$ (6) Å^3	Block, colorless
$Z = 4$	$0.32 \times 0.29 \times 0.26$ mm

Data collection

Bruker SMART CCD area-detector diffractometer	phi and ω scans
	Absorption correction: multi-scan
Radiation source: fine-focus sealed tube	(*SADABS*; Bruker, 2005)
Graphite monochromator	$T_{min} = 0.925$, $T_{max} = 0.939$

9185 measured reflections
3542 independent reflections
3407 reflections with $I > 2\sigma(I)$
$R_{int} = 0.085$

$\theta_{max} = 28.3°$, $\theta_{min} = 2.4°$
$h = -13 \rightarrow 11$
$k = -14 \rightarrow 14$
$l = -16 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.127$
$S = 1.03$
3542 reflections
200 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0549P)^2 + 1.5672P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.65$ e Å^{-3}
$\Delta\rho_{min} = -0.89$ e Å^{-3}
Absolute structure: Flack (1983), 1484 Friedel pairs
Absolute structure parameter: 0.05 (13)

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}
C1	0.8710 (3)	0.2998 (3)	0.0944 (2)	0.0351 (7)
H1A	0.9248	0.2527	0.1396	0.053*
H1B	0.9017	0.3800	0.0931	0.053*
H1C	0.8722	0.2679	0.0235	0.053*
C2	0.6394 (3)	0.3609 (2)	0.0958 (2)	0.0283 (6)
H2	0.6415	0.4128	0.0382	0.034*
C3	0.5369 (3)	0.3342 (2)	0.1548 (2)	0.0249 (5)
H3	0.4549	0.3641	0.1460	0.030*
C4	0.6999 (3)	0.2335 (2)	0.21741 (19)	0.0204 (5)
H4	0.7497	0.1826	0.2583	0.025*
C5	0.4948 (3)	0.2031 (3)	0.3150 (2)	0.0228 (5)
H5A	0.4678	0.2657	0.3630	0.027*
H5B	0.5440	0.1461	0.3558	0.027*
C6	0.3787 (2)	0.1426 (2)	0.26935 (19)	0.0192 (5)
C7	0.2587 (3)	0.1657 (2)	0.3101 (2)	0.0236 (5)
H7	0.2489	0.2225	0.3632	0.028*
C8	0.1529 (3)	0.1050 (3)	0.2724 (2)	0.0234 (5)
H8	0.0729	0.1216	0.3008	0.028*
C9	0.1643 (2)	0.0194 (2)	0.19242 (19)	0.0191 (5)

C10	0.2861 (2)	−0.0017 (2)	0.14997 (18)	0.0190 (5)
H10	0.2960	−0.0569	0.0956	0.023*
C11	0.3914 (2)	0.0584 (2)	0.1878 (2)	0.0199 (5)
H11	0.4714	0.0429	0.1590	0.024*
C12	0.0501 (3)	−0.0449 (2)	0.1563 (2)	0.0230 (5)
H12	−0.0282	−0.0177	0.1812	0.028*
C13	0.0480 (3)	−0.1377 (3)	0.0917 (2)	0.0260 (5)
H13A	0.1239	−0.1681	0.0647	0.031*
H13B	−0.0294	−0.1725	0.0733	0.031*
F1	0.67298 (19)	0.98396 (17)	0.85265 (12)	0.0364 (4)
F2	0.75104 (15)	0.85036 (13)	0.96990 (13)	0.0245 (3)
F3	0.54994 (15)	0.92291 (15)	0.99001 (14)	0.0297 (4)
F4	0.70480 (17)	0.97546 (17)	1.10592 (12)	0.0305 (4)
F5	0.62797 (17)	1.11050 (14)	0.98847 (14)	0.0309 (4)
F6	0.82848 (16)	1.03763 (16)	0.96874 (17)	0.0359 (4)
N1	0.7402 (2)	0.2980 (2)	0.13540 (17)	0.0247 (5)
N2	0.5760 (2)	0.25420 (19)	0.23092 (17)	0.0191 (4)
P1	0.68904 (6)	0.98115 (6)	0.97936 (5)	0.01785 (15)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0343 (15)	0.0449 (17)	0.0259 (13)	−0.0109 (14)	0.0098 (12)	−0.0043 (13)
C2	0.0468 (16)	0.0210 (12)	0.0170 (11)	−0.0073 (12)	−0.0057 (12)	0.0022 (10)
C3	0.0371 (14)	0.0197 (12)	0.0179 (11)	0.0005 (11)	−0.0092 (11)	0.0012 (9)
C4	0.0257 (12)	0.0206 (12)	0.0149 (10)	−0.0049 (10)	−0.0021 (9)	−0.0001 (8)
C5	0.0254 (12)	0.0298 (13)	0.0130 (10)	−0.0031 (11)	−0.0006 (10)	0.0008 (10)
C6	0.0227 (11)	0.0222 (12)	0.0128 (10)	0.0002 (10)	−0.0020 (9)	0.0007 (9)
C7	0.0295 (13)	0.0235 (12)	0.0180 (10)	0.0022 (11)	0.0003 (10)	−0.0052 (10)
C8	0.0218 (12)	0.0274 (13)	0.0212 (11)	0.0045 (10)	0.0049 (10)	−0.0042 (10)
C9	0.0244 (12)	0.0188 (11)	0.0142 (9)	0.0014 (9)	−0.0009 (9)	0.0024 (9)
C10	0.0266 (12)	0.0191 (12)	0.0114 (9)	0.0024 (9)	0.0013 (9)	−0.0005 (8)
C11	0.0197 (11)	0.0246 (12)	0.0154 (10)	0.0046 (10)	0.0016 (9)	0.0024 (9)
C12	0.0210 (11)	0.0288 (13)	0.0192 (11)	−0.0004 (10)	0.0015 (10)	0.0016 (10)
C13	0.0262 (12)	0.0297 (14)	0.0223 (12)	−0.0030 (11)	−0.0030 (10)	−0.0017 (11)
F1	0.0519 (11)	0.0436 (10)	0.0136 (7)	0.0145 (9)	−0.0002 (7)	−0.0030 (7)
F2	0.0282 (8)	0.0221 (7)	0.0234 (7)	0.0037 (6)	−0.0019 (7)	−0.0001 (6)
F3	0.0214 (7)	0.0343 (9)	0.0336 (9)	−0.0042 (6)	−0.0004 (7)	0.0058 (7)
F4	0.0384 (9)	0.0382 (9)	0.0149 (7)	0.0029 (8)	0.0031 (6)	0.0037 (6)
F5	0.0343 (8)	0.0240 (8)	0.0343 (9)	0.0064 (7)	0.0045 (8)	0.0040 (7)
F6	0.0238 (8)	0.0317 (9)	0.0521 (11)	−0.0055 (7)	−0.0052 (8)	−0.0098 (8)
N1	0.0333 (12)	0.0256 (11)	0.0153 (9)	−0.0103 (10)	0.0007 (9)	−0.0018 (9)
N2	0.0234 (10)	0.0191 (10)	0.0147 (9)	−0.0025 (8)	−0.0026 (8)	0.0011 (8)
P1	0.0191 (3)	0.0204 (3)	0.0141 (3)	0.0009 (2)	−0.0008 (2)	0.0002 (2)

Geometric parameters (Å, º)

C1—N1	1.464 (4)	C7—H7	0.9300
C1—H1A	0.9600	C8—C9	1.398 (4)
C1—H1B	0.9600	C8—H8	0.9300
C1—H1C	0.9600	C9—C10	1.404 (3)
C2—C3	1.339 (4)	C9—C12	1.471 (4)
C2—N1	1.367 (4)	C10—C11	1.379 (4)
C2—H2	0.9300	C10—H10	0.9300
C3—N2	1.377 (3)	C11—H11	0.9300
C3—H3	0.9300	C12—C13	1.324 (4)
C4—N1	1.330 (3)	C12—H12	0.9300
C4—N2	1.330 (3)	C13—H13A	0.9300
C4—H4	0.9300	C13—H13B	0.9300
C5—N2	1.473 (3)	F1—P1	1.6001 (17)
C5—C6	1.508 (4)	F2—P1	1.6156 (16)
C5—H5A	0.9700	F3—P1	1.6046 (17)
C5—H5B	0.9700	F4—P1	1.5988 (16)
C6—C7	1.383 (4)	F5—P1	1.5964 (17)
C6—C11	1.403 (4)	F6—P1	1.5998 (18)
C7—C8	1.387 (4)		
N1—C1—H1A	109.5	C11—C10—C9	120.9 (2)
N1—C1—H1B	109.5	C11—C10—H10	119.5
H1A—C1—H1B	109.5	C9—C10—H10	119.5
N1—C1—H1C	109.5	C10—C11—C6	120.5 (2)
H1A—C1—H1C	109.5	C10—C11—H11	119.7
H1B—C1—H1C	109.5	C6—C11—H11	119.7
C3—C2—N1	107.6 (2)	C13—C12—C9	126.3 (3)
C3—C2—H2	126.2	C13—C12—H12	116.9
N1—C2—H2	126.2	C9—C12—H12	116.9
C2—C3—N2	107.0 (3)	C12—C13—H13A	120.0
C2—C3—H3	126.5	C12—C13—H13B	120.0
N2—C3—H3	126.5	H13A—C13—H13B	120.0
N1—C4—N2	108.3 (2)	C4—N1—C2	108.6 (2)
N1—C4—H4	125.9	C4—N1—C1	125.3 (3)
N2—C4—H4	125.9	C2—N1—C1	126.0 (3)
N2—C5—C6	111.8 (2)	C4—N2—C3	108.5 (2)
N2—C5—H5A	109.3	C4—N2—C5	125.9 (2)
C6—C5—H5A	109.3	C3—N2—C5	125.6 (2)
N2—C5—H5B	109.3	F5—P1—F4	90.40 (10)
C6—C5—H5B	109.3	F5—P1—F6	90.51 (10)
H5A—C5—H5B	107.9	F4—P1—F6	90.25 (10)
C7—C6—C11	118.9 (2)	F5—P1—F1	90.63 (10)
C7—C6—C5	120.6 (2)	F4—P1—F1	178.83 (11)
C11—C6—C5	120.5 (2)	F6—P1—F1	90.30 (11)
C6—C7—C8	120.6 (2)	F5—P1—F3	90.19 (10)
C6—C7—H7	119.7	F4—P1—F3	89.69 (10)

C8—C7—H7	119.7	F6—P1—F3	179.30 (10)
C7—C8—C9	121.2 (2)	F1—P1—F3	89.74 (10)
C7—C8—H8	119.4	F5—P1—F2	179.86 (11)
C9—C8—H8	119.4	F4—P1—F2	89.71 (9)
C8—C9—C10	117.8 (2)	F6—P1—F2	89.39 (9)
C8—C9—C12	119.5 (2)	F1—P1—F2	89.27 (9)
C10—C9—C12	122.7 (2)	F3—P1—F2	89.91 (9)

Hydrogen-bond geometry (Å, º)

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
C4—H4⋯F1[i]	0.93	2.36	3.266 (3)	164
C3—H3⋯F6[ii]	0.93	2.25	3.044 (3)	143

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